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SECRETARIO DE ESTADO

POR:   
SECRETARIA AUXILIAR DE SERVICIOS



Enmiendas al Reglamento para el Control de la Contaminación Atmosférica de la Junta de Calidad Ambiental para cumplir con los requisitos para Planes Estatales de las Secciones 111(d) y 129 de la Ley Federal de Aire Limpio para implantar las Guías de Emisiones para Incineradores de Desperdicios de Hospitales y Médico Infecciosos

Versión 2001



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**ESTADO LIBRE ASOCIADO DE PUERTO RICO**  
**OFICINA DE LA GOBERNADORA**  
**Junta de Calidad Ambiental**

De acuerdo con la Ley sobre Política Pública Ambiental (Ley Número 9 del 18 de junio de 1970, según enmendada) y la Ley Federal de Aire Limpio Secciones 111 (d) y 129, según enmendadas (42 U.S.C.A. §§ 7411 (d) y 7429) se han enmendado las

**REGLAS 102 Y 405 DEL**  
**REGLAMENTO PARA EL CONTROL DE LA CONTAMINACIÓN**  
**ATMOSFÉRICA**

Estas enmiendas fueron promulgadas por la Resolución R-2001-02-07 del 20 de febrero del 2001 con el propósito de proteger la calidad natural del aire y para prevenir, eliminar y controlar la contaminación atmosférica; para establecer normas y requisitos para la prevención, eliminación y control de la contaminación atmosférica debido a las emisiones generadas por los Incineradores de Desperdicios Biomédicos/Hospitalarios.

Jorge Marrero Huertas  
Vice -Presidente

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Miembro Asociado

Miguel A. Morales Ramos  
Presidente Interino



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## REVISIÓN A LA REGLA 102 - DEFINICIONES

### **Administrador**

Significa el Administrador de la Agencia Federal de Protección Ambiental (APA).

### **Administrador de la Instalación**

Significa la persona a cargo de comprar, mantener y operar el IDBH o el representante del dueño u operador responsable del manejo del IDBH. Los títulos alternos pueden incluir director de la instalación o vicepresidente de servicios de apoyo.

### **Agente Infeccioso (Para propósitos de la Regla 405)**

Significa cualquier organismo (como un virus o bacteria) capaz de transmitirse por medio de invasión y reproducción en tejidos corporales y capaz de causar enfermedades o impactos adversos a la salud en humanos.

### **Amperaje o Caballaje Mínimo**

Significa el 90 por ciento del promedio de tres horas más alto, del caballaje o amperaje al lavador de gases húmedo (tomado por lo menos una vez cada minuto) medido durante la prueba de funcionamiento más reciente para demostrar cumplimiento con los límites de emisión aplicables.

### **APA**

Significa la Agencia Federal de Protección Ambiental.

### **Área Estadística Metropolitana Estándar (AEME)**

Significa cualquiera de las áreas registradas en el Boletín de OMB, Núm. 93-17 titulado Definiciones Estadísticas Revisadas para las Áreas Metropolitanas (*Revised Statistical Definitions for Metropolitan Areas*) del 30 de junio de 1993.

### **Caída en presión mínima a través del lavador de gases húmedo**

Significa el 90 por ciento del promedio de tres horas más alto de la caída en presión a través del lavador de gases húmedo (tomado por lo menos una vez cada minuto) medido durante la prueba de funcionamiento más reciente para demostrar cumplimiento con el límite de emisión de materia particulada (MP).

### **Cámara Primaria**

Significa la cámara de un IDBH que recibe desperdicios, en la cual se encienden los desperdicios y de donde se remueven las cenizas.

### Cámara Secundaria

Significa un componente del IDBH que recibe los gases de combustión de la cámara primaria y en donde se completa el proceso de combustión.

### Cantidad Máxima de Carga

Significa:

- (1) Para IDBH continuos e intermitentes, 110 por ciento de la cantidad de carga promedio de tres horas más baja medida durante la prueba de funcionamiento más reciente para demostrar cumplimiento con todos los límites de emisión aplicables.
- (2) Para IDBH por tandas, 110 por ciento de la cantidad diaria de carga más baja medida durante la prueba de funcionamiento más reciente para demostrar cumplimiento con todos los límites de emisión aplicables.

### Capacidad Máxima por diseño para la quema de desperdicios

Significa:

- (1) Para IDBH intermitentes y continuos,  

$$C = P_v \times 15,000 / 8,500$$

donde:

C	=	capacidad del IDBH, lbs/hora
$P_v$	=	volumen de la cámara primaria, $ft^3$
15,000	=	factor de la cantidad de descarga de calor de la cámara primaria, $Utb/ft^3/hora$
8,500	=	valor estándar de quema de desperdicios, $Utb/lb$

- (2) Para IDBH por tandas,  

$$C = P_v \times 4.5 / 8$$

donde:

C	=	capacidad del IDBH, lbs/hora
$P_v$	=	volumen de la cámara primaria, $ft^3$
4.5	=	densidad de los desperdicios, $lbs/ft^3$
8	=	horas de operación típicas de un IDBH por tandas, horas

### Comienzo de Proceso de Incineración (*Startup*)

Significa el periodo de tiempo entre la activación del sistema y la primera carga a la unidad. Para los IDBH por tandas, el comienzo del proceso de incineración será el período de tiempo entre la activación del sistema y el momento en que se enciende el desperdicio.

### Condición Estándar

Significa una temperatura de 20°C y una presión de 101.3 kilopascales.

### **Contaminante Designado**

Significa cualquier contaminante atmosférico cuyas emisiones están sujetas a un estándar de funcionamiento para fuentes estacionarias nuevas, pero para el cual no se hayan emitido criterios de calidad de aire y no esté incluido en la lista publicada bajo la Sección 108(a) o 112(b)(1)(A) de la Ley.

### **Chimenea Auxiliar (*Bypass Stack*)**

Significa aparato utilizado para descargar gases de combustión para evitar un daño severo al equipo de control de contaminación atmosférica u otro equipo.

### **Desperdicios Biomédicos (Para propósitos de la Regla 405)**

Significa cualquier desperdicio generado en el diagnóstico, tratamiento o inmunización de seres humanos o animales en investigaciones relacionadas o en la producción o prueba de productos biológicos mencionados a continuación:

- (1) Cultivos y cepas de agentes infecciosos y productos biológicos asociados incluyendo: cultivos de laboratorios médicos y patológicos; cultivos y cepas de agentes infecciosos de laboratorios industriales y de investigación; desperdicios de la producción de productos biológicos; vacunas vivas o atenuadas descartadas; platos de cultivos y mecanismos para transferir, inocular y mezclar cultivos.
- (2) Desperdicios patológicos humanos incluyendo tejidos, órganos, partes del cuerpo y fluidos corporales que son removidos durante cirugías o autopsia u otros procedimientos médicos y muestras de fluidos corporales y sus envases.
- (3) Sangre humana y productos derivados de sangre incluyendo:
  - (i) sangre humana desechada en su estado líquido;
  - (ii) productos derivados de sangre;
  - (iii) materiales saturados y/o goteando sangre humana; o
  - (iv) materiales que estuvieron saturados y/o goteando sangre humana que al presente se encuentran cubiertos de sangre seca incluyendo sueros, plasma y otros componentes sanguíneos y sus recipientes, los cuales han sido usados o se han intentado utilizar en el cuidado de pacientes, análisis y prueba de laboratorio o el desarrollo de productos farmacéuticos. Se incluyen en esta categoría las bolsas intravenosas.
- (4) Objetos filosos que han sido utilizados en el cuidado o tratamiento de pacientes humanos o animales, o en laboratorios médicos, de investigación o industriales, incluyendo agujas hipodérmicas, jeringuillas (con o sin aguja), probetas, hojas de escalpelos, ampollitas, agujas adheridas a tubos y platos de cultivo (independientemente de la presencia de agentes infecciosos). Este tipo de desperdicio incluye otros tipos de objetos de cristal que estuvieron en contacto con agentes infecciosos, tales como laminillas de microscopio desechadas y cubre objetos.

- (5) Desperdicios de animales incluyendo restos de animales contaminados, partes del cuerpo y cualquier material colocado en el lugar donde se mantienen o duermen animales cuando se conoce que los mismos han estado expuestos a agentes infecciosos durante trabajos de investigación (incluyendo los realizados en hospitales veterinarios), en el desarrollo de productos biológicos o en pruebas realizadas a productos farmacéuticos.
- (6) Desperdicios de aislamiento incluyendo biológicos y materiales desechados contaminados con sangre, excreciones, exudados o secreciones de seres humanos que son aislados para proteger a otros de la posible transmisión de enfermedades contagiosas, o de animales de los cuales se tenga conocimiento fueron infectados con enfermedades contagiosas.
- (7) Objetos cortantes o punzantes desechados que no hayan sido utilizados incluyendo: agujas hipodérmicas, agujas de sutura, jeringuillas y hojas de escalpelo.

La definición de desperdicios biomédicos no incluye desperdicios peligrosos identificados o registrados bajo las reglamentaciones en el 40 CRF 261; desperdicios domésticos, según definidos en el 40 CRF 261.4(b)(1); cenizas producto de la incineración de desperdicios biomédicos, una vez se completa el proceso de incineración; cuerpos, restos y partes anatómicas humanas que van a ser enterrados o cremados; y materiales provenientes de sistema de alcantarillado doméstico identificados en el 40 CRF 261.4 (a)(1).

#### **Desperdicios de Bajo Nivel de Radioactividad**

Significa desperdicios que contienen núclidos radioactivos que principalmente emitan radiación beta y gama o ambas, en concentraciones o cantidades que excedan los estándares federales o estatales aplicables para escapes no limitados. Los desperdicios de bajo nivel de radioactividad no son desperdicios de un alto nivel de radioactividad, combustible nuclear consumido o material derivado según definidos por la Ley Federal de Energía Atómica de 1954 (42 U.S.C. 2014 (e)(2)).

#### **Desperdicios Hospitalarios**

Significa desperdicios generados en un hospital, excepto artículos no usados devueltos al fabricante. La definición de desperdicios hospitalarios no incluye cuerpos, partes anatómicas y restos humanos a ser enterrados en un cementerio o cremados.

#### **Desperdicios Patológicos (Para propósitos de la Regla 405)**

Significa desperdicios que contengan sólo restos de seres humanos o animales, partes anatómicas y/o tejidos, las bolsas o contenedores utilizados para recoger y transportar el desperdicio y cualquier material colocado en el lugar donde se mantienen o duermen animales (si aplica).

#### **Desperdicios Quimioterapéuticos**

Significa desperdicios generados en la producción o uso de agentes antineoplásicos utilizados para detener o invertir el crecimiento de células malignas.

**Día de Operación**

Significa un periodo de 24 horas, entre las 12:00 de la medianoche y la medianoche del próximo día durante el cual se quemó cualquier cantidad de desperdicios biomédicos y hospitalarios en el IDBH.

**Dioxinas/furanos**

Significa emisiones combinadas de tetra- a octa-clorodibenzo-para dioxinas y dibenzofuranos medidas por el Método de Referencia 23 de la APA.

**Fase de Aire Máximo (*High-air Phase*)**

Significa la etapa en el ciclo de operación de una tanda cuando la cámara primaria alcanza y mantiene las temperaturas máximas de operación.

**Filtro de Tela o Colector de polvo (*Baghouse*)**

Significa sistema de control de contaminación añadido que remueve materia particulada (MP) y emisiones de metales no vaporosos al pasar el gas de la caldera a través de bolsas filtrantes.

**Fin del Proceso de Incineración (*Shutdown*) (Para propósitos de la Regla 405)**

Significa período que sigue luego que se ha consumido todo el desperdicio en la cámara primaria. Para los IDBH continuos, el fin del proceso de incineración comenzará no menos de 2 horas luego de cargar el incinerador por última vez. Para los IDBH intermitentes, el fin del proceso de incineración comenzará no menos de 4 horas luego de cargar el incinerador por última vez. Para los IDBH por tandas, el fin del proceso de incineración comenzará no menos de 5 horas luego de completada la fase de aire máximo en la combustión.

**Fluidos Corporales (Para propósitos de la Regla 405)**

Significa líquidos emanados o derivados de seres humanos y limitados a sangre: dialítico, amniótico, cerebro espinal, sinovial, pleural, fluidos peritoneales y pericardiales, semen y secreciones vaginales.

**Hospital**

Significa cualquier instalación que tenga una facultad médica organizada, mantenga por lo menos 6 camillas para enfermos residentes y donde la función principal de la institución sea proveer servicios de diagnóstico y terapéuticos a pacientes y cuidado continuo a pacientes humanos que no están relacionados y que permanezcan en la institución un promedio en exceso de 24 horas por admisión. Esta definición no incluye instalaciones que se dediquen exclusivamente a proveer servicios de asilo o convalecencia a pacientes humanos que generalmente no estén enfermos de gravedad pero que requieren supervisión médica continua.

**IDBH Continuo**

Significa un IDBH diseñado de forma que se puedan cargar desperdicios y remover las cenizas durante el proceso de combustión.

**IDBH Grande**

Significa:

- (1) Excepto según se provee en el inciso (2).
  - (i) Un IDBH cuya capacidad máxima por diseño para la quema de desperdicios es mayor de 500 libras por hora; o
  - (ii) Un IDBH continuo o intermitente cuya cantidad de carga máxima de desperdicios es mayor de 500 libras por hora; o
  - (iii) Un IDBH por tandas cuya cantidad máxima de carga de desperdicios es mayor de 4,000 libras por día.
- (2) Los siguientes no son IDBH grandes:
  - (i) Un IDBH continuo o intermitente cuya cantidad máxima de carga de desperdicios es menor o igual a 500 libras por hora;
  - (ii) Un IDBH por tandas cuya cantidad máxima de carga de desperdicios es menor o igual a 4,000 libras por día.

**IDBH Intermitente**

Significa un IDBH diseñado de forma tal que se puedan cargar desperdicios pero no remover las cenizas durante el proceso de combustión.

**IDBH Mediano**

Significa:

- (1) Excepto según se provee en el inciso (2).
  - (i) Un IDBH cuya capacidad máxima por diseño para la quema de desperdicios es mayor de 200 libras por hora pero menor o igual a 500 libras por hora; o
  - (ii) Un IDBH continuo o intermitente cuya cantidad máxima de carga de desperdicios es mayor de 200 libras por hora pero menor o igual a 500 libras por hora; o
  - (iii) Un IDBH por tandas cuya cantidad máxima de carga de desperdicios es mayor de 1,600 libras por día pero menor o igual a 4,000 libras por día.
- (2) Los siguientes no son IDBH medianos:
  - (i) Un IDBH continuo o intermitente cuya cantidad máxima de carga de desperdicios es menor o igual a 200 libras por hora o mayor de 500 libras por hora; o

- (ii) Un IDBH por tandas cuya cantidad máxima de carga de desperdicios es mayor de 4,000 libras por día pero menor o igual a 1,600 libras por día.

### **IDBH Pequeño**

Significa:

- (1) Excepto según se provee en el inciso (2),
  - (i) Un IDBH cuya capacidad máxima por diseño para la quema de desperdicios es menor o igual a 200 libras por hora; o
  - (ii) Un IDBH continuo o intermitente cuya cantidad máxima de carga de desperdicios es menor o igual a 200 libras por hora; o
  - (iii) Un IDBH por tandas cuya cantidad máxima de carga de desperdicios es menor o igual a 1,600 libras por día.
- (2) Los siguientes no son IDBH pequeños:
  - (i) Un IDBH continuo o intermitente cuya cantidad máxima de carga de desperdicios es mayor de 200 libras por hora; o
  - (ii) Un IDBH por tandas cuya cantidad máxima de carga de desperdicios es mayor de 1,600 libras por día.

### **Incinerador de Desperdicios Biomédicos y Hospitalarios o IDBH o Unidad de IDBH**

Significa cualquier equipo que queme cualquier cantidad de desperdicios biomédicos o desperdicios hospitalarios.

### **Incinerador de Desperdicios Biomédicos y Hospitalarios por tandas o IDBH por tandas**

Significa un IDBH diseñado de forma tal que no se puedan cargar desperdicios ni remover las cenizas durante el proceso de combustión.

### **Incinerador de quema combinada (*Co-fired combustors*)**

Significa una unidad que quema desperdicios de hospital y/o médico/infecciosos con otros combustibles o desperdicios (ej. carbón, desperdicios sólidos municipales) y que está sujeta a requisitos ejecutables que limiten la unidad a quemar un flujo de carga de desperdicios compuesto de 10 por ciento o menos de desperdicios de hospital y/o médico/infecciosos basado en un período trimestral. Para propósitos de esta definición, se considerará a los desperdicios patológicos, quimioterapéuticos y de bajo nivel de radioactividad como *otros* desperdicios al momento de calcular el porcentaje de desperdicios de hospital y médico/infecciosos quemados.

### **Instalación Afectada**

Significa, con relación a una fuente estacionaria, cualquier equipo al cual le aplica un estándar.

### **Instalación Designada**

Significa cualquier instalación existente que emita un contaminante designado y que, de ser una instalación afectada, estaría sujeta a un estándar de funcionamiento para ese contaminante.

### **Instalación Existente**

Significa con relación a una fuente estacionaria, cualquier equipo del tipo para el cual se haya promulgado un estándar en el 40 CRF Parte 60 y cuya construcción o modificación haya comenzado antes de la fecha en que se propuso este estándar; o cualquier aparato que pudiese ser alterado de tal manera que sea de este tipo.

### **JCA**

Significa la Junta, la Junta de Calidad Ambiental del Gobierno de Puerto Rico.

### **Junta**

Significa la Junta de Calidad Ambiental (JCA) del Gobierno de Puerto Rico.

### **Lavador de gases húmedo**

Significa sistema de control de contaminación añadido el cual utiliza un licor alcalino que extrae impurezas para recoger materia particulada (incluyendo orgánicos condensados y metales no vaporosos) y/o para absorber y neutralizar los gases ácidos.

### **Lavador de gases seco**

Significa sistema de control de contaminación añadido el cual inyecta un absorbente seco alcalino (inyección seca) o rocía un absorbente alcalino (rociado seco) el cual reacciona y neutraliza los gases ácidos en el flujo de escape del IDBH formando un material seco en polvo.

### **Mal Funcionamiento (Para propósitos de la Regla 405)**

Significa cualquier fallo repentino, infrecuente y que no sea razonablemente prevenible en un equipo de control de contaminación atmosférica, en un equipo de proceso o en un proceso para operar en la forma normal o usual. Los fallos causados en parte por un pobre mantenimiento u operación descuidada no serán considerados como mal funcionamiento. Durante los períodos de mal funcionamiento el operador deberá operar dentro de los parámetros establecidos hasta donde le sea posible y deberá continuar con el muestreo de todos los parámetros de operación aplicables hasta que todos los desperdicios hayan sido quemados o hasta que cese el mal funcionamiento, lo que suceda primero.

### **Materia Particulada o MP (Para propósitos de la Regla 405)**

Significa el total de materia particulada emitida por un IDBH medido de acuerdo con los Métodos de Referencia 5 ó 29 de la APA.

### **Modificación o IDBH Modificado (Para propósitos de la Regla 405)**

Significa cualquier cambio a una unidad de IDBH luego de la fecha de efectividad de estos estándares, siempre y cuando:

- (1) Los costos acumulativos de las modificaciones durante el período de vida de la unidad excedan en un 50 por ciento el costo original de la construcción e instalación de la unidad (sin incluir el costo de cualquier compra de terreno en relación con la construcción o instalación), computado de acuerdo con los costos actuales; o
- (2) El cambio requiera un cambio físico o un cambio en el método de operación de la unidad el cual aumente la cantidad de cualquier contaminante atmosférico emitido por la unidad para el cual se hayan establecido estándares bajo las secciones 129 ó 111 de la Ley.

### **Operación**

Significa período durante el cual se queman desperdicios en el incinerador, excluyendo aquellos periodos definidos como comienzo y fin del proceso de incineración.

### **Operador de Incinerador de Desperdicios Biomédicos y Hospitalarios u Operador de IDBH**

Significa cualquier persona que opere, controle o supervise las operaciones diarias de un IDBH.

### **pH Mínimo del Licor del Lavador de gases**

Significa el 90 por ciento del promedio de tres horas más alto del pH del licor del lavador de gases a la entrada del lavador de gases húmedo (tomado por lo menos una vez cada minuto) medido durante la prueba de funcionamiento más reciente para demostrar cumplimiento con el límite de emisión de HCl.

### **Pirólisis**

Significa la gasificación endotérmica de los desperdicios biomédicos y hospitalarios por medio de energía externa.

### **Productos Biológicos (Para propósitos de la Regla 405)**

Significa preparación hecha con organismos vivos y sus productos incluyendo vacunas, cultivos, etcétera para utilizarse en el diagnóstico, inmunización o tratamiento de seres humanos o animales o en la investigación relacionada con éstos.

### **Productos Sanguíneos (Para propósitos de la Regla 405)**

Significa cualquier producto derivado de sangre humana incluyendo, pero sin limitarse a, plasma, plaquetas, glóbulos blancos o rojos y otros productos derivados, como el interferón, etcétera.

**Razón de Flujo Mínima del Absorbente de Ácido Hidroclórico (HCl)**

Significa el 90 por ciento de la razón de flujo del absorbente de HCl promedio de tres horas más alto (tomado por lo menos una vez cada hora) medido durante la prueba de funcionamiento más reciente para demostrar el cumplimiento con los límites de emisión de HCl.

**Razón de Flujo Mínima del Licor del Lavador de gases**

Significa el 90 por ciento del promedio de tres horas más alto de la razón de flujo del licor en la entrada del lavador de gases húmedo (tomado por lo menos una vez cada minuto) medido durante la prueba de funcionamiento más reciente para demostrar cumplimiento con todos los límites de emisión aplicables.

**Razón de Flujo Mínima del Absorbente de Dioxinas/furanos**

Significa el 90 por ciento del promedio de tres horas más alto de la razón de flujo del absorbente de dioxina/furano (tomado por lo menos una vez cada hora) medido durante la prueba de funcionamiento más reciente para demostrar el cumplimiento con el límite de emisión de Hg.

**Sistema Continuo de Muestreo de Emisiones o SCME**

Significa sistema de muestreo para medir y registrar de forma continua las emisiones de contaminantes provenientes de una instalación afectada.

**Temperatura Máxima a la Entrada del Filtro de Tela**

Significa el 110 por ciento de la temperatura promedio de tres horas más baja en la entrada del filtro de tela (tomada por lo menos una vez cada minuto) medida durante la prueba de funcionamiento más reciente para demostrar cumplimiento con el límite de emisión de dioxina/furano.

**Temperatura Máxima de los Gases de Chimenea**

Significa el 110 por ciento de la temperatura promedio de tres horas más baja en la salida del lavador de gases húmedo (tomada por lo menos una vez cada minuto) medida durante la prueba de funcionamiento más reciente para demostrar cumplimiento con el límite de emisión de mercurio (Hg).

**Temperatura Mínima de la Cámara Secundaria**

Significa el 90 por ciento del promedio de tres horas más alto de la temperatura en la cámara secundaria (tomado por lo menos una vez cada minuto) medido durante la prueba de funcionamiento más reciente para demostrar cumplimiento con los límites de emisión de MP, CO o de dioxinas/furanos.

## Revisión a la Regla 405 INCINERACIÓN

### (a) Requisitos para los Incineradores de Desperdicios Sólidos No-Peligrosos

(a)(1) Aplicabilidad - Esta Sección (a) de la Regla 405 aplica a todos los incineradores de desperdicios sólidos no peligrosos existentes, nuevos y modificados no cubiertos por la Sección (b) de esta Regla 405.

(a)(1)(A) Los incineradores existentes a la fecha de adopción de esta Regla deberán cumplir con esta sección dentro de un término de 6 meses a partir de la fecha de efectividad de esta regla y deberán completar una prueba de funcionamiento para demostrar el cumplimiento con los límites establecidos en esta Sección (a).

(a)(1)(B) Los incineradores nuevos deberán completar una prueba de funcionamiento para demostrar el cumplimiento con los límites establecidos en esta Sección (a) dentro de un término de 180 días a partir de la fecha de la aprobación del primer permiso de operación.

(a)(1)(C) Todos los incineradores afectados por esta Sección (a) deberán completar una prueba de funcionamiento para demostrar el cumplimiento con la regla cada 5 años luego de la primera prueba de funcionamiento.

(a)(1)(D) Esta regla no aplicará a incineradores de desperdicios sólidos no-peligrosos, excepto por los siguientes requisitos:

(a)(1)(D)(i) Deberá cumplir con la limpieza diaria periódica de la cámara de combustión después de la última actividad de incineración del día y antes de comenzar a cargar nuevamente esta cámara.

(a)(1)(D)(ii) Deberán cumplir con un plan de mantenimiento para la cámara de decantación para evitar excedencias del 20% del límite de opacidad según requerido bajo la Regla 403.

(a)(1)(D)(iii) Deberán segregar los desperdicios y reciclar o disponer de los plásticos, vidrios, metales y baterías en un centro de acopio de material reciclable de tal manera que los mismos no sean incinerados.

(a)(1)(D)(iv) Deberán cumplir con cualquier otro requisito aplicable para los incineradores domésticos de desperdicios sólidos no-peligrosos establecidos por la Junta.

- (a)(1)(E) Los incineradores de desperdicios sólidos no peligrosos existentes con una capacidad de 15 ton/día o menor que hayan obtenido un permiso de fuente de emisión previamente y que hayan realizado las pruebas de cumplimiento dentro de los pasados 5 años, no se les requerirá realizar una prueba de funcionamiento inicial para materia particulada (MP) si su determinación de cumplimiento anterior demostró cumplimiento con el estándar establecido por esta regla en el párrafo (a)(2). Para los incineradores con una capacidad de 15 ton/día o menor que deben cumplir con la Sección (a)(1)(C), la próxima prueba será requerida dentro de 5 años después de la fecha de efectividad de esta regla.
- (a)(2) Ningún incinerador de desperdicios sólidos no-peligrosos causará o permitirá la emisión de materia particulada (MP) en exceso de 0.40 libras por cada 100 libras (4 gm/kg) de carga de desperdicios.
- (a)(3) Cualquier persona que opere un incinerador de desperdicios sólidos no-peligrosos deberá someter a la Junta una certificación para demostrar que ha recibido el adiestramiento adecuado para operar tales incineradores y el equipo relacionado, la cual deberá ser similar a los requisitos para la certificación y adiestramiento de operadores en la Sección (b)(3).
- (a)(4) Cualquier incinerador afectado por esta regla deberá cumplir con los requisitos aplicables bajo la Regla 106.
- (a)(5) Cualquier incinerador de desperdicios sólidos no-peligrosos que tenga que cumplir con cualquier reglamento o requisito aplicable bajo las Normas de Funcionamiento para Nuevas Fuentes Estacionarias (NFNFE), Normas Nacionales de Emisión para Contaminantes Atmosféricos Peligrosos (NNECAP) o las normas de Tecnología de Control Máxima Obtenible (TCMO) está exento de cumplir con la Sección (a) de esta regla.

**(b) Guías de Emisión e Itinerario de Cumplimiento para los Incineradores de Desperdicios Biomédicos y Hospitalarios (IDBH)**

**(b)(1) Aplicabilidad**

- (b)(1)(A) La instalación designada a la cual le aplica esta sección es cada incinerador individual de desperdicios biomédicos y hospitalarios (IDBH) cuya construcción fue comenzada el 20 de junio de 1996 o antes, excepto según se provee en los párrafos (b)(1)(B) a (b)(1)(H) de esta sección.
- (b)(1)(B) Un incinerador no está sujeto a esta sección durante aquellos períodos cuando sólo queme desperdicios patológicos, desperdicios de bajo nivel de radioactividad y/o desperdicios quimioterapéuticos, siempre que el dueño u operador del incinerador:
- (b)(1)(B)(i) Notifique a la JCA su reclamo de exención con copia a la APA; y
- (b)(1)(B)(ii) Mantenga expedientes trimestrales de los períodos de tiempo en que sólo se quemen desperdicios patológicos, desperdicios de bajo nivel de radioactividad y/o desperdicios quimioterapéuticos; y

- (b)(1)(B)(iii) Mantener tales registros que demuestren que las fuentes logran y mantienen la exención por al menos 5 años consecutivos y que los sometán cuando sean requeridos dentro de 10 días laborables.
- (b)(1)(C) Cualquier incinerador de quema combinada no está sujeto a esta sección, si el dueño u operador del incinerador:
  - (b)(1)(C)(i) Notifica a la APA su reclamo de exención con copia a la JCA; y
  - (b)(1)(C)(ii) Provea un estimado del peso relativo de desperdicios hospitalarios, desperdicios médicos/infecciosos y otros combustibles y/o desperdicios a ser quemados; y
  - (b)(1)(C)(iii) Mantener expedientes trimestrales sobre el peso relativo de desperdicios hospitalarios y los desperdicios médico/infecciosos quemados y de otros combustibles y/o desperdicios quemados en el incinerador; y
  - (b)(1)(C)(iv) Mantener tales registros que demuestren que las fuentes logran y mantienen la exención por al menos 5 años consecutivos y que los sometán cuando sean requeridos dentro de 10 días laborables.
- (b)(1)(D) Cualquier incinerador que requiere tener un permiso bajo la Sección 3005 de la Ley Federal de Disposición de Desperdicios Sólidos no está sujeto a esta sección.
- (b)(1)(E) Cualquier incinerador que cumpla con los requisitos de aplicabilidad bajo las subpartes Cb, Ea o Eb del 40 CRF Parte 60 (estándares o guías para ciertos incineradores de desperdicios municipales) no está sujeto a esta sección.
- (b)(1)(F) Cualquier unidad de pirólisis no está sujeta a esta sección.
- (b)(1)(G) Los hornos de cemento que quemen desperdicios de hospital y/o desperdicios médico/infecciosos no están sujetos a esta sección.
- (b)(1)(H) Cualquier cambio físico u operacional realizado a un IDBH existente con el único propósito de cumplir con las guías de emisión aquí establecidas no será considerado como una modificación ni tendrá el efecto de convertir el IDBH en uno sujeto a las disposiciones de la subparte Ec del 40 CRF Parte 60.
- (b)(1)(I) Toda instalación designada sujeta a esta sección deberá operar conforme a un permiso de operación Título V según los requisitos de la Parte VI del Reglamento para el Control de la Contaminación Atmosférica, no más tarde del 15 de septiembre de 2000.

**(b)(2) Límites de Emisión**

- (b)(2)(A) Ningún dueño u operador de una instalación designada causará la emisión de gases a la atmósfera desde ésta en exceso de los límites presentados en la Tabla 1 en o después de la fecha en la cual la prueba de funcionamiento inicial es completada o se requiere que se complete bajo esta regla, lo que ocurra primero, excepto como se provee en el párrafo (b)(2)(B) de esta sección.

**TABLA 1. LÍMITE DE EMISIÓN PARA IDBH  
PEQUEÑOS, MEDIANOS Y GRANDES**

Contaminante	Unidades (7 por ciento de oxígeno, en base seca)	Límites de Emisión		
		Tamaño del IDBH		
		Pequeño	Mediano	Grande
Materia particulada	miligramos por metro cúbico seco estándar (gramo por pie cúbico seco estándar)	115 (0.05)	69 (0.03)	34 (0.015)
Monóxido de carbono	partes por millón por volumen	40	40	40
Dioxinas/furanos	nanogramos por metro cúbico seco estándar (gramos por billón de pie cúbico seco estándar) o nanogramos por metro cúbico seco estándar total ET (granos por billón de pie cúbico seco estándar)	125 (55) ó 2.3 (1.0)	125 (55) ó 2.3 (1.0)	125 (55) ó 2.3 (1.0)
Ácido hidroc্লórico	partes por millón por volumen o por ciento de reducción	100 ó 93%	100 ó 93%	100 ó 93%
Bióxido de azufre	partes por millón por volumen	55	55	55
Óxidos de nitrógeno	partes por millón por volumen	250	250	250
Plomo	miligramos por metro cúbico seco estándar (granos por mil de pie cúbico seco estándar) o por ciento de reducción	1.2 (0.52) ó 70%	1.2 (0.52) ó 70%	1.2 (0.52) ó 70%
Cadmio	miligramos por metro cúbico seco estándar (granos por mil de pie cúbico seco estándar) o por ciento de reducción	0.16 (0.07) ó 65%	0.16 (0.07) ó 65%	0.16 (0.07) ó 65%
Mercurio	miligramos por metro cúbico seco estándar (granos por mil de pie cúbico seco estándar) o por ciento de reducción	0.55 (0.24) ó 85%	0.55 (0.24) ó 85%	0.55 (0.24) ó 85%

(b)(2)(B) Cualquier IDBH pequeño localizado a más de 50 millas del límite del Área Estadística Metropolitana Estándar (AEMS) más cercana, y que queme menos de 2,000 libras de desperdicios hospitalarios y médico/infecciosos por semana, no podrá emitir ningún gas desde su chimenea en exceso de los límites presentados en la Tabla 2. El límite de 2,000 libras por semana no aplicará durante pruebas de funcionamiento.

(b)(2)(C) Ningún dueño u operador de una instalación designada podrá causar la emisión de gases a la atmósfera desde su chimenea que tengan una opacidad mayor de 10 por ciento (promedio bloque de 6 minutos) en o después de que la prueba inicial de funcionamiento es completada o se requiere que se complete bajo esta regla, lo que ocurra primero.

**(b)(3) Requisitos de adiestramiento y certificación de operadores**

(b)(3)(A) Ningún dueño u operador de una instalación designada podrá permitir la operación de la instalación designada en ningún momento en que no se encuentre accesible, ya sea en la instalación o disponible dentro de una hora, un operador de IDBH adiestrado y calificado. El operador de IDBH adiestrado y calificado podrá operar el IDBH directamente o ser el supervisor directo de uno o más operadores del IDBH.

(b)(3)(B) El adiestramiento y calificación del operador deberá obtenerse a través de un programa aprobado por la Junta o que cumpla con los requisitos incluidos en los párrafos (b)(3)(C) a (b)(3)(G) de esta sección.

(b)(3)(C) El adiestramiento se podrá obtener por medio de un curso de adiestramiento de operadores de IDBH que incluya, como mínimo, las siguientes condiciones:

(b)(3)(C)(i) 24 horas de adiestramiento sobre los siguientes temas:

(b)(3)(C)(i)(1) Preocupaciones ambientales incluyendo destrucción de patógenos y los tipos de emisiones;

(b)(3)(C)(i)(2) Principios básicos de la combustión incluyendo los productos de la combustión;

(b)(3)(C)(i)(3) Operación del tipo de incinerador a ser utilizado por el operador incluyendo comienzo adecuado del proceso de incineración, carga de desperdicios y procedimientos sobre el fin del proceso de incineración;

(b)(3)(C)(i)(4) Controles de combustión y muestreo;

**TABLE 2. LÍMITES DE EMISIÓN PARA IDBH PEQUEÑOS QUE CUMPLAN CON LOS REQUISITOS DE RURAL**

<b>Contaminante</b>	<b>Unidades (7 por ciento de oxígeno, en base seca)</b>	<b>Límites de Emisión IDBH</b>
Materia particulada	miligramos por metro cúbico seco estándar (gramos por pie cúbico seco estándar)	197 (0.086)
Monóxido de carbono	partes por millón por volumen	40
Dioxinas/furanos	nanogramos por metro cúbico seco estándar (granos por billón de pie cúbico seco estándar) o nanogramos por metro cúbico seco estándar total ET (gramos por billón de pie cúbico seco estándar)	800 (350) ó 15 (6.6)
Ácido hidroclicóric	partes por millón por volumen	3100
Bióxido de azufre	partes por millón por volumen	55
Óxidos de nitrógeno	partes por millón por volumen	250
Plomo	miligramos por metro cúbico seco estándar (granos por mil de pie cúbico seco estándar)	10 (4.4)
Cadmio	miligramos por metro cúbico seco estándar (granos por mil de pie cúbico seco estándar)	4 (1.7)
Mercurio	miligramos por metro cúbico seco estándar (granos por mil de pie cúbico seco estándar)	7.5 (3.3)

- (b)(3)(C)(i)(5) Operación de equipo de control de contaminación atmosférica y factores que afectan el funcionamiento (si aplica);
- (b)(3)(C)(i)(6) Métodos para el muestreo de contaminantes (sistemas continuos para el muestreo de emisiones y muestreo de parámetros de operación del IDBH y de los equipos de control de contaminación atmosférica) y procedimientos de calibración del equipo (donde apliquen);
- (b)(3)(C)(i)(7) Inspección y mantenimiento del IDBH, de los equipos de control de contaminación atmosférica y de los sistemas continuos para el muestreo de emisiones;
- (b)(3)(C)(i)(8) Acciones para corregir el mal funcionamiento u otras condiciones que puedan conducir al mal funcionamiento;
- (b)(3)(C)(i)(9) Procedimientos para el manejo y caracterización de cenizas;
- (b)(3)(C)(i)(10) Reglamentaciones federales, estatales y locales aplicables;
- (b)(3)(C)(i)(11) Procedimientos de seguridad en el trabajo;
- (b)(3)(C)(i)(12) Inspecciones previas al comienzo del proceso de incineración y
- (b)(3)(C)(i)(13) Requisitos para mantener expedientes.
- (b)(3)(C)(ii) Un examen diseñado y administrado por el instructor.
- (b)(3)(C)(iii) Materiales de referencia distribuidos a los participantes en donde se cubran los tópicos del curso.
- (b)(3)(D) La calificación se obtendrá por:
  - (b)(3)(D)(i) Completar un curso de adiestramiento que satisfaga los criterios bajo el párrafo (b)(C)(3) de esta sección y
  - (b)(3)(D)(ii) Seis meses de experiencia como un operador de IDBH, seis meses de experiencia como supervisor directo de un operador de IDBH o la realización efectiva de dos ciclos de quema bajo la observación de dos operadores calificados.
- (b)(3)(E) La calificación es válida desde la fecha en que se apruebe el examen o se completen los requisitos de experiencia, lo que ocurra más tarde.
- (b)(3)(F) Para mantener la calificación, el operador de IDBH adiestrado y calificado deberá completar y aprobar un repaso anual o un curso de renovación de por lo menos cuatro horas de duración en donde se cubra, como mínimo, lo siguiente:

- (b)(3)(F)(i) Actualización de la reglamentación aplicable;
- (b)(3)(F)(ii) Operación del incinerador incluyendo comienzo y fin del proceso de incineración;
- (b)(3)(F)(iii) Inspección y mantenimiento;
- (b)(3)(F)(iv) Respuestas a situaciones de mal funcionamiento o condiciones que puedan conducir al mal funcionamiento; y
- (b)(3)(F)(v) Discusión de problemas de operación experimentados por los participantes.
- (b)(3)(G) Se podrá renovar una calificación que haya caducado por medio de los siguientes métodos:
  - (b)(3)(G)(i) En caso de que la calificación haya caducado hace menos de tres años, el operador del IDBH deberá completar y aprobar un repaso anual o curso de renovación según descrito en el párrafo (b)(3)(F) de esta sección.
  - (b)(3)(G)(ii) En caso de que la calificación haya caducado hace más de tres años, el operador del IDBH deberá completar y aprobar un curso que contenga los criterios mínimos descritos en el párrafo (b)(3)(C) de esta sección.
- (b)(3)(H) El dueño u operador de una instalación designada deberá mantener en la instalación los documentos que contengan lo siguiente:
  - (b)(3)(H)(i) Resumen de los estándares aplicables bajo esta regla;
  - (b)(3)(H)(ii) Descripción de la teoría básica de combustión aplicable a un IDBH;
  - (b)(3)(H)(iii) Procesos para recibir, manejar y cargar desperdicios;
  - (b)(3)(H)(iv) Procedimientos para el comienzo y fin del proceso de incineración y para el mal funcionamiento de los IDBH;
  - (b)(3)(H)(v) Procedimientos para mantener niveles apropiados de suministros de aire para la combustión;
  - (b)(3)(H)(vi) Procedimientos para operar el IDBH y los sistemas de control de contaminación atmosférica asociados dentro de los estándares establecidos bajo esta regla;
  - (b)(3)(H)(vii) Procedimientos para responder a un mal funcionamiento periódico o a condiciones que puedan conducir al mal funcionamiento;
  - (b)(3)(H)(viii) Procedimientos para muestrear las emisiones del IDBH;

- (b)(3)(H)(ix) Procedimientos para someter informes y mantener expedientes y
- (b)(3)(H)(x) Procedimientos para manejar las cenizas.
- (b)(3)(I) El dueño u operador de una instalación designada deberá establecer un programa para revisar anualmente la información registrada en el párrafo (b)(3)(H) de esta sección con cada operador de IDBH. Este programa de revisión del expediente del IDBH deberá ser implementado por cada operador.
- (b)(3)(I)(i) La revisión inicial de la información registrada en el párrafo (b)(3)(H) de esta sección deberá realizarse dentro de 6 meses después de la fecha de efectividad de esta regla o antes de asumir responsabilidades que afecten la operación del IDBH, lo que ocurra más tarde.
- (b)(3)(I)(ii) Las revisiones subsiguientes a la información registrada en el párrafo (b)(3)(H) de esta sección deben ser realizadas anualmente.
- (b)(3)(J) La información registrada en el párrafo (b)(3)(H) de esta sección deberá mantenerse en un lugar accesible para todos los operadores del IDBH. Esta información conjuntamente con los expedientes de adiestramiento deben estar disponibles para inspección por la APA o su representante de cumplimiento delegado cuando lo solicite.

**(b)(4) Plan de manejo de desperdicios**

El dueño u operador de una instalación designada deberá preparar un plan de manejo de desperdicios. El plan de manejo de desperdicios deberá identificar la posibilidad y la manera de separar ciertos componentes de desperdicios sólidos del flujo de desperdicios de cuidado de la salud de manera que se pueda reducir la cantidad de emisiones tóxicas proveniente de los desperdicios incinerados. Un plan de manejo de desperdicios puede incluir, pero no está limitado, al reciclaje de elementos tales como papel, cartón, plásticos, cristal, baterías o metales; o a la compra de productos reciclables o reciclados. Un plan de manejo de desperdicios puede incluir metas o estrategias diferentes para las diferentes áreas o departamentos de la instalación y no necesita incluir metas nuevas de manejo para cada flujo de desperdicios. Este deberá identificar, hasta donde sea posible, medidas adicionales que sean razonables y que estén disponibles para el manejo de desperdicios, tomando en consideración la efectividad de medidas de manejo de desperdicios ya implantadas, los costos de medidas adicionales, las reducciones de emisiones que se esperan lograr y cualquier otro impacto ambiental o de energía que puedan tener. La publicación de la Asociación Americana de Hospitales titulada *An Ounce of Prevention: Waste Reduction Strategies for Health Care Facilities* deberá ser considerada en el desarrollo del plan de manejo de desperdicios.

**(b)(5) Requisitos de Inspección**

(b)(5)(A) Cualquier IDBH pequeño que cumpla con los requisitos para ser considerado como rural según la Sección (b)(2)(B) y que esté sujeto a los límites de emisión de la Tabla 2 de esta regla, deberá realizar una inspección inicial de su equipo dentro de un año después de la aprobación del Plan Estatal o después de la fecha de publicación del Plan Federal de la APA, lo que ocurra primero, según es requerido en esta sección.

(b)(5)(A)(i) La inspección deberá incluir como mínimo lo siguiente:

(b)(5)(A)(i)(1) Inspeccionar que todos los mecheros, ensamblaje de los encendedores pilotos y sensores operen de manera correcta, limpiar el sensor de la llama de los encendedores pilotos, según sea necesario;

(b)(5)(A)(i)(2) Asegurarse de que el aire de las cámaras de combustión primarias y secundarias esté ajustado correctamente y ajustarlo según sea necesario;

(b)(5)(A)(i)(3) Inspeccionar los goznes y los cerrojos de las puertas y lubricarlos según sea necesario;

(b)(5)(A)(i)(4) Inspeccionar que el regulador del tiro de chimenea, abanicos y ventiladores operen correctamente;

(b)(5)(A)(i)(5) Inspeccionar que las puertas y las justas de las puertas sellen correctamente;

(b)(5)(A)(i)(6) Inspeccionar que los motores funcionen de forma correcta;

(b)(5)(A)(i)(7) Inspeccionar el revestimiento refractorio de la cámara primaria; limpiar y reparar/reemplazar el mismo según sea necesario;

(b)(5)(A)(i)(8) Inspeccionar el cuerpo del incinerador para indicadores de corrosión y/o partes calientes;

(b)(5)(A)(i)(9) Inspeccionar la cámara secundaria/terciaria y la chimenea, limpiarlas según sea necesario;

(b)(5)(A)(i)(10) Inspeccionar que el cargador mecánico, que incluye los interruptores de límite, opere correctamente, si aplica;

(b)(5)(A)(i)(11) Inspeccionar visualmente la parrilla de desperdicios y reparar o sellar, según sea necesario;

(b)(5)(A)(i)(12) En el ciclo de quema que se realice luego de la inspección, documentar que el incinerador está funcionando correctamente y hacer cualquier ajuste necesario;

- (b)(5)(A)(i)(13) Inspeccionar que el equipo de control de contaminación atmosférica opere correctamente, si aplica;
- (b)(5)(A)(i)(14) Inspeccionar que el sistema de desperdicios de calderas opere correctamente, si aplica;
- (b)(5)(A)(i)(15) Inspeccionar los componentes de la chimenea auxiliar;
- (b)(5)(A)(i)(16) Asegurar que los pares termoeléctricos, los sistemas de alimentación de absorbentes y cualquier otro equipo de muestreo se calibren correctamente; y
- (b)(5)(A)(i)(17) Observar, en términos generales, que el equipo se mantenga en buenas condiciones de operación.
- (b)(5)(A)(ii) Todas las reparaciones necesarias deben ser realizadas dentro de 10 días de operación a partir de la inspección del equipo, a menos que el dueño u operador obtenga una aprobación por escrito de la Agencia Estatal que establezca una fecha alterna para la cual todas las reparaciones necesarias en la instalación designada deben ser completadas.
- (b)(5)(B) Cualquier IDBH pequeño que cumpla con los requisitos para ser considerado como rural según la Sección (b)(2)(B) y que esté sujeto a los límites de emisión de la Tabla 2 de esta regla, deberá realizar una inspección al equipo anualmente (en o antes del aniversario de la inspección anterior) según se resume en los párrafos (b)(5)(A)(i) y (b)(5)(A)(ii) de esta sección.

**(b)(6) Requisitos de Cumplimiento y de Pruebas de Funcionamiento**

- (b)(6)(A) Los límites de emisión bajo esta regla aplican en todo momento, excepto durante el comienzo y fin del proceso de incineración, o durante períodos de mal funcionamiento, siempre que no se carguen desperdicios hospitalarios o desperdicios médico/infecciosos en la instalación designada durante estos períodos.
- (b)(6)(B) El dueño u operador de una instalación designada deberá realizar una prueba inicial de funcionamiento, para determinar el cumplimiento con los límites de emisión utilizando los procedimientos y métodos de prueba registrados en los párrafos (b)(6)(B)(i) a (b)(6)(B)(ix) de esta sección, según es requerido bajo esta regla. El uso de una chimenea auxiliar durante una prueba de funcionamiento invalidará esta prueba.
  - (b)(6)(B)(i) Todas las pruebas de funcionamiento consistirán de un mínimo de tres corridas de prueba realizadas bajo condiciones representativas de la operación.
  - (b)(6)(B)(ii) El tiempo mínimo para tomar la muestra será de una hora por corrida, a menos que se indique lo contrario.

- (b)(6)(B)(iii) Se utilizará el Método de Referencia 1 de la APA del apéndice A del 40 CRF Parte 60 e incorporado a este reglamento en su apéndice, para seleccionar el lugar de la muestra y la cantidad de puntos transversales.
- (b)(6)(B)(iv) Se utilizará el Método de Referencia 3 ó 3A de la APA del apéndice A del 40 CRF Parte 60 e incorporado a este reglamento en su apéndice, para realizar los análisis de composición de los gases, que incluye la medida de concentración de oxígeno. Se utilizará el Método de Referencia 3 ó 3A de la APA del apéndice A del 40 CRF Parte 60 simultáneamente con cada método de referencia.
- (b)(6)(B)(v) Las concentraciones de contaminantes se ajustarán a un 7 por ciento de oxígeno utilizando la siguiente ecuación:

$$C_{\text{ajustada}} = C_{\text{medida}} (20.9 - 7) / (20.9 - \%O_2)$$

donde:

- $C_{\text{ajustada}}$  = concentración de contaminante ajustada a un 7 por ciento oxígeno;
- $C_{\text{medida}}$  = concentración de contaminante medida en una base seca;
- $(20.9 - 7)$  = 20.9 por ciento oxígeno - 7 por ciento oxígeno (base de corrección de oxígeno definida);
- 20.9 = concentración de oxígeno en el aire, por ciento; y
- $\%O_2$  = concentración de oxígeno medida en una base seca, por ciento.

- (b)(6)(B)(vi) Se utilizará el Método de Referencia 5 ó 29 de la APA del apéndice A del 40 CRF Parte 60 e incorporado a este reglamento en su apéndice, para medir las emisiones de materia particulada.
- (b)(6)(B)(vii) Se utilizará el Método de Referencia 9 de la APA del apéndice A del 40 CRF Parte 60 e incorporado a este reglamento en su apéndice, para medir opacidad de la chimenea.
- (b)(6)(B)(viii) Se utilizará el Método de Referencia 10 ó 10B de la APA del apéndice A del 40 CRF Parte 60 e incorporado a este reglamento en su apéndice, para medir las emisiones de CO.
- (b)(6)(B)(ix) Se utilizará el Método de Referencia 23 de la APA del apéndice A del 40 CRF Parte 60 e incorporado a este reglamento en su apéndice, para medir emisiones totales de dioxinas/furanos. El tiempo mínimo para tomar la muestra será de cuatro horas por corrida de prueba. En el caso de que la instalación designada seleccione los estándares de equivalencias tóxicas para dioxinas/furanos, se utilizará el siguiente procedimiento para determinar el cumplimiento:
  - (b)(6)(B)(ix)(1) Medirá la concentración de cada tetra- a octa-congénere de dioxinas/furanos emitidos utilizando el Método de Referencia 23 de la APA.

- (b)(6)(B)(ix)(2) Para cada congénere de dioxinas/furanos medido de acuerdo con el párrafo (b)(6)(B)(ix)(1) de esta sección, multiplicará la concentración de congénere por el factor de equivalencia tóxica correspondiente especificado en la Tabla 3 de esta regla.
- (b)(6)(B)(ix)(3) Sumará los productos calculados de acuerdo con el párrafo (b)(6)(B)(ix)(2) de esta sección, para obtener la concentración total de dioxinas/furanos emitidos en términos de su equivalencia tóxica.
- (b)(6)(B)(x) Se utilizará el Método de Referencia 26 de la APA del apéndice A del 40 CRF Parte 60 e incorporado a este reglamento en su apéndice, para medir emisiones de HCl. Si la instalación designada ha seleccionado los estándares de reducción de porcentaje para HCl bajo la Sección (b) (2) de esta regla, el por ciento de reducción de emisiones de HCl ( $\%R_{HCl}$ ) se calcula utilizando la siguiente fórmula:

$$(\%R_{HCl}) = \left( \frac{E_i - E_o}{E_i} \right) \times 100$$

donde:

- $\%R_{HCl}$  = porcentaje de reducción alcanzado para emisiones de HCl;  
 $E_i$  = concentración de emisiones de HCl medida en la entrada del equipo de control, corregida a un 7 por ciento de oxígeno (base seca); y  
 $E_o$  = concentración de emisiones de HCl medida en la salida del equipo de control, corregida a un 7 por ciento de oxígeno (base seca).

- (b)(6)(B)(xi) Se utilizará el Método de Referencia 29 de la APA del apéndice A del 40 CRF Parte 60 e incorporado en el apéndice de este reglamento para medir emisiones de Pb, Cd y Hg. Si la instalación designada ha seleccionado los estándares de reducción de porcentaje para metales bajo la Sección (b) (2) de esta regla, el por ciento de reducción de emisiones ( $\%R_{metal}$ ) se calcula utilizando la siguiente fórmula:

$$(\%R_{Metal}) = \left( \frac{E_i - E_o}{E_i} \right) \times 100$$

donde:

- $\%R_{metal}$  = porcentaje de reducción alcanzado para emisiones de metales (Pb, Cd, o Hg);  
 $E_i$  = concentración de emisiones de metales (Pb, Cd o Hg) medida en la entrada del equipo de control, corregida a un 7 por ciento de oxígeno (base seca); y  
 $E_o$  = concentración de emisiones de metales (Pb, Cd o Hg) medida en la salida del equipo de control, corregida a un 7 por ciento de oxígeno (base seca).

**TABLA 3. FACTORES DE EQUIVALENCIA TÓXICA**

CONGÉNERE DIOXINAS/FURANOS	FACTOR DE EQUIVALENCIA TÓXICA
2,3,7,8 - tetraclorodibenzo-p-dioxina	1
1,2,3,7,8 - pentaclorodibenzo-p-dioxina	0.5
1,2,3,4,7,8 - hexaclorodibenzo-p-dioxina	0.1
1,2,3,7,8,9 - hexaclorodibenzo-p-dioxina	0.1
1,2,3,6,7,8 - hexaclorodibenzo-p-dioxina	0.1
1,2,3,4,6,7,8 - heptaclorodibenzo-p-dioxina	0.01
octaclorodibenzo-p-dioxina	0.001
dibenzofurano 2,3,7,8 - tetraclorinado	0.1
dibenzofurano 2,3,4,7,8 - pentaclorinado	0.5
dibenzofurano 1,2,3,7,8 - pentaclorinado	0.05
dibenzofurano 1,2,3,4,7,8 - hexaclorinado	0.1
dibenzofurano 1,2,3,6,7,8 - hexaclorinado	0.1
dibenzofurano 1,2,3,7,8,9 - hexaclorinado	0.1
dibenzofurano 2,3,4,6,7,8 - hexaclorinado	0.1
dibenzofurano 1,2,3,4,6,7,8 - heptaclorinado	0.01
dibenzofurano 1,2,3,4,7,8,9 - heptaclorinado	0.01
dibenzofurano octaclorinado	0.001

- (b)(6)(C) Próximo a la fecha en que la prueba inicial de funcionamiento es completada o se requiere que se complete bajo esta regla, lo que ocurra primero, el dueño u operador de la instalación deberá:
- (b)(6)(C)(i) Determinar el cumplimiento con los límites de opacidad por medio de una prueba anual de funcionamiento (no más tarde de 12 meses luego de la prueba de funcionamiento anterior) utilizando los procedimientos aplicables y los métodos de prueba establecidos en el párrafo (b)(6)(B) de esta sección.
  - (b)(6)(C)(ii) Determinar el cumplimiento con los límites de emisión para MP, CO y HCl por medio de una prueba de funcionamiento anual (no más tarde de 12 meses luego de la prueba de funcionamiento anterior) utilizando los procedimientos aplicables y los métodos de prueba establecidos en el párrafo (b)(6)(B) de esta sección. En el caso de que tres pruebas de funcionamiento indiquen que la instalación está en cumplimiento con los límites de emisión para un contaminante (MP, CO o HCl) durante un período de tres años, el dueño u operador no tendrá que realizar la prueba de funcionamiento para este contaminante por los próximos dos años. Como mínimo, se deberá realizar una prueba de funcionamiento para MP, CO y HCl cada tercer año (no más tarde de 36 meses luego de la prueba de funcionamiento anterior). Si la prueba de funcionamiento realizada cada tres años indica que la instalación está en cumplimiento con el límite de emisión para un contaminante (PM, CO o HCl), el dueño u operador no tendrá que realizar una prueba de funcionamiento para este contaminante por 2 años adicionales. Si cualquier prueba de funcionamiento realizada indica que la instalación no está en cumplimiento con el límite de emisión respectivo, se tendrá que realizar una prueba de funcionamiento anual para ese contaminante hasta que todas las pruebas de funcionamiento por un período de tres años indiquen que la instalación se encuentra en cumplimiento con el límite de emisión. El uso de una chimenea auxiliar durante cualquier prueba de funcionamiento invalidará la prueba.
  - (b)(6)(C)(iii) Las instalaciones que utilicen un sistema continuo de muestreo de emisiones (SCME) para demostrar el cumplimiento con cualquiera de los límites de emisión bajo la Sección (b) (2) de esta regla deberá:
    - (b)(6)(C)(iii)(1) Determinar el cumplimiento con los límites de emisión apropiados utilizando un promedio continuo de 12 horas, el cual es calculado cada hora como el promedio de las 12 horas previas de operación (excluyendo comienzo y fin del proceso de incineración o mal funcionamiento).
    - (b)(6)(C)(iii)(2) Operar todos los SCME de acuerdo con los procedimientos aplicables bajo los apéndices B y F del 40 CRF Parte 60 y en el Apéndice de este Reglamento.
  - (b)(6)(D) El dueño u operador de una instalación designada equipada con un lavador de gases seco seguido por un filtro de tela, un lavador de gases húmedo o un lavador de gases seco seguido de un filtro de tela y un lavador de gases húmedo deberá:

- (b)(6)(D)(i) Establecer los parámetros de operación máximos y mínimos indicados en la Tabla 4 de esta regla para cada sistema de control, como parámetros específicos de operación en la instalación durante la prueba inicial de funcionamiento para determinar el cumplimiento con los límites de emisión; y
- (b)(6)(D)(ii) Próxima a la fecha en que la prueba inicial de funcionamiento es completada o requiere ser completada bajo esta regla, lo que ocurra primero, asegurar que la instalación designada no opere por encima de ninguno de los parámetros máximos ni por debajo de ninguno de los parámetros mínimos de operación establecidos en la Tabla 4 y medidos como promedios continuos de 3 horas (el cual es calculado cada hora como el promedio de las 3 horas previas de operación) en todo momento excepto durante períodos de comienzo y fin del proceso de incineración o períodos de mal funcionamiento. Los límites de los parámetros de operación no aplican durante las pruebas de funcionamiento. La operación por encima o por debajo de los parámetros de operación establecidos constituirá una violación a estos parámetros.
- (b)(6)(E) Para instalaciones designadas que estén equipadas con un lavador de gases seco seguido por un filtro de tela; excepto como se provee en el párrafo (b)(6)(H) de esta sección.
- (b)(6)(E)(i) La operación de la instalación designada por encima de la cantidad máxima de carga y por debajo de la temperatura mínima de la cámara secundaria simultáneamente (cada uno medido en un promedio continuo de 3 horas) constituirá una violación al límite de emisión de CO.
- (b)(6)(E)(ii) La operación de la instalación designada por encima de la temperatura máxima en la entrada del filtro de tela, por encima de la cantidad máxima de carga y por debajo de la razón de flujo mínima de absorbentes de dioxinas/furanos simultáneamente (cada uno medido en un promedio continuo de 3 horas) constituirá una violación al límite de emisión para dioxinas/furanos.
- (b)(6)(E)(iii) La operación de la instalación designada por encima de la cantidad máxima de carga y por debajo de la razón de flujo mínima de absorbentes de HCl simultáneamente (cada uno medido en un promedio continuo de 3 horas) constituirá una violación al límite de emisión de HCl.

**TABLA 4. PARÁMETROS DE OPERACIÓN A SER MONITOREADOS Y FRECUENCIAS MÍNIMAS PARA MEDICIONES Y REGISTROS**

Parámetros de operación a ser muestreados	Frecuencia mínima		Sistema de control		
	Medición de datos	Registro de datos	Lavador de gases seco seguido por un filtro de tela	Lavador de gases húmedo	Lavador de gases seco seguido de un filtro de tela y lavador de gases húmedo
<b>Parámetros máximos de operación</b>					
Cantidad máxima de carga	continuo	1 x hora	✓	✓	✓
Temperatura máxima en la entrada del filtro de tela	continuo	1 x minuto	✓		✓
Temperatura máxima de los gases de chimenea	continuo	1 x minuto		✓	✓
<b>Parámetros mínimos de operación</b>					
Temperatura mínima en la cámara secundaria	continuo	1 x minuto	✓	✓	✓
Razón de flujo mínima de absorbentes de dioxinas/furanos	cada hora	1 x minuto	✓		✓
Razón de flujo mínima de absorbentes de HCl	cada hora	1 x hora	✓		✓
Razón de flujo mínima de absorbentes de mercurio (Hg)	cada hora	1 x hora	✓		✓
Caída en presión mínima a través del lavador de gases húmedo o caballaje o amperaje mínimo al lavador de gases húmedo	continuo	1 x minuto		✓	✓
Razón de flujo mínima del licor del lavador de gases	continuo	1 x minuto		✓	✓
pH mínimo del licor del lavador de gases	continuo	1 x minuto		✓	✓

- (b)(6)(E)(iv) La operación de la instalación designada por encima de la cantidad máxima de carga y por debajo de la razón de flujo mínima de absorbentes de Hg simultáneamente (cada uno medido en un promedio continuo de 3 horas) constituirá una violación al límite de emisión de Hg.
- (b)(6)(E)(v) El uso de una chimenea auxiliar (excepto durante el comienzo y fin del proceso de incineración o durante el mal funcionamiento) constituirá una violación a los límites de emisión para MP, dioxinas/furanos, HCl, Pb, Cd y Hg.
- (b)(6)(F) Para instalaciones designadas que estén equipadas con un lavador de gases húmedo, excepto como se provee en el párrafo (b)(6)(H) de esta sección:
- (b)(6)(F)(i) La operación de la instalación designada por encima de la cantidad máxima de carga y por debajo de la caída en presión mínima a través del lavador de gases húmedo o por debajo del caballaje o amperaje mínimo al sistema simultáneamente (cada uno medido en un promedio continuo de 3 horas) constituirá una violación al límite de emisión de MP.
- (b)(6)(F)(ii) La operación de la instalación designada por encima de la cantidad máxima de carga y por debajo de la temperatura mínima de la cámara secundaria simultáneamente (cada uno medido en un promedio continuo de 3 horas) constituirá una violación al límite de emisión de CO.
- (b)(6)(F)(iii) La operación de la instalación designada por encima de la cantidad máxima de carga, por debajo de la temperatura mínima de la cámara secundaria y por debajo de la razón de flujo mínima del licor del lavador de gases simultáneamente (cada uno medido en un promedio continuo de 3 horas) constituirá una violación al límite de emisión de dioxinas/furanos.
- (b)(6)(F)(iv) La operación de la instalación designada por encima de la cantidad máxima de carga y por debajo del pH mínimo del licor del lavador de gases simultáneamente (cada uno medido en un promedio continuo de 3 horas) constituirá una violación al límite de emisión de HCl.
- (b)(6)(F)(v) La operación de la instalación designada por encima de la temperatura máxima de los gases de chimenea y por encima de la cantidad máxima de carga simultáneamente (cada uno medido en un promedio continuo de 3 horas) constituirá una violación a los límites de emisión para Hg.
- (b)(6)(F)(vi) El uso de una chimenea auxiliar (excepto durante el comienzo y fin del proceso de incineración o durante el mal funcionamiento) constituirá una violación a los límites de emisión para MP, dioxinas/furanos, HCl, Pb, Cd y Hg.
- (b)(6)(G) Para instalaciones designadas que estén equipadas con un lavador de gases seco seguido por un filtro de tela y un lavador de gases húmedo, excepto como se provee en el párrafo (b)(6)(H) de esta sección:

- (b)(6)(G)(i) La operación de la instalación designada por encima de la cantidad máxima de carga y por debajo de la temperatura mínima de la cámara secundaria simultáneamente (cada uno medido en un promedio continuo de 3 horas) constituirá una violación al límite de emisión de CO.
- (b)(6)(G)(ii) La operación de la instalación designada por encima de la temperatura máxima a la entrada del filtro de tela, por encima la cantidad máxima de carga y por debajo de la razón de flujo mínima de sorbentes de dioxinas/furanos simultáneamente (cada uno medido en un promedio continuo de 3 horas) constituirá una violación al límite de emisión de dioxinas/furanos.
- (b)(6)(G)(iii) La operación de la instalación designada por encima de la cantidad máxima de carga y por debajo del pH mínimo del licor del lavador de gases simultáneamente (cada uno medido en un promedio continuo de 3 horas) constituirá una violación al límite de emisión de HCl.
- (b)(6)(G)(iv) La operación de la instalación designada por encima de la cantidad máxima de carga y por debajo de la razón de flujo mínima de absorbentes de Hg simultáneamente (cada uno medido en un promedio continuo de 3 horas) constituirá una violación al límite de emisión de Hg.
- (b)(6)(G)(v) El uso de una chimenea auxiliar (excepto durante el comienzo y fin del proceso de incineración o durante el mal funcionamiento) constituirá una violación a los límites de emisión para MP, dioxinas/furanos, HCl, Pb, Cd y Hg.
- (b)(6)(H) El dueño u operador de una instalación designada podrá repetir una prueba de funcionamiento dentro de los 30 días desde la violación de un parámetro de operación aplicable para demostrar que la instalación no está en violación de los límites de emisión aplicables. Las pruebas de funcionamiento repetidas según este párrafo se realizarán utilizando los mismos parámetros de operación que indicaron una violación bajo el párrafo (b)(6)(E), (b)(6)(F) o (b)(6)(G) de esta sección.
- (b)(6)(I) El dueño u operador de una instalación designada que utiliza un equipo de control de contaminación atmosférica que no sea un lavador de gases seco seguido por un filtro de tela, un lavador de gases húmedo, o un lavador de gases seco seguido por un filtro de tela y un lavador de gases húmedo para cumplir con los límites de emisión bajo la Sección (b)(2) de esta regla, deberá solicitar al Administrador otros parámetros específicos para la instalación, a ser establecidos durante la prueba inicial de funcionamiento y monitoreados de aquí en adelante. El dueño u operador no realizará la prueba inicial de funcionamiento hasta que su solicitud haya sido aprobada por el Administrador.
- (b)(6)(J) El dueño u operador de una instalación designada podrá repetir la prueba de funcionamiento en cualquier momento para establecer nuevos valores a los parámetros de operación. El Administrador solicitará una prueba de funcionamiento repetida en cualquier momento.

(b)(6)(K) Cualquier IDBH pequeño que cumpla con los requisitos para ser considerado como rural según la Sección (b)(2)(B) y que esté sujeta a los límites de emisión establecidos en la Tabla 2 de esta regla, deberá cumplir con los siguientes requisitos de cumplimiento y pruebas de funcionamiento:

(b)(6)(K)(i) Realizar la prueba de funcionamiento según los requisitos de las secciones (b)(6)(A), (b)(6)(B)(i) a (b)(6)(B)(ix), (b)(6)(B)(xi) (sólo Hg) y (b)(6)(C)(i) de esta regla. El límite de 2,000 lbs/sem bajo la Sección (b)(2)(B) de esta regla no aplica durante las pruebas de funcionamiento.

(b)(6)(K)(ii) Establecer la cantidad máxima de carga y la temperatura mínima de la cámara secundaria como parámetros específicos de la instalación durante la prueba inicial de funcionamiento realizada para determinar el cumplimiento con los límites de emisión aplicables.

(b)(6)(K)(iii) Asegurar que la instalación designada no opere por encima de la cantidad máxima de carga o por debajo de la temperatura mínima de la cámara secundaria, medidos en todo momento como promedios continuos de 3 horas (calculados cada hora como el promedio de las 3 horas previas de operación), excluyendo el comienzo y fin del proceso de incineración, o períodos de mal funcionamiento; próximo a la fecha en que la prueba inicial de funcionamiento es completada o requiere ser completada bajo esta regla, lo que ocurra primero. Los límites de los parámetros de operación no aplican durante las pruebas de funcionamiento. La operación por encima de la cantidad máxima de carga o por debajo de la temperatura mínima de la cámara secundaria constituirá una violación a los parámetros de operación establecidos.

(b)(6)(K)(iv) La operación de la instalación designada por encima de la cantidad máxima de carga o por debajo de la temperatura mínima de la cámara secundaria simultáneamente (cada uno medido en un promedio continuo de 3 horas) constituirá una violación a los límites de emisión de MP, CO y dioxinas/furanos; excepto como se provee en el párrafo (b)(6)(K)(v) de esta sección.

(b)(6)(K)(v) El dueño u operador de una instalación designada realizará una prueba de funcionamiento repetida dentro de 30 días de la violación de un parámetro de operación aplicable para demostrar que la instalación no está en violación de los límites aplicables de emisión. La prueba de funcionamiento que se repita según este párrafo debe ser realizada utilizando los parámetros de operación idénticos a los que indicaron la violación bajo el párrafo (b)(6)(K)(iv).

#### **(b)(7) Requisitos de Muestreo**

(b)(7)(A) El dueño u operador de una instalación designada deberá instalar, calibrar (según las especificaciones del fabricante), mantener y operar equipos (o establecer métodos) para monitorear los parámetros máximos y mínimos aplicables establecidos en la Tabla 4 de modo que estos equipos (o métodos) midan y registren los valores para estos parámetros

de operación con la frecuencia indicada en la Tabla 4 en todo momento, excepto durante el comienzo o fin del proceso de incineración.

- (b)(7)(B) El dueño u operador de una instalación designada deberá instalar, calibrar (según las especificaciones del fabricante), mantener y operar un equipo o método para medir el uso de la chimenea auxiliar incluyendo la fecha, hora y duración.
- (b)(7)(C) El dueño u operador de una instalación designada que utilice un equipo que no sea un lavador de gases seco seguido por un filtro de tela, un lavador de gases húmedo o un lavador de gases seco seguido por un filtro de tela y un lavador de gases húmedo, para cumplir con los límites de emisión bajo la Sección (b)(2) de esta regla, deberá instalar, calibrar (según las especificaciones del fabricante), mantener y operar el equipo necesario para medir los parámetros específicos de la instalación desarrolladoras de acuerdo con la Sección (b)(6)(I) de esta regla.
- (b)(7)(D) El dueño u operador de una instalación designada deberá obtener datos de monitoreo en todo momento durante la operación del IDBH, excepto durante períodos de mal funcionamiento, calibración o reparación. Como mínimo, se deberá obtener datos de muestreo válidos para el 75 por ciento de las horas de operación por día y para el 90 por ciento de los días de operación por trimestre en que por la instalación designada o quemé desperdicios de hospital y/o médico/infecciosos.
- (b)(7)(E) Cualquier IDBH pequeño que cumpla con los requisitos para ser considerado como rural según la Sección (b)(2)(B) y que esté sujeto a los límites de emisión establecidos en la Tabla 2 de esta regla, deberá cumplir con los siguientes requisitos de monitoreo:
  - (b)(7)(E)(i) Instalar, calibrar (según las especificaciones del fabricante), mantener y operar un equipo para medir y registrar de forma continua la temperatura de la cámara secundaria, cuyos resultados deberán registrarse, como mínimo, una vez cada minuto durante toda la operación.
  - (b)(7)(E)(ii) Instalar, calibrar (según las especificaciones del fabricante), mantener y operar un equipo para medir y registrar automáticamente, la fecha, hora y peso de cada carga alimentada al IDBH.
  - (b)(7)(E)(iii) El dueño u operador de una instalación designada deberá obtener datos de monitoreo en todo momento durante la operación del IDBH, excepto durante períodos de mal funcionamiento del equipo de muestreo, calibración o reparación. Como mínimo, se deberá obtener datos de monitoreo válidos para el 75 por ciento de las horas de operación por día y para el 90 por ciento de los días de operación por trimestre en que la instalación designada quemen desperdicios hospitalarios y/o médico/infecciosos.

**(b)(8) Requisitos para someter informes y mantener expedientes**

- (b)(8)(A) El dueño u operador de una instalación designada deberá mantener la siguiente información (según sea aplicable) por un período de por lo menos 5 años:

- (b)(8)(A)(i) Fecha de cada expediente;
- (b)(8)(A)(ii) Registros con la siguiente información:
- (b)(8)(A)(ii)(1) Las concentraciones de cualquier contaminante registrado en la Sección (b)(2) de esta regla o mediciones de opacidad según es determinado por el sistema continuo de muestreo de emisiones, (si aplica);
  - (b)(8)(A)(ii)(2) Las fechas, horas y peso de las cargas al IDBH y las cantidades de carga por hora;
  - (b)(8)(A)(ii)(3) Temperatura en la entrada del filtro de tela durante cada minuto de la operación, si aplica;
  - (b)(8)(A)(ii)(4) La cantidad y tipo de absorbentes de dioxinas/furanos utilizados durante cada hora de operación, según sea aplicable;
  - (b)(8)(A)(ii)(5) La cantidad y tipo de absorbente de Hg utilizado durante cada hora de operación, según sea aplicable;
  - (b)(8)(A)(ii)(6) La cantidad y tipo de absorbente de HCl utilizado durante cada hora de operación, según sea aplicable;
  - (b)(8)(A)(ii)(7) Las temperaturas registradas en la cámara secundaria durante cada minuto de operación;
  - (b)(8)(A)(ii)(8) La razón de flujo del licor a la entrada del lavador de gases húmedo durante cada minuto de operación, según sea aplicable;
  - (b)(8)(A)(ii)(9) El caballaje o amperaje al lavador de gases húmedo durante cada minuto de operación, según sea aplicable;
  - (b)(8)(A)(ii)(10) La caída en presión a través del sistema del lavador de gases húmedo durante cada minuto de operación, según sea aplicable;
  - (b)(8)(A)(ii)(11) La temperatura en la salida del lavador de gases húmedo durante cada minuto de operación, según sea aplicable;
  - (b)(8)(A)(ii)(12) El pH en la entrada del lavador de gases húmedo durante cada minuto de operación, según sea aplicable;
  - (b)(8)(A)(ii)(13) Los registros que indiquen el uso de chimeneas auxiliares, incluyendo las fechas, las horas y las duraciones y
  - (b)(8)(A)(ii)(14) Para instalaciones designadas que cumplan con las secciones (b)(6)(I) y (b)(7)(C) de esta regla, el dueño u operador mantendrá los datos de los parámetros de operación recolectados.

- (b)(8)(A)(iii) Identificación de los días para los cuales no se ha obtenido datos sobre las tasas de emisión o los parámetros de operación especificados bajo el párrafo (b)(8)(A)(ii) de esta sección, con una identificación de las tasas de emisión o los parámetros no medidos, razones por las cuales no se obtuvieron los datos y una descripción de las acciones correctivas tomadas.
- (b)(8)(A)(iv) Identificación de los días, horas y duración de los períodos de mal funcionamiento, una descripción del mal funcionamiento y las medidas correctivas tomadas.
- (b)(8)(A)(v) Identificación de los días en los cuales los datos sobre las tasas de emisión o los parámetros de operación especificados en el párrafo (b)(8)(A)(ii) de esta sección se excedieron, con una descripción de las excedencias, las razones de estas excedencias y una descripción de las medidas correctivas tomadas.
- (b)(8)(A)(vi) Los resultados de las pruebas de funcionamiento inicial, anual y cualquier otra realizada con el propósito de determinar el cumplimiento con los límites de emisión y/o para establecer parámetros de operación, si aplica.
- (b)(8)(A)(vii) Expedientes donde se indiquen los nombres de los operadores del IDBH que hayan completado la revisión de la información en (b)(3)(H) según requerido por la Sección (b)(3)(I), incluyendo la fecha de la revisión inicial y todas las revisiones anuales subsiguientes.
- (b)(8)(A)(viii) Expedientes donde se indiquen los nombres de los operadores del IDBH que hayan completado los requisitos de adiestramiento para operadores, incluyendo documentación sobre el adiestramiento y las fechas en que éste se tomó;
- (b)(8)(A)(ix) Expedientes donde se indiquen los nombres de los operadores del IDBH que cumplan los criterios para la calificación bajo la Sección (b)(3) de esta regla y las fechas de sus calificaciones; y
- (b)(8)(A)(x) Registros sobre la calibración de cualquier equipo de muestreo según requerido bajo las secciones (b)(7)(A), (B) y (C) de esta regla.
- (b)(8)(B) El dueño u operador de una instalación designada deberá someter la información descrita en los párrafos (b)(8)(B)(i) a (b)(8)(B)(iii) de esta sección no más tarde de 60 días luego de la prueba inicial de funcionamiento. Todos los informes deberán estar firmados por el administrador de la instalación.
- (b)(8)(B)(i) Los datos sobre la prueba inicial de funcionamiento registrados bajo la Sección (b)(6)(B)(i) a la (b)(6)(B)(xi), si aplica.
- (b)(8)(B)(ii) Los valores establecidos para los parámetros de operación específicos de la instalación, según la Sección (b)(6)(D) o (I), si aplica.

- (b)(8)(B)(iii) El plan de manejo de desperdicios según se especifica en la Sección (b)(4) de esta regla.
- (b)(8)(C) Presentará un informe anual dentro de un año después de someter la información en el párrafo (b)(8)(B) de esta sección e informes subsiguientes deben ser presentados no más tarde de 12 meses después del informe anterior (el dueño u operador de una instalación designada deberá someter estos informes semestralmente, una vez la unidad esté sujeta a los requisitos de permiso bajo el Título V de la Ley Federal de Aire Limpio). El informe anual incluirá la información que se indica en los párrafos (b)(8)(C)(i) a (b)(8)(C)(vii) de esta sección. Todos los informes deben ser firmados por el administrador de la instalación.
- (b)(8)(C)(i) Los valores para los parámetros de operación específicos de la instalación establecidos según la Sección (b)(6)(D) o (I) de esta regla, si aplica.
- (b)(8)(C)(ii) El parámetro de operación máximo más alto y el parámetro de operación mínimo más bajo para cada parámetro de operación registrado en el año que es informado, según la Sección (b)(6)(D) o (I) de esta regla, si aplica.
- (b)(8)(C)(iii) El parámetro de operación máximo más alto y el parámetro de operación mínimo más bajo según sea aplicable, para cada parámetro de operación registrado en el año anterior al que es informado según la Sección (b)(6)(D) o (I), para proveerle a la JCA un resumen del funcionamiento de la instalación designada por un período de 2 años.
- (b)(8)(C)(iv) Toda la información registrada bajo los párrafos (b)(8)(A)(iii) a (b)(8)(A)(v) de esta sección durante el año que se informa.
- (b)(8)(C)(v) Toda la información registrada bajo los párrafos (b)(8)(A)(iii) a (b)(8)(A)(v) de esta sección durante el año que antecede al año que se informa, para proveerle a la JCA un resumen del funcionamiento de la instalación designada por un período de 2 años.
- (b)(8)(C)(vi) Si se ha realizado una prueba de funcionamiento durante el período a ser informado, los resultados de esta prueba.
- (b)(8)(C)(vii) Si no se informaron excedencias o mal funcionamiento bajo los párrafos (b)(8)(A)(iii) a (b)(8)(A)(v) de esta sección durante el año que se informa, una declaración de que no ocurrieron excedencias durante el período de informe.
- (b)(8)(C)(viii) Cualquier uso de chimeneas auxiliares, la duración, razón para el mal funcionamiento y las medidas correctivas tomadas.

- (b)(8)(D) El dueño u operador de la instalación designada deberá someter informes semestrales sobre cualquier información registrada bajo los párrafos (b) (8) (A) (iii) al (b) (8) (A) (v) de esta sección no más tarde de 60 días luego del período de informe. El período para someter el primer informe semestral termina en 6 meses después de que se someta la información en el párrafo (b)(8)(B) de esta sección. Los informes subsiguientes deberán someterse no más tarde de 6 meses consecutivos después del informe anterior. Todos los informes serán firmados por el administrador de la instalación.
- (b)(8)(E) Todos los expedientes especificados en el párrafo (b)(8)(A) de esta sección deberán mantenerse en la instalación, ya sea en papel o en un formato de computadora, a menos que se apruebe un formato alternativo por la APA.
- (b)(8)(F) Todo IDBH pequeño que cumpla con los requisitos para ser considerado como rural según la Sección (b)(2)(B) y que esté sujeto a los límites de emisión establecidos en la Tabla 2 de esta regla, deberá cumplir con los siguientes requisitos:
- (b)(8)(F)(i) Mantener los registros de las inspecciones anuales del equipo, de cualquier trabajo de mantenimiento requerido y cualquier reparación que no haya sido completada dentro de los 10 días después de la inspección (o el límite de tiempo establecido por la Junta); y
- (b)(8)(F)(ii) Someter un informe anual que contenga la información registrada bajo el párrafo (b)(8)(F)(i) de esta sección, en un término no mayor de 60 días después del año en el cual los datos son recolectados. Los informes subsiguientes serán enviados no más tarde de 12 meses consecutivos luego del informe anterior. El dueño u operador deberá someter estos informes semestralmente, una vez la unidad esté sujeta a los requisitos de permiso bajo el Título V de la Ley. El informe será firmado por el administrador de la instalación.
- (b)(8)(G) Otros informes
- (b)(8)(G)(i) Todas las instalaciones designadas que se les requiera cumplir con la Regla 405 (b)(9)(E) informará a la JCA sus logros hacia el cumplimiento con los incrementos de progreso dentro de 10 días después de lograr cada uno de los incrementos de progreso del itinerario de cumplimiento.

**(b)(9) Términos para el Cumplimiento**

- (b)(9)(A) Todas las instalaciones deberán cumplir con todos los requisitos aplicables del Plan Estatal en o antes de un año luego de que la APA apruebe el Plan Estatal o después de la fecha de publicación del Plan Federal de la APA, lo que ocurra primero, independientemente de si la instalación designada es identificada en el inventario del Plan Estatal requerido por la Sección 60.25 (a) del 40 CRF Parte 60 Subparte B y en el Apéndice de este Reglamento, excepto como se provea en los párrafos (E) y (F) de esta sección.

- (b)(9)(B) Todo incinerador que queme sólo desperdicios patológicos, desperdicios de bajo nivel de radioactividad y/o quimioterapéuticos, y cualquier incinerador de hornos múltiples, deberá reclamar una exención a la APA, no más tarde de 12 meses luego de que la APA apruebe el Plan Estatal o después de la fecha de publicación del Plan Federal de la APA, lo que ocurra primero, con copia a la JCA.
- (b)(9)(C) Toda instalación designada que no haya solicitado la exención a tiempo, o que no se le haya otorgado una exención de cumplimiento más allá de la fecha de cumplimiento como se especifica en el párrafo (A) de esta sección y que no esté en cumplimiento con todos los requisitos del Plan Estatal un año después de que la APA apruebe el Plan Estatal o después de la fecha de publicación del Plan Federal de la APA, lo que ocurra primero, deberá cesar sus operaciones hasta la fecha en que instalación designada cumpla con todos los requisitos aplicables, o cese sus operaciones permanentemente.
- (b)(9)(D) Cualquier instalación designada que cese sus operaciones o que opte por cesar operaciones en o antes de la fecha de cumplimiento indicada en (b)(9)(A) deberá estar en cumplimiento con todos los requisitos aplicables, incluyendo el realizar los cambios necesarios a los controles de contaminación atmosférica, antes de reanudar sus operaciones, excepto si éste es parte de un itinerario de cumplimiento según se especifica en el párrafo (b)(9)(E).
- (b)(9)(E) Las extensiones otorgadas a instalaciones designadas para cumplir con los requisitos aplicables para la instalación de equipo de control necesario o de tecnología alterna para el tratamiento de desperdicios, más allá de la fecha especificada en (b)(9)(A), deberá incluir los incrementos de progreso hacia el cumplimiento mensurables y legalmente ejecutables. Los incrementos de progreso se harán de la siguiente manera:
- (b)(9)(E)(i) Someterá un plan final de control en donde se describan los controles o la tecnología que utilizará la fuente para cumplir con los límites de emisión y otros requisitos no más tarde de 12 meses luego de que la APA apruebe el Plan Estatal o después de la fecha de publicación del Plan Federal de la APA, lo que ocurra primero, para controlar las emisiones de los IDBH. Este plan de control deberá ser sometido al mismo tiempo que la solicitud de extensión de cumplimiento.
- (b)(9)(E)(ii) Otorgación de contratos para sistema de control o modificaciones de proceso u órdenes para comprar componentes. Este paso debe ser completado no más tarde de 15 meses después de la aprobación del Plan Estatal por la APA o después de la fecha de publicación del Plan Federal de la APA, lo que ocurra primero.
- (b)(9)(E)(iii) Comienzo de la construcción en el lugar o la instalación del equipo de control de contaminación atmosférica o cambios en el proceso. Este paso debe ser completado no más tarde de 18 meses después de la aprobación del Plan Estatal por la APA o después de la fecha de publicación del Plan Federal de la APA, lo que ocurra primero.

- (b)(9)(E)(iv) Terminación de la construcción en el lugar o instalación del equipo de control o cambios en el proceso. Este paso debe ser completado no más tarde de 21 meses después de la aprobación del Plan Estatal por la APA o después de la fecha de publicación del Plan Federal de la APA, lo que ocurra primero.
- (b)(9)(E)(v) El cumplimiento final debe ser logrado el 15 de septiembre de 2002 o antes.
- (b)(9)(E)(vi) Una prueba inicial de funcionamiento siguiendo los procedimientos y requisitos establecidos en la Sección (b)(6) deberá completarse dentro de 180 días luego de que el cumplimiento final haya sido logrado.
- (b)(9)(F) Toda instalación designada que solicite una extensión de cumplimiento más allá de la fecha de cumplimiento como se especifica en (b)(9)(A), deberá someter la siguiente información antes de someter una solicitud de permiso de operación Título V completa y a tiempo.
  - (b)(9)(F)(i) Documentación de los análisis realizados para justificar la necesidad de una extensión, incluyendo una explicación de por qué se necesita más de un año después de que la APA apruebe el Plan Estatal para cumplir con todos los requisitos del plan. La documentación deberá incluir una evaluación de la alternativa para transportar el desperdicio de afuera a una instalación de disposición y tratamiento de desperdicios médicos comerciales en una base temporal o permanente.
  - (b)(9)(F)(ii) Documentación de los pasos para los incrementos de progreso mensurables y ejecutables según requeridos en (b)(9)(E) a ser tomados hacia el cumplimiento con las guías de emisión.
- (b)(9)(G) Los requisitos para el adiestramiento y certificación de operadores como se especifica bajo la Sección (b)(3) y los requisitos de inspección para los IDBH pequeños que cumplan con los requisitos de rural según se especifican bajo la Sección (b)(5) de esta regla, no podrán incluirse dentro de la solicitud de extensión de cumplimiento, y deberán cumplirse dentro de un año después de la aprobación del Plan Estatal por la APA o después de la fecha de publicación del Plan Federal de la APA, lo que ocurra primero.
- (b)(9)(H) Disposiciones para aprobar o denegar las extensiones de cumplimiento.
  - (b)(9)(H)(i) Las extensiones más allá de la fecha límite para cumplimiento pueden ser otorgadas o denegadas a las instalaciones designadas que planifiquen instalar el equipo de control necesario para cumplir con los requisitos de esta regla y para las instalaciones que planifiquen instalar una tecnología alterna para el tratamiento de desperdicios en el lugar, la cual no está disponible para la instalación hasta después de un año, siempre que las instalaciones designadas cumplan con los procedimientos de solicitud de extensión especificados en esta regla.

- (b)(9)(H)(ii) Las solicitudes para extensiones de cumplimiento que no se sometan a tiempo (ésto significa el 15 de septiembre de 2000 o antes) serán denegadas automáticamente.
- (b)(9)(H)(iii) Las solicitudes para extensiones de cumplimiento deberán ser sometidas por escrito al Director del Programa de Aire.
- (b)(9)(H)(iv) Las solicitudes para extensiones de cumplimiento serán aprobadas o denegadas dentro de 30 días después de recibir la solicitud con la información requerida.

*APÉNDICES*



***40 CFR SECTION 60.25(a)***

## Subpart B—Adoption and Submittal of State Plans for Designated Facilities

SOURCE: 40 FR 53346, Nov. 17, 1975, unless otherwise noted.

### § 60.20 Applicability.

The provisions of this subpart apply to States upon publication of a final guideline document under § 60.22(a).

### § 60.21 Definitions.

Terms used but not defined in this subpart shall have the meaning given them in the Act and in subpart A:

(a) *Designated pollutant* means any air pollutant, emissions of which are subject to a standard of performance for new stationary sources but for which air quality criteria have not been issued, and which is not included on a list published under section 108(a) or section 112(b)(1)(A) of the Act.

(b) *Designated facility* means any existing facility (see § 60.2(aa)) which emits a designated pollutant and which would be subject to a standard of performance for that pollutant if the existing facility were an affected facility (see § 60.2(e)).

(c) *Plan* means a plan under section 111(d) of the Act which establishes emission standards for designated pollutants from designated facilities and provides for the implementation and enforcement of such emission standards.

(d) *Applicable plan* means the plan, or most recent revision thereof, which has been approved under § 60.27(b) or promulgated under § 60.27(d).

(e) *Emission guideline* means a guideline set forth in subpart C of this part, or in a final guideline document published under § 60.22(a), which reflects the degree of emission reduction achievable through the application of the best system of emission reduction which (taking into account the cost of such reduction) the Administrator has determined has been adequately demonstrated for designated facilities.

(f) *Emission standard* means a legally enforceable regulation setting forth an allowable rate of emissions into the atmosphere, or prescribing equipment specifications for control of air pollution emissions.

(g) *Compliance schedule* means a legally enforceable schedule specifying a date or dates by which a source or category of sources must comply with specific emission standards contained in a plan or with any increments of progress to achieve such compliance.

(h) *Increments of progress* means steps to achieve compliance which must be taken by an owner or operator of a designated facility, including:

(1) Submittal of a final control plan for the designated facility to the appropriate air pollution control agency;

(2) Awarding of contracts for emission control systems or for process modifications, or issuance of orders for the purchase of component parts to accomplish emission control or process modification;

(3) Initiation of on-site construction or installation of emission control equipment or process change;

(4) Completion of on-site construction or installation of emission control equipment or process change; and

(5) Final compliance.

(i) *Region* means an air quality control region designated under section 107 of the Act and described in part 81 of this chapter.

(j) *Local agency* means any local governmental agency.

### § 60.22 Publication of guideline documents, emission guidelines, and final compliance times.

(a) Concurrently upon or after proposal of standards of performance for the control of a designated pollutant from affected facilities, the Administrator will publish a draft guideline document containing information pertinent to control of the designated pollutant from designated facilities. Notice of the availability of the draft guideline document will be published in the FEDERAL REGISTER and public comments on its contents will be invited. After consideration of public comments and upon or after promulgation of standards of performance for control of a designated pollutant from affected facilities, a final guideline document will be published and notice of its availability will be published in the FEDERAL REGISTER.

(b) Guideline documents published under this section will provide information for the development of State plans, such as:

(1) Information concerning known or suspected endangerment of public health or welfare caused, or contributed to, by the designated pollutant.

(2) A description of systems of emission reduction which, in the judgment of the Administrator, have been adequately demonstrated.

(3) Information on the degree of emission reduction which is achievable with each system, together with information on the costs and environmental effects of applying each system to designated facilities.

(4) Incremental periods of time normally expected to be necessary for the design, installation, and startup of identified control systems.

(5) An emission guideline that reflects the application of the best system of emission reduction (considering the cost of such reduction) that has

## § 60.23

been adequately demonstrated for designated facilities, and the time within which compliance with emission standards of equivalent stringency can be achieved. The Administrator will specify different emission guidelines or compliance times or both for different sizes, types, and classes of designated facilities when costs of control, physical limitations, geographical location, or similar factors make subcategorization appropriate. (6) Such other available information as the Administrator determines may contribute to the formulation of State plans.

(c) Except as provided in paragraph (d)(1) of this section, the emission guidelines and compliance times referred to in paragraph (b)(5) of this section will be proposed for comment upon publication of the draft guideline document, and after consideration of comments will be promulgated in subpart C of this part with such modifications as may be appropriate.

(d)(1) If the Administrator determines that a designated pollutant may cause or contribute to endangerment of public welfare, but that adverse effects on public health have not been demonstrated, he will include the determination in the draft guideline document and in the FEDERAL REGISTER notice of its availability. Except as provided in paragraph (d)(2) of this section, paragraph (c) of this section shall be inapplicable in such cases.

(2) If the Administrator determines at any time on the basis of new information that a prior determination under paragraph (d)(1) of this section is incorrect or no longer correct, he will publish notice of the determination in the FEDERAL REGISTER, revise the guideline document as necessary under paragraph (a) of this section, and propose and promulgate emission guidelines and compliance times under paragraph (c) of this section.

[40 FR 53346, Nov. 17, 1975, as amended at 54 FR 52189, Dec. 20, 1989]

### § 60.23 Adoption and submittal of State plans; public hearings.

(a)(1) Within nine months after notice of the availability of a final guideline document is published under § 60.22(a), each State shall adopt and submit to the Administrator, in accordance with § 60.4, a plan for the control of the designated pollutant to which the guideline document applies.

(2) Within nine months after notice of the availability of a final revised guideline document is published as provided in § 60.22(d)(2), each State shall adopt and submit to the Administrator any plan revision necessary to meet the requirements of this subpart.

(b) If no designated facility is located within a State, the State shall submit a letter of certification to that effect to the Administrator within the time

specified in paragraph (a) of this section. Such certification shall exempt the State from the requirements of this subpart for that designated pollutant.

(c)(1) Except as provided in paragraphs (c)(2) and (c)(3) of this section, the State shall, prior to the adoption of any plan or revision thereof, conduct one or more public hearings within the State on such plan or plan revision.

(2) No hearing shall be required for any change to an increment of progress in an approved compliance schedule unless the change is likely to cause the facility to be unable to comply with the final compliance date in the schedule.

(3) No hearing shall be required on an emission standard in effect prior to the effective date of this subpart if it was adopted after a public hearing and is at least as stringent as the corresponding emission guideline specified in the applicable guideline document published under § 60.22(a).

(d) Any hearing required by paragraph (c) of this section shall be held only after reasonable notice. Notice shall be given at least 30 days prior to the date of such hearing and shall include:

(1) Notification to the public by prominently advertising the date, time, and place of such hearing in each region affected;

(2) Availability, at the time of public announcement, of each proposed plan or revision thereof for public inspection in at least one location in each region to which it will apply;

(3) Notification to the Administrator;

(4) Notification to each local air pollution control agency in each region to which the plan or revision will apply; and

(5) In the case of an interstate region, notification to any other State included in the region.

(e) The State shall prepare and retain, for a minimum of 2 years, a record of each hearing for inspection by any interested party. The record shall contain, as a minimum, a list of witnesses together with the text of each presentation.

(f) The State shall submit with the plan or revision:

(1) Certification that each hearing required by paragraph (c) of this section was held in accordance with the notice required by paragraph (d) of this section; and

(2) A list of witnesses and their organizational affiliations, if any, appearing at the hearing and a brief written summary of each presentation or written submission.

(g) Upon written application by a State agency (through the appropriate Regional Office), the Administrator may approve State procedures designed to insure public participation in the matters for which hearings are required and public notification of the opportunity to participate if, in the judgment of the Administrator, the procedures, although dif-

ferent from the requirements of this subpart, in fact provide for adequate notice to and participation of the public. The Administrator may impose such conditions on his approval as he deems necessary. Procedures approved under this section shall be deemed to satisfy the requirements of this subpart regarding procedures for public hearings.

[40 FR 53346, Nov. 17, 1975, as amended at 60 FR 65414, Dec. 19, 1995]

EFFECTIVE DATE NOTE: At 60 FR 65414, Dec. 19, 1995, § 60.23 was amended by revising paragraph (a)(1) without establishing an effective date. For the convenience of the reader, the existing text continues to be carried within the body of the section and the new text is set forth below:

**§ 60.23 Adoption and submittal of State plans: public hearings.**

(a)(1) Unless otherwise specified in the applicable subpart, within 9 months after notice of the availability of a final guideline document is published under § 60.22(a), each State shall adopt and submit to the Administrator, in accordance with § 60.4 of subpart A of this part, a plan for the control of the designated pollutant to which the guideline document applies.

**§ 60.24 Emission standards and compliance schedules.**

(a) Each plan shall include emission standards and compliance schedules.

(b)(1) Emission standards shall prescribe allowable rates of emissions except when it is clearly impracticable. Such cases will be identified in the guideline documents issued under § 60.22. Where emission standards prescribing equipment specifications are established, the plan shall, to the degree possible, set forth the emission reductions achievable by implementation of such specifications, and may permit compliance by the use of equipment determined by the State to be equivalent to that prescribed.

(2) Test methods and procedures for determining compliance with the emission standards shall be specified in the plan. Methods other than those specified in appendix A to this part may be specified in the plan if shown to be equivalent or alternative methods as defined in § 60.2 (t) and (u).

(3) Emission standards shall apply to all designated facilities within the State. A plan may contain emission standards adopted by local jurisdictions provided that the standards are enforceable by the State.

(c) Except as provided in paragraph (f) of this section, where the Administrator has determined that a designated pollutant may cause or contribute to endangerment of public health, emission standards shall be no less stringent than the corresponding emission guideline(s) specified in subpart C of

this part, and final compliance shall be required as expeditiously as practicable but no later than the compliance times specified in subpart C of this part.

(d) Where the Administrator has determined that a designated pollutant may cause or contribute to endangerment of public welfare but that adverse effects on public health have not been demonstrated, States may balance the emission guidelines, compliance times, and other information provided in the applicable guideline document against other factors of public concern in establishing emission standards, compliance schedules, and variances. Appropriate consideration shall be given to the factors specified in § 60.22(b) and to information presented at the public hearing(s) conducted under § 60.23(c).

(e)(1) Any compliance schedule extending more than 12 months from the date required for submittal of the plan shall include legally enforceable increments of progress to achieve compliance for each designated facility or category of facilities. Increments of progress shall include, where practicable, each increment of progress specified in § 60.21(h) and shall include such additional increments of progress as may be necessary to permit close and effective supervision of progress toward final compliance.

(2) A plan may provide that compliance schedules for individual sources or categories of sources will be formulated after plan submittal. Any such schedule shall be the subject of a public hearing held according to § 60.23 and shall be submitted to the Administrator within 60 days after the date of adoption of the schedule but in no case later than the date prescribed for submittal of the first semiannual report required by § 60.25(e).

(f) On a case-by-case basis for particular designated facilities, or classes of facilities, States may provide for the application of less stringent emission standards or longer compliance schedules than those otherwise required by paragraph (c) of this section, provided that the State demonstrates with respect to each such facility (or class of facilities):

(1) Unreasonable cost of control resulting from plant age, location, or basic process design;

(2) Physical impossibility of installing necessary control equipment; or

(3) Other factors specific to the facility (or class of facilities) that make application of a less stringent standard or final compliance time significantly more reasonable.

(g) Nothing in this subpart shall be construed to preclude any State or political subdivision thereof from adopting or enforcing (1) emission standards more stringent than emission guidelines specified in subpart C of this part or in applicable guideline documents or (2) compliance schedules requiring

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final compliance at earlier times than those specified in subpart C or in applicable guideline documents.

[40 FR 53346, Nov. 17, 1975]

EFFECTIVE DATE NOTE: At 60 FR 65414, Dec. 19, 1995, § 60.24 was amended by revising paragraph (f) introductory text without establishing an effective date. For the convenience of the reader, the existing text continues to be carried within the body of the section and the new text is set forth below:

### § 60.24 Emission standards and compliance schedules.

(f) Unless otherwise specified in the applicable subpart on a case-by-case basis for particular designated facilities or classes of facilities, States may provide for the application of less stringent emissions standards or longer compliance schedules than those otherwise required by paragraph (c) of this section, provided that the State demonstrates with respect to each such facility (or class of facilities):

### § 60.25 Emission inventories, source surveillance, reports.

(a) Each plan shall include an inventory of all designated facilities, including emission data for the designated pollutants and information related to emissions as specified in appendix D to this part. Such data shall be summarized in the plan, and emission rates of designated pollutants from designated facilities shall be correlated with applicable emission standards. As used in this subpart, "correlated" means presented in such a manner as to show the relationship between measured or estimated amounts of emissions and the amounts of such emissions allowable under applicable emission standards.

(b) Each plan shall provide for monitoring the status of compliance with applicable emission standards. Each plan shall, as a minimum, provide for:

(1) Legally enforceable procedures for requiring owners or operators of designated facilities to maintain records and periodically report to the State information on the nature and amount of emissions from such facilities, and/or such other information as may be necessary to enable the State to determine whether such facilities are in compliance with applicable portions of the plan.

(2) Periodic inspection and, when applicable, testing of designated facilities.

(c) Each plan shall provide that information obtained by the State under paragraph (b) of this section shall be correlated with applicable emission standards (see § 60.25(a)) and made available to the general public.

(d) The provisions referred to in paragraphs (b) and (c) of this section shall be specifically identified. Copies of such provisions shall be submitted with the plan unless:

(1) They have been approved as portions of a preceding plan submitted under this subpart or as portions of an implementation plan submitted under section 110 of the Act, and

(2) The State demonstrates:

(i) That the provisions are applicable to the designated pollutant(s) for which the plan is submitted, and

(ii) That the requirements of § 60.26 are met.

(e) The State shall submit reports on progress in plan enforcement to the Administrator on an annual (calendar year) basis, commencing with the first full report period after approval of a plan or after promulgation of a plan by the Administrator. Information required under this paragraph must be included in the annual report required by § 51.321 of this chapter.

(f) Each progress report shall include:

(1) Enforcement actions initiated against designated facilities during the reporting period, under any emission standard or compliance schedule of the plan.

(2) Identification of the achievement of any increment of progress required by the applicable plan during the reporting period.

(3) Identification of designated facilities that have ceased operation during the reporting period.

(4) Submission of emission inventory data as described in paragraph (a) of this section for designated facilities that were not in operation at the time of plan development but began operation during the reporting period.

(5) Submission of additional data as necessary to update the information submitted under paragraph (a) of this section or in previous progress reports.

(6) Submission of copies of technical reports on all performance testing on designated facilities conducted under paragraph (b)(2) of this section, complete with concurrently recorded process data.

[40 FR 53346, Nov. 17, 1975, as amended at 44 FR 65071, Nov. 9, 1979]

### § 60.26 Legal authority.

(a) Each plan shall show that the State has legal authority to carry out the plan, including authority to:

(1) Adopt emission standards and compliance schedules applicable to designated facilities.

(2) Enforce applicable laws, regulations, standards, and compliance schedules, and seek injunctive relief.

(3) Obtain information necessary to determine whether designated facilities are in compliance with applicable laws, regulations, standards, and

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compliance schedules, including authority to require recordkeeping and to make inspections and conduct tests of designated facilities.

(4) Require owners or operators of designated facilities to install, maintain, and use emission monitoring devices and to make periodic reports to the State on the nature and amounts of emissions from such facilities; also authority for the State to make such data available to the public as reported and as correlated with applicable emission standards.

(b) The provisions of law or regulations which the State determines provide the authorities required by this section shall be specifically identified. Copies of such laws or regulations shall be submitted with the plan unless:

(1) They have been approved as portions of a preceding plan submitted under this subpart or as portions of an implementation plan submitted under section 110 of the Act, and

(2) The State demonstrates that the laws or regulations are applicable to the designated pollutant(s) for which the plan is submitted.

(c) The plan shall show that the legal authorities specified in this section are available to the State at the time of submission of the plan. Legal authority adequate to meet the requirements of paragraphs (a)(3) and (4) of this section may be delegated to the State under section 114 of the Act.

(d) A State governmental agency other than the State air pollution control agency may be assigned responsibility for carrying out a portion of a plan if the plan demonstrates to the Administrator's satisfaction that the State governmental agency has the legal authority necessary to carry out that portion of the plan.

(e) The State may authorize a local agency to carry out a plan, or portion thereof, within the local agency's jurisdiction if the plan demonstrates to the Administrator's satisfaction that the local agency has the legal authority necessary to implement the plan or portion thereof, and that the authorization does not relieve the State of responsibility under the Act for carrying out the plan or portion thereof.

### § 60.27 Actions by the Administrator.

(a) The Administrator may, whenever he determines necessary, extend the period for submission of any plan or plan revision or portion thereof.

(b) After receipt of a plan or plan revision, the Administrator will propose the plan or revision for approval or disapproval. The Administrator will, within four months after the date required for submission of a plan or plan revision, approve or disapprove such plan or revision or each portion thereof.

(c) The Administrator will, after consideration of any State hearing record, promptly prepare and

publish proposed regulations setting forth a plan, or portion thereof, for a State if:

(1) The State fails to submit a plan within the time prescribed;

(2) The State fails to submit a plan revision required by § 60.23(a)(2) within the time prescribed; or

(3) The Administrator disapproves the State plan or plan revision or any portion thereof, as unsatisfactory because the requirements of this subpart have not been met.

(d) The Administrator will, within six months after the date required for submission of a plan or plan revision, promulgate the regulations proposed under paragraph (c) of this section with such modifications as may be appropriate unless, prior to such promulgation, the State has adopted and submitted a plan or plan revision which the Administrator determines to be approvable.

(e)(1) Except as provided in paragraph (e)(2) of this section, regulations proposed and promulgated by the Administrator under this section will prescribe emission standards of the same stringency as the corresponding emission guideline(s) specified in the final guideline document published under § 60.22(a) and will require final compliance with such standards as expeditiously as practicable but no later than the times specified in the guideline document.

(2) Upon application by the owner or operator of a designated facility to which regulations proposed and promulgated under this section will apply, the Administrator may provide for the application of less stringent emission standards or longer compliance schedules than those otherwise required by this section in accordance with the criteria specified in § 60.24(f).

(f) If a State failed to hold a public hearing as required by § 60.23(c), the Administrator will provide opportunity for a hearing within the State prior to promulgation of a plan under paragraph (d) of this section.

### § 60.28 Plan revisions by the State.

(a) Plan revisions which have the effect of delaying compliance with applicable emission standards or increments of progress or of establishing less stringent emission standards shall be submitted to the Administrator within 60 days after adoption in accordance with the procedures and requirements applicable to development and submission of the original plan.

(b) More stringent emission standards, or orders which have the effect of accelerating compliance, may be submitted to the Administrator as plan revisions in accordance with the procedures and requirements applicable to development and submission of the original plan.

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(c) A revision of a plan, or any portion thereof, shall not be considered part of an applicable plan until approved by the Administrator in accordance with this subpart.

**§ 60.29 Plan revisions by the Administrator.**

After notice and opportunity for public hearing in each affected State, the Administrator may revise any provision of an applicable plan if:

- (a) The provision was promulgated by the Administrator, and
- (b) The plan, as revised, will be consistent with the Act and with the requirements of this subpart.

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changes must have prior approval of the Administrator. An owner employing such methods or deviations from the test methods without obtaining prior approval does so at the risk of subsequent disapproval and retesting with approved methods.

Within the test methods, certain specific equipment or procedures are recognized as being acceptable or potentially acceptable and are specifically identified in the methods. The items identified as acceptable options may be used without approval but must be identified in the test report. The potentially approvable options are cited as "subject to the approval of the Administrator" or as "or equivalent." Such potentially approvable techniques or alternatives may be used at the discretion of the owner without prior approval. However, detailed descriptions for applying these potentially approvable techniques or alternatives are not provided in the test methods. Also, the potentially approvable options are not necessarily acceptable in all applications. Therefore, an owner electing to use such potentially approvable techniques or alternatives is responsible for: (1) assuring that the techniques or alternatives are in fact applicable and are properly executed; (2) including a written description of the alternative method in the test report (the written method must be clear and must be capable of being performed without additional instruction, and the degree of detail should be similar to the detail contained in the test methods); and (3) providing any rationale or supporting data necessary to show the validity of the alternative in the particular application. Failure to meet these requirements can result in the Administrator's disapproval of the alternative.

#### METHOD 1—SAMPLE AND VELOCITY TRAVERSES FOR STATIONARY SOURCES

##### 1. Principle and Applicability

1.1 Principle. To aid in the representative measurement of pollutant emissions and/or total volumetric flow rate from a stationary source, a measurement site where the effluent stream is flowing in a known direction is selected, and the cross-section of the stack is divided into a number of equal areas. A tra-

verse point is then located within each of these equal areas.

1.2 Applicability. This method is applicable to flowing gas streams in ducts, stacks, and flues. The method cannot be used when: (1) flow is cyclonic or swirling (see Section 2.4), (2) a stack is smaller than about 0.30 meter (12 in.) in diameter, or 0.071 m<sup>2</sup> (113 in.<sup>2</sup>) cross-sectional area, or (3) the measurement site is less than two stack or duct diameters downstream or less than a half diameter upstream from a flow disturbance.

The requirements of this method must be considered before construction of a new facility from which emissions will be measured; failure to do so may require subsequent alterations to the stack or deviation from the standard procedure. Cases involving variants are subject to approval by the Administrator, U.S. Environmental Protection Agency.

##### 2. Procedure

2.1 Selection of Measurement Site. Sampling or velocity measurement is performed at a site located at least eight stack or duct diameters downstream and two diameters upstream from any flow disturbance such as a bend, expansion, or contraction in the stack, or from a visible flame. If necessary, an alternative location may be selected, at a position at least two stack or duct diameters downstream and a half diameter upstream from any flow disturbance. For a rectangular cross section, an equivalent diameter ( $D_e$ ) shall be calculated from the following equation, to determine the upstream and downstream distances:

EC16NO91.248

where  $L$ =length and  $W$ =width.

An alternative procedure is available for determining the acceptability of a measurement location not meeting the criteria above. This procedure, determination of gas flow angles at the sampling points and comparing the results with acceptability criteria, is described in Section 2.5.

2.2 Determining the Number of Traverse Points.

EC01JN92.073

2.2.1 Particulate Traverses. When the eight- and two-diameter criterion can be met, the minimum number of traverse points shall be: (1) twelve, for circular or rectangular stacks with diameters (or equivalent diameters) greater than 0.61 meter (24 in.); (2) eight, for circular stacks with diameters between 0.30 and 0.61 meter (12-24 in.); (3) nine, for rectangular stacks with equivalent diameters between 0.30 and 0.61 meter (12-24 in.).

When the eight- and two-diameter criterion cannot be met, the minimum number of traverse points is determined from Figure 1-1. Before referring to the figure, however, determine the distances from the chosen measurement site to the nearest upstream and downstream disturbances, and divide each distance by the stack diameter or equivalent diameter, to determine the distance in terms of the number of duct diameters. Then, determine from Figure 1-1 the minimum number of traverse points that

corresponds: (1) to the number of duct diameters upstream; and (2) to the number of diameters downstream. Select the higher of the two minimum numbers of traverse points, or a greater value, so that for circular stacks the number is a multiple of 4, and for rectangular stacks, the number is one of those shown in Table 1-1.

TABLE 1-1. CROSS-SECTION LAYOUT FOR RECTANGULAR STACKS

Number of traverse points	Matrix layout
9	3x3
12	4x3
16	4x4
20	5x4
25	5x5
30	6x5
36	6x6
42	7x6
49	7x7

EC01JN92.074

2.2.2 Velocity (Non-Particulate) Traverses. When velocity or volumetric flow rate is to be determined (but not particulate matter), the same procedure as that for particulate traverses (Section 2.2.1) is followed, except that Figure 1-2 may be used instead of Figure 1-1.

2.3 Cross-sectional Layout and Location of Traverse Points.

2.3.1 Circular Stacks. Locate the traverse points on two perpendicular diameters according to Table 1-2 and the example shown in Figure 1-3. Any equation (for examples, see Citations 2 and 3 in the Bibliography) that gives the same values as those in Table 1-2 may be used in lieu of Table 1-2.

For particulate traverses, one of the diameters must be in a plane containing the greatest expected concentration variation, e.g., after bends, one diameter shall be in the plane of the bend. This requirement becomes less critical as the distance from the disturbance increases; therefore, other diameter locations may be used, subject to approval of the Administrator.

In addition for stacks having diameters greater than 0.61 m (24 in.) no traverse points shall be located within 2.5 centimeters (1.00 in.) of the stack walls; and for stack diameters equal to or less than 0.61 m (24 in.), no traverse points shall be located within 1.3 cm (0.50 in.) of the stack walls. To meet these criteria, observe the procedures given below.

2.3.1.1 Stacks With Diameters Greater Than 0.61 m (24 in.). When any of the traverse points as located in Section 2.3.1 fall within 2.5 cm (1.00 in.) of the stack walls, relocate them away from the stack walls to: (1) a distance of 2.5 cm (1.00 in.); or (2) a distance equal to the nozzle inside diameter, whichever is larger. These relocated traverse points (on each end of a diameter) shall be the "adjusted" traverse points.

Whenever two successive traverse points are combined to form a single adjusted traverse point, treat the adjusted point as two separate traverse points, both in the sampling (or velocity measurement) procedure, and in recording the data.

EC01JN82.075

TABLE 1-2. LOCATION OF TRAVERSE POINTS IN CIRCULAR STACKS  
 (Percent of stack diameter from inside wall to traverse point)

Traverse point number on a diameter	Number of traverse points on a diameter—																									
	2	4	6	8	10	12	14	16	18	20	22	24														
1	14.6	6.7	4.4	3.2	2.6	2.1	1.8	1.6	1.4	1.3	1.1	1.1														
2	85.4	25.0	14.6	10.5	8.2	6.7	5.7	4.9	4.4	3.9	3.5	3.2														
3		75.0	29.6	19.4	14.6	11.8	9.9	8.6	7.6	6.7	6.0	5.5														
4			70.4	32.3	22.6	17.7	14.6	12.5	10.9	9.7	8.7	7.9														
5				85.4	67.7	34.2	25.0	20.1	16.9	14.6	12.6	11.6	10.5													
6					80.6	65.8	35.6	26.9	22.0	18.8	16.6	14.6	13.2													
7						89.5	77.4	64.4	36.6	28.3	23.6	20.4	18.0	16.1												
8							86.8	85.4	76.0	63.4	37.5	29.6	25.0	21.8	19.4											
9								91.5	82.3	73.1	62.5	38.2	30.6	26.2	23.0											
10									97.4	88.2	79.9	71.7	61.8	38.8	31.5	27.2										
11										93.3	85.4	78.0	70.4	61.2	39.3	32.8										
12											97.9	90.1	83.1	75.4	69.4	60.7	39.8									
13												94.3	87.5	81.2	75.0	68.5	60.2									
14													98.2	91.5	85.4	79.6	73.8	67.7								
15														95.1	89.1	83.5	78.2	72.8								
16															98.4	92.5	87.1	82.0	77.0							
17																95.6	90.3	85.4	80.6							
18																	98.6	93.3	88.4	83.9						
19																		96.1	91.3	86.6	82.1					
20																			98.7	94.0	89.5	85.1				
21																				96.5	92.1	87.6	83.1			
22																					98.9	94.5	90.1	85.6		
23																						96.8	92.8	88.1	83.6	
24																							98.8	94.8	90.3	85.8

2.3.1.2 Stacks With Diameters Equal to or Less Than 0.61 m (24 in.). Follow the procedure in Section 2.3.1.1, noting only that any "adjusted" points should be relocated away from the stack walls to: (1) a distance of 1.3 cm (0.50 in.); or (2) a distance equal to the nozzle inside diameter, whichever is larger.

2.3.2 Rectangular Stacks. Determine the number of traverse points as explained in Sections 2.1 and 2.2 of this method. From Table 1-1, determine the grid configuration. Divide the stack cross-section into as many equal rectangular elemental areas as traverse points, and then locate a traverse point

at the centroid of each equal area according to the example in Figure 1-4.

If the tester desires to use more than the minimum number of traverse points, expand the "minimum number of traverse points" matrix (see Table 1-1) by adding the extra traverse points along one or the other or both legs of the matrix; the final matrix need not be balanced. For example, if a 4x3 "minimum number of points" matrix were expanded to 36 points, the final matrix could be 9x4 or 12x3, and would not necessarily have to be 6x6. After constructing the final matrix, divide the stack cross-section into as many equal rectangular, elemental areas as

traverse points, and locate a traverse point at the centroid of each equal area.

The situation of traverse points being too close to the stack walls is not expected to arise with rectangular stacks. If this problem should ever arise, the Administrator must be contacted for resolution of the matter.

2.4 Verification of Absence of Cyclonic Flow. In most stationary sources, the direction of stack gas flow is essentially parallel to the stack walls. However, cyclonic flow may exist (1) after such devices as cyclones and inertial demisters following venturi scrubbers, or (2) in stacks having tangential inlets or other duct configurations which tend to induce swirling; in these instances, the presence or absence of cyclonic flow at the sampling location must be determined. The following techniques are acceptable for this determination.

EC01JN82.076

Level and zero the manometer. Connect a Type S pitot tube to the manometer. Position the Type S pitot tube at each traverse point, in succession, so that the planes of the face openings of the pitot tube are perpendicular to the stack cross-sectional plane; when the Type S pitot tube is in this position, it is at "0° reference." Note the differential pressure ( $\Delta p$ ) reading at each traverse point. If a null (zero) pitot reading is obtained at 0° reference at a given traverse point, an acceptable flow condition exists at that point. If the pitot reading is not zero at 0° reference, rotate the pitot tube (up to  $\pm 90^\circ$  yaw angle), until a null reading is obtained. Carefully determine and record the value of the rotation angle ( $\alpha$ ) to the nearest degree. After the null technique has been applied at each traverse point, calculate the average of the absolute values of  $\alpha$ ; assign  $\alpha$  values of 0° to those points for which no rotation was required, and include these in the overall average. If the average value of  $\alpha$  is greater than 20°, the overall flow condition in the stack is unacceptable and alternative methodology, subject to the approval of the Administrator, must be used to perform accurate sample and velocity traverses.

The alternative procedure described in Section 2.5 may be used to determine the rotation angles in lieu of the procedure described above.

2.5 Alternative Measurement Site Selection Procedure. This alternative applies to sources where measurement locations are less than 2 equivalent stack or duct diameters downstream or less than  $\frac{1}{2}$  duct diameter upstream from a flow disturbance. The alternative should be limited to ducts larger than 24 in. in diameter where blockage and wall effects are minimal. A directional flow-sensing probe is used to measure pitch and yaw angles of the gas flow at 40 or more traverse points; the resultant angle is calculated and compared with acceptable criteria for mean and standard deviation.

NOTE: Both the pitch and yaw angles are measured from a line passing through the traverse point and parallel to the stack axis. The pitch angle is the angle of the gas flow component in the plane that INCLUDES the traverse line and is parallel to the stack axis. The yaw angle is the angle of the gas flow component in the plane PERPENDICULAR to the traverse line at the traverse point and is measured from the line passing through the traverse point and parallel to the stack axis.

#### 2.5.1 Apparatus.

2.5.1.1 Directional Probe. Any directional probe, such as United Sensor Type DA Three-Dimensional Directional Probe, capable of measuring both the pitch and yaw angles of gas flows is acceptable. (NOTE: Mention of trade name or specific products does not constitute endorsement by the U.S. Environmental Protection Agency.) Assign an identification number to the directional probe, and permanently mark or engrave the number on the body of the probe. The pressure holes of directional probes are susceptible to plugging when used in particulate-laden gas streams. Therefore, a system for cleaning the pressure holes by "back-purging" with pressurized air is required.

2.5.1.2 Differential Pressure Gauges. Inclined manometers, U-tube manometers, or other differential pressure gauges (e.g., magnetic gauges) that meet the specifications described in Method 2, section 2.2.

NOTE: If the differential pressure gauge produces both negative and positive readings, then both negative and positive pressure readings shall be calibrated at a minimum of three points as specified in Method 2, section 2.2.

2.5.2 Traverse Points. Use a minimum of 40 traverse points for circular ducts and 42 points for rectangular ducts for the gas flow angle determinations. Follow section 2.3 and Table 1-1 or 1-2 for the location and layout of the traverse points. If the measurement location is determined to be acceptable according to the criteria in this alternative

procedure, use the same traverse point number and locations for sampling and velocity measurements.

#### 2.5.3 Measurement Procedure.

2.5.3.1 Prepare the directional probe and differential pressure gauges as recommended by the manufacturer. Capillary tubing or surge tanks may be used to dampen pressure fluctuations. It is recommended, but not required, that a pretest leak check be conducted. To perform a leak check, pressurize or use suction on the impact opening until a reading of at least 7.6 cm (3 in.) H<sub>2</sub>O registers on the differential pressure gauge, then plug the impact opening. The pressure of a leak-free system will remain stable for at least 15 seconds.

2.5.3.2 Level and zero the manometers. Since the manometer level and zero may drift because of vibrations and temperature changes, periodically check the level and zero during the traverse.

2.5.3.3 Position the probe at the appropriate locations in the gas stream, and rotate until zero deflection is indicated for the yaw angle pressure gauge. Determine and record the yaw angle. Record the pressure gauge readings for the pitch angle, and determine the pitch angle from the calibration curve. Repeat this procedure for each traverse point. Complete a "back-purge" of the pressure lines and the impact openings prior to measurements of each traverse point.

A post-test check as described in section 2.5.3.1 is required. If the criteria for a leak-free system are not met, repair the equipment, and repeat the flow angle measurements.

2.5.4 Calculate the resultant angle at each traverse point, the average resultant angle, and the standard deviation using the following equations. Complete the calculations retaining at least one extra significant figure beyond that of the acquired data. Round the values after the final calculations.

2.5.4.1 Calculate the resultant angle at each traverse point:

$$R_i = \arccosine [(\cosine Y_i)(\cosine P_i)]$$

Eq. 1-2

Where:

R<sub>i</sub>=Resultant angle at traverse point i, degree.

Y<sub>i</sub>=Yaw angle at traverse point i, degree.

P<sub>i</sub>=Pitch angle at traverse point i, degree.

2.5.4.2 Calculate the average resultant for the measurements:

EC16N091.107

Where:

R=Average resultant angle, degree.

n=Total number of traverse points.

2.5.4.3 Calculate the standard deviations:

EN30AU93.031

Eq. 1-4

Where:

S<sub>d</sub>=Standard deviation, degree.

2.5.5 The measurement location is acceptable if R<sub>d</sub> ≤ 20° and S<sub>d</sub> ≤ 10°.

2.5.6 Calibration. Use a flow system as described in Sections 4.1.2.1 and 4.1.2.2 of Method 2. In addition, the flow system shall have the capacity to generate two test-section velocities: one between 365 and 730 m/min (1200 and 2400 ft/min) and one between 730 and 1100 m/min (2400 and 3600 ft/min).

2.5.6.1 Cut two entry ports in the test section. The axis through the entry ports shall be perpendicular to each other and intersect in the centroid of the test section. The ports should be elongated slots parallel to the axis of the test section and of sufficient length to allow measurement of pitch angles while maintaining the pitot head position at the test-section centroid. To facilitate alignment of the directional probe during calibration, the test section should be constructed of plexiglass or some other transparent material. All calibration measurements should be made at the same point in the test section, preferably at the centroid of the test section.

2.5.6.2 To ensure that the gas flow is parallel to the central axis of the test section, follow the procedure in Section 2.4 for cyclonic flow determination to measure the gas flow angles at the centroid of the test section from two test ports located 90° apart. The gas flow angle measured in each port must be ±2° of 0°. Straightening vanes should be installed, if necessary, to meet this criterion.

2.5.6.3 Pitch Angle Calibration. Perform a calibration traverse according to the manufacturer's recommended protocol in 5° increments for angles from -60° to +60° at one velocity in each of the two ranges specified above. Average the pressure ratio values obtained for each angle in the two flow ranges, and plot a calibration curve with the average values of the pressure ratio (or other suitable measurement factor as recommended by the manufacturer) versus the pitch angle. Draw a smooth line through the data points. Plot also the data values for each traverse point. Determine the differences between the measured data values and the angle from the calibration curve at the same pressure ratio. The difference at each comparison must be within 2° for angles between 0° and 40° and within 3° for angles between 40° and 60°.

2.5.6.4 Yaw Angle Calibration. Mark the three-dimensional probe to allow the determination of the yaw position of the probe. This is usually a line extending the length of the probe and aligned with the impact opening. To determine the accuracy of measurements of the yaw angle, only the zero or null position need be calibrated as follows. Place the directional probe in the test section, and rotate the probe until the zero position is found. With a protractor or other angle measuring device, measure the angle indicated by the yaw angle indicator on the three-dimensional probe. This should be within  $2^\circ$  of  $0^\circ$ . Repeat this measurement for any other points along the length of the pitot where yaw angle measurements could be read in order to account for variations in the pitot markings used to indicate pitot head positions.

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### METHOD 1A—SAMPLE AND VELOCITY TRAVERSES FOR STATIONARY SOURCES WITH SMALL STACKS OR DUCTS

#### 1. Applicability and Principle

1.1 The applicability and principle of this method are identical to Method 1, except this method's applicability is limited to stacks or ducts less than about 0.30 meter (12 in.) in diameter or  $0.071 \text{ m}^2$  ( $113 \text{ in.}^2$ ) in cross-sectional area, but equal to or greater than about 0.10 meter (4 in.) in diameter or  $0.0081 \text{ m}^2$  ( $12.57 \text{ in.}^2$ ) in cross-sectional area.

1.2 In these small diameter stacks or ducts, the conventional Method 5 stack assembly (consisting of a Type S pitot tube attached to a sampling probe, equipped with a nozzle and thermocouple) blocks a significant portion of the cross section of the duct and causes inaccurate measurements. Therefore, for particulate matter (PM) sampling in small stacks or ducts, the gas velocity is measured using a standard pitot tube downstream of the actual emission sampling site. The straight run of duct between the PM sampling and velocity measurement sites allows the flow profile, temporarily disturbed by the presence of the sampling probe, to redevelop and stabilize.

1.3 The cross-sectional layout and location of traverse points and the verification of the absence of cyclonic flow are the same as in Method 1, Sections 2.3 and 2.4, respectively. Differences from Method 1, except as noted, are given below.

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ER14MY89.044

**METHOD 3—GAS ANALYSIS FOR THE  
DETERMINATION OF DRY MOLECULAR WEIGHT**

**1. APPLICABILITY AND PRINCIPLE**

*1.1 Applicability.*

1.1.1 This method is applicable for determining carbon dioxide (CO<sub>2</sub>) and oxygen (O<sub>2</sub>) concentrations and dry molecular weight of

a sample from a gas stream of a fossil-fuel combustion process. The method may also be applicable to other processes where it has been determined that compounds other than CO<sub>2</sub>, O<sub>2</sub>, carbon monoxide (CO), and nitrogen (N<sub>2</sub>) are not present in concentrations sufficient to affect the results.

1.1.2 Other methods, as well as modifications to the procedure described herein, are

also applicable for some or all of the above determinations. Examples of specific methods and modifications include: (1) A multi-point sampling method using an Orsat analyzer to analyze individual grab samples obtained at each point; (2) a method using CO<sub>2</sub> or O<sub>2</sub> and stoichiometric calculations to determine dry molecular weight; and (3) assigning a value of 30.0 for dry molecular weight, in lieu of actual measurements, for processes burning natural gas, coal, or oil. These methods and modifications may be used, but are subject to the approval of the Administrator, U.S. Environmental Protection Agency (EPA).

1.1.3 Note. Mention of trade names or specific products does not constitute endorsements by EPA.

1.2 Principle. A gas sample is extracted from a stack by one of the following methods: (1) Single-point, grab sampling; (2) single-point, integrated sampling; or (3) multi-point, integrated sampling. The gas sample is analyzed for percent CO<sub>2</sub>, percent O<sub>2</sub>, and if necessary, for percent CO. For dry molecular weight determination, either an Orsat or a Fyrite analyzer may be used for the analysis.

## 2. APPARATUS

As an alternative to the sampling apparatus and systems described herein, other

sampling systems (e.g., liquid displacement) may be used, provided such systems are capable of obtaining a representative sample and maintaining a constant sampling rate, and are, otherwise, capable of yielding acceptable results. Use of such systems is subject to the approval of the Administrator.

### 2.1 Grab Sampling (Figure 3-1).

2.1.1 Probe. Stainless steel or borosilicate glass tubing equipped with an in-stack or out-stack filter to remove particulate matter (a plug of glass wool is satisfactory for this purpose). Any other materials, inert to O<sub>2</sub>, CO<sub>2</sub>, CO, and N<sub>2</sub> and resistant to temperature at sampling conditions, may be used for the probe. Examples of such materials are aluminum, copper, quartz glass, and Teflon.

2.1.2 Pump. A one-way squeeze bulb, or equivalent, to transport the gas sample to the analyzer.

### 2.2 Integrated Sampling (Figure 3-2).

2.2.1 Probe. Same as in Section 2.1.1.

2.2.2 Condenser. An air-cooled or water-cooled condenser, or other condenser no greater than 250 ml that will not remove O<sub>2</sub>, CO<sub>2</sub>, CO, and N<sub>2</sub>, to remove excess moisture which would interfere with the operation of the pump and flowmeter.

2.2.3 Valve. A needle valve, to adjust sample gas flow rate.

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**2.2.4 Pump.** A leaf-free, diaphragm-type pump, or equivalent, to transport sample gas to the flexible bag. Install a small surge tank between the pump and rate meter to eliminate the pulsation effect of the diaphragm pump on the rotameter.

**2.2.5 Rate Meter.** A rotameter, or equivalent rate meter, capable of measuring flow rate to within 2 percent of the selected flow

rate. A flow rate range of 500 to 1000 cc/min is suggested.

**2.2.6 Flexible Bag.** Any leak-free plastic (e.g., Tedlar, Mylar, Teflon) or plastic-coated aluminum (e.g., aluminized Mylar) bag, or equivalent, having a capacity consistent with the selected flow rate and time length of the test run. A capacity in the range of 55 to 90 liters is suggested. To leak check the



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Figure 3-3. Sampling rate data.

## 6. LEAK-CHECK PROCEDURE FOR ANALYZER

Moving an Orsat analyzer frequently causes it to leak. Therefore, an Orsat analyzer should be thoroughly leak checked on site before the flue gas sample is introduced into it. The procedure for leak checking an Orsat analyzer is as follows:

6.1 Bring the liquid level in each pipette up to the reference mark on the capillary tubing, and then close the pipette stopcock.

6.2 Raise the leveling bulb sufficiently to bring the confining liquid meniscus onto the graduated portion of the burette, and then close the manifold stopcock.

### 6.3 Record the meniscus position.

6.4 Observe the meniscus in the burette and the liquid level in the pipette for movement over the next 4 minutes.

6.5 For the Orsat analyzer to pass the leak check, two conditions must be met:

6.5.1 The liquid level in each pipette must not fall below the bottom of the capillary tubing during this 4-minute interval.

6.5.2 The meniscus in the burette must not change by more than 0.2 ml during this 4-minute interval.

6.6 If the analyzer fails the leak-check procedure, check all rubber connections and stopcocks to determine whether they might be the cause of the leak. Disassemble, clean, and regrease leaking stopcocks. Replace leaking rubber connections. After the analyzer is reassembled, repeat the leak-check procedure.

## 7. CALCULATIONS

### 7.1 Nomenclature

$M_d$  = Dry molecular weight, g/g-mole (lb/lb-mole).

%CO<sub>2</sub> = Percent CO<sub>2</sub> by volume, dry basis.

%O<sub>2</sub> = Percent O<sub>2</sub> by volume, dry basis.

%CO = Percent CO by volume, dry basis.

%N<sub>2</sub> = Percent N<sub>2</sub> by volume, dry basis.

0.280 = Molecular weight of N<sub>2</sub> or CO, divided by 100.

0.320 = Molecular weight of O<sub>2</sub> divided by 100.

0.440 = Molecular weight of CO<sub>2</sub> divided by 100.

7.2 Dry Molecular Weight. Use Equation 3-1 to calculate the dry molecular weight of the stack gas.

$$M_d = 0.440(\%CO_2) + 0.320(\%O_2) + 0.280(\%N_2 + \%CO) \quad \text{Eq. 3-1}$$

NOTE. The above equation does not consider argon in air (about 0.9 percent, molecular weight of 39.9). A negative error of about 0.4 percent is introduced. The tester may choose to include argon in the analysis using procedures subject to approval of the Administrator.

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## METHOD 3A—DETERMINATION OF OXYGEN AND CARBON DIOXIDE CONCENTRATIONS IN EMISSIONS FROM STATIONARY SOURCES (INSTRUMENTAL ANALYZER PROCEDURE)

### 1. Applicability and Principle

1.1 Applicability. This method is applicable to the determination of oxygen (O<sub>2</sub>) and carbon dioxide (CO<sub>2</sub>) concentrations in emissions from stationary sources only when specified within the regulations.

1.2 Principle. A sample is continuously extracted from the effluent stream; a portion of the sample stream is conveyed to an instrumental analyzer(s) for determination of O<sub>2</sub> and CO<sub>2</sub> concentration(s). Performance specifications and test procedures are provided to ensure reliable data.

### 2. Range and Sensitivity

Same as Method 6C, Sections 2.1 and 2.2, except that the span of the monitoring system shall be selected such that the average O<sub>2</sub> or CO<sub>2</sub> concentration is not less than 20 percent of the span.

### 3. Definitions

3.1 Measurement System. The total equipment required for the determination of the O<sub>2</sub> or CO<sub>2</sub> concentration. The measurement system consists of the same major subsystems as defined in Method 6C, Sections 3.1.1, 3.1.2, and 3.1.3.

3.2 Span, Calibration Gas, Analyzer Calibration Error, Sampling System Bias, Zero Drift, Calibration Drift, Response Time, and Calibration Curve. Same as Method 6C, Sections 3.2 through 3.8, and 3.10.

3.3 Interference Response. The output response of the measurement system to a component in the sample gas, other than the gas component being measured.

### 4. Measurement System Performance Specifications

Same as Method 6C, Sections 4.1 through 4.4.

### 5. Apparatus and Reagents

5.1 Measurement System. Any measurement system for O<sub>2</sub> or CO<sub>2</sub> that meets the specifications of this method. A schematic of an acceptable measurement system is shown in Figure 6C-1 of Method 6C. The essential components of the measurement system are described below:

5.1.1 Sample Probe. A leak-free probe, of sufficient length to traverse the sample points.

5.1.2 Sample Line. Tubing, to transport the sample gas from the probe to the moisture removal system. A heated sample line is not required for systems that measure the O<sub>2</sub> or CO<sub>2</sub> concentration on a dry basis, or transport dry gases.

5.1.3 Sample Transport Line, Calibration Value Assembly, Moisture Removal System, Particulate Filter, Sample Pump, Sample Flow Rate Control, Sample Gas Manifold, and Data Recorder. Same as Method 6C, Sections 5.1.3 through 5.1.9, and 5.1.11, except that the requirements to use stainless steel, Teflon, and nonreactive glass filters do not apply.

5.1.4 Gas Analyzer. An analyzer to determine continuously the O<sub>2</sub> or CO<sub>2</sub> concentration in the sample gas stream. The analyzer shall meet the applicable performance specifications of Section 4. A means of controlling the analyzer flow rate and a device for determining proper sample flow rate (e.g., precision rotameter, pressure gauge downstream of all flow controls, etc.) shall be provided at the analyzer. The requirements for measuring and controlling the analyzer flow rate are not applicable if data are presented that demonstrate the analyzer is insensitive to flow variations over the range encountered during the test.

5.2 Calibration Gases. The calibration gases for CO<sub>2</sub> analyzers shall be CO<sub>2</sub> in N<sub>2</sub> or CO<sub>2</sub> in air. Alternatively, CO<sub>2</sub>/SO<sub>2</sub>, O<sub>2</sub>/SO<sub>2</sub>, or O<sub>2</sub>/CO<sub>2</sub>/SO<sub>2</sub> gas mixtures in N<sub>2</sub> may be used. Three calibration gases, as specified Section 5.3.1 through 5.3.3 of Method 6C, shall be used. For O<sub>2</sub> monitors that cannot analyze zero gas, a calibration gas concentration equivalent to less than 10 percent of the span may be used in place of zero gas.

#### 6. Measurement System Performance Test Procedures

Perform the following procedures before measurement of emissions (Section 7).

6.1 Calibration Concentration Verification. Follow Section 6.1 of Method 6C, except if calibration gas analysis is required, use Method 3 and change the acceptance criteria for agreement among Method 3 results to 5 percent (or 0.2 percent by volume, whichever is greater).

6.2 Interference Response. Conduct an interference response test of the analyzer prior to its initial use in the field. Thereafter, recheck the measurement system if changes are made in the instrumentation that could

alter the interference response (e.g., changes in the type of gas detector). Conduct the interference response in accordance with Section 5.4 of Method 20.

6.3 Measurement System Preparation, Analyzer Calibration Error, and Sampling System Bias Check. Follow Sections 6.2 through 6.4 of Method 6C.

#### 7. Emission Test Procedure

7.1 Selection of Sampling Site and Sampling Points. Select a measurement site and sampling points using the same criteria that are applicable to tests performed using Method 3.

7.2 Sample Collection. Position the sampling probe at the first measurement point, and begin sampling at the same rate as used during the sampling system bias check. Maintain constant rate sampling (i.e., ±10 percent) during the entire run. The sampling time per run shall be the same as for tests conducted using Method 3 plus twice the system response time. For each run, use only those measurements obtained after twice the response time of the measurement system has elapsed to determine the average effluent concentration.

7.3 Zero and Calibration Drift Test. Follow Section 7.4 of Method 6C.

#### 8. Quality Control Procedures

The following quality control procedures are recommended when the results of this method are used for an emission rate correction factor, or excess air determination. The tester should select one of the following options for validating measurement results:

8.1 If both O<sub>2</sub> and CO<sub>2</sub> are measured using Method 3A, the procedures described in Section 4.4 of Method 3 should be followed to validate the O<sub>2</sub> and CO<sub>2</sub> measurement results.

8.2 If only O<sub>2</sub> is measured using Method 3A, measurements of the sample stream CO<sub>2</sub> concentration should be obtained at the sample by-pass vent discharge using an Orsat or Fyrite analyzer, or equivalent. Duplicate samples should be obtained concurrent with at least one run. Average the duplicate Orsat or Fyrite analysis results for each run. Use the average CO<sub>2</sub> values for comparison with the O<sub>2</sub> measurements in accordance with the procedures described in Section 4.4 of Method 3.

8.3 If only CO<sub>2</sub> is measured using Method 3A, concurrent measurements of the sample stream CO<sub>2</sub> concentration should be obtained using an Orsat or Fyrite analyzer as described in Section 8.2. For each run, differences greater than 0.5 percent between the Method 3A results and the average of the duplicate Fyrite analysis should be investigated.

#### 9. Emission Calculation

For all CO<sub>2</sub> analyzers, and for O<sub>2</sub> analyzers that can be calibrated with zero gas, follow

Section 8 of Method 6C, except express all concentrations as percent, rather than ppm.

For O<sub>2</sub> analyzers that use a low-level calibration gas in place of a zero gas, calculate

the effluent gas concentration using Equation 3A-1.

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Where:

$C_{gas}$  = Effluent gas concentration, dry basis, percent.

$C_{ms}$  = Actual concentration of the upscale calibration gas, percent.

$C_{ml}$  = Actual concentration of the low-level calibration gas, percent.

$C_{ms}$  = Average of initial and final system calibration bias check responses for the upscale calibration gas, percent.

$C_{ml}$  = Average of initial and final system calibration bias check responses for the low-level gas, percent.

$C_0$  = Average gas concentration indicated by the gas analyzer, dry basis, percent.

#### 10. Bibliography

Same as bibliography of Method 6C.

### METHOD 3B—GAS ANALYSIS FOR THE DETERMINATION OF EMISSION RATE CORRECTION FACTOR OR EXCESS AIR

#### 1. APPLICABILITY AND PRINCIPLE

##### 1.1 Applicability

1.1.1 This method is applicable for determining carbon dioxide (CO<sub>2</sub>), oxygen (O<sub>2</sub>), and carbon monoxide (CO) concentrations of a sample from a gas stream of a fossil-fuel combustion process for excess air or emission rate correction factor calculations.

1.1.2 Other methods, as well as modifications to the procedure described herein, are also applicable for all of the above determinations. Examples of specific methods and modifications include: (1) A multi-point sampling method using an Orsat analyzer to analyze individual grab samples obtained at each point, and (2) a method using CO<sub>2</sub> or O<sub>2</sub> and stoichiometric calculations to determine excess air. These methods and modifications may be used, but are subject to the approval of the Administrator, U.S. Environmental Protection Agency (EPA).

1.1.3 Note. Mention of trade names or specific products does not constitute endorsement by EPA.

1.2 Principle. A gas sample is extracted from a stack by one of the following methods: (1) Single-point, grab sampling; (2) single-point, integrated sampling; or (3) multi-point, integrated sampling. The gas sample is analyzed for percent CO<sub>2</sub>, percent O<sub>2</sub>, and, if necessary, percent CO. An Orsat analyzer

must be used for excess air or emission rate correction factor determinations.

#### 2. APPARATUS

The alternative sampling systems are the same as those mentioned in Section 2 of Method 3.

2.1 Grab Sampling and Integrated Sampling. Same as in Sections 2.1 and 2.2, respectively, of Method 3.

2.2 Analysis. An Orsat analyzer only. For low CO<sub>2</sub> (less than 4.0 percent) or high O<sub>2</sub> (greater than 15.0 percent) concentrations, the measuring burettes of the Orsat must have at least 0.1 percent subdivisions. For Orsat maintenance and operation procedures, follow the instructions recommended by the manufacturer, unless otherwise specified herein.

#### 3. PROCEDURES

Each of the three procedures below shall be used only when specified in an applicable subpart of the standards. The use of these procedures for other purposes must have specific prior approval of the Administrator.

NOTE.—A Fyrite-type combustion gas analyzer is not acceptable for excess air or emission rate correction factor determinations, unless approved by the Administrator. If both percent CO<sub>2</sub> and percent O<sub>2</sub> are measured, the analytical results of any of the three procedures given below may be used for calculating the dry molecular weight (see Method 3).

##### 3.1 Single-Point, Grab Sampling and Analytical Procedure.

3.1.1 The sampling point in the duct shall be as described in Section 3.1 of Method 3.

3.1.2 Set up the equipment as shown in Figure 3-1 of Method 3, making sure all connections ahead of the analyzer are tight. Leak check the Orsat analyzer according to the procedure described in Section 6 of Method 3. This leak check is mandatory.

3.1.3 Place the probe in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line long enough to allow at least five exchanges. Draw a sample into the analyzer. For emission rate correction factor determinations, immediately analyze the sample, as outlined

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METHOD 5—DETERMINATION OF PARTICULATE EMISSIONS FROM STATIONARY SOURCES

1. Principle and Applicability

1.1 Principle. Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at a temperature in the range of  $120 \pm 14$  °C ( $248 \pm 25$  °F) or such other temperature as specified by an applicable subpart of the standards or approved by Administrator, U.S. Environmental Protection Agency, for a particular application. The particulate mass, which includes any material that condenses at or above the filtration temperature, is de-

termined gravimetrically after removal of uncombined water.

1.2 Applicability. This method is applicable for the determination of particulate emissions from stationary sources.

2. Apparatus

2.1 Sampling Train. A schematic of the sampling train used in this method is shown in Figure 5-1. Complete construction details are given in APTD-0581 (Citation 2 in Bibliography); commercial models of this train are also available. For changes from APTD-0581 and for allowable modifications of the train shown in Figure 5-1, see the following subsections.

The operating and maintenance procedures for the sampling train are described in APTD-0576 (Citation 3 in Bibliography). Since correct usage is important in obtaining valid results, all users should read APTD-0576 and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein. The sampling train consists of the following components:

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2.1.1 Probe Nozzle. Stainless steel (316) or glass with sharp, tapered leading edge. The angle of taper shall be  $\leq 30^\circ$  and the taper shall be on the outside to preserve a con-

stant internal diameter. The probe nozzle shall be of the button-hook or elbow design, unless otherwise specified by the Administrator. If made of stainless steel, the nozzle

shall be constructed from seamless tubing; other materials of construction may be used, subject to the approval of the Administrator.

A range of nozzle sizes suitable for isokinetic sampling should be available, e.g., 0.32 to 1.27 cm ( $\frac{1}{8}$  to  $\frac{1}{2}$  in.)—or larger if higher volume sampling trains are used—inside diameter (ID) nozzles in increments of 0.16 cm ( $\frac{1}{16}$  in.). Each nozzle shall be calibrated according to the procedures outlined in Section 5.

2.1.2 Probe Liner. Borosilicate or quartz glass tubing with a heating system capable of maintaining a gas temperature at the exit end during sampling of  $120 \pm 14$  °C ( $248 \pm 25$  °F), or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator for a particular application. (The tester may opt to operate the equipment at a temperature lower than that specified.) Since the actual temperature at the outlet of the probe is not usually monitored during sampling, probes constructed according to APTD-0581 and utilizing the calibration curves of APTD-0576 (or calibrated according to the procedure outlined in APTD-0576) will be considered acceptable.

Either borosilicate or quartz glass probe liners may be used for stack temperatures up to about 480 °C (900 °F); quartz liners shall be used for temperatures between 480 and 900 °C (900 and 1,650 °F). Both types of liners may be used at higher temperatures than specified for short periods of time, subject to the approval of the Administrator. The softening temperature for borosilicate is 820 °C (1,508 °F), and for quartz it is 1,500 °C (2,732 °F).

Whenever practical, every effort should be made to use borosilicate or quartz glass probe liners. Alternatively, metal liners (e.g., 316 stainless steel, Incoloy 825,<sup>2</sup> or other corrosion resistant metals) made of seamless tubing may be used, subject to the approval of the Administrator.

2.1.3 Pitot Tube. Type S, as described in Section 2.1 of Method 2, or other device approved by the Administrator. The pitot tube shall be attached to the probe (as shown in Figure 5-1) to allow constant monitoring of the stack gas velocity. The impact (high pressure) opening plane of the pitot tube shall be even with or above the nozzle entry plane (see Method 2, Figure 2-8b) during sampling. The Type S pitot tube assembly shall have a known coefficient, determined as outlined in Section 4 of Method 2.

2.1.4 Differential Pressure Gauge. Inclined manometer or equivalent device (two), as described in Section 2.2 of Method 2. One manometer shall be used or velocity head ( $\Delta P$ ) readings, and the other, for orifice differential pressure readings.

<sup>2</sup>Mention of trade names or specific product does not constitute endorsement by the Environmental Protection Agency.

2.1.5 Filter Holder. Borosilicate glass, with a glass frit filter support and a silicone rubber gasket. Other materials of construction (e.g., stainless steel, Teflon, Viton) may be used, subject to approval of the Administrator. The holder design shall provide a positive seal against leakage from the outside or around the filter. The holder shall be attached immediately at the outlet of the probe (or cyclone, if used).

2.1.6 Filter Heating System. Any heating system capable of maintaining a temperature around the filter holder during sampling of  $120 \pm 14$  °C ( $248 \pm 25$  °F), or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator for a particular application. Alternatively, the tester may opt to operate the equipment at a temperature lower than that specified. A temperature gauge capable of measuring temperature to within 3 °C (5.4 °F) shall be installed so that the temperature around the filter holder can be regulated and monitored during sampling. Heating systems other than the one shown in APTD-0581 may be used.

2.1.7 Condenser. The following system shall be used to determine the stack gas moisture content: Four impingers connected in series with leak-free ground glass fittings or any similar leak-free non-contaminating fittings. The first, third, and fourth impingers shall be of the Greenburg-Smith design, modified by replacing the tip with 1.3 cm ( $\frac{1}{2}$  in.) ID glass tube extending to about 1.3 cm ( $\frac{1}{2}$  in.) from the bottom of the flask. The second impinger shall be of the Greenburg-Smith design with the standard tip. Modifications (e.g., using flexible connections between the impingers, using materials other than glass, or using flexible vacuum lines to connect the filter holder to the condenser) may be used, subject to the approval of the Administrator. The first and second impingers shall contain known quantities of water (Section 4.1.3), the third shall be empty, and the fourth shall contain a known weight of silica gel, or equivalent desiccant. A thermometer, capable of measuring temperature to within 1 °C (2 °F) shall be placed at the outlet of the fourth impinger for monitoring purposes.

Alternatively, any system that cools the sample gas stream and allows measurement of the water condensed and moisture leaving the condenser, each to within 1 ml or 1 g may be used, subject to the approval of the Administrator. Acceptable means are to measure the condensed water either gravimetrically or volumetrically and to measure the moisture leaving the condenser by: (1) monitoring the temperature and pressure at the exit of the condenser and using Dalton's law of partial pressures; or (2) passing the sample gas stream through a tared silica gel (or equivalent desiccant) trap with exit gases

kept below 20 °C (68 °F) and determining the weight gain.

If means other than silica gel are used to determine the amount of moisture leaving the condenser, it is recommended that silica gel (or equivalent) still be used between the condenser system and pump to prevent moisture condensation in the pump and metering devices and to avoid the need to make corrections for moisture in the metered volume.

NOTE: If a determination of the particulate matter collected in the impingers is desired in addition to moisture content, the impinger system described above shall be used, without modification. Individual States or control agencies requiring this information shall be contacted as to the sample recovery and analysis of the impinger contents.

2.1.8 Metering System. Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3 °C (5.4 °F), dry gas meter capable of measuring volume to within 2 percent, and related equipment, as shown in Figure 5-1. Other metering systems capable of maintaining sampling rates within 10 percent of isokinetic and of determining sample volumes to within 2 percent may be used, subject to the approval of the Administrator. When the metering system is used in conjunction with a pitot tube, the system shall enable checks of isokinetic rates.

Sampling trains utilizing metering systems designed for higher flow rates than that described in APTD-0581 or APDT-0578 may be used provided that the specifications of this method are met.

2.1.9 Barometer. Mercury aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases the barometric reading may be obtained from a nearby National Weather Service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

2.1.10 Gas Density Determination Equipment. Temperature sensor and pressure gauge, as described in Sections 2.3 and 2.4 of Method 2, and gas analyzer, if necessary, as described in Method 3. The temperature sensor shall, preferably, be permanently attached to the pitot tube or sampling probe in a fixed configuration, such that the tip of the sensor extends beyond the leading edge of the probe sheath and does not touch any metal. Alternatively, the sensor may be attached just prior to use in the field. Note, however, that if the temperature sensor is attached in the field, the sensor must be placed in an interference-free arrangement with respect to the Type S pitot tube openings (see Method 2, Figure 2-7). As a second

alternative, if a difference of not more than 1 percent in the average velocity measurement is to be introduced, the temperature gauge need not be attached to the probe or pitot tube. (This alternative is subject to the approval of the Administrator.)

2.2 Sample Recovery. The following items are needed.

2.2.1 Probe-Liner and Probe-Nozzle Brushes. Nylon bristle brushes with stainless steel wire handles. The probe brush shall have extensions (at least as long as the probe) of stainless steel, Nylon, Teflon, or similarly inert material. The brushes shall be properly sized and shaped to brush out the probe liner and nozzle.

2.2.2 Wash Bottles—Two. Glass wash bottles are recommended; polyethylene wash bottles may be used at the option of the tester. It is recommended that acetone not be stored in polyethylene bottles for longer than a month.

2.2.3 Glass Sample Storage Containers. Chemically resistant, borosilicate glass bottles, for acetone washes, 500 ml or 1000 ml. Screw cap liners shall either be rubber-backed Teflon or shall be constructed so as to be leak-free and resistant to chemical attack by acetone. (Narrow mouth glass bottles have been found to be less prone to leakage.) Alternatively, polyethylene bottles may be used.

2.2.4 Petri Dishes. For filter samples, glass or polyethylene, unless otherwise specified by the Administrator.

2.2.5 Graduated Cylinder and/or Balance. To measure condensed water to within 1 ml or 1 g. Graduated cylinders shall have subdivisions no greater than 2 ml. Most laboratory balances are capable of weighing to the nearest 0.5 g or less. Any of these balances is suitable or use here and in Section 2.3.4.

2.2.6 Plastic Storage Containers. Air-tight containers to store silica gel.

2.2.7 Funnel and Rubber Policeman. To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

2.2.8 Funnel. Glass or polyethylene, to aid in sample recovery.

2.3 Analysis. For analysis, the following equipment is needed.

2.3.1 Glass Weighing Dishes.

2.3.2 Desiccator.

2.3.3 Analytical Balance. To measure to within 0.1 mg.

2.3.4 Balance. To measure to within 0.5 g.

2.3.5 Beakers, 250 ml.

2.3.6 Hygrometer. To measure the relative humidity of the laboratory environment.

2.3.7 Temperature Gauge. To measure the temperature of the laboratory environment.

### 3. Reagents

3.1 Sampling. The reagents used in sampling are as follows:

3.1.1 **Filters.** Glass fiber filters, without organic binder, exhibiting at least 99.95 percent efficiency (<0.05 percent penetration) on 0.3-micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM Standard Method D2886-71 (Reapproved 1978) (incorporated by reference—see §60.17). Test data from the supplier's quality control program are sufficient for this purpose. In sources containing SO<sub>2</sub> or SO<sub>3</sub>, the filter material must be of a type that is unreactive to SO<sub>2</sub> or SO<sub>3</sub>. Citation 10 in Bibliography, may be used to select the appropriate filter.

3.1.2 **Silica Gel.** Indicating type, 6 to 16 mesh. If previously used, dry at 175 °C (350 °F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to the approval of the Administrator.

3.1.3 **Water.** When analysis of the material caught in the impingers is required, deionized distilled water shall be used. Run blanks prior to field use to eliminate a high blank on test samples.

3.1.4 **Crushed Ice.**

3.1.5 **Stopcock Grease.** Acetone-insoluble, heat-stable silicone grease. This is not necessary if screw-on connectors with Teflon sleeves, or similar, are used. Alternatively, other types of stopcock grease may be used, subject to the approval of the Administrator.

3.2 **Sample Recovery.** Acetone-reagent grade, ≤0.001 percent residue, in glass bottles—is required. Acetone from metal containers generally has a high residue blank and should not be used. Sometimes, suppliers transfer acetone to glass bottles from metal containers; thus, acetone blanks shall be run prior to field use and only acetone with low blank values (≤0.001 percent) shall be used. In no case shall a blank value of greater than 0.001 percent of the weight of acetone used be subtracted from the sample weight.

3.3 **Analysis.** Two reagents are required for the analysis:

3.3.1 **Acetone.** Same as 3.2.

3.3.2 **Desiccant.** Anhydrous calcium sulfate, indicating type. Alternatively, other types of desiccants may be used, subject to the approval of the Administrator.

#### 4. Procedure

4.1 **Sampling.** The complexity of this method is such that, in order to obtain reliable results, testers should be trained and experienced with the test procedures.

4.1.1 **Pretest Preparation.** It is suggested that sampling equipment be maintained according to the procedure described in APTD-0578.

Weigh several 200 to 300 g portions of silica gel in air-tight containers to the nearest 0.5 g. Record the total weight of the silica gel plus container, on each container. As an alternative, the silica gel need not be

preweighed, but may be weighed directly in the impinger or sampling holder just prior to train assembly.

Check filters visually against light for irregularities and flaws or pinhole leaks. Label filters of the proper diameter on the back side near the edge using numbering machine ink. As an alternative, label the shipping containers (glass or plastic petri dishes) and keep the filters in these containers at all times except during sampling and weighing.

Desiccate the filters at 20±5.6 °C (68±10 °F) and ambient pressure for at least 24 hours and weigh at intervals of at least 6 hours to a constant weight, i.e., 0.5 mg change from previous weighing; record results to the nearest 0.1 mg. During each weighing the filter must not be exposed to the laboratory atmosphere for a period greater than 2 minutes and a relative humidity above 50 percent. Alternatively (unless otherwise specified by the Administrator), the filters may be oven dried at 105 °C (220 °F) for 2 to 3 hours, desiccated for 2 hours, and weighed. Procedures other than those described, which account for relative humidity effects, may be used, subject to the approval of the Administrator.

4.1.2 **Preliminary Determinations.** Select the sampling site and the minimum number of sampling points according to Method 1 or as specified by the Administrator. Determine the stack pressure, temperature, and the range of velocity heads using Method 2. It is recommended that a leak-check of the pitot lines (see Method 2, Section 3.1) be performed. Determine the moisture content using Approximation Method 4 or its alternatives for the purpose of making isokinetic sampling rate settings. Determine the stack gas dry molecular weight, as described in Method 2, Section 3.8; if integrated Method 3 sampling is used for molecular weight determination, the integrated bag sample shall be taken simultaneously with, and for the same total length of time as, the particulate sample run.

Select a nozzle size based on the range of velocity heads, such that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see Section 2.2 of Method 2).

Select a suitable probe liner and probe length such that all traverse points can be sampled. For large stacks, consider sampling from opposite sides of the stack to reduce the length of probes.

Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the specific industry such that (1) the sampling time per point is not less than 2 min (or some greater time interval as specified by the Administrator), and (2) the sample volume taken (corrected to standard conditions)

will exceed the required minimum total gas sample volume. The latter is based on an approximate average sampling rate.

It is recommended that the number of minutes sampled at each point be an integer or an integer plus one-half minute, in order to avoid timekeeping errors. The sampling time at each point shall be the same.

In some circumstances, e.g., batch cycles, it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the Administrator's approval must first be obtained.

4.1.3 Preparation of Collection Train. During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin.

Place 100 ml of water in each of the first two impingers, leave the third impinger empty, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the fourth impinger. More silica gel may be used, but care should be taken to ensure that it is not entrained and carried out from the impinger during sampling. Place the container in a clean place for later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 g and recorded.

Using a tweezer or clean disposable surgical gloves, place a labeled (identified) and weighed filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as to prevent the sample gas stream from circumventing the filter. Check the filter for tears after assembly is completed.

When glass liners are used, install the selected nozzle using a Viton A O-ring when stack temperatures are less than 260 °C (500 °F) and an asbestos string gasket when temperatures are higher. See APTD-0576 for details. Other connecting systems using either 316 stainless steel or Teflon ferrules may be used. When metal liners are used, install the nozzle as above or by a leak-free direct mechanical connection. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

Set up the train as in Figure 3-1, using (if necessary) a very light coat of silicone grease on all ground glass joints, greasing only the outer portion (see APTD-0576) to avoid possibility of contamination by the silicone grease. Subject to the approval of the Administrator, a glass cyclone may be used between the probe and filter holder when the total particulate catch is expected to exceed 100 mg or when water droplets are present in the stack gas.

Place crushed ice around the impingers.

#### 4.1.4 Leak-Check Procedures.

4.1.4.1 Pretest Leak-Check. A pretest leak-check is recommended, but not required. If the tester opts to conduct the pretest leak-check, the following procedure shall be used.

After the sampling train has been assembled, turn on and set the filter and probe heating systems at the desired operating temperatures. Allow time for the temperatures to stabilize. If a Viton A O-ring or other leak-free connection is used in assembling the probe nozzle to the probe liner, leak-check the train at the sampling site by plugging the nozzle and pulling a 380 mm Hg (15 in. Hg) vacuum.

NOTE: A lower vacuum may be used, provided that it is not exceeded during the test.

If an asbestos string is used, do not connect the probe to the train during the leak-check. Instead, leak-check the train by first plugging the inlet to the filter holder (cyclone, if applicable) and pulling a 380 mm Hg (15 in. Hg) vacuum (see Note immediately above). Then connect the probe to the train and leak-check at about 25 mm Hg (1 in. Hg) vacuum; alternatively, the probe may be leak-checked with the rest of the sampling train, in one step, at 380 mm Hg (15 in. Hg) vacuum. Leakage rates in excess of 4 percent of the average sampling rate or 0.00057 m<sup>3</sup>/min (0.02 cfm), whichever is less, are unacceptable.

The following leak-check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with bypass valve fully open and coarse adjust valve, completely closed. Partially open the coarse adjust valve and slowly close the bypass valve until the desired vacuum is reached. Do not reverse direction of bypass valve; this will cause water to back up into the filter holder. If the desired vacuum is exceeded, either leak-check at this higher vacuum or end the leak-check as shown below and start over.

When the leak-check is completed, first slowly remove the plug from the inlet to the probe, filter holder, or cyclone (if applicable) and immediately turn off the vacuum pump. This prevents the water in the impingers from being forced backward into the filter holder and silica gel from being entrained backward into the third impinger.

4.1.4.2 Leak-Checks During Sample Run. If, during the sampling run, a component (e.g., filter assembly or impinger) change becomes necessary, a leak-check shall be conducted immediately before the change is made. The leak-check shall be done according to the procedure outlined in Section 4.1.4.1 above, except that it shall be done at a vacuum equal to or greater than the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than 0.00057 m<sup>3</sup>/min (0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and

no correction will need to be applied to the total volume of dry gas metered; if, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and plan to correct the sample volume as shown in Section 6.3 of this method, or shall void the sampling run.

Immediately after component changes, leak-checks are optional; if such leak-checks are done, the procedure outlined in Section 4.1.4.1 above shall be used.

**4.1.4.3 Post-test Leak-Check.** A leak-check is mandatory at the conclusion of each sampling run. The leak-check shall be done in accordance with the procedures outlined in Section 4.1.4.1, except that it shall be conducted at a vacuum equal to or greater than the maximum value reached during the sampling run. If the leakage rate is found to be no greater than  $0.00057 \text{ m}^3/\text{min}$  (0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction need be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and correct the sample volume as shown in Section 6.3 of this method, or shall void the sampling run.

**4.1.5 Particulate Train Operation.** During the sampling run, maintain an isokinetic sampling rate (within 10 percent of true isokinetic unless otherwise specified by the Administrator) and a temperature around the filter of  $120 \pm 14 \text{ }^\circ\text{C}$  ( $248 \pm 25 \text{ }^\circ\text{F}$ ), or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator.

For each run, record the data required on a data sheet such as the one shown in Figure

5-2. Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, before and after each leak-check, and when sampling is halted. Take other readings required by Figure 5-2 at least once at each sample point during each time increment and additional readings when significant changes (20 percent variation in velocity head readings) necessitate additional adjustments in flow rate. Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse.

Clean the portholes prior to the test run to minimize the chance of sampling deposited material. To begin sampling, remove the nozzle cap, verify that the filter and probe heating systems are up to temperature, and that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs are available, which aid in the rapid adjustment of the isokinetic sampling rate without excessive computations. These nomographs are designed for use when the Type S pitot tube coefficient is  $0.85 \pm 0.02$ , and the stack gas equivalent density (dry molecular weight) is equal to 29.4. APTD-0576 details the procedure for using the nomographs. If  $C_p$  and  $M_d$  are outside the above stated ranges do not use the nomographs unless appropriate steps (see Citation 7 in Bibliography) are taken to compensate for the deviations.



When the stack is under significant negative pressure (height of Impinger stem), take care to close the coarse adjust valve before inserting the probe into the stack to prevent water from backing into the filter holder. If necessary, the pump may be turned on with the coarse adjust valve closed.

When the probe is in position, block off the openings around the probe and porthole to prevent unrepresentative dilution of the gas stream.

Traverse the stack cross-section, as required by Method 1 or as specified by the Administrator, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the portholes; this minimizes the chance of extracting deposited material.

During the test run, make periodic adjustments to keep the temperature around the filter holder at the proper level; add more ice and, if necessary, salt to maintain a temperature of less than 20 °C (68 °F) at the condenser/silica gel outlet. Also, periodically check the level and zero of the manometer.

If the pressure drop across the filter becomes too high, making isokinetic sampling difficult to maintain, the filter may be replaced in the midst of a sample run. It is recommended that another complete filter assembly be used rather than attempting to change the filter itself. Before a new filter assembly is installed, conduct a leak-check (see Section 4.1.4.2). The total particulate weight shall include the summation of all filter assembly catches.

A single train shall be used for the entire sample run, except in cases where simultaneous sampling is required in two or more separate ducts or at two or more different locations within the same duct, or, in cases where equipment failure necessitates a change of trains. In all other situations, the use of two or more trains will be subject to the approval of the Administrator.

Note that when two or more trains are used, separate analyses of the front-half and (if applicable) Impinger catches from each train shall be performed, unless identical nozzle sizes were used on all trains, in which case, the front-half catches from the individual trains may be combined (as may the Impinger catches) and one analysis of front-half catch and one analysis of Impinger catch may be performed. Consult with the Administrator for details concerning the calculation of results when two or more trains are used.

At the end of the sample run, turn off the coarse adjust valve, remove the probe and nozzle from the stack, turn off the pump, record the final dry gas meter reading, and conduct a post-test leak-check, as outlined in Section 4.1.4.3. Also, leak-check the pitot lines as described in Method 2, Section 3.1;

the lines must pass this leak-check, in order to validate the velocity head data.

**4.1.5 Calculation of Percent Isokinetic.** Calculate percent isokinetic (see Calculations, Section 8) to determine whether the run was valid or another test run should be made. If there was difficulty in maintaining isokinetic rates due to source conditions, consult with the Administrator for possible variance on the isokinetic rates.

**4.2 Sample Recovery.** Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period. Allow the probe to cool.

When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it to prevent losing or gaining particulate matter. Do not cap off the probe tip tightly while the sampling train is cooling down as this would create a vacuum in the filter holder, thus drawing water from the Impingers into the filter holder.

Before moving the sample train to the cleanup site, remove the probe from the sample train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate that might be present. Wipe off the silicone grease from the filter inlet where the probe was fastened and cap it. Remove the umbilical cord from the last Impinger and cap the Impinger. If a flexible line is used between the first Impinger or condenser and the filter holder, disconnect the line at the filter holder and let any condensed water or liquid drain into the Impingers or condenser. After wiping off the silicone grease, cap off the filter holder outlet and Impinger inlet. Either ground-glass stoppers, plastic caps, or serum caps may be used to close these openings.

Transfer the probe and Impinger assembly to the cleanup area. This area should be clean and protected from the wind so that the chances of contaminating or losing the sample will be minimized.

Save a portion of the acetone used for cleanup as a blank. Take 200 ml of this acetone directly from the wash bottle being used and place it in a glass sample container labeled "acetone blank."

Inspect the train prior to and during disassembly and note any abnormal conditions. Treat the samples as follows:

**Container No. 1.** Carefully remove the filter from the filter holder and place it in its identified petri dish container. Use a pair of tweezers and/or clean disposable surgical gloves to handle the filter. If it is necessary to fold the filter, do so such that the particulate cake is inside the fold. Carefully transfer to the petri dish any particulate matter and/or filter fibers which adhere to the filter holder gasket, by using a dry Nylon bristle brush and/or a sharp-edged blade. Seal the container.

**Container No. 2.** Taking care to see that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover particulate matter or any condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder by washing these components with acetone and placing the wash in a glass container. Distilled water may be used instead of acetone when approved by the Administrator and shall be used when specified by the Administrator; in these cases, save a water blank and follow the Administrator's directions on analysis. Perform the acetone rinses as follows:

Carefully remove the probe nozzle and clean the inside surface by rinsing with acetone from a wash bottle and brushing with a Nylon bristle brush. Brush until the acetone rinse shows no visible particles, after which make a final rinse of the inside surface with acetone.

Brush and rinse the inside parts of the Swagelok fitting with acetone in a similar way until no visible particles remain.

Rinse the probe liner with acetone by tilting and rotating the probe while squirting acetone into its upper end so that all inside surfaces will be wetted with acetone. Let the acetone drain from the lower end into the sample container. A funnel (glass or polyethylene) may be used to aid on transferring liquid washes to the container. Follow the acetone rinse with a probe brush. Hold the probe in an inclined position, squirt acetone into the upper end as the probe brush is being pushed with a twisting action through the probe; hold a sample container underneath the lower end of the probe, and catch any acetone and particulate matter which is brushed from the probe. Run the brush through the probe three times or more until no visible particulate matter is carried out with the acetone or until none remains in the probe liner on visual inspection. With stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times since metal probes have small crevices in which particulate matter can be entrapped. Rinse the brush with acetone, and quantitatively collect these washings in the sample container. After the brushing, make a final acetone rinse of the probe as described above.

It is recommended that two people clean the probe to minimize sample losses. Between sampling runs, keep brushes clean and protected from contaminations.

After ensuring that all joints have been wiped clean of silicone grease, clean the inside of the front half of the filter holder by rubbing the surfaces with a Nylon bristle brush and rinsing with acetone. Rinse each surface three times or more if needed to remove visible particulate. Make a final rinse of the brush and filter holder. Carefully rinse out the glass cyclone, also (if applicable).

After all acetone washings and particulate matter have been collected in the sample container, tighten the lid on the sample container so that acetone will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether or not leakage occurred during transport. Label the container to clearly identify its contents.

**Container No. 3.** Note the color of the indicating silica gel to determine if it has been completely spent and make a notation of its condition. Transfer the silica gel from the fourth impinger to its original container and seal. A funnel may make it easier to pour the silica gel without spilling. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere to the impinger wall and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, follow the procedure for container No. 3 in Section 4.3.

**Impinger Water.** Treat the impingers as follows; Make a notation of any color or film in the liquid catch. Measure the liquid which is in the first three impingers to within  $\pm 1$  ml by using a graduated cylinder or by weighing it to within  $\pm 0.5$  g by using a balance (if one is available). Record the volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas.

Discard the liquid after measuring and recording the volume or weight, unless analysis of the impinger catch is required (see Note, Section 2.1.7).

If a different type of condenser is used, measure the amount of moisture condensed either volumetrically or gravimetrically.

Whenever possible, containers should be shipped in such a way that they remain upright at all times.

**4.3 Analysis.** Record the data required on a sheet such as the one shown in Figure 5-3. Handle each sample container as follows:

FIGURE 5-3—ANALYTICAL DATA

Plant \_\_\_\_\_  
 Date \_\_\_\_\_  
 Run No. \_\_\_\_\_  
 Filter No. \_\_\_\_\_  
 Amount liquid lost during transport \_\_\_\_\_  
 Acetone blank volume, ml \_\_\_\_\_  
 Acetone wash volume, ml \_\_\_\_\_  
 Acetone blank concentration, mg/mg (Equation 5-4) \_\_\_\_\_  
 Acetone wash blank, mg (Equation 5-5) \_\_\_\_\_

Container number	Weight of particulate collected, mg		
	Final weight	Tare weight	Weight gain
1.			

Container number	Weight of particulate collected, mg		
	Final weight	Tare weight	Weight gain
2.			
Total.			
Less acetone blank.			
Weight of particulate matter.			
	Volume of liquid water collected		
	Impinger volume, ml	Silica gel weight, g	
Final.			
Initial.			
Liquid collected.			
Total volume collected .....		g*	ml

\*Convert weight of water to volume by dividing total weight increase by density of water (1 g/ml).

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**Container No. 1.** Leave the contents in the shipping container or transfer the filter and any loose particulate from the sample container to a tared glass weighing dish. Desiccate for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh to a constant weight and report the results to the nearest 0.1 mg. For purposes of this Section, 4.3, the term "constant weight" means a difference of no more than 0.5 mg or 1 percent of total weight less tare weight, whichever is greater, between two consecutive weighings, with no less than 6 hours of desiccation time between weighings.

Alternatively, the sample may be oven dried at 105 °C (220 °F) for 2 to 3 hours, cooled in the desiccator, and weighed to a constant weight, unless otherwise specified by the Administrator. The tester may also opt to oven dry the sample at 105 °C (220 °F) for 2 to 3 hours, weigh the sample, and use this weight as a final weight.

**Container No. 2.** Note the level of liquid in the container and confirm on the analysis sheet whether or not leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in this container either volumetrically to ±1 ml or gravimetrically to ±0.5 g. Transfer the contents to a tared 250-ml beaker and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a constant

weight. Report the results to the nearest 0.1 mg.

**Container No. 3.** Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance. This step may be conducted in the field.

**"Acetone Blank" Container.** Measure acetone in this container either volumetrically or gravimetrically. Transfer the acetone to a tared 250-ml beaker and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 mg.

**NOTE:** At the option of the tester, the contents of Container No. 2 as well as the acetone blank container may be evaporated at temperatures higher than ambient. If evaporation is done at an elevated temperature, the temperature must be below the boiling point of the solvent; also, to prevent "bumping," the evaporation process must be closely supervised, and the contents of the beaker must be swirled occasionally to maintain an even temperature. Use extreme care, as acetone is highly flammable and has a low flash point.

**4.4 Quality Control Procedures.** The following quality control procedures are suggested to check the volume metering system calibration values at the field test site prior to sample collection. These procedures are optional for the tester.

**4.4.1 Meter Orifice Check.** Using the calibration data obtained during the calibration procedure described in Section 5.3, determine the  $\Delta H@$  for the metering system orifice. The  $\Delta H@$  is the orifice pressure differential in units of in. H<sub>2</sub>O that correlates to 0.75 cfm of air at 528° R and 29.92 in. Hg. The  $\Delta H@$  is calculated as follows:

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Where:

$\Delta H$  = Average pressure differential across the orifice meter, in. H<sub>2</sub>O.

$T_m$  = Absolute average dry gas meter temperature, ° R.

$P_m$  = Barometric pressure, in. Hg.

$\theta$  = Total sampling time, min.

$Y$  = Dry gas meter calibration factor, dimensionless.

$V_m$  = Volume of gas sample as measured by dry gas meter, dcf.

$0.0319 = (0.0367 \text{ in. Hg}^0 \text{ R}) \times (0.75 \text{ cfm})^2$ .

Before beginning the field test (a set of three runs usually constitutes a field test), operate the metering system (i.e., pump, volume meter, and orifice) at the  $\Delta H@$  pressure differential for 10 minutes. Record the volume collected, the dry gas meter temperature, and the barometric pressure. Calculate a dry gas meter calibration check value,  $Y_c$ , as follows:

EN30AU83.032

Eq. 5-10

Where:

$Y_c$  = Dry gas meter calibration check value, dimensionless.

10 = 10 minutes of run time.

Compare the  $Y_c$  value with the dry gas meter calibration factor  $Y$  to determine that:

$0.97Y < Y_c < 1.03Y$

If the  $Y_c$  value is not within this range, the volume metering system should be investigated before beginning the test.

4.4.2 Calibrated Critical Orifice. A calibrated critical orifice, calibrated against a wet test meter or spirometer and designed to be inserted at the inlet of the sampling meter box may be used as a quality control check by following the procedure of Section 7.2.

#### 5. Calibration

Maintain a laboratory log of all calibrations.

5.1 Probe Nozzle. Probe nozzles shall be calibrated before their initial use in the field. Using a micrometer, measure the inside diameter of the nozzle to the nearest 0.025 mm (0.001 in.). Make three separate measurements using different diameters each time, and obtain the average of the measurements. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in.). When nozzles become nicked, dented, or corroded, they shall be reshaped, sharpened, and recalibrated before use. Each nozzle shall be permanently and uniquely identified.

5.2 Pitot Tube. The Type S pitot tube assembly shall be calibrated according to the procedure outlined in Section 4 of Method 2.

#### 5.3 Metering System.

5.3.1 Calibration Prior to Use. Before its initial use in the field, the metering system shall be calibrated as follows: Connect the metering system inlet to the outlet of a wet test meter that is accurate to within 1 percent. Refer to Figure 5.5. The wet test meter should have a capacity of 30 liters/rev (1 ft<sup>3</sup>/rev). A spirometer of 400 liters (14 ft<sup>3</sup>) or more capacity, or equivalent, may be used for this calibration, although a wet test meter is usually more practical. The wet test meter should be periodically calibrated with a spirometer or a liquid displacement meter to ensure the accuracy of the wet test meter. Spirometers or wet test meters of other sizes may be used, provided that the specified accuracies of the procedure are maintained. Run the metering system pump for about 15 minutes with the orifice manometer indicating a median reading as expected in field use to allow the pump to warm up and to permit the interior surface of the wet test meter to be thoroughly wetted. Then, at each of a minimum of three orifice manometer settings, pass an exact quantity of gas through the wet test meter and note the gas volume indicated by the dry gas meter. Also note the barometric pressure, and the temperatures of the wet test meter, the inlet of the dry gas meter, and the outlet of the dry gas meter. Select the highest and lowest orifice settings to bracket the expected field operating range of the orifice. Use a minimum volume of 0.15 m<sup>3</sup> (5 cf) at all orifice settings. Record all the data on a form similar to Figure 5.6, and calculate  $Y$ , the dry gas meter calibration factor, and  $\Delta H_{@}$ , the orifice calibration factor, at each orifice setting as shown on Figure 5.6. Allowable tolerances for individual  $Y$  and  $\Delta H_{@}$  values are given in Figure 5.6. Use the average of the  $Y$  values in the calculations in Section 6.

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Before calibrating the metering system, it is suggested that a leak-check be conducted. For metering systems having diaphragm pumps, the normal leak-check procedure will not detect leakages within the pump. For these cases the following leak-check procedure is suggested: make a 10-minute calibra-

tion run at  $0.00057 \text{ m}^3/\text{min}$  (0.02 cfm); at the end of the run, take the difference of the measured wet test meter and dry gas meter volumes; divide the difference by 10, to get the leak rate. The leak rate should not exceed  $0.00057 \text{ m}^3/\text{min}$  (0.02 cfm).

5.3.2 Calibration After Use. After each field use, the calibration of the metering system shall be checked by performing three calibration runs at a single, intermediate orifice setting (based on the previous field test), with the vacuum set at the maximum value reached during the test series. To adjust the vacuum, insert a valve between the wet test meter and the inlet of the metering system. Calculate the average value of the dry gas meter calibration factor. If the value has changed by more than 5 percent, recalibrate the meter over the full range of orifice settings, as previously detailed.

Alternative procedures, e.g., rechecking the orifice meter coefficient may be used, subject to the approval of the Administrator.

5.3.3 Acceptable Variation in Calibration. If the dry gas meter coefficient values obtained before and after a test series differ by more than 5 percent, the test series shall either be voided, or calculations for the test series shall be performed using whichever meter coefficient value (i.e., before or after) gives the lower value of total sample volume.

5.4 Probe Heater Calibration. The probe heating system shall be calibrated before its initial use in the field.

Use a heat source to generate air heated to selected temperatures that approximate those expected to occur in the sources to be sampled. Pass this air through the probe at a typical sample flow rate while measuring the probe inlet and outlet temperatures at various probe heater settings. For each air temperature generated, construct a graph of probe heating system setting versus probe outlet temperature. The procedure outlined in APTD-0576 can also be used. Probes constructed according to APTD-0581 need not be

calibrated if the calibration curves in APTD-0576 are used. Also, probes with outlet temperature monitoring capabilities do not require calibration.

5.5 Temperature Gauges. Use the procedure in Section 4.3 of Method 2 to calibrate in-stack temperature gauges. Dial thermometers, such as are used for the dry gas meter and condenser outlet, shall be calibrated against mercury-in-glass thermometers.

5.6 Leak Check of Metering System Shown in Figure 5-1. That portion of the sampling train from the pump to the orifice meter should be leak checked prior to initial use and after each shipment. Leakage after the pump will result in less volume being recorded than is actually sampled. The following procedure is suggested (see Figure 5-4): Close the main valve on the meter box. Insert a one-hole rubber stopper with rubber tubing attached into the orifice exhaust pipe. Disconnect and vent the low side of the orifice manometer. Close off the low side orifice tap. Pressurize the system to 13 to 18 cm (5 to 7 in.) water column by blowing into the rubber tubing. Pinch off the tubing and observe the manometer for one minute. A loss of pressure on the manometer indicates a leak in the meter box; leaks, if present, must be corrected.

5.7 Barometer. Calibrate against a mercury barometer.

#### 6. Calculations

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after the final calculation. Other forms of the equations may be used as long as they give equivalent results.

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6.1 Nomenclature.  
 $A_n$ =Cross-sectional area of nozzle,  $m^2(ft^2)$ .  
 $B_{ws}$ =Water vapor in the gas stream, proportion by volume.

$C_a$ =Acetone blank residue concentration,  $mg/mg$ .

$c_p$ =Concentration of particulate matter in stack gas, dry basis, corrected to standard conditions, g/dscm (g/dscf).  
 $I$ =Percent of isokinetic sampling.  
 $L_a$ =Maximum acceptable leakage rate for either a pretest leak check or for a leak check following a component change; equal to 0.00057 m<sup>3</sup>/min (0.02 cfm) or 4 percent of the average sampling rate, whichever is less.  
 $L_p$ =Individual leakage rate observed during the leak check conducted prior to the "i<sup>th</sup>" component change (i=1, 2, 3... n), m<sup>3</sup>/min (cfm).  
 $L_{p'}$ =Leakage rate observed during the post-test leak check, m<sup>3</sup>/min (cfm).  
 $m_a$ =Mass of residue of acetone after evaporation, mg.  
 $m_p$ =Total amount of particulate matter collected, mg.  
 $M_w$ =Molecular weight of water, 18.0 g/g-mole (18.0lb/lb-mole).  
 $P_{bar}$ =Barometric pressure at the sampling site, mm Hg (in. Hg).  
 $P_s$ =Absolute stack gas pressure, mm Hg (in. Hg).  
 $P_{std}$ =Standard absolute pressure, 760 mm Hg (29.92 in. Hg).  
 $R$ =Ideal gas constant, 0.06236 mm Hg-m<sup>3</sup>/K-g-mole (21.85 in. Hg-ft<sup>3</sup>/R-lb-mole).  
 $T_m$ =Absolute average dry gas meter temperature (see Figure 5-2), °K (°R).  
 $T_s$ =Absolute average stack gas temperature (see Figure 5-2), °K (°R).  
 $T_{std}$ =Standard absolute temperature, 293°K (528°R).  
 $V_a$ =Volume of acetone blank, ml.  
 $V_{aw}$ =Volume of acetone used in wash, ml.  
 $V_k$ =Total volume of liquid collected in impingers and silica gel (see Figure 5-3), ml.  
 $V_m$ =Volume of gas sample as measured by dry gas meter, dcm (dscf).  
 $V_{m(std)}$ =Volume of gas sample measured by the dry gas meter, corrected to standard conditions, dscm (dscf).  
 $V_{w(std)}$ =Volume of water vapor in the gas sample, corrected to standard conditions, scm (scf).  
 $v_s$ =Stack gas velocity, calculated by Method 2, Equation 2-9, using data obtained from Method 5, m/sec (ft/sec).  
 $W_a$ =Weight of residue in acetone wash, mg.  
 $Y$ =Dry gas meter calibration factor.  
 $\Delta H$ =Average pressure differential across the orifice meter (see Figure 5-2), mm H<sub>2</sub>O (in. H<sub>2</sub>O).  
 $\phi_a$ =Density of acetone, mg/ml (see label on bottle).  
 $\phi_w$ =Density of water, 0.9982 g/ml (0.002201 lb/ml).  
 $\epsilon$ =Total sampling time, min.  
 $\epsilon_i$ =Sampling time interval, from the beginning of a run until the first component change, min.  
 $\epsilon_{i'}$ =Sampling time interval, between two successive component changes, beginning

with the interval between the first and second changes, min.  
 $\epsilon_{i''}$ =Sampling time interval, from the final (i<sup>th</sup>) component change until the end of the sampling run, min.  
 13.6=Specific gravity of mercury.  
 60=Sec/min.  
 100=Conversion to percent.  
 6.2 Average Dry Gas Meter Temperature and Average Orifice Pressure Drop. See data sheet (Figure 5-2).  
 6.3 Dry Gas Volume. Correct the sample volume measured by the dry gas meter to standard conditions (20 °C, 760 mm Hg or 68 °F, 29.92 in. Hg) by using Equation 5-1.

EC01JN82.105

Where:  
 $K_1=0.3858$  °K/mm Hg for metric units  
 $=17.64$  °R/in. Hg for English units  
 NOTE: Equation 5-1 can be used as written unless the leakage rate observed during any of the mandatory leak checks (i.e., the post-test leak check or leak checks conducted prior to component changes) exceeds  $L_a$ . If  $L_p$  or  $i$  exceeds  $L_a$ , Equation 5-1 must be modified as follows:  
 (a) Case I. No component changes made during sampling run. In this case, replace  $V_m$  in Equation 5-1 with the expression:  
 $[V_m - (L_p - L_a)\theta]$   
 (b) Case II. One or more component changes made during the sampling run. In this case, replace  $V_m$  in Equation 5-1 by the expression:

EC01JN82.106

and substitute only for those leakage rates ( $L_i$  or  $L_p$ ) which exceed  $L_a$ .  
 6.4 Volume of Water Vapor.

EC01JN82.107

Where:  
 $K_2=0.001333$  m<sup>3</sup>/ml for metric units  
 $=0.04707$  ft<sup>3</sup>/ml for English units.  
 8.5 Moisture Content.

EC16NO91.126

NOTE: In saturated or water droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one from the Impinger analysis (Equation 5-3), and a second from the assumption of saturated conditions. The lower of the two values of  $B_{ws}$  shall be considered correct. The procedure for determining the moisture content based upon assumption of saturated conditions is given in the Note of Section 1.2 of Method 4. For the purposes of this method, the average stack gas temperature from Figure 5-2 may be used to make this determination, provided that the accuracy of the in-stack temperature sensor is  $\pm 1^\circ\text{C}$  ( $2^\circ\text{F}$ ).

6.6 Acetone Blank Concentration.

EC16NO91.127

6.7 Acetone Wash Blank.

EC16NO91.129

Where:

$K_3 = 0.003454 \text{ mm Hg} \cdot \text{m}^3/\text{ml} \cdot ^\circ\text{K}$  for metric units.  
 $= 0.002869 \text{ in. Hg} \cdot \text{ft}^3/\text{ml} \cdot ^\circ\text{R}$  for English units.

6.11.2 Calculation From Intermediate Values.

EC01JN92.108

Where:

$K_4 = 4.320$  for metric units  
 $= 0.09450$  for English units.

6.12 Acceptable Results. If 90 percent  $\leq I \leq$  110 percent, the results are acceptable. If the particulate results are low in comparison to the standard, and  $I$  is over 110 percent or less than 90 percent, the Administrator may accept the results. Citation 4 in the bibliography section can be used to make acceptability judgments. If  $I$  is judged to be unacceptable, reject the particulate results and repeat the test.

6.13 Stack Gas Velocity and Volumetric Flow Rate. Calculate the average stack gas

EC16NO91.128

6.8 Total Particulate Weight. Determine the total particulate catch from the sum of the weights obtained from Containers 1 and 2 less the acetone blank (see Figure 5-3).

NOTE: Refer to Section 4.1.5 to assist in calculation of results involving two or more filter assemblies or two or more sampling trains.

6.9 Particulate Concentration.

$$C_p = (0.001 \text{ g/mg}) (m_p / V_m (\text{std}))$$

Eq. 5-8

6.10 Conversion Factors:

From	To	Multiply by
scf	m <sup>3</sup>	0.02832
g	mg	0.001
g/m <sup>3</sup>	gr/ft <sup>3</sup>	15.43
g/m <sup>3</sup>	lb/ft <sup>3</sup>	$2.205 \times 10^{-3}$
g/m <sup>3</sup>	g/m <sup>3</sup>	35.31

6.11 Isokinetic Variation.

6.11.1 Calculation From Raw Data.

velocity and volumetric flow rate, if needed, using data obtained in this method and the equations in Sections 5.2 and 5.3 of Method 2.

7. Alternative Procedures

7.1 Dry Gas Meter as a Calibration Standard. A dry gas meter may be used as a calibration standard for volume measurements in place of the wet test meter specified in Section 5.3, provided that it is calibrated initially and recalibrated periodically as follows:

7.1.1 Standard Dry Gas Meter Calibration.

7.1.1.1 The dry gas meter to be calibrated and used as a secondary reference meter should be of high quality and have an appropriately sized capacity, e.g., 3 liters/rev (0.1 ft<sup>3</sup>/rev). A spirometer (400 liters or more capacity), or equivalent, may be used for this calibration, although a wet test meter is usually more practical. The wet test meter should have a capacity of 30 liters/rev (1 ft<sup>3</sup>/rev) and capable of measuring volume to within  $\pm 1.0$  percent; wet test meters should be checked against a spirometer or a liquid displacement meter to ensure the accuracy of the wet test meter. Spirometers or wet test meters of other sizes may be used, provided that the specified accuracies of the procedure are maintained.

7.1.1.2 Set up the components as shown in Figure 5.7. A spirometer, or equivalent, may

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be used in place of the wet test meter in the system. Run the pump for at least 5 minutes at a flow rate of about 10 liters/min (0.35 cfm) to condition the interior surface of the wet test meter. The pressure drop indicated by the manometer at the inlet side of the dry

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gas meter should be minimized [no greater than 100 mm H<sub>2</sub>O (4 in. H<sub>2</sub>O) at a flow rate of 30 liters/min (1.0 cfm)]. This can be accomplished by using large diameter tubing connections and straight pipe fittings.

EC01.JN92.109

7.1.1.3 Collect the data as shown in the example data sheet (see Figure 5-8). Make triplicate runs at each of the flow rates and at no less than five different flow rates. The

range of flow rates should be between 10 and 34 liters/min (0.35 and 1.2 cfm) or over the expected operating range.

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7.1.1.4 Calculate flow rate,  $Q$ , for each run using the wet test meter gas volume,  $V_w$ , and the run time. 8. Calculate the dry gas meter coefficient,  $Y_{ds}$ , for each run. These calculations are as follows:

EC16N091.130

Where:

$K_1=0.3858$  for International system of units (SI); 17.64 for English units.

$V_w$ =Wet test meter volume, liters (ft<sup>3</sup>).

$V_{ds}$ =Dry gas meter volume, liters (ft<sup>3</sup>).

$t_{ds}$ =Average dry gas meter temperature, °C (°F).

$t_{std}$ =273° C for SI units; 460° F for English units.

$t_w$ =Average wet test meter temperature, °C (°F).

$P_{bar}$ =Barometric pressure, mm Hg (in. Hg).

$\Delta p$ =Dry gas meter inlet differential pressure, mm H<sub>2</sub>O (in. H<sub>2</sub>O).

$\theta$ =Run time, min.

7.1.1.5 Compare the three  $Y_{ds}$  values at each of the flow rates and determine the maximum and minimum values. The difference between the maximum and minimum values at each flow rate should be no greater than 0.030. Extra sets of triplicate runs may be made in order to complete this requirement. In addition, the meter coefficients should be between 0.95 and 1.05. If these specifications cannot be met in three sets of successive triplicate runs, the meter is not suitable as a calibration standard and should not be used as such. If these specifications are met, average the three  $Y_{ds}$  values at each flow rate resulting in five average meter coefficients,  $Y_{ds}$ .

7.1.1.6 Prepare a curve of meter coefficient,  $Y_{ds}$ , versus flow rate,  $Q$ , for the dry gas meter. This curve shall be used as a reference when the meter is used to calibrate other dry gas meters and to determine whether recalibration is required.

7.1.2 Standard Dry Gas Meter Recalibration.

7.1.2.1 Recalibrate the standard dry gas meter against a wet test meter or spirometer annually or after every 200 hours of operation, whichever comes first. This requirement is valid provided the standard dry gas meter is kept in a laboratory and, if transported, cared for as any other laboratory instrument. Abuse to the standard meter may cause a change in the calibration and will require more frequent recalibrations.

7.1.2.2 As an alternative to full recalibration, a two-point calibration check may be made. Follow the same procedure and equipment arrangement as for a full recalibration, but run the meter at only two flow rates [suggested rates are 14 and 28 liters/min (0.5 and 1.0 cfm)]. Calculate the meter coefficients for these two points, and compare the values with the meter calibration curve. If the two coefficients are within  $\pm 1.5$  percent of the calibration curve values at the same flow rates, the meter need not be recalibrated until the next date for a recalibration check.

7.2 Critical Orifices As Calibration Standards. Critical orifices may be used as calibration standards in place of the wet test meter specified in Section 5.3, provided that they are selected, calibrated, and used as follows:

7.2.1 Section of Critical Orifices.

7.2.1.1 The procedure that follows describes the use of hypodermic needles or stainless steel needle tubings which have been found suitable for use as critical orifices. Other materials and critical orifice designs may be used provided the orifices act as true critical orifices; i.e., a critical vacuum can be obtained, as described in Section 7.2.2.3. Select five critical orifices that are appropriately sized to cover the range of flow rates between 10 and 34 liters/min or the expected operating range. Two of the critical orifices should bracket the expected operating range.

A minimum of three critical orifices will be needed to calibrate a Method 5 dry gas meter (DGM); the other two critical orifices can serve as spares and provide better selection for bracketing the range of operating flow rates. The needle sizes and tubing lengths shown below give the following approximate flow rates:

Gauge/cm	Flow rate (liters/min)	Gauge/cm	Flow rate (liters/min)
12/7.6	32.66	14/2.5	19.94
12/10.2	30.02	14/3.1	17.27
13/2.5	26.77	14/7.6	16.14
13/5.1	23.50	15/3.2	14.16
13/7.6	22.37	15/7.6	11.61
13/10.2	20.67	15/10.2	10.48

7.2.1.2 These needles can be adapted to a Method 5 type sampling train as follows: Insert a serum bottle stopper, 13- by 20-mm sleeve type, into a 1/8-inch Swagelok quick connect. Insert the needle into the stopper as shown in Figure 5-9.

EC01JN92.111

**7.2.2 Critical Orifice Calibration.** The procedure described in this section uses the Method 5 meter box configuration with a DGM as described in Section 2.1.8 to calibrate the critical orifices. Other schemes may be used, subject to the approval of the Administrator.

**7.2.2.1 Calibration of Meter Box.** The critical orifices must be calibrated in the same configuration as they will be used; i.e., there should be no connections to the inlet of the orifice.

**7.2.2.1.1** Before calibrating the meter box, leak check the system as follows: Fully open the coarse adjust valve, and completely close the by-pass valve. Plug the inlet. Then turn on the pump, and determine whether there is any leakage. The leakage rate shall be zero;

i.e., no detectable movement of the DGM dial shall be seen for 1 minute.

**7.2.2.1.2** Check also for leakages in that portion of the sampling train between the pump and the orifice meter. See Section 5.8 for the procedure; make any corrections, if necessary. If leakage is detected, check for cracked gaskets, loose fittings, worn O-rings, etc., and make the necessary repairs.

**7.2.2.1.3** After determining that the meter box is leakless, calibrate the meter box according to the procedure given in Section 5.3. Make sure that the wet test meter meets the requirements stated in Section 7.1.1.1. Check the water level in the wet test meter. Record the DGM calibration factor, Y.

**7.2.2.2 Calibration of Critical Orifices.** Set up the apparatus as shown in Figure 5-10.

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7.2.2.2.1 Allow a warm-up time of 15 minutes. This step is important to equilibrate the temperature conditions through the DGM.

7.2.2.2.2 Leak check the system as in Section 7.2.2.1.1. The leakage rate shall be zero.

7.2.2.2.3 Before calibrating the critical orifice, determine its suitability and the appropriate operating vacuum as follows: Turn on the pump, fully open the coarse adjust valve, and adjust the by-pass valve to give a vacuum reading corresponding to about half of atmospheric pressure. Observe the meter box orifice manometer reading, H. Slowly increase the vacuum reading until a stable reading is obtained on the meter box orifice manometer. Record the critical vacuum for each orifice.

Orifices that do not reach a critical value shall not be used.

7.2.2.2.4 Obtain the barometric pressure using a barometer as described in Section 2.1.9. Record the barometric pressure,  $P_{bar}$ , in mm Hg (In. Hg).

7.2.2.2.5 Conduct duplicate runs at a vacuum of 25 to 50 mm Hg (1 to 2 in. Hg) above the critical vacuum. The runs shall be at least 5 minutes each. The DGM volume readings shall be in increments of 0.00283 m<sup>3</sup> (0.1 ft<sup>3</sup>) or in increments of complete revolutions of the DGM. As a guideline, the times should not differ by more than 3.0 seconds (this includes allowance for changes in the DGM temperatures) to achieve  $\pm 0.5$  percent in K'. Record the information listed in Figure 5-11.

7.2.2.2.6 Calculate K' using Equation 5-9.

EC16NO91.131

Where:

EC16NO91.132

$T_{amb}$  = Absolute ambient temperature, °K (°R).  
Average the K' values. The individual K' values should not differ by more than ±0.5 percent from the average.

7.2.3 Using the Critical Orifices as Calibration Standards.

7.2.3.1 Record the barometric pressure.  
Date \_\_\_\_\_ Train ID \_\_\_\_\_ DGM cal. factor \_\_\_\_\_  
Critical orifice ID \_\_\_\_\_

Dry gas meter		Run number	
		1	2
Final reading .....	m <sup>3</sup> (ft <sup>3</sup> ) .....	.....	.....
Initial reading .....	m <sup>3</sup> (ft <sup>3</sup> ) .....	.....	.....
Difference, $V_{in}$ .....	m <sup>3</sup> (ft <sup>3</sup> ) .....	.....	.....
Inlet/Outlet temperatures:			
Initial .....	°C (°F) .....	/	/
Final .....	°C (°F) .....	/	/
Avg. Temperature, $t_c$ .....	°C (°F) .....	.....	.....
Time, $\Theta$ .....	min/sec .....	/	/

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where:

$V_{cor(Std)}$  = Volume of gas sample passed through the critical orifice, corrected to standard conditions, dsm<sup>3</sup> (dscf).

$K_1$  = 0.3858 °K/mm Hg for metric units  
= 17.64 °R/in. Hg for English units.

7.2.3.4 Average the DGM calibration values for each of the flow rates. The calibration

Dry gas meter		Run number	
		1	2
Orifice man. rdg., $\Delta H$ .....	mm (in.) H <sub>2</sub> O .....	.....	.....
Bar. pressure, $P_{bar}$ .....	mm (in.) Hg .....	.....	.....
Ambient temperature, $t_{amb}$ .....	°C (°F) .....	.....	.....
Pump vacuum .....	mm (in.) Hg .....	.....	.....
K' factor .....	.....	.....	.....
Average .....	.....	.....	.....

Figure 5-11. Data sheet for determining K' factor.

7.2.3.2 Calibrate the metering system according to the procedure outlined in Sections 7.2.2.2.1 to 7.2.2.2.5. Record the information listed in Figure 5.12.

7.2.3.3 Calculate the standard volumes of air passed through the DGM and the critical orifices, and calculate the DGM calibration factor, Y, using the equations below:

tion factor, Y, at each of the flow rates should not differ by more than ±2 percent from the average.

7.2.3.5 To determine the need for recalibrating the critical orifices, compare the DGM Y factors obtained from two adjacent orifices each time a DGM is calibrated; for example, when checking 13/2.5, use orifices 12/10.2 and 13/5.1. If any critical orifice yields a DGM Y factor differing by more than 2 percent from the others, recalibrate the critical orifice according to Section 7.2.2.2.

Date \_\_\_\_\_ Train ID \_\_\_\_\_ Critical orifice ID \_\_\_\_\_ Critical orifice K' factor \_\_\_\_\_

Dry gas meter		Run number	
		1	2
Final reading	ml (l)		
Initial reading	ml (l)		
Difference, $V_{(2)}$	m <sup>3</sup> (ft <sup>3</sup> )		
Inlet/outlet temperatures:			
Initial	°C (°F)	/	/
Final	°C (°F)	/	/
Avg. Temperature, $t_m$	°C (°F)		
Time, $\Theta$	min/sec	/	/
Orifice man. rdg., $\Delta H$	mm (in.)		
Bar. pressure, $P_{bar}$	mm (in.) Hg		
Ambient temperature, $t_{amb}$	°C (°F)		
Pump vacuum	mm (in.) Hg		
$V_{(2)(20)}$	m <sup>3</sup> (ft <sup>3</sup> )		
$V_{(2)(20)}$	m <sup>3</sup> (ft <sup>3</sup> )		
DGM cal. factor, Y			

Figure 5-12. Data sheet for determining DGM Y factor.

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METHOD 5A—DETERMINATION OF PARTICULATE EMISSIONS FROM THE ASPHALT PROCESSING AND ASPHALT ROOFING INDUSTRY

1. Applicability and Principle

1.1 Applicability. This method applies to the determination of particulate emissions from asphalt roofing industry process saturators, blowing stills, and other sources as specified in the regulations.

1.2 Principle. Particulate matter is withdrawn isokinetically from the source and collected on a glass filter fiber maintained at a temperature of 42 ±10 °C (108 ±18 °F). The particulate mass, which includes any material that condenses at or above the filtration temperature, is determined gravimetrically after removal of uncombined water.

2. Apparatus

2.1 Sampling Train. The sampling train configuration is the same as shown in Figure 5-1 of Method 5. The sampling train consists of the following components:

2.1.1 Probe Nozzle, Pitot Tube, Differential Pressure Gauge, Filter Holder, Condenser, Metering System, Barometer, and Gas Density Determination Equipment. Same as Method 5, Sections 2.1.1, 2.1.3 to 2.1.5, and 2.1.7 to 2.1.10, respectively.

2.1.2 Probe Liner. Same as in Method 5, Section 2.1.2, with the note that at high stack gas temperatures (greater than 230 °C (480 °F)), water-cooled probes may be required to control the probe exit temperature to 42 ±10 °C (108 ±18 °F).

2.1.3 Precollector Cyclone. Borosilicate glass following the construction details shown in Air Pollution Technical Document-0581, "Construction Details of Isokinetic Source-Sampling Equipment".

NOTE: The tester shall use the cyclone when the stack gas moisture is greater than 10 percent. The tester shall not use the precollector cyclone under other, less severe conditions.

2.1.4 Filter Heating System. Any heating (or cooling) system capable of maintaining a sample gas temperature at the exit end of the filter holder during sampling at 42 ±10° C (108 ±18° F). Install a temperature gauge capable of measuring temperature within 3° C (5.4° F) at the exit side of the filter holder so that the sensing tip of the temperature

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EC16NO91.248

Where:

$K_1=0.003484$  mm Hg-m<sup>3</sup>/ml-°K for metric units.

$=0.002876$  in. Hg-ft<sup>3</sup>/ml-°R for English units.

6.7.2 Calculation from Intermediate Values.

EC01JN82.153

where:

$K_2=4.320$  for metric units.

$=0.09450$  for English units

6.8 Acceptable Results. If 90 percent < I < 110 percent, the results are acceptable. If the results are low in comparison to the standards and I is beyond the acceptable range, the Administrator may opt to accept the results. Use Citation 4 in the Bibliography of Method 5 to make judgments. Otherwise, reject the results and repeat the test.

6.9 Stack Gas Velocity and Volumetric Flow Rate. Calculate the average stack gas velocity and volumetric flow rate, if needed, using data obtained in this method and equations in Sections 5.2 and 5.3 of Method 2.

6.10 Relative Error (RE) for QA Audit Samples. Same as in Method 6, Section 6.4.

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7. Annual Book of ASTM Standards. Part 31; Water, Atmospheric Analysis. pp. 40-42. American Society for Testing and Materials. Philadelphia, Pa. 1974.

#### METHOD 9—VISUAL DETERMINATION OF THE OPACITY OF EMISSIONS FROM STATIONARY SOURCES

Many stationary sources discharge visible emissions into the atmosphere; these emissions are usually in the shape of a plume. This method involves the determination of plume opacity by qualified observers. The method includes procedures for the training and certification of observers, and procedures to be used in the field for determination of plume opacity. The appearance of a plume as viewed by an observer depends upon a number of variables, some of which may be controllable and some of which may not be controllable in the field. Variables which can be controlled to an extent to which they no longer exert a significant influence upon plume appearance include: Angle of the observer with respect to the plume; angle of the observer with respect to the sun; point of observation of attached and detached steam plume; and angle of the observer with respect to a plume emitted from a rectangular stack with a large length to width ratio. The method includes specific criteria applicable to these variables.

Other variables which may not be controllable in the field are luminescence and color contrast between the plume and the background against which the plume is viewed. These variables exert an influence upon the appearance of a plume as viewed by an observer, and can affect the ability of the observer to accurately assign opacity values to the observed plume. Studies of the theory of plume opacity and field studies have demonstrated that a plume is most visible and presents the greatest apparent opacity when viewed against a contrasting background. It follows from this, and is confirmed by field trials, that the opacity of a plume, viewed under conditions where a contrasting background is present can be assigned with the greatest degree of accuracy. However, the potential for a positive error is also the greatest when a plume is viewed under such contrasting conditions. Under conditions presenting a less contrasting background, the apparent opacity of a plume is less and

approaches zero as the color and luminescence contrast decrease toward zero. As a result, significant negative bias and negative errors can be made when a plume is viewed under less contrasting conditions. A negative bias decreases rather than increases the possibility that a plant operator will be cited for a violation of opacity standards due to observer error.

Studies have been undertaken to determine the magnitude of positive errors which can be made by qualified observers while reading plumes under contrasting conditions and using the procedures set forth in this method. The results of these studies (field trials) which involve a total of 769 sets of 25 readings each are as follows:

(1) For black plumes (133 sets at a smoke generator), 100 percent of the sets were read with a positive error<sup>1</sup> of less than 7.5 percent opacity; 99 percent were read with a positive error of less than 5 percent opacity.

(2) For white plumes (170 sets at a smoke generator, 168 sets at a coal-fired power plant, 299 sets at a sulfuric acid plant), 99 percent of the sets were read with a positive error of less than 7.5 percent opacity; 85 percent were read with a positive error of less than 5 percent opacity.

The positive observational error associated with an average of twenty-five readings is therefore established. The accuracy of the method must be taken into account when determining possible violations of applicable opacity standards.

#### 1. Principle and Applicability

1.1 Principle. The opacity of emissions from stationary sources is determined visually by a qualified observer.

1.2 Applicability. This method is applicable for the determination of the opacity of emissions from stationary sources pursuant to §60.11(b) and for qualifying observers for visually determining opacity of emissions.

#### 2. Procedures

The observer qualified in accordance with section 3 of this method shall use the following procedures for visually determining the opacity of emissions:

2.1 Position. The qualified observer shall stand at a distance sufficient to provide a clear view of the emissions with the sun oriented in the 140° sector to his back. Consistent with maintaining the above requirement, the observer shall, as much as possible, make his observations from a position such that his line of vision is approximately perpendicular to the plume direction, and when observing opacity of emissions from rectangular outlets (e.g., roof monitors, open

baghouses, noncircular stacks), approximately perpendicular to the longer axis of the outlet. The observer's line of sight should not include more than one plume at a time when multiple stacks are involved, and in any case the observer should make his observations with his line of sight perpendicular to the longer axis of such a set of multiple stacks (e.g., stub stacks on baghouses).

2.2 Field Records. The observer shall record the name of the plant, emission location, type facility, observer's name and affiliation, a sketch of the observer's position relative to the source, and the date on a field data sheet (Figures 9-1). The time, estimated distance to the emission location, approximate wind direction, estimated wind speed, description of the sky condition (presence and color of clouds), and plume background are recorded on a field data sheet at the time opacity readings are initiated and completed.

2.3 Observations. Opacity observations shall be made at the point of greatest opacity in that portion of the plume where condensed water vapor is not present. The observer shall not look continuously at the plume, but instead shall observe the plume momentarily at 15-second intervals.

2.3.1 Attached Steam Plumes. When condensed water vapor is present within the plume as it emerges from the emission outlet, opacity observations shall be made beyond the point in the plume at which condensed water vapor is no longer visible. The observer shall record the approximate distance from the emission outlet to the point in the plume at which the observations are made.

2.3.2 Detached Steam Plume. When water vapor in the plume condenses and becomes visible at a distinct distance from the emission outlet, the opacity of emissions should be evaluated at the emission outlet prior to the condensation of water vapor and the formation of the steam plume.

2.4 Recording Observations. Opacity observations shall be recorded to the nearest 5 percent at 15-second intervals on an observational record sheet. (See Figure 9-2 for an example.) A minimum of 24 observations shall be recorded. Each momentary observation recorded shall be deemed to represent the average opacity of emissions for a 15-second period.

2.5 Data Reduction. Opacity shall be determined as an average of 24 consecutive observations recorded at 15-second intervals. Divide the observations recorded on the record sheet into sets of 24 consecutive observations. A set is composed of any 24 consecutive observations. Sets need not be consecutive in time and in no case shall two sets overlap. For each set of 24 observations, calculate the average by summing the opacity of the 24 observations and dividing this sum

<sup>1</sup> For a set, positive error = average opacity determined by observers' 25 observations—average opacity determined from transmissometer's 25 recordings.

by 24. If an applicable standard specifies an averaging time requiring more than 24 observations, calculate the average for all observations made during the specified time period. Record the average opacity on a record sheet. (See Figure 9-1 for an example.)

### 3. Qualifications and Testing

3.1 Certification Requirements. To receive certification as a qualified observer, a candidate must be tested and demonstrate the ability to assign opacity readings in 5 percent increments to 25 different black plumes and 25 different white plumes, with an error not to exceed 15 percent opacity on any one reading and an average error not to exceed 7.5 percent opacity in each category. Candidates shall be tested according to the procedures described in section 3.2. Smoke generators used pursuant to section 3.2 shall be equipped with a smoke meter which meets the requirements of section 3.3.

The certification shall be valid for a period of 6 months, at which time the qualification procedure must be repeated by any observer in order to retain certification.

3.2 Certification Procedure. The certification test consists of showing the candidate a complete run of 50 plumes—25 black plumes and 25 white plumes—generated by a smoke generator. Plumes within each set of 25 black and 25 white runs shall be presented in random order. The candidate assigns an opacity value to each plume and records his observation on a suitable form. At the completion of each run of 50 readings, the score of the candidate is determined. If a candidate fails to qualify, the complete run of 50 readings must be repeated in any retest. The smoke test may be administered as part of a smoke school or training program, and may be preceded by training or familiarization runs of the smoke generator during which candidates are shown black and white plumes of known opacity.

3.3 Smoke Generator Specifications. Any smoke generator used for the purposes of section 3.2 shall be equipped with a smoke meter installed to measure opacity across the diameter of the smoke generator stack. The smoke meter output shall display in-stack opacity based upon a pathlength equal to the stack exit diameter, on a full 0 to 100 percent chart recorder scale. The smoke meter optical design and performance shall meet the specifications shown in Table 9-1. The smoke meter shall be calibrated as prescribed in section 3.3.1 prior to the conduct of each smoke reading test. At the com-

pletion of each test, the zero and span drift shall be checked and if the drift exceeds  $\pm 1$  percent opacity, the condition shall be corrected prior to conducting any subsequent test runs. The smoke meter shall be demonstrated, at the time of installation, to meet the specifications listed in Table 9-1. This demonstration shall be repeated following any subsequent repair or replacement of the photocell or associated electronic circuitry including the chart recorder or output meter, or every 6 months, whichever occurs first.

TABLE 9-1—SMOKE METER DESIGN AND PERFORMANCE SPECIFICATIONS

Parameter	Specification
a. Light source .....	Incandescent lamp operated at nominal rated voltage.
b. Spectral response of photocell .....	Photopic (daylight spectral response of the human eye—Citation 3).
c. Angle of view .....	19° maximum total angle.
d. Angle of projection .....	18° maximum total angle.
e. Calibration error .....	$\pm 3\%$ opacity, maximum.
f. Zero and span drift .....	$\pm 1\%$ opacity, 30 minutes
g. Response time .....	5 seconds.

3.3.1 Calibration. The smoke meter is calibrated after allowing a minimum of 30 minutes warmup by alternately producing simulated opacity of 0 percent and 100 percent. When stable response at 0 percent or 100 percent is noted, the smoke meter is adjusted to produce an output of 0 percent or 100 percent, as appropriate. This calibration shall be repeated until stable 0 percent and 100 percent readings are produced without adjustment. Simulated 0 percent and 100 percent opacity values may be produced by alternately switching the power to the light source on and off while the smoke generator is not producing smoke.

3.3.2 Smoke Meter Evaluation. The smoke meter design and performance are to be evaluated as follows:

3.3.2.1 Light Source. Verify from manufacturer's data and from voltage measurements made at the lamp, as installed, that the lamp is operated within  $\pm 5$  percent of the nominal rated voltage.

3.3.2.2 Spectral Response of Photocell. Verify from manufacturer's data that the photocell has a photopic response; i.e., the spectral sensitivity of the cell shall closely approximate the standard spectral-luminosity curve for photopic vision which is referenced in (b) of Table 9-1.

EC01.JN92.164

FIGURE 9-2—OBSERVATION RECORD

Page \_\_\_\_ of \_\_\_\_

Company .....	Observer .....
Location .....	Type facility .....
Test Number .....	Point of emissions .....
Date .....	

Hr.	Min.	Seconds				Steam plume (check if applicable)		Comments
		0	15	30	45	Attached	Detached	
	0							
	1							
	2							
	3							
	4							
	5							
	6							
	7							
	8							
	9							
	10							
	11							
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	22							
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	29							

FIGURE 9-2—OBSERVATION RECORD—(CONTINUED)

Page \_\_\_\_ of \_\_\_\_

Company .....  
 Location .....  
 Test Number .....  
 Date .....

Observer .....  
 Type facility .....  
 Point of emissions .....

Hr.	Min.	Seconds				Steam plume (check if applicable)		Comments
		0	15	30	45	Attached	Detached	
	30							

Hr.	Min.	Seconds				Steam plume (check if applicable)		Comments
		0	15	30	45	Attached	Detached	
	31							
	32							
	33							
	34							
	35							
	36							
	37							
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3.3.2.3 Angle of View. Check construction geometry to ensure that the total angle of view of the smoke plume, as seen by the photocell, does not exceed 15°. The total angle of view may be calculated from:  $\theta = 2 \tan^{-1} d/2L$ , where  $\theta$ =total angle of view;  $d$ =the sum of the photocell diameter+the diameter of the limiting aperture; and  $L$ =the distance from the photocell to the limiting aperture. The limiting aperture is the point in the path between the photocell and the smoke plume

where the angle of view is most restricted. In smoke generator smoke meters this is normally an orifice plate.

3.3.2.4 Angle of Projection. Check construction geometry to ensure that the total angle of projection of the lamp on the smoke plume does not exceed 15°. The total angle of projection may be calculated from:  $\theta = 2 \tan^{-1} d/2L$ , where  $\theta$ = total angle of projection;  $d$ = the sum of the length of the lamp

filament + the diameter of the limiting aperture; and  $L$  = the distance from the lamp to the limiting aperture.

3.3.2.5 Calibration Error. Using neutral-density filters of known opacity, check the error between the actual response and the theoretical linear response of the smoke meter. This check is accomplished by first calibrating the smoke meter according to 3.3.1 and then inserting a series of three neutral-density filters of nominal opacity of 20, 50, and 75 percent in the smoke meter pathlength. Filters calibrated within  $\pm 2$  percent shall be used. Care should be taken when inserting the filters to prevent stray light from affecting the meter. Make a total of five nonconsecutive readings for each filter. The maximum error on any one reading shall be 3 percent opacity.

3.3.2.6 Zero and Span Drift. Determine the zero and span drift by calibrating and operating the smoke generator in a normal manner over a 1-hour period. The drift is measured by checking the zero and span at the end of this period.

3.3.2.7 Response Time. Determine the response time by producing the series of five simulated 0 percent and 100 percent opacity values and observing the time required to reach stable response. Opacity values of 0 percent and 100 percent may be simulated by alternately switching the power to the light source off and on while the smoke generator is not operating.

#### 4. Bibliography.

1. Air Pollution Control District Rules and Regulations, Los Angeles County Air Pollution Control District, Regulation IV, Prohibitions, Rule 50.

2. Weisburd, Melvin I., Field Operations and Enforcement Manual for Air, U.S. Environmental Protection Agency, Research Triangle Park, NC. APTD-1100, August 1972, pp. 4.1-4.36.

3. Condon, E.U., and Odishaw, H., Handbook of Physics, McGraw-Hill Co., New York, NY, 1958, Table 3.1, p. 6-52.

#### ALTERNATE METHOD 1—DETERMINATION OF THE OPACITY OF EMISSIONS FROM STATIONARY SOURCES REMOTELY BY LIDAR

This alternate method provides the quantitative determination of the opacity of an emissions plume remotely by a mobile lidar system (laser radar; Light Detection and Ranging). The method includes procedures for the calibration of the lidar and procedures to be used in the field for the lidar determination of plume opacity. The lidar is used to measure plume opacity during either day or nighttime hours because it contains its own pulsed light source or transmitter. The operation of the lidar is not dependent upon ambient lighting conditions (light, dark, sunny or cloudy).

The lidar mechanism or technique is applicable to measuring plume opacity at numerous wavelengths of laser radiation. However, the performance evaluation and calibration test results given in support of this method apply only to a lidar that employs a ruby (red light) laser [Reference 5.1].

#### 1. Principle and Applicability

1.1 Principle. The opacity of visible emissions from stationary sources (stacks, roof vents, etc.) is measured remotely by a mobile lidar (laser radar).

1.2 Applicability. This method is applicable for the remote measurement of the opacity of visible emissions from stationary sources during both nighttime and daylight conditions, pursuant to 40 CFR §60.11(b). It is also applicable for the calibration and performance verification of the mobile lidar for the measurement of the opacity of emissions. A performance/design specification for a basic lidar system is also incorporated into this method.

#### 1.3 Definitions.

**Azimuth angle:** The angle in the horizontal plane that designates where the laser beam is pointed. It is measured from an arbitrary fixed reference line in that plane.

**Backscatter:** The scattering of laser light in a direction opposite to that of the incident laser beam due to reflection from particulates along the beam's atmospheric path which may include a smoke plume.

**Backscatter signal:** The general term for the lidar return signal which results from laser light being backscattered by atmospheric and smoke plume particulates.

**Convergence distance:** The distance from the lidar to the point of overlap of the lidar receiver's field-of-view and the laser beam.

**Elevation angle:** The angle of inclination of the laser beam referenced to the horizontal plane.

**Far region:** The region of the atmosphere's path along the lidar line-of-sight beyond or behind the plume being measured.

**Lidar:** Acronym for Light Detection and Ranging.

**Lidar range:** The range or distance from the lidar to a point of interest along the lidar line-of-sight.

**Near region:** The region of the atmospheric path along the lidar line-of-sight between the lidar's convergence distance and the plume being measured.

**Opacity:** One minus the optical transmittance of a smoke plume, screen target, etc.

**Pick interval:** The time or range intervals in the lidar backscatter signal whose minimum average amplitude is used to calculate opacity. Two pick intervals are required, one in the near region and one in the far region.

**Plume:** The plume being measured by lidar.

**Plume signal:** The backscatter signal resulting from the laser light pulse passing through a plume.

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runs and the results of each should be recorded. The requirements of Section 3.3.1 must be fulfilled for each of the three runs.

Once the conditions of the annual calibration are fulfilled the lidar shall be subjected to the routine verification for three separate complete runs. The requirements of Section 3.3.2 must be fulfilled for each of the three runs and the results should be recorded. The Administrator may request that the results of the performance evaluation be submitted for review.

#### 5. References

5.1 The Use of Lidar for Emissions Source Opacity Determination, U.S. Environmental Protection Agency, National Enforcement Investigations Center, Denver, CO. EPA-330/1-79-003-R, Arthur W. Dybdahl, current edition [NTIS No. PB81-246662].

5.2 Field Evaluation of Mobile Lidar for the Measurement of Smoke Plume Opacity, U.S. Environmental Protection Agency, National Enforcement Investigations Center, Denver, CO. EPA/NEIC-TS-128, February 1978.

5.3 Remote Measurement of Smoke Plume Transmittance Using Lidar, C. S. Cook, G. W. Bethke, W. D. Conner (EPA/RTP). Applied Optics 11, pg 1742, August 1972.

5.4 Lidar Studies of Stack Plumes in Rural and Urban Environments, EPA-650/4-73-002, October 1973.

5.5 American National Standard for the Safe Use of Lasers ANSI Z 136.1-176, March 8, 1978.

5.6 U.S. Army Technical Manual TB MED 279, Control of Hazards to Health from Laser Radiation, February 1969.

5.7 Laser Institute of America Laser Safety Manual, 4th Edition.

5.8 U.S. Department of Health, Education and Welfare, Regulations for the Administration and Enforcement of the Radiation Control for Health and Safety Act of 1968, January 1976.

5.9 Laser Safety Handbook, Alex Mallow, Leon Chabot, Van Nostrand Reinhold Co., 1978.

#### METHOD 10—DETERMINATION OF CARBON MONOXIDE EMISSIONS FROM STATIONARY SOURCES

##### 1. Principle and Applicability

1.1 Principle. An integrated or continuous gas sample is extracted from a sampling point and analyzed for carbon monoxide (CO) content using a Luft-type nondispersive infrared analyzer (NDIR) or equivalent.

1.2 Applicability. This method is applicable for the determination of carbon monoxide emissions from stationary sources only when specified by the test procedures for determining compliance with new source performance standards. The test procedure will indicate whether a continuous or an integrated sample is to be used.

##### 2. Range and Sensitivity

2.1 Range. 0 to 1,000 ppm.

2.2 Sensitivity. Minimum detectable concentration is 20 ppm for a 0 to 1,000 ppm span.

##### 3. Interferences

Any substance having a strong absorption of infrared energy will interfere to some extent. For example, discrimination ratios for water (H<sub>2</sub>O) and carbon dioxide (CO<sub>2</sub>) are 3.5 percent H<sub>2</sub>O per 7 ppm CO and 10 percent CO<sub>2</sub> per 10 ppm CO, respectively, for devices measuring in the 1,500 to 3,000 ppm range. For devices measuring in the 0 to 100 ppm range, interference ratios can be as high as 3.5 percent H<sub>2</sub>O per 25 ppm CO and 10 percent CO<sub>2</sub> per 50 ppm CO. The use of silica gel and ascarite traps will alleviate the major interference problems. The measured gas volume must be corrected if these traps are used.

##### 4. Precision and Accuracy

4.1 Precision. The precision of most NDIR analyzers is approximately  $\pm 2$  percent of span.

4.2 Accuracy. The accuracy of most NDIR analyzers is approximately  $\pm 5$  percent of span after calibration.

##### 5. Apparatus

5.1 Continuous Sample (Figure 10-1).

5.1.1 Probe. Stainless steel or sheathed Pyrex<sup>1</sup> glass, equipped with a filter to remove particulate matter.

5.1.2 Air-Cooled Condenser or Equivalent. To remove any excess moisture.

5.2 Integrated Sample (Figure 10-2).

5.2.1 Probe. Stainless steel or sheathed Pyrex glass, equipped with a filter to remove particulate matter.

5.2.2 Air-Cooled Condenser or Equivalent. To remove any excess moisture.

5.2.3 Valve. Needle valve, or equivalent, to adjust flow rate.

5.2.4 Pump. Leak-free diaphragm type, or equivalent, to transport gas.

5.2.5 Rate Meter. Rotameter, or equivalent, to measure a flow range from 0 to 1.0 liter per min (0.035 cfm).

5.2.6 Flexible Bag. Tedlar, or equivalent, with a capacity of 60 to 90 liters (2 to 3 ft<sup>3</sup>). Leak-test the bag in the laboratory before using by evacuating bag with a pump followed by a dry gas meter. When evacuation is complete, there should be no flow through the meter.

5.2.7 Pitot Tube. Type S, or equivalent, attached to the probe so that the sampling rate can be regulated proportional to the stack gas velocity when velocity is varying with the time or a sample traverse is conducted.

5.3 Analysis (Figure 10-3).

<sup>1</sup>Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

5.3.1 Carbon Monoxide Analyzer. Nondispersive infrared spectrometer, or equivalent. This instrument should be demonstrated, preferably by the manufacturer, to meet or exceed manufacturer's specifications and those described in this method.

5.3.2 Drying Tube. To contain approximately 200 g of silica gel.

5.3.3 Calibration Gas. Refer to section 6.1.

5.3.4 Filter. As recommended by NDIR manufacturer.

EC01JN82.177

EC01JN82.178

5.3.5 CO<sub>2</sub> Removal Tube. To contain approximately 500 g of ascarite.

5.3.6 Ice Water Bath. For ascarite and silica gel tubes.

5.3.7 Valve. Needle valve, or equivalent, to adjust flow rate

5.3.8 Rate Meter. Rotameter or equivalent to measure gas flow rate of 0 to 1.0 liter per min (0.035 cfm) through NDIR.

5.3.9 Recorder (*optional*). To provide permanent record of NDIR readings.

#### 6. Reagents

6.1 Calibration Gases. Known concentration of CO in nitrogen (N<sub>2</sub>) for instrument span, prepurified grade of N<sub>2</sub> for zero, and two additional concentrations corresponding approximately to 60 percent and 30 percent span. The span concentration shall not exceed 1.5 times the applicable source performance standard. The calibration gases shall be certified by the manufacturer to be within  $\pm 2$  percent of the specified concentration.

EC01JN82.178

6.2 Silica Gel. Indicating type, 6 to 16 mesh, dried at 175 °C (347 °F) for 2 hours.

6.3 Ascarite. Commercially available.

#### 7. Procedure

##### 7.1 Sampling.

7.1.1 Continuous Sampling. Set up the equipment as shown in Figure 10-1 making sure all connections are leak free. Place the probe in the stack at a sampling point and purge the sampling line. Connect the analyzer and begin drawing sample into the analyzer. Allow 5 minutes for the system to stabilize, then record the analyzer reading as required by the test procedure. (See section 7.2 and 8). CO<sub>2</sub> content of the gas may be determined by using the Method 3 integrated sample procedure, or by weighing the ascarite CO<sub>2</sub> removal tube and computing CO<sub>2</sub> concentration from the gas volume sampled and the weight gain of the tube.

7.1.2 Integrated Sampling. Evacuate the flexible bag. Set up the equipment as shown in Figure 10-2 with the bag disconnected. Place the probe in the stack and purge the sampling line. Connect the bag, making sure that all connections are leak free. Sample at a rate proportional to the stack velocity. CO<sub>2</sub> content of the gas may be determined by using the Method 3 integrated sample procedures, or by weighing the ascarite CO<sub>2</sub> removal tube and computing CO<sub>2</sub> concentration from the gas volume sampled and the weight gain of the tube.

7.2 CO Analysis. Assemble the apparatus as shown in Figure 10-3, calibrate the instrument, and perform other required operations as described in section 8. Purge analyzer with N<sub>2</sub> prior to introduction of each sample. Direct the sample stream through the instrument for the test period, recording the readings. Check the zero and span again after the test to assure that any drift or malfunction is detected. Record the sample data on Table 10-1.

#### 8. Calibration

Assemble the apparatus according to Figure 10-3. Generally an instrument requires a warm-up period before stability is obtained. Follow the manufacturer's instructions for specific procedure. Allow a minimum time of 1 hour for warm-up. During this time check

the sample conditioning apparatus, i.e., filter, condenser, drying tube, and CO<sub>2</sub> removal tube, to ensure that each component is in good operating condition. Zero and calibrate the instrument according to the manufacturer's procedures using, respectively, nitrogen and the calibration gases.

TABLE 10-1—FIELD DATA

Comments	
Location .....	
Test .....	
Date .....	
Operator .....	
Clock time	Rotameter setting, liters per minute (cubic feet per minute)

### 9. Calculation

Calculate the concentration of carbon monoxide in the stack using Equation 10-1.

$$C_{CO \text{ stack}} = C_{CO \text{ NDIR}}(1 - F_{CO_2})$$

Eq. 10-1

Where:

$C_{CO \text{ stack}}$ —Concentration of CO in stack, ppm by volume (dry basis).

$C_{CO \text{ NDIR}}$ —Concentration of CO measured by NDIR analyzer, ppm by volume (dry basis).

$F_{CO_2}$ —Volume fraction of CO<sub>2</sub> in sample, i.e., percent CO<sub>2</sub> from Orsat analysis divided by 100.

### 10. Alternative Procedures

10.1 Interference Trap. The sample conditioning system described in Method 10A, sections 2.1.2 and 4.2, may be used as an alternative to the silica gel and ascarite traps.

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### ADDENDA

#### A. PERFORMANCE SPECIFICATIONS FOR NDIR CARBON MONOXIDE ANALYZERS

Range (minimum) .....	0-1000 ppm.
Output (minimum) .....	0-10mV.
Minimum detectable sensitivity.	20 ppm.
Rise time, 90 percent (maximum).	30 seconds.
Fall time, 90 percent (maximum).	30 seconds.
Zero drift (maximum) .....	10% in 8 hours.
Span drift (maximum) .....	10% in 8 hours.
Precision (minimum) .....	±2% of full scale.
Noise (maximum) .....	±1% of full scale.
Linearity (maximum deviation) .....	2% of full scale.
Interference rejection ratio .....	CO <sub>2</sub> —1000 to 1, H <sub>2</sub> O—500 to 1.

#### B. Definitions of Performance Specifications.

**Range**— The minimum and maximum measurement limits.

**Output**— Electrical signal which is proportional to the measurement; intended for connection to readout or data processing devices. Usually expressed as millivolts or milliamps full scale at a given impedance.

**Full scale**— The maximum measuring limit for a given range.

**Minimum detectable sensitivity**— The smallest amount of input concentration that can be detected as the concentration approaches zero.

**Accuracy**— The degree of agreement between a measured value and the true value; usually expressed as ± percent of full scale.

**Time to 90 percent response**— The time interval from a step change in the input concentration at the instrument inlet to a reading of 90 percent of the ultimate recorded concentration.

**Rise Time (90 percent)**—The interval between initial response time and time to 90 percent response after a step increase in the inlet concentration.

**Fall Time (90 percent)**—The interval between initial response time and time to 90 percent response after a step decrease in the inlet concentration.

**Zero Drift**— The change in instrument output over a stated time period, usually 24 hours, of unadjusted continuous operation when the input concentration is zero; usually expressed as percent full scale.

**Span Drift**— The change in instrument output over a stated time period, usually 24 hours, of unadjusted continuous operation when the input concentration is a stated upscale value; usually expressed as percent full scale.

**Precision**— The degree of agreement between repeated measurements of the same concentration, expressed as the average deviation of the single results from the mean.

**Noise**—Spontaneous deviations from a mean output not caused by input concentration changes.

**Linearity**—The maximum deviation between an actual instrument reading and the reading predicted by a straight line drawn between upper and lower calibration points.

**METHOD 10A—DETERMINATION OF CARBON MONOXIDE EMISSIONS IN CERTIFYING CONTINUOUS EMISSION MONITORING SYSTEMS AT PETROLEUM REFINERIES**

### 1. Applicability and Principle

**1.1 Applicability.** This method applies to the measurement of carbon monoxide (CO) at petroleum refineries. This method serves as the reference method in the relative accuracy test for nondispersive Infrared (NDIR) CO continuous emission monitoring systems (CEMS's) that are required to be installed in petroleum refineries on fluid catalytic cracking unit catalyst regenerators [40 CFR Part 60.105(a)(2)].

**1.2 Principle.** An integrated gas sample is extracted from the stack, passed through an alkaline permanganate solution to remove sulfur and nitrogen oxides, and collected in a Tedlar bag. The CO concentration in the sample is measured spectrophotometrically using the reaction of CO with p-sulfaminobenzoic acid.

#### 1.3 Range and Sensitivity.

**1.3.1 Range.** Approximately 3 to 1800 ppm CO. Samples having concentrations below 400 ppm are analyzed at 425 nm, and samples having concentrations above 400 ppm are analyzed at 600 nm.

**1.3.2 Sensitivity.** The detection limit is 3 ppm based on three times the standard deviation of the mean reagent blank values.

**1.4 Interferences.** Sulfur oxides, nitric oxide, and other acid gases interfere with the col-

orimetric reaction. They are removed by passing the sampled gas through an alkaline potassium permanganate scrubbing solution. Carbon dioxide (CO<sub>2</sub>) does not interfere, but, because it is removed by the scrubbing solution, its concentration must be measured independently and an appropriate volume correction made to the sampled gas.

#### 1.5 Precision, Accuracy, and Stability.

**1.5.1 Precision.** The estimated intralaboratory standard deviation of the method is 3 percent of the mean for gas samples analyzed in duplicate in the concentration range of 39 to 412 ppm. The interlaboratory precision has not been established.

**1.5.2 Accuracy.** The method contains no significant biases when compared to an NDIR analyzer calibrated with National Bureau of Standards (NBS) standards.

**1.5.3 Stability.** The individual components of the colorimetric reagent are stable for at least 1 month. The colorimetric reagent must be used within 2 days after preparation to avoid excessive blank correction. The samples in the Tedlar<sup>1</sup> bag should be stable for at least 1 week if the bags are leak-free.

### 2. Apparatus

**2.1 Sampling.** The sampling train is shown in Figure 10A-1, and component parts are discussed below:

<sup>1</sup>Mention of trade names or commercial products in this publication does not constitute the endorsement or recommendation for use by the Environmental Protection Agency.

EC01.JN92.180

**2.1.1 Probe.** Stainless steel, sheathed Pyrex glass, or equivalent, equipped with a glass wool plug to remove particulate matter.

**2.1.2 Sample Conditioning System.** Three Greenburg-Smith impingers connected in series with leak-free connections.

**2.1.3 Pump.** Leak-free pump with stainless steel and Teflon parts to transport sample at a flow rate of 300 ml/min to the flexible bag.

**2.1.4 Surge Tank.** Installed between the pump and the rate meter to eliminate the pulsation effect of the pump on the rate meter.

**2.1.5 Rate Meter.** Rotameter, or equivalent, to measure flow rate at 300 ml/min. Calibrate according to Section 3.2.

**2.1.6 Flexible Bag.** Tedlar, or equivalent, with a capacity of 10 liters and equipped with a sealing quick-connect plug. The bag must be leak-free according to Section 4.1. For protection, it is recommended that the bag be enclosed with a rigid container.

**2.1.7 Valves.** Stainless-steel needle valve to adjust flow rate, and stainless-steel three-way valve, or equivalent.

**2.1.8 CO<sub>2</sub> Analyzer.** Method 3 or its approved alternative to measure CO<sub>2</sub> concentration to within 0.5 percent.

**2.1.9 Volume Meter.** Dry gas meter, calibrated and capable of measuring the sample volume under rotameter calibration conditions of 300 ml/min for 10 minutes.

**2.1.10 Pressure Gauge.** A water filled U-tube manometer, or equivalent, of about 28 cm (12 in.) to leak-check the flexible bag.

## 2.2 Analysis.

**2.2.1 Spectrophotometer.** Single- or double-beam to measure absorbance at 425 and 600 nm. Slit width should not exceed 20 nm.

**2.2.2 Spectrophotometer Cells.** 1-cm pathlength.

**2.2.3 Vacuum Gauge.** U-tube mercury manometer, 1 meter (39 in.), with 1-mm divisions, or other gauge capable of measuring pressure to within 1 mm Hg.

**2.2.4 Pump.** Capable of evacuating the gas reaction bulb to a pressure equal to or less than 40 mm Hg absolute, equipped with coarse and fine flow control valves.

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*2.25 Barometer.* Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 1 mm Hg.

*2.26 Reaction Bulbs.* Pyrex glass, 100 ml with Teflon stopcock (Figure 10A-2), leak-

free at 40 mm Hg, designed so that 10 ml of the colorimetric reagent can be added and removed easily and accurately. Commercially available gas sample bulbs such as Supelco Catalog No. 2-2181 may also be used.

EC01JN82.181

*2.2.7 Manifold.* Stainless steel, with connections for three reaction bulbs and the appropriate connections for the manometer and sampling bag as shown in Figure 10A-3.

*2.2.8 Pipets.* Class A, 10-ml size.

*2.2.9 Shaker Table.* Reciprocating-stroke type such as Eberbach Corporation, Model 6015. A rocking arm or rotary-motion type

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shaker may also be used. The shaker must be large enough to accommodate at least six gas sample bulbs simultaneously. It may be necessary to construct a table top extension

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for most commercial shakers to provide sufficient space for the needed bulbs (Figure 10A-4).

2.2.10 *Valve.* Stainless steel shut-off valve.

EC01/N92.182

2.2.11 *Analytical Balance.* Capable of accurately weighing to 0.1 mg.

3. Reagents

Unless otherwise indicated, all reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available, otherwise, the best available grade shall be used.

3.1 Sampling.

3.1.1 *Water.* Deionized distilled, as described in Method 6, Section 3.1.1.

3.1.2 *Alkaline Permanganate Solution, 0.25 M  $KMnO_4$ /1.5 M NaOH.* Dissolve 40 g  $KMnO_4$  and 60 g NaOH in water, and dilute to 1 liter.

3.2 Analysis.

3.2.1 *Water.* Same as in Section 3.1.1.

3.2.2 *1 M Sodium Hydroxide (NaOH) Solution.* Dissolve 40 g NaOH in approximately 800 ml of water, cool, and dilute to 1 liter.

3.2.3 *0.1 M Silver Nitrate ( $AgNO_3$ ) Solution.* Dissolve 8.5 g  $AgNO_3$  in water, and dilute to 500 ml.

3.2.4 *0.1 M Para-Sulfamino benzoic Acid (p-SABA) Solution.* Dissolve 10.0 g p-SABA in 0.1 M NaOH (prepared by diluting 50 ml of 1 M NaOH to 500 ml), and dilute to 500 ml with 0.1 M NaOH.

EC01JN92.183

**3.2.5 Colorimetric Solution.** To a flask, add 100 ml of p-SABA solution and 100 ml of  $\text{AgNO}_3$  solution. Mix, and add 50 ml of 1 M NaOH with shaking. The resultant solution should be clear and colorless. This solution is acceptable for use for a period of 2 days.

**3.2.6 Standard Gas Mixtures.** Traceable to NBS standards and containing between 50 and 1000 ppm CO in nitrogen. At least two concentrations are needed to span each calibration range used (Section 5.3).

The calibration gases shall be certified by the manufacturer to be within 2 percent of the specified concentrations.

#### 4. Procedure

**4.1 Sample Bag Leak-checks.** While a bag leak-check is required after bag use, it should also be done before the bag is used for

sample collection. The bag should be leak-checked in the inflated and deflated condition according to the following procedures.

Connect the bag to a water manometer, and pressurize the bag to 5 to 10 cm  $\text{H}_2\text{O}$  (2 to 4 in.  $\text{H}_2\text{O}$ ). Allow the bag to stand for 60 minutes. Any displacement in the water manometer indicates a leak. Now, evacuate the bag with a leakless pump that is connected on the downstream side of a flow-indicating device such as a 0-to 100-ml/min rotameter or an impinger containing water. When the bag is completely evacuated, no flow should be evident if the bag is leak-free.

**4.2 Sampling.** Evacuate the Tedlar bag completely using a vacuum pump. Assemble the apparatus as shown in Figure 10A-1. Loosely pack glass wool in the tip of the probe. Place 400 ml of alkaline permanganate

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solution in the first two impingers and 250 ml in the third. Connect the pump to the third impinger, and follow this with the surge tank, rate meter, and three-way valve. Do not connect the Tedlar bag to the system at this time.

Leak-check the sampling system by placing a vacuum gauge at or near the probe inlet, plugging the probe inlet, opening the three-way valve, and pulling a vacuum of approximately 250 mm Hg on the system while observing the rate meter for flow. If flow is indicated on the rate meter, do not proceed further until the leak is found and corrected.

Purge the system with sample gas by inserting the probe into the stack and drawing sample through the system at 300 ml/min  $\pm$ 10 percent for 5 minutes. Connect the evacuated Tedlar bag to the system, record the starting time, and sample at a rate of 300 ml/min for 30 minutes, or until the Tedlar bag is nearly full. Record the sampling time, the barometric pressure, and the ambient temperature. Purge the system as described above immediately before each sample.

The scrubbing solution is adequate for removing sulfur and nitrogen oxides from 50 liters of stack gas when the concentration of

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each is less than 1,000 ppm and the CO<sub>2</sub> concentration is less than 15 percent. Replace the scrubber solution after every fifth sample.

**4.3 Carbon Dioxide Measurement.** Measure the CO<sub>2</sub> content in the stack to the nearest 0.5 percent each time a CO sample is collected. A simultaneous grab sample analyzed by the Fyrite analyzer is acceptable.

**4.4 Analysis.** Assemble the system shown in Figure 10A-3, and record the information required in Table 10A-1 as it is obtained. Pipet 10.0 ml of the colorimetric reagent into each gas reaction bulb, and attach the bulbs to the system. Open the stopcocks to the reaction bulbs, but leave the valve to the Tedlar bag closed. Turn on the pump, fully open the coarse-adjust flow valve, and slowly open the fine-adjust valve until the pressure is reduced to at least 40 mm Hg. Now close the coarse adjust valve, and observe the manometer to be certain that the system is leak-free. Wait a minimum of 2 minutes. If the pressure has increased less than 1 mm, proceed as described below. If a leak is present, find and correct it before proceeding further.

EC01JN92.184

Record the vacuum pressure ( $P_v$ ) to the nearest 1 mm Hg, and close the reaction bulb stopcocks. Open the Tedlar bag valve, and allow the system to come to atmospheric pressure. Close the bag valve, open the pump coarse adjust valve, and evacuate the system again. Repeat this fill/evacuation procedure at least twice to flush the manifold completely. Close the pump coarse adjust valve, open the Tedlar bag valve, and let the system fill to atmospheric pressure. Open the stopcocks to the reaction bulbs, and let the entire system come to atmospheric pressure.

Close the bulb stopcocks, remove the bulbs, record the room temperature and barometric pressure ( $P_{bar}$ , to nearest mm Hg), and place the bulbs on the shaker table with their main axis either parallel to or perpendicular to the plane of the table top. Purge the bulb-filling system with ambient air for several minutes between samples. Shake the samples for exactly 2 hours.

Immediately after shaking, measure the absorbance (A) of each bulb sample at 425 nm if the concentration is less than or equal to 400 ppm CO or at 600 nm if the concentration

is above 400 ppm. This may be accomplished with multiple bulb sets by sequentially collecting sets and adding to the shaker at staggered intervals, followed by sequentially removing sets from the shaker for absorbance measurement after the two-hour designated intervals have elapsed.

Use a small portion of the sample to rinse a spectrophotometer cell several times before taking an aliquot for analysis. If one cell is used to analyze multiple samples, rinse the cell several times between samples with water.

Prepare and analyze standards and a reagent blank as described in Section 5.3. Use water as the reference. Reject the analysis if the blank absorbance is greater than 0.1. All conditions should be the same for analysis of samples and standards. Measure the absorbances as soon as possible after shaking is completed. Determine the CO concentration of each bag sample using the calibration curve for the appropriate concentration range as discussed in Section 5.3.

#### 5. Calibration

**5.1 Bulb Calibration.** Weigh the empty bulb to the nearest 0.1 g. Fill the bulb to the stopcock with water, and again weigh to the nearest 0.1 g. Subtract the tare weight, and calculate the volume in liters to three significant figures using the density of water (at the measurement temperature). Record the volume on the bulb; alternatively, mark an identification number on the bulb, and record the volume in a notebook.

**5.2 Rate Meter Calibration.** Assemble the system as shown in Figure 10A-1 (the impingers may be removed), and attach a volume meter to the probe inlet. Set the rotameter at 300 ml/min, record the volume meter reading, start the pump, and pull gas through the system for 10 minutes. Record the final volume meter reading. Repeat the procedure and average the results to determine the volume of gas that passed through the system.

**5.3 Spectrophotometer Calibration Curve.** The calibration curve is established by taking at least two sets of three bulbs of known CO collected from Tedlar bags through the analysis procedure. Reject the standard set where any of the individual bulb absorbances differ from the set mean by more than 10 percent. Collect the standards as described in Section 4.2. Prepare standards to span the 0- to 400- or 400- to 1000-ppm range. If any samples span both concentration ranges, prepare a calibration curve for each range. A set of three bulbs containing colorimetric reagent but no CO should serve as a reagent blank and be taken through the analysis procedure.

Calculate the average absorbance for each set (3 bulbs) of standards using Equation 10A-1 and Table 10A-1. Construct a graph of average absorbance for each standard against its corresponding concentration in

ppm. Draw a smooth curve through the points. The curve should be linear over the two concentration ranges discussed in Section 1.3.1.

#### 6. Calculations

Carry out calculations retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

##### 6.1 Nomenclature.

A=Sample absorbance, uncorrected for the reagent blank.  
 $A_r$ =Absorbance of the reagent blank.  
 $A_v$ =Average sample absorbance per liter, units/liter.  
 $B_w$ =Moisture content in the bag sample.  
 C=CO concentration in the stack gas, dry basis, ppm.  
 $C_b$ =CO concentration of the bag sample, dry basis, ppm.  
 $C_s$ =CO concentration from the calibration curve, ppm.  
 F=Volume fraction of CO<sub>2</sub> in the stack.  
 n=Number of reaction bulbs used per bag sample.  
 $P_{bar}$ =Barometric pressure, mm Hg.  
 $P_r$ =Residual pressure in the sample bulb after evacuation, mm Hg.  
 $P_w$ =Vapor pressure of H<sub>2</sub>O in the bag (from Table 10A-2), mm Hg.  
 $V_b$ =Volume of the sample bulb, liters.  
 $V_r$ =Volume of reagent added to the sample bulb, 0.0100 liter.

##### 6.2 Average Sample Absorbance per Liter.

Average the three absorbance values for each bulb set. Then calculate  $A_v$  for each set of gas bulbs using Equation 10A-1. Use  $A_v$  to determine the CO concentration from the calibration curve ( $C_s$ ).

EC16N091.169

NOTE: A and  $A_r$  must be at the same wavelength.

##### 6.3 CO Concentration in the Bag.

Calculate  $C_b$  using Equations 10A-2 and 10A-3. If condensate is visible in the Tedlar bag, calculate  $B_w$  using Table 10A-2 and the temperature and barometric pressure in the analysis room. If condensate is not visible, calculate  $B_w$  using the temperature and barometric pressure recorded at the sampling site.

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***METHOD 10 B 40 CFR PART 60 APPENDIX A***

EC16NO91.170

EC16NO91.171

EC01JN92.185

#### 7. BIBLIOGRAPHY

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#### METHOD 10B—DETERMINATION OF CARBON MONOXIDE EMISSIONS FROM STATIONARY SOURCES

##### 1. Applicability and Principle

1.1 Applicability. This method applies to the measurement of carbon monoxide (CO) emissions at petroleum refineries and from other sources when specified in an applicable subpart of the regulations.

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1.2 Principle. An integrated gas sample is extracted from the sampling point and analyzed for CO. The sample is passed through a conditioning system to remove interferences and collected in a Tedlar bag. The CO is separated from the sample by gas chromatography (GC) and catalytically reduced to methane (CH<sub>4</sub>) prior to analysis by flame ionization detection FID. The analytical portion of this method is identical to applicable sections in Method 25 detailing CO measurement. The oxidation catalyst required in Method 25 is not needed for sample analysis. Complete Method 25 analytical systems are acceptable alternatives when calibrated for CO and operated by the Method 25 analytical procedures.

NOTE: Mention of trade names or commercial products in this method does not constitute the endorsement or recommendation for use by the Environmental Protection Agency.

1.3 Interferences. Carbon dioxide (CO<sub>2</sub>) and organics potentially can interfere with the analysis. Carbon dioxide is primarily removed from the sample by the alkaline permanganate conditioning system; any residual CO<sub>2</sub> and organics are separated from the CO by GC.

### 2. Apparatus

2.1 Sampling. Same as in Method 10A, section 2.1.

2.2 Analysis.

2.2.1 Gas Chromatographic (GC) Analyzer. A semicontinuous GC/FID analyzer capable of quantifying CO in the sample and containing at least the following major components.

2.2.1.1 Separation Column. A column that separates CO from CO<sub>2</sub> and organic compounds that may be present. A 1/8-in. OD stainless-steel column packed with 5.5 ft of 60/80 mesh Carbosieve S-II (available from Supelco) has been used successfully for this purpose. The column listed in Addendum 1 of Method 25 is also acceptable.

2.2.1.2 Reduction Catalyst. Same as in Method 25, section 2.3.2.

2.2.1.3 Sample Injection System. Same as in Method 25, section 2.3.4, equipped to accept a sample line from the Tedlar bag.

2.2.1.4 Flame Ionization Detector. Linearity meeting the specifications in section 2.3.5.1 of Method 25 where the linearity check is carried out using standard gases containing 20-, 200-, and 1,000-ppm CO. The minimal instrument range shall span 10 to 1,000 ppm CO.

2.2.1.5 Data Recording System. Same as in Method 25, section 2.3.6.

3. Reagents

3.1 Sampling. Same as in Method 10A, section 3.1.

3.2 Analysis.

3.2.1 Carrier, Fuel, and Combustion Gases. Same as in Method 25, sections 3.2.1, 3.2.2, and 3.2.3.

3.2.2 Linearity and Calibration Gases. Three standard gases with nominal CO concentrations of 20-, 200-, and 1,000-ppm CO in nitrogen.

3.2.3 Reduction Catalyst Efficiency Check Calibration Gas. Standard CH<sub>4</sub> gas with a concentration of 1,000 ppm in air.

### 4. Procedure

4.1 Sample Bag Leak-checks, Sampling, and CO<sub>2</sub> Measurement. Same as in Method 10A, sections 4.1, 4.2, and 4.3.

4.2 Preparation for Analysis. Before putting the GC analyzer into routine operation, conduct the calibration procedures listed in section 5. Establish an appropriate carrier flow rate and detector temperature for the specific instrument used.

4.3 Sample Analysis. Purge the sample loop with sample, and then inject the sample. Analyze each sample in triplicate, and calculate the average sample area (A). Determine the bag CO concentration according to section 6.2.

### 5. Calibration

5.1 Carrier Gas Blank Check. Analyze each new tank of carrier gas with the GC analyzer according to section 4.3 to check for contamination. The corresponding concentration must be less than 5 ppm for the tank to be acceptable for use.

5.2 Reduction Catalyst Efficiency Check. Prior to initial use, the reduction catalyst shall be tested for reduction efficiency. With the heated reduction catalyst bypassed, make triplicate injections of the 1,000-ppm CH<sub>4</sub> gas (section 3.2.3) to calibrate the analyzer. Repeat the procedure using 1,000-ppm CO (section 3.2.2) with the catalyst in operation. The reduction catalyst operation is acceptable if the CO response is within 5 percent of the certified gas value.

5.3 Analyzer Linearity Check and Calibration. Perform this test before the system is first placed into operation. With the reduction catalyst in operation, conduct a linearity check of the analyzer using the standards specified in section 3.2.2. Make triplicate injections of each calibration gas, and then calculate the average response factor (area/ppm) for each gas, as well as the overall mean of the response factor values. The instrument linearity is acceptable if the average response factor of each calibration gas is within 2.5 percent of the overall mean value and if the relative standard deviation (calculated in section 6.9 of Method 25) for each set of triplicate injections is less than 2 percent. Record the overall mean of the response factor values as the calibration response factor (R).

## 6. Calculations

Carry out calculations retaining at least one extra decimal figure beyond that of the acquired data. Round off results only after the final calculation.

## 6.1 Nomenclature.

A=Average sample area.  
 $B_w$ =Moisture content in the bag sample, fraction.  
 C=CO concentration in the stack gas, dry basis, ppm.  
 $C_b$ =CO concentration in the bag sample, dry basis, ppm.  
 F=Volume fraction of CO<sub>2</sub> in the stack, fraction.  
 $P_{bar}$ =Barometric pressure, mm Hg.  
 $P_w$ =Vapor pressure H<sub>2</sub>O in the bag (from Table 10-2, Method 10A), mm Hg.  
 R=Mean calibration response factor, area/ppm.

6.2 CO Concentration in the Bag. Calculate  $C_b$  using Equations 10B-1 and 10B-2. If condensate is visible in the Tedlar bag, calculate  $B_w$  using Table 10A-1 of Method 10A and the temperature and barometric pressure in the analysis room. If condensate is not visible, calculate  $B_w$  using the temperature and barometric pressure at the sampling site.

EC18N091.172

EC18N091.173

## 6.3 CO Concentration in the Stack.

$C=C_b(1-F)$   
 Eq. 10B-3

## 7. Bibliography

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## METHOD 11—DETERMINATION OF HYDROGEN SULFIDE CONTENT OF FUEL GAS STREAMS IN PETROLEUM REFINERIES

## 1. Principle and Applicability

1.1 Principle. Hydrogen sulfide (H<sub>2</sub>S) is collected from a source in a series of midgett impingers and absorbed in pH 3.0 cadmium sulfate (CdSO<sub>4</sub>) solution to form cadmium sulfide (CdS). The latter compound is then measured iodometrically. An impinger containing hydrogen peroxide is included to remove SO<sub>2</sub> as an interfering species. This method is a revision of the H<sub>2</sub>S method originally published in the FEDERAL REGISTER, Volume 39, No. 47, dated Friday, March 8, 1974.

1.2 Applicability. This method is applicable for the determination of the hydrogen sulfide content of fuel gas streams at petroleum refineries.

## 2. Range and Sensitivity

The lower limit of detection is approximately 8 mg/m<sup>3</sup> (6 ppm). The maximum of the range is 740 mg/m<sup>3</sup> (520 ppm).

## 3. Interferences

Any compound that reduces iodine or oxidizes iodide ion will interfere in this procedure, provided it is collected in the cadmium sulfate impingers. Sulfur dioxide in concentrations of up to 2,800 mg/m<sup>3</sup> is eliminated by the hydrogen peroxide solution. Thiols precipitate with hydrogen sulfide. In the absence of H<sub>2</sub>S, only co-traces of thiols are collected. When methane- and ethane-thiols at a total level of 300 mg/m<sup>3</sup> are present in addition to H<sub>2</sub>S, the results vary from 2 percent low at an H<sub>2</sub>S concentration of 400 mg/m<sup>3</sup> to 14 percent high at an H<sub>2</sub>S concentration of 100 mg/m<sup>3</sup>. Carbon oxysulfide at a concentration of 20 percent does not interfere. Certain carbonyl-containing compounds react with iodine and produce recurring end points. However, acetaldehyde and acetone at concentrations of 1 and 3 percent, respectively, do not interfere.

Entrained hydrogen peroxide produces a negative interference equivalent to 100 percent of that of an equimolar quantity of hydrogen sulfide. Avoid the ejection of hydrogen peroxide into the cadmium sulfate impingers.

## 4. Precision and Accuracy

Collaborative testing has shown the within-laboratory coefficient of variation to be 2.2 percent and the overall coefficient of variation to be 5 percent. The method bias was shown to be -4.8 percent when only H<sub>2</sub>S was present. In the presence of the interferences cited in section 3, the bias was positive at low H<sub>2</sub>S concentration and negative at higher concentrations. At 230 mg H<sub>2</sub>S/m<sup>3</sup>, the level of the compliance standard, the bias

***METHOD 23 40 CFR PART 60 APPENDIX A***

EC01JN62.248

**METHOD 23—DETERMINATION OF POLY-  
CHLORINATED DIBENZO-P-DIOXINS AND POLY-  
CHLORINATED DIBENZOFURANS FROM STA-  
TIONARY SOURCES**

*1. Applicability and Principle*

1.1 **Applicability.** This method is applica-  
ble to the determination of polychlorinated  
dibenzo-p-dioxins (PCDD's) and polychlor-  
inated dibenzofurans (PCDF's) from sta-  
tionary sources.

1.2 **Principle.** A sample is withdrawn from  
the gas stream isokinetically and collected

in the sample probe, on a glass fiber filter,  
and on a packed column of adsorbent mate-  
rial. The sample cannot be separated into a  
particle vapor fraction. The PCDD's and  
PCDF's are extracted from the sample, sepa-  
rated by high resolution gas chroma-  
tography, and measured by high resolution  
mass spectrometry.

*2. Apparatus*

2.1 **Sampling.** A schematic of the sam-  
pling train used in this method is shown in  
Figure 23-1. Sealing greases may not be used

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In assembling the train. The train is identical to that described in section 2.1 of Meth-

od 5 of this appendix with the following additions:

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2.1.1 Nozzle. The nozzle shall be made of nickel, nickel-plated stainless steel, quartz, or borosilicate glass.

2.1.2 Sample Transfer Lines. The sample transfer lines, if needed, shall be heat traced, heavy walled TFE (1/4 in. OD with 1/8 in. wall) with connecting fittings that are capable of

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forming leak-free, vacuum-tight connections without using sealing greases. The line shall be as short as possible and must be maintained at 120 °C.

2.1.1 Filter Support. Teflon or Teflon-coated wire.

2.1.2 Condenser. Glass, coil type with compatible fittings. A schematic diagram is shown in Figure Z3-2.

2.1.3 Water Bath. Thermostatically controlled to maintain the gas temperature exiting the condenser at < 20 °C (68 °F).

2.1.4 Adsorbent Module. Glass container to hold the solid adsorbent. A schematic diagram is shown in Figure Z3-2. Other physical configurations of the resin trap/condenser assembly are acceptable. The connecting fit-

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tings shall form leak-free, vacuum tight seals. No sealant greases shall be used in the sampling train. A coarse glass frit is included to retain the adsorbent.

2.2 Sample Recovery.

2.2.1 Fitting Caps. Ground glass, Teflon tape, or aluminum foil (Section 2.2.6) to cap off the sample exposed sections of the train.

2.2.2 Wash Bottles. Teflon, 500-ml.

2.2.3 Probe-Liner Probe-Nozzle, and Filter-Holder Brushes. Inert bristle brushes with pre-cleaned stainless steel or Teflon handles. The probe brush shall have extensions of stainless steel or Teflon, at least as long as the probe. The brushes shall be properly sized and shaped to brush out the nozzle, probe liner, and transfer line, if used.

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2.2.4 Filter Storage Container. Sealed filter holder, wide-mouth amber glass jar with Teflon-lined cap, or glass petri dish.

2.2.5 Balance. Triple beam.

2.2.6 Aluminum Foil. Heavy duty, hexane-rinsed.

2.2.7 Metal Storage Container. Air tight container to store silica gel.

2.2.8 Graduated Cylinder. Glass, 250-ml with 2-ml graduation.

2.2.9 Glass Sample Storage Container. Amber glass bottle for sample glassware washes, 500- or 1000-ml, with leak free Teflon-lined caps.

### 2.3 Analysis.

2.3.1 Sample Container. 125- and 250-ml flint glass bottles with Teflon-lined caps.

2.3.2 Test Tube. Glass.

2.3.3 Soxhlet Extraction Apparatus. Capable of holding 43 x 123 mm extraction thimbles.

2.3.4 Extraction Thimble. Glass, pre-cleaned cellulosic, or glass fiber.

2.3.5 Pasteur Pipettes. For preparing liquid chromatographic columns.

2.3.6 Reacti-vials. Amber glass, 2-ml, sterilized prior to use.

2.3.7 Rotary Evaporator. Buchi/Brinkman RF-121 or equivalent.

2.3.8 Nitrogen Evaporative Concentrator. N-Evap Analytical Evaporator Model III or equivalent.

2.3.9 Separatory Funnels. Glass, 2-liter.

2.3.10 Gas Chromatograph. Consisting of the following components:

2.3.10.1 Oven. Capable of maintaining the separation column at the proper operating temperature  $\pm$  °C and performing programmed increases in temperature at rates of at least 40 °C/min.

2.3.10.2 Temperature Gauge. To monitor column oven, detector, and exhaust temperatures  $\pm$  1 °C.

2.3.10.3 Flow System. Gas metering system to measure sample, fuel, combustion gas, and carrier gas flows.

2.3.10.4 Capillary Columns. A fused silica column, 60 x 0.25 mm inside diameter (ID), coated with DB-5 and a fused silica column, 30 m x 0.25 mm ID coated with DB-225. Other column systems may be used provided that the user is able to demonstrate using calibration and performance checks that the column system is able to meet the specifications of section 6.1.2.2.

2.3.11 Mass Spectrometer. Capable of routine operation at a resolution of 1:10000 with a stability of  $\pm$  5 ppm.

2.3.12 Data System. Compatible with the mass spectrometer and capable of monitoring at least five groups of 25 ions.

2.3.13 Analytical Balance. To measure within 0.1 mg.

## 3. Reagents

### 3.1 Sampling.

3.1.1 Filters. Glass fiber filters, without organic binder, exhibiting at least 99.95 percent efficiency (<0.05 percent penetration) on 0.3-micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM Standard Method D 2986-71 (Reapproved 1978) (incorporated by reference—see § 60.17).

3.1.1.1 Precleaning. All filters shall be cleaned before their initial use. Place a glass extraction thimble and 1 g of silica gel and a plug of glass wool into a Soxhlet apparatus, charge the apparatus with toluene, and reflux for a minimum of 3 hours. Remove the toluene and discard it, but retain the silica gel. Place no more than 50 filters in the thimble onto the silica gel bed and top with the cleaned glass wool. Charge the Soxhlet with toluene and reflux for 16 hours. After extraction, allow the Soxhlet to cool, remove the filters, and dry them under a clean N<sub>2</sub> stream. Store the filters in a glass petri dish sealed with Teflon tape.

3.1.2 Adsorbent Resin. Amberlite XAD-2 resin. Thoroughly cleaned before initial use.

3.1.2.1 Cleaning Procedure. This procedure may be carried out in a giant Soxhlet extractor. An all-glass filter thimble containing an extra-course frit is used for extraction of XAD-2. The frit is recessed 10-15 mm above a crenelated ring at the bottom of the thimble to facilitate drainage. The resin must be carefully retained in the extractor cup with a glass wool plug and a stainless steel ring because it floats on methylene chloride. This process involves sequential extraction in the following order.

Solvent	Procedure
Water .....	Initial rinse: Place resin in a beaker, rinse once with water, and discard. Fill with water a second time, let stand overnight, and discard.
Water .....	Extract with water for 8 hours.
Methanol .....	Extract for 22 hours.
Methylene Chloride .....	Extract for 22 hours.
Toluene .....	Extract for 22 hours.

### 3.1.2.2 Drying.

3.1.2.2.1 Drying Column. Pyrex pipe, 10.2 cm ID by 0.8 m long, with suitable retainers.

3.1.2.2.2 Procedure. The adsorbent must be dried with clean inert gas. Liquid nitrogen from a standard commercial liquid nitrogen cylinder has proven to be a reliable source of large volumes of gas free from organic contaminants. Connect the liquid nitrogen cylinder to the column by a length of cleaned copper tubing, 0.85 cm ID, coiled to pass through a heat source. A convenient heat source is a water-bath heated from a steam line. The final nitrogen temperature should only be warm to the touch and not over 40 °C. Continue flowing nitrogen through the adsorbent until all the residual solvent is removed. The flow rate should be sufficient to gently agitate the particles but not so excessive as the cause the particles to fracture.

3.1.2.3 Quality Control Check. The adsorbent must be checked for residual toluene.

3.1.2.3.1 Extraction. Weigh 1.0 g sample of dried resin into a small vial, add 3 ml of toluene, cap the vial, and shake it well.

3.1.2.3.2 Analysis. Inject a 2  $\mu$ l sample of the extract into a gas chromatograph operated under the following conditions:

Column: 6 ft  $\times$   $\frac{1}{8}$  in stainless steel containing 10 percent OV-101 on 100/120 Supelcoport.

Carrier Gas: Helium at a rate of 30 ml/min.

Detector: Flame ionization detector operated at a sensitivity of  $4 \times 10^{-11}$  A/mV.

Injection Port Temperature: 250  $^{\circ}$ C.

Detector Temperature: 305  $^{\circ}$ C.

Oven Temperature: 30  $^{\circ}$ C for 4 min; programmed to rise at 40  $^{\circ}$ C/min until it reaches 250  $^{\circ}$ C; return to 30  $^{\circ}$ C after 17 minutes.

Compare the results of the analysis to the results from the reference solution. Prepare the reference solution by injection 2.5  $\mu$ l of methylene chloride into 100 ml of toluene. This corresponds to 100  $\mu$ g of methylene chloride per g of adsorbent. The maximum acceptable concentration is 1000  $\mu$ g/g of adsorbent. If the adsorbent exceeds this level, drying must be continued until the excess methylene chloride is removed.

3.1.2.4 Storage. The adsorbent must be used within 4 weeks of cleaning. After cleaning, it may be stored in a wide mouth amber glass container with a Teflon-lined cap or placed in one of the glass adsorbent modules tightly sealed with glass stoppers. If precleaned adsorbent is purchased in sealed containers, it must be used within 4 weeks after the seal is broken.

3.1.3 Glass Wool. Cleaned by sequential immersion in three aliquots of methylene chloride, dried in a 110  $^{\circ}$ C oven, and stored in a methylene chloride-washed glass jar with a Teflon-lined screw cap.

3.1.4 Water. Deionized distilled and stored in a methylene chloride-rinsed glass container with a Teflon-lined screw cap.

3.1.5 Silica Gel. Indicating type, 6 to 18 mesh. If previously used, dry at 175  $^{\circ}$ C (350  $^{\circ}$ F) for two hours. New silica gel may be used as received. Alternately other types of desiccants (equivalent or better) may be used, subject to the approval of the Administrator.

3.1.6 Chromic Acid Cleaning Solution. Dissolve 20 g of sodium dichromate in 15 ml of water, and then carefully add 400 ml of concentrated sulfuric acid.

## 3.2 Sample Recovery.

3.2.2 Acetone. Pesticide quality.

3.2.2 Methylene Chloride. Pesticide quality.

3.2.3 Toluene. Pesticide quality.

## 3.3 Analysis.

3.3.1 Potassium Hydroxide. ACS grade, 2-percent (weight/volume) in water.

3.3.2 Sodium Sulfate. Granulated, reagent grade. Purify prior to use by rinsing with methylene chloride and oven drying. Store the cleaned material in a glass container with a Teflon-lined screw cap.

3.3.3 Sulfuric Acid. Reagent grade.

3.3.4 Sodium Hydroxide. 1.0 N. Weigh 40 g of sodium hydroxide into a 1-liter volumetric flask. Dilute to 1 liter with water.

3.3.5 Hexane. Pesticide grade.

3.3.6 Methylene Chloride. Pesticide grade.

3.3.7 Benzene. Pesticide Grade.

3.3.8 Ethyl Acetate.

3.3.9 Methanol. Pesticide Grade.

3.3.10 Toluene. Pesticide Grade.

3.3.11 Nonane. Pesticide Grade.

3.3.12 Cyclohexane. Pesticide Grade.

3.3.13 Basic Alumina. Activity grade 1, 100-200 mesh. Prior to use, activate the alumina by heating for 16 hours at 130  $^{\circ}$ C before use. Store in a desiccator. Pre-activated alumina may be purchased from a supplier and may be used as received.

3.3.14 Silica Gel. Bio-Sil A, 100-200 mesh. Prior to use, activate the silica gel by heating for at least 30 minutes at 180  $^{\circ}$ C. After cooling, rinse the silica gel sequentially with methanol and methylene chloride. Heat the rinsed silica gel at 50  $^{\circ}$ C for 10 minutes, then increase the temperature gradually to 180  $^{\circ}$ C over 25 minutes and maintain it at this temperature for 90 minutes. Cool at room temperature and store in a glass container with a Teflon-lined screw cap.

3.3.15 Silica Gel Impregnated with Sulfuric Acid. Combine 100 g of silica gel with 44 g of concentrated sulfuric acid in a screw capped glass bottle and agitate thoroughly. Disperse the solids with a stirring rod until a uniform mixture is obtained. Store the mixture in a glass container with a Teflon-lined screw cap.

3.3.16 Silica Gel Impregnated with Sodium Hydroxide. Combine 39 g of 1 N sodium hydroxide with 100 g of silica gel in a screw capped glass bottle and agitate thoroughly. Disperse solids with a stirring rod until a uniform mixture is obtained. Store the mixture in glass container with a Teflon-lined screw cap.

3.3.17 Carbon/Celite. Combine 10.7 g of AX-21 carbon with 124 g of Celite 545 in a 250-ml glass bottle with a Teflon-lined screw cap. Agitate the mixture thoroughly until a uniform mixture is obtained. Store in the glass container.

3.3.18 Nitrogen. Ultra high purity.

3.3.19 Hydrogen. Ultra high purity.

3.3.20 Internal Standard Solution. Prepare a stock standard solution containing the isotopically labelled PCDD's and PCDF's at the concentrations shown in Table 1 under the heading "Internal Standards" in 10 ml of nonane.

3.3.21 Surrogate Standard Solution. Prepare a stock standard solution containing the isotopically labelled PCDD's and PCDF's at the concentrations shown in Table 1 under the heading "Surrogate Standards" in 10 ml of nonane.

3.3.22 Recovery Standard Solution. Prepare a stock standard solution containing the isotopically labelled PCDD's and PCDF's



at the concentrations shown in Table 1 under the heading "Recovery Standards" in 10 ml of nonane.

#### 4. Procedure

4.1 Sampling. The complexity of this method is such that, in order to obtain reliable results, testers should be trained and experienced with the test procedures.

##### 4.1.1 Pretest Preparation.

4.1.1.1 Cleaning Glassware. All glass components of the train upstream of and including the adsorbent module, shall be cleaned as described in section 3A of the "Manual of Analytical Methods for the Analysis of Pesticides in Human and Environmental Samples." Special care shall be devoted to the removal of residual silicone grease sealants on ground glass connections of used glassware. Any residue shall be removed by soaking the glassware for several hours in a chromic acid cleaning solution prior to cleaning as described above.

4.1.1.2 Adsorbent Trap. The traps must be loaded in a clean area to avoid contamination. They may not be loaded in the field. Fill a trap with 20 to 40 g of XAD-2. Follow the XAD-2 with glass wool and tightly cap both ends of the trap. Add 100  $\mu$ l of the surrogate standard solution (section 3.3.21) to each trap.

4.1.1.3 Sample Train. It is suggested that all components be maintained according to the procedure described in APTD-0576.

4.1.1.4 Silica Gel. Weigh several 200 to 300 g portions of silica gel in an air tight container to the nearest 0.5 g. Record the total weight of the silica gel plus container, on each container. As an alternative, the silica gel may be weighed directly in its impinger or sampling holder just prior to sampling.

4.1.1.5 Filter. Check each filter against light for irregularities and flaws or pinhole leaks. Pack the filters flat in a clean glass container.

4.1.2 Preliminary Determinations. Same as section 4.1.2 of Method 5.

##### 4.1.3 Preparation of Collection Train.

4.1.3.1 During preparation and assembly of the sampling train, keep all train openings where contamination can enter, sealed until just prior to assembly or until sampling is about to begin.

NOTE: Do not use sealant grease in assembling the train.

4.1.3.2 Place approximately 100 ml of water in the second and third impingers, leave the first and fourth impingers empty, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the fifth impinger.

4.1.3.3 Place the silica gel container in a clean place for later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 g and recorded.

4.1.3.4 Assemble the train as shown in Figure 23-1.

4.1.3.5 Turn on the adsorbent module and condenser coil recirculating pump and begin monitoring the adsorbent module gas entry temperature. Ensure proper sorbent temperature gas entry temperature before proceeding and before sampling is initiated. It is extremely important that the XAD-2 adsorbent resin temperature never exceed 50 °C because thermal decomposition will occur. During testing, the XAD-2 temperature must not exceed 20 °C for efficient capture of the PCDD's and PCDF's.

4.1.4 Leak-Check Procedure. Same as Method 5, section 4.1.4.

4.1.5 Sample Train Operation. Same as Method 5, section 4.1.5.

4.2 Sample Recovery. Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period. Seal the nozzle end of the sampling probe with Teflon tape or aluminum foil.

When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe. Remove the probe from the train and close off both ends with aluminum foil. Seal off the inlet to the train with Teflon tape, a ground glass cap, or aluminum foil.

Transfer the probe and impinger assembly to the cleanup area. This area shall be clean and enclosed so that the chances of losing or contaminating the sample are minimized. Smoking, which could contaminate the sample, shall not be allowed in the cleanup area.

Inspect the train prior to and during disassembly and note any abnormal conditions, e.g., broken filters, colored impinger liquid, etc. Treat the samples as follows:

4.2.1 Container No. 1. Either seal the filter holder or carefully remove the filter from the filter holder and place it in its identified container. Use a pair of cleaned tweezers to handle the filter. If it is necessary to fold the filter, do so such that the particulate cake is inside the fold. Carefully transfer to the container any particulate matter and filter fibers which adhere to the filter holder gasket, by using a dry inert bristle brush and a sharp-edged blade. Seal the container.

4.2.2 Adsorbent Module. Remove the module from the train, tightly cap both ends, label it, cover with aluminum foil, and store it on ice for transport to the laboratory.

4.2.3 Container No. 2. Quantitatively recover material deposited in the nozzle, probe transfer lines, the front half of the filter holder, and the cyclone, if used, first, by brushing while rinsing three times each with acetone and then, by rinsing the probe three times with methylene chloride. Collect all the rinses in Container No. 2.

Rinse the back half of the filter holder three times with acetone. Rinse the connecting line between the filter and the condenser three times with acetone. Soak the connecting line with three separate portions of methylene chloride for 5 minutes each. If using a separate condenser and adsorbent trap, rinse the condenser in the same manner as the connecting line. Collect all the rinses in Container No. 2 and mark the level of the liquid on the container.

4.2.4 Container No. 3. Repeat the methylene chloride-rinsing described in Section 4.2.3 using toluene as the rinse solvent. Collect the rinses in Container No. 3 and mark the level of the liquid on the container.

4.2.5 Impinger Water. Measure the liquid in the first three impingers to within  $\pm 1$  ml by using a graduated cylinder or by weighing it to within  $\pm 0.5$  g by using a balance. Record the volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas.

Discard the liquid after measuring and recording the volume or weight.

4.2.7 Silica Gel. Note the color of the indicating silica gel to determine if it has been completely spent and make a mention of its condition. Transfer the silica gel from the fifth impinger to its original container and seal.

### 5. Analysis

All glassware shall be cleaned as described in section 3A of the "Manual of Analytical Methods for the Analysis of Pesticides in Human and Environmental Samples." All samples must be extracted within 30 days of collection and analyzed within 45 days of extraction.

#### 5.1 Sample Extraction.

5.1.1 Extraction System. Place an extraction thimble (section 2.3.4), 1 g of silica gel, and a plug of glass wool into the Soxhlet apparatus, charge the apparatus with toluene, and reflux for a minimum of 3 hours. Remove the toluene and discard it, but retain the silica gel. Remove the extraction thimble from the extraction system and place it in a glass beaker to catch the solvent rinses.

5.1.2 Container No. 1 (Filter). Transfer the contents directly to the glass thimble of the extraction system and extract them simultaneously with the XAD-2 resin.

5.1.3 Adsorbent Cartridge. Suspend the adsorbent module directly over the extraction thimble in the beaker (See section 5.1.1). The glass frit of the module should be in the up position. Using a Teflon squeeze bottle containing toluene, flush the XAD-2 into the thimble onto the bed of cleaned silica gel. Thoroughly rinse the glass module catching the rinsings in the beaker containing the thimble. If the resin is wet, effective extraction can be accomplished by loosely packing the resin in the thimble. Add the XAD-2 glass wool plug into the thimble.

5.1.4 Container No. 2 (Acetone and Methylene Chloride). Concentrate the sample to a volume of about 1-5 ml using the rotary evaporator apparatus, at a temperature of less than 37 °C. Rinse the sample container three times with small portions of methylene chloride and add these to the concentrated solution and concentrate further to near dryness. This residue contains particulate matter removed in the rinse of the train probe and nozzle. Add the concentrate to the filter and the XAD-2 resin in the Soxhlet apparatus described in section 5.1.1.

5.1.5 Extraction. Add 100  $\mu$ l of the internal standard solution (Section 3.3.20) to the extraction thimble containing the contents of the adsorbent cartridge, the contents of Container No. 1, and the concentrate from section 5.1.4. Cover the contents of the extraction thimble with the cleaned glass wool plug to prevent the XAD-2 resin from floating into the solvent reservoir of the extractor. Place the thimble in the extractor, and add the toluene contained in the beaker to the solvent reservoir. Pour additional toluene to fill the reservoir approximately 2/3 full. Add Teflon boiling chips and assemble the apparatus. Adjust the heat source to cause the extractor to cycle three times per hour. Extract the sample for 16 hours. After extraction, allow the Soxhlet to cool. Transfer the toluene extract and three 10-ml rinses to the rotary evaporator. Concentrate the extract to approximately 10 ml. At this point the analyst may choose to split the sample in half. If so, split the sample, store one half for future use, and analyze the other according to the procedures in sections 5.2 and 5.3. In either case, use a nitrogen evaporative concentrator to reduce the volume of the sample being analyzed to near dryness. Dissolve the residue in 5 ml of hexane.

5.1.6 Container No. 3 (Toluene Rinse). Add 100  $\mu$ l of the Internal Standard solution (section 3.3.2) to the contents of the container. Concentrate the sample to a volume of about 1-5 ml using the rotary evaporator apparatus at a temperature of less than 37 °C. Rinse the sample container apparatus at a temperature of less than 37 °C. Rinse the sample container three times with small portions of toluene and add these to the concentrated solution and concentrate further to near dryness. Analyze the extract separately according to the procedures in sections 5.2 and 5.3, but concentrate the solution in a rotary evaporator apparatus rather than a nitrogen evaporative concentrator.

#### 5.2 Sample Cleanup and Fractionation.

5.2.1 Silica Gel Column. Pack one end of a glass column, 20 mm x 230 mm, with glass wool. Add in sequence, 1 g silica gel, 2 g of sodium hydroxide impregnated silica gel, 1 g silica gel, 4 g of acid-modified silica gel, and 1 g of silica gel. Wash the column with 30 ml

of hexane and discard it. Add the sample extract, dissolved in 5 ml of hexane to the column with two additional 5-ml rinses. Elute the column with an additional 80 ml of hexane and retain the entire eluate. Concentrate this solution to a volume of about 1 ml using the nitrogen evaporative concentrator (section 2.3.7).

**5.2.2 Basic Alumina Column.** Shorten a 25-ml disposable Pasteur pipette to about 16 ml. Pack the lower section with glass wool and 12 g of basic alumina. Transfer the concentrated extract from the silica gel column to the top of the basic alumina column and elute the column sequentially with 120 ml of 0.5 percent methylene chloride in hexane followed by 120 ml of 35 percent methylene chloride in hexane. Discard the first 120 ml of eluate. Collect the second 120 ml of eluate and concentrate it to about 0.5 ml using the nitrogen evaporative concentrator.

**5.2.3 AX-21 Carbon/Celite 545 Column.** Remove the bottom 0.5 in. from the tip of a 9-ml disposable Pasteur pipette. Insert a glass fiber filter disk in the top of the pipette 2.5 cm from the constriction. Add sufficient carbon/celite mixture to form a 2 cm column. Top with a glass wool plug. In some cases AX-21 carbon fines may wash through the glass wool plug and enter the sample. This may be prevented by adding a celite plug to the exit end of the column. Rinse the column in sequence with 2 ml of 50 percent benzene in ethyl acetate, 1 ml of 50 percent methylene chloride in cyclohexane, and 2 ml of hexane. Discard these rinses. Transfer the concentrate in 1 ml of hexane from the basic alumina column to the carbon/celite column along with 1 ml of hexane rinse. Elute the column sequentially with 2 ml of 50 percent methylene chloride in hexane and 2 ml of 50 percent benzene in ethyl acetate and discard these eluates. Invert the column and elute in the reverse direction with 13 ml of toluene. Collect this eluate. Concentrate the eluate in a rotary evaporator at 50 °C to about 1 ml. Transfer the concentrate to a Reacti-vial using a toluene rinse and concentrate to a volume of 200 µl using a stream of N<sub>2</sub>. Store extracts at room temperature, shielded from light, until the analysis is performed.

**5.3 Analysis.** Analyze the sample with a gas chromatograph coupled to a mass spectrometer (GC/MS) using the instrumental parameters in sections 5.3.1 and 5.3.2. Immediately prior to analysis, add a 20 µl aliquot of the Recovery Standard solution from Table 1 to each sample. A 2 µl aliquot of the extract is injected into the GC. Sample extracts are first analyzed using the DB-5 capillary column to determine the concentration of each isomer of PCDD's and PCDF's (tetra-through octa-). If tetra-chlorinated dibenzofurans are detected in this analysis, then analyze another aliquot of the sample in a separate run, using the DB-225 column to measure the 2,3,7,8 tetra-chloro

dibenzofuran isomer. Other column systems may be used, provided that the user is able to demonstrate using calibration and performance checks that the column system is able to meet the specifications of section 6.1.2.2.

**5.3.1 Gas Chromatograph Operating Conditions.**

**5.3.1.1 Injector.** Configured for capillary column, splitless, 250 °C.

**5.3.1.2 Carrier Gas.** Helium, 1-2 ml/min.

**5.3.1.3 Oven.** Initially at 150 °C. Raise by at least 40 °C/min to 190 °C and then at 3 °C/min up to 300 °C.

**5.3.2 High Resolution Mass Spectrometer.**

**5.3.2.1 Resolution.** 10000 m/e.

**5.3.2.2 Ionization Mode.** Electron impact.

**5.3.2.3 Source Temperature** 250 °C.

**5.3.2.4 Monitoring Mode.** Selected ion monitoring. A list of the various ions to be monitored is summarized in Table 3.

**5.3.2.5 Identification Criteria.** The following identification criteria shall be used for the characterization of polychlorinated dibenzodioxins and dibenzofurans.

1. The integrated ion-abundance ratio ( $M/M+2$  or  $M+2/M+4$ ) shall be within 15 percent of the theoretical value. The acceptable ion-abundance ratio ranges for the identification of chlorine-containing compounds are given in Table 4.

2. The retention time for the analytes must be within 3 seconds of the corresponding <sup>13</sup>C-labeled internal standard, surrogate or alternate standard.

3. The monitored ions, shown in Table 3 for a given analyte, shall reach their maximum within 2 seconds of each other.

4. The identification of specific isomers that do not have corresponding <sup>13</sup>C-labeled standards is done by comparison of the relative retention time (RRT) of the analyte to the nearest internal standard retention time with reference (i.e., within 0.005 RRT units) to the comparable RRT's found in the continuing calibration.

5. The signal to noise ratio for all monitored ions must be greater than 2.5.

6. The confirmation of 2, 3, 7, 8-TCDD and 2, 3, 7, 8-TCDF shall satisfy all of the above identification criteria.

7. For the identification of PCDF's, no signal may be found in the corresponding PCDF channels.

**5.3.2.6 Quantification.** The peak areas for the two ions monitored for each analyte are summed to yield the total response for each analyte. Each internal standard is used to quantify the indigenous PCDD's or PCDF's in its homologous series. For example, the <sup>13</sup>C<sub>12</sub>-2,3,7,8-tetra chlorinated dibenzodioxin is used to calculate the concentrations of all other tetra chlorinated isomers. Recoveries of the tetra- and penta- internal standards are calculated using the <sup>13</sup>C<sub>12</sub>-1,2,3,4-TCDD. Recoveries of the hexa- through octa- internal standards are calculated using <sup>13</sup>C<sub>12</sub>-

1,2,3,7,8,9-HxCDD. Recoveries of the surrogate standards are calculated using the corresponding homolog from the internal standard.

#### 6. Calibration

Same as Method 5 with the following additions.

##### 6.1 GC/MS System.

6.1.1 Initial Calibration. Calibrate the GC/MS system using the set of five standards shown in Table 2. The relative standard deviation for the mean response factor from each of the unlabeled analytes (Table 2) and of the internal, surrogate, and alternate standards shall be less than or equal to the values in Table 5. The signal to noise ratio for the GC signal present in every selected ion current profile shall be greater than or equal to 2.5. The ion abundance ratios shall be within the control limits in Table 4.

##### 6.1.2 Daily Performance Check.

6.1.2.1 Calibration Check. Inject on  $\mu$ l of solution Number 3 from Table 2. Calculate the relative response factor (RRF) for each compound and compare each RRF to the corresponding mean RRF obtained during the initial calibration. The analyzer performance is acceptable if the measured RRF's for the labeled and unlabeled compounds for the daily run are within the limits of the mean values shown in Table 5. In addition, the ion-abundance ratios shall be within the allowable control limits shown in Table 4.

6.1.2.2 Column Separation Check. Inject a solution of a mixture of PCDD's and PCDF's that documents resolution between 2,3,7,8-TCDD and other TCDD isomers. Resolution is defined as a valley between peaks that is less than 25 percent of the lower of the two peaks. Identify and record the retention time windows for each homologous series.

Perform a similar resolution check on the confirmation column to document the resolution between 2,3,7,8 TCDF and other TCDF isomers.

6.2 Lock Channels. Set mass spectrometer lock channels as specified in Table 3. Monitor the quality control check channels specified in Table 3 to verify instrument stability during the analysis.

#### 7. Quality Control

7.1 Sampling Train Collection Efficiency Check. Add 100  $\mu$ l of the surrogate standards in Table 1 to the adsorbent cartridge of each train before collecting the field samples.

7.2 Internal Standard Percent Recoveries. A group of nine carbon labeled PCDD's and PCDF's representing the tetra-through octachlorinated homologues, is added to every sample prior to extraction. The role of the internal standards is to quantify the native PCDD's and PCDF's present in the sample as well as to determine the overall method efficiency. Recoveries of the internal

standards must be between 40 to 130 percent for the tetra-through hexachlorinated compounds while the range is 25 to 130 percent for the higher hepta- and octachlorinated homologues.

7.3 Surrogate Recoveries. The five surrogate compounds in Table 2 are added to the resin in the adsorbent sampling cartridge before the sample is collected. The surrogate recoveries are measured relative to the internal standards and are a measure of collection efficiency. They are not used to measure native PCDD's and PCDF's. All recoveries shall be between 70 and 130 percent. Poor recoveries for all the surrogates may be an indication of breakthrough in the sampling train. If the recovery of all standards is below 70 percent, the sampling runs must be repeated. As an alternative, the sampling runs do not have to be repeated if the final results are divided by the fraction of surrogate recovery. Poor recoveries of isolated surrogate compounds should not be grounds for rejecting an entire set of the samples.

7.4 Toluene QA Rinse. Report the results of the toluene QA rinse separately from the total sample catch. Do not add it to the total sample.

#### 8. Quality Assurance

8.1 Applicability. When the method is used to analyze samples to demonstrate compliance with a source emission regulation, an audit sample must be analyzed, subject to availability.

8.2 Audit Procedure. Analyze an audit sample with each set of compliance samples. The audit sample contains tetra through octa isomers of PCDD and PCDF. Concurrently, analyze the audit sample and a set of compliance samples in the same manner to evaluate the technique of the analyst and the standards preparation. The same analyst, analytical reagents, and analytical system shall be used both for the compliance samples and the EPA audit sample.

8.3 Audit Sample Availability. Audit samples will be supplied only to enforcement agencies for compliance tests. The availability of audit samples may be obtained by writing: Source Test Audit Coordinator (MD-77B), Quality Assurance Division, Atmospheric Research and Exposure Assessment Laboratory, U.S. Environmental Protection Agency, Research Triangle Park, NC 27711, or by calling the Source Test Audit Coordinator (STAC) at (919) 541-7834. The request for the audit sample must be made at least 30 days prior to the scheduled compliance sample analysis.

8.4 Audit Results. Calculate the audit sample concentration according to the calculation procedure described in the audit instructions included with the audit sample. Fill in the audit sample concentration and the analyst's name on the audit response form included with the audit instructions.

Send one copy to the EPA Regional Office or the appropriate enforcement agency and a second copy to the STAC. The EPA Regional office or the appropriate enforcement agency will report the results of the audit to the laboratory being audited. Include this response with the results of the compliance samples in relevant reports to the EPA Regional Office or the appropriate enforcement agency.

### 9. Calculations

Same as Method 5, section 6 with the following additions.

#### 9.1 Nomenclature.

$A_{ni}$ =Integrated ion current of the noise at the retention time of the analyte.

$A^*_{ci}$ =Integrated ion current of the two ions characteristic of the internal standard  $i$  in the calibration standard.

$A_{cij}$ =Integrated ion current of the two ions characteristic of compound  $i$  in the  $j$ th calibration standard.

$A^*_{cij}$ =Integrated ion current of the two ions characteristic of the internal standard  $i$  in the  $j$ th calibration standard.

$A_{cni}$ =Integrated ion current of the two ions characteristic of surrogate compound  $i$  in the calibration standard.

$A_i$ =Integrated ion current of the two ions characteristic of compound  $i$  in the sample.

$A^*_i$ =Integrated ion current of the two ions characteristic of internal standard  $i$  in the sample.

$A_{ri}$ =Integrated ion current of the two ions characteristic of the recovery standard.

$A_{si}$ =Integrated ion current of the two ions characteristic of surrogate compound  $i$  in the sample.

$C_i$ =Concentration of PCDD or PCDF  $i$  in the sample,  $\text{pg}/\text{M}^3$ .

$C_T$ =Total concentration of PCDD's or PCDF's in the sample,  $\text{pg}/\text{M}^3$ .

$m_{ci}$ =Mass of compound  $i$  in the calibration standard injected into the analyzer,  $\text{pg}$ .

$m_{ri}$ =Mass of recovery standard in the calibration standard injected into the analyzer,  $\text{pg}$ .

$m_{si}$ =Mass of surrogate compound in the calibration standard,  $\text{pg}$ .

$\text{RRF}_i$ =Relative response factor.

$\text{RRF}_{ri}$ =Recovery standard response factor.

$\text{RRF}_{si}$ =Surrogate compound response factor.

#### 9.2 Average Relative Response Factor.

EC16NO91.219

9.3 Concentration of the PCDD's and PCDF's.

EC16NO91.220

9.4 Recovery Standard Response Factor.

EC16NO91.221

9.5 Recovery of Internal Standards ( $R^*$ ).

EC16NO91.222

9.6 Surrogate Compound Response Factor.

EC16NO91.223

9.7 Recovery of Surrogate Compounds ( $R_s$ ).

EC16NO91.224

9.8 Minimum Detectable Limit (MDL).

EC16NO91.225

9.9 Total Concentration of PCDD's and PCDF's in the Sample.

EC16NO91.226

Any PCDD's or PCDF's that are reported as nondetected (below the MDL) shall be counted as zero for the purpose of calculating the total concentration of PCDD's and PCDF's in the sample.

### 10. Bibliography

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3. Thompson, J. R. (ed.). Analysis of Pesticide Residues in Human and Environmental Samples. U.S. Environmental Protection Agency. Research Triangle Park, NC, 1974.

4. Triangle Laboratories. Case Study: Analysis of Samples for the Presence of Tetra Through Octachloro-p-Dibenzodioxins and Dibenzofurans. Research Triangle Park, NC. 1988. 26 p.

5. U.S. Environmental Protection Agency. Method 8290—The Analysis of Polychlorinated Dibenzo-p-dioxin and Polychlorinated Dibenzofurans by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry. In: Test Methods for Evaluating Solid Waste. Washington, DC. SW-846.

TABLE 1—COMPOSITION OF THE SAMPLE FORTIFICATION AND RECOVERY STANDARDS SOLUTIONS—Continued

Analyte	Concentration (pg/μl)
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDD	100
<sup>13</sup> C <sub>12</sub> -2,3,4,7,8-PeCDF	100
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDF	100
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8,9-HpCDF	100
Recovery Standards:	
<sup>13</sup> C <sub>12</sub> -1,2,3,4-TCDD	500
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8,9-HxCDD	500

TABLE 1—COMPOSITION OF THE SAMPLE FORTIFICATION AND RECOVERY STANDARDS SOLUTIONS

Analyte	Concentration (pg/μl)
Internal Standards:	
<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDD	100
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDD	100
<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-HxCDD	100
<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDD	100
<sup>13</sup> C <sub>12</sub> -OCDD	100
<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDF	100
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDF	100
<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-HxCDF	100
<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDF	100
Surrogate Standards:	
<sup>37</sup> Cl <sub>2</sub> -2,3,7,8-TCDD	100

TABLE 2—COMPOSITION OF THE INITIAL CALIBRATION SOLUTIONS

Compound	Concentrations (pg/μL)				
	Solution No.				
	1	2	3	4	5
Alternate Standard:					
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8,9-HxCDF	2.5	5	25	250	500
Recovery Standards:					
<sup>13</sup> C <sub>12</sub> -1,2,3,4-TCDD	100	100	100	100	100
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8,9-HxCDD	100	100	100	100	100

TABLE 3—ELEMENTAL COMPOSITIONS AND EXACT MASSES OF THE IONS MONITORED BY HIGH RESOLUTION MASS SPECTROMETRY FOR PCDD'S AND PCDF'S

Descriptor No.	Accurate mass	Ion type	Elemental composition	Analyte	
2	292.9825	LOCK	CF <sub>11</sub>	PFK	
	303.9016	M	C <sub>12</sub> H <sub>4</sub> Cl <sub>2</sub> O	TCDF	
	305.8987	M+2	C <sub>12</sub> H <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O	TCDF	
	315.9419	M	<sup>12</sup> C <sub>12</sub> H <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O	TCDF (S)	
	317.9389	M+2	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O	TCDF (S)	
	319.8965	M	C <sub>12</sub> H <sub>3</sub> Cl <sub>2</sub> O <sub>2</sub>	TCDD	
	321.8936	M+2	C <sub>12</sub> H <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	TCDD	
	327.8847	M	C <sub>12</sub> H <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	TCDD (S)	
	330.9792	QC	CF <sub>13</sub>	PFK	
	331.9368	M	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	TCDD (S)	
	333.9339	M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	TCDD (S)	
	339.8597	M+2	C <sub>12</sub> H <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	PeCDF	
	341.8567	M+4	C <sub>12</sub> H <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O	PeCDF	
	351.9000	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O	PeCDF (S)	
	353.8970	M+4	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O	PeCDF (S)	
	355.8546	M+2	C <sub>12</sub> H <sub>2</sub> <sup>37</sup> Cl <sub>3</sub> O <sub>2</sub>	PeCDD	
	357.8516	M+4	C <sub>12</sub> H <sub>2</sub> <sup>37</sup> Cl <sub>3</sub> O <sub>2</sub>	PeCDD	
	367.8949	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>37</sup> Cl <sub>3</sub> O <sub>2</sub>	PeCDD (S)	
	369.8919	M+4	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>37</sup> Cl <sub>3</sub> O <sub>2</sub>	PeCDD (S)	
	375.8364	M+2	C <sub>12</sub> H <sub>2</sub> <sup>37</sup> Cl <sub>3</sub> O	HxCDFE	
	409.7974	M+2	C <sub>12</sub> H <sub>2</sub> <sup>37</sup> Cl <sub>3</sub> O	HxCDFE	
	3	373.8208	M+2	C <sub>12</sub> H <sub>2</sub> <sup>37</sup> Cl <sub>3</sub> O	HxCDF
		375.8178	M+4	C <sub>12</sub> H <sub>2</sub> <sup>37</sup> Cl <sub>3</sub> O	HxCDF
		383.8639	M	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>37</sup> Cl <sub>3</sub> O	HxCDF (S)
		385.8610	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>37</sup> Cl <sub>3</sub> O	HxCDF (S)
		389.8157	M+2	C <sub>12</sub> H <sub>2</sub> <sup>37</sup> Cl <sub>4</sub> O <sub>2</sub>	HxCDD
		391.8127	M+4	C <sub>12</sub> H <sub>2</sub> <sup>37</sup> Cl <sub>4</sub> O <sub>2</sub>	HxCDD
392.9760		LOCK	CF <sub>15</sub>	PFK	
401.8559		M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>37</sup> Cl <sub>4</sub> O <sub>2</sub>	HxCDD (S)	
403.8529		M+4	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>37</sup> Cl <sub>4</sub> O <sub>2</sub>	HxCDD (S)	
446.7855		M+4	C <sub>12</sub> H <sub>2</sub> <sup>37</sup> Cl <sub>5</sub> O <sub>2</sub>	OCDFE	
430.9729		QC	CF <sub>17</sub>	PFK	
4		407.7618	M+2	C <sub>12</sub> H <sub>2</sub> <sup>37</sup> Cl <sub>5</sub> O	HpCDF
	409.7789	M+4	C <sub>12</sub> H <sub>2</sub> <sup>37</sup> Cl <sub>5</sub> O	HpCDF	

TABLE 3—ELEMENTAL COMPOSITIONS AND EXACT MASSES OF THE IONS MONITORED BY HIGH RESOLUTION MASS SPECTROMETRY FOR PCDD'S AND PCDF'S—Continued

Descriptor No.	Accurate mass	Ion type	Elemental composition	Analyte
	417.8253	M	<sup>13</sup> C <sub>12</sub> H <sup>15</sup> Cl <sub>7</sub> O	HpCDF (S)
	419.8220	M+2	<sup>13</sup> C <sub>12</sub> H <sup>15</sup> Cl <sub>6</sub> <sup>37</sup> ClO	HpCDF (S)
	423.7786	M+2	C <sub>12</sub> H <sup>13</sup> Cl <sub>5</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD
	425.7737	M+4	<sup>13</sup> C <sub>12</sub> H <sup>13</sup> Cl <sub>5</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD
	435.8169	M+2	<sup>13</sup> C <sub>12</sub> H <sup>13</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD (S)
	437.8140	M+4	<sup>13</sup> C <sub>12</sub> H <sup>13</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HpCDD (S)
	479.7165	M+4	C <sub>12</sub> H <sup>13</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	NCPDE
	430.8729	LOCK	CF <sub>17</sub>	PFK
	441.7428	M+2	C <sub>12</sub> <sup>37</sup> Cl <sub>7</sub> O	OCDF
	443.7389	M+4	C <sub>12</sub> <sup>37</sup> Cl <sub>6</sub> <sup>37</sup> ClO	OCDF
	457.7377	M+2	C <sub>12</sub> <sup>37</sup> Cl <sub>5</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD
	459.7348	M+4	C <sub>12</sub> <sup>37</sup> Cl <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD
	469.7779	M+2	<sup>13</sup> C <sub>12</sub> <sup>37</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)
	471.7760	M+4	<sup>13</sup> C <sub>12</sub> <sup>37</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD (S)
	513.6775	M+4	C <sub>12</sub> <sup>37</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	DCDFE
	442.8728	QC	C <sub>10</sub> F <sub>17</sub>	PFK

(a) The following nucleic masses were used:  
 H = 1.007825  
 C = 12.000000  
<sup>13</sup>C = 13.003355  
 F = 18.9984  
 O = 15.994915  
<sup>35</sup>Cl = 34.968853  
<sup>37</sup>Cl = 36.965903  
 S = Labeled Standard  
 QC = Ion selected for monitoring instrument stability during the GC/MS analysis.

TABLE 4—ACCEPTABLE RANGES FOR ION-ABUNDANCE RATIOS OF PCDD'S AND PCDF'S

No. of chlorine atoms	Ion type	Theoretical ratio	Control limits	
			Lower	Upper
4	M/M+2	0.77	0.65	0.89
5	M+2/M+4	1.65	1.32	1.78
6	M+2/M+4	1.24	1.05	1.43
6+	M/M+2	0.51	0.43	0.59
7 <sup>b</sup>	M/M+2	0.44	0.37	0.51
7	M+2/M+4	1.04	0.88	1.20
8	M+2/M+4	0.89	0.76	1.02

<sup>a</sup> Used only for <sup>13</sup>C-HxCDF.  
<sup>b</sup> Used only for <sup>13</sup>C-HpCDF.

TABLE 5—MINIMUM REQUIREMENTS FOR INITIAL AND DAILY CALIBRATION RESPONSE FACTORS

Compound	Relative response factors	
	Initial calibration RSD	Daily calibration % difference
Unlabeled		
Analytes:		
2,3,7,8-TCDD	25	25
2,3,7,8-TCDF	25	25
1,2,3,7,8-PeCDD	25	25
1,2,3,7,8-PeCDF	25	25
2,3,4,7,8-PeCDF	25	25
1,2,4,6,7,8-HxCDD	25	25
1,2,3,6,7,8-HxCDD	25	25
1,2,3,7,8,9-HxCDD	25	25
1,2,3,4,7,8-HxCDF	25	25
1,2,3,6,7,8-HxCDF	25	25
1,2,3,7,8,9-HxCDF	25	25
2,3,4,6,7,8-HxCDF	25	25
1,2,3,4,6,7,8-HpCDD	25	25
1,2,3,4,6,7,8-HpCDF	25	25
OCDD	25	25

TABLE 5—MINIMUM REQUIREMENTS FOR INITIAL AND DAILY CALIBRATION RESPONSE FACTORS—Continued

Compound	Relative response factors	
	Initial calibration RSD	Daily calibration % difference
OCDF	30	30
Internal		
Standards:		
<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDD	25	25
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDD	30	30
<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-HxCDD	25	25
<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDD	30	30
<sup>13</sup> C <sub>12</sub> -OCDD	30	30
<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDF	30	30
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDF	30	30
<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-HxCDF	30	30
<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDF	30	30
Surrogate		
Standards:		
<sup>37</sup> Cl <sub>4</sub> -2,3,7,8-TCDD	25	25
<sup>13</sup> C <sub>12</sub> -2,3,4,7,8-PeCDF	25	25
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDD	25	25
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8,9-HpCDF	25	25
Alternate		
Standard:		
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8,9-HxCDF	25	25

METHOD 24—DETERMINATION OF VOLATILE MATTER CONTENT, WATER CONTENT, DENSITY, VOLUME SOLIDS, AND WEIGHT SOLIDS OF SURFACE COATINGS

1. Applicability and Principle

***METHOD 26 40 CFR PART 60 APPENDIX A***

6. Operational Checks and Calibration

Maintain a record of performance of each item.

6.1 Use the procedures in Section 6.1.1 to calibrate the headspace analyzer and FID and check for linearity before the system is first placed in operation, after any shutdown longer than 6 months, and after any modification of the system.

6.1.1 Calibration and Linearity. Use the procedures in Section 6.2.1 of Method 18 of Part 60, Appendix A, to prepare the standards and calibrate the flowmeters, using propane as the standard gas. Fill the calibration standard vials halfway (±5 percent) with de-ionized water. Purge and fill the airspace with calibration standard. Prepare a minimum of three calibration standards in triplicate at concentrations that will bracket the applicable cutoff. For a cutoff of 5.2 kPa, prepare nominal concentrations of 30,000, 50,000, and 70,000 ppm as propane. For a cutoff of 27.6 kPa, prepare nominal concentrations of 200,000, 300,000, and 400,000 ppm as propane.

6.1.1.1 Use the procedures in Section 5.2.3 to measure the FID response of each standard. Use a linear regression analysis to calculate the values for the slope (k) and the y-intercept (b). Use the procedures in Sections 7.2 and 7.3 to test the calibration and the linearity.

6.1.2 Daily FID Calibration Check. Check the calibration at the beginning and at the end of the daily runs by using the following procedures. Prepare two calibration standards at the nominal cutoff concentration using the procedures in Section 6.1.1. Place one at the beginning and one at the end of the daily run. Measure the FID response of the daily calibration standard and use the values for k and b from the most recent calibration to calculate the concentration of the daily standard. Use an equation similar to 25E-2 to calculate the percent difference between the daily standard and  $C_s$ . If the difference is within 5 percent, then the previous values for k and b may be used. Otherwise, use the procedures in Section 6.1.1 to recalibrate the FID.

7. Calculations

7.1 Nomenclature.

- A = Measurement of the area under the response curve, counts.
- b = y-intercept of the linear regression line.
- $C_s$  = Measured vapor phase organic concentration of sample, ppm as propane.
- $C_{ms}$  = Average measured vapor phase organic concentration of standard, ppm as propane.
- $C_m$  = Measured vapor phase organic concentration of standard, ppm as propane.
- $C_c$  = Calculated standard concentration, ppm as propane.
- k = Slope of the linear regression line.

- $P_{atm}$  = Atmospheric pressure at analysis conditions, mm Hg (in. Hg).
- $P^*$  = Organic vapor pressure in the sample, kPa (psi).
- $\beta = 1.333 \times 10^{-7} \text{ kPa}/[(\text{mm Hg})(\text{ppm})]$ . ( $4.91 \times 10^{-7} \text{ psi}/[(\text{in. Hg})(\text{ppm})]$ )

7.2 Linearity. Use the following equation to calculate the measured standard concentration for each standard vial.

$$C_m = k A + b \quad \text{Eq. 25E-1}$$

7.2.1 Calculate the average measured standard concentration ( $C_{ms}$ ) for each set of triplicate standards and use the following equation to calculate the percent difference (PD) between  $C_{ms}$  and  $C_c$ .

ER08DE94.001

The instrument linearity is acceptable if the percent difference is within five for each standard.

7.3 Relative Standard Deviation (RSD). Use the following equation to calculate the RSD for each triplicate set of standards.

ER06DE94.002

The calibration is acceptable if the RSD is within five for each standard concentration.

7.4 Concentration of organics in the headspace. Use the following equation to calculate the concentration of vapor phase organics in each sample.

$$C_s = k A + b \quad \text{Eq. 25E-4}$$

7.5 Vapor Pressure of Organics in the Headspace Sample. Use the following equation to calculate the vapor pressure of organics in the sample.

$$P^* = \beta P_{atm} C_s \quad \text{Eq. 25E-5}$$

METHOD 26—DETERMINATION OF HYDROGEN CHLORIDE EMISSIONS FROM STATIONARY SOURCES

1. Applicability, Principle, Interferences, Precision, Bias, and Stability

1.1 Applicability. This method is applicable for determining emissions of hydrogen halides (HX) [hydrogen chloride (HCl), hydrogen bromide (HBr), and hydrogen fluoride (HF)] and halogens ( $X_2$ ) [chlorine ( $Cl_2$ ) and bromine ( $Br_2$ )] from stationary sources. Sources, such as those controlled by wet scrubbers, that emit acid particulate matter must be sampled using Method 26A.

NOTE: Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.]

1.2 Principle. An integrated sample is extracted from the source and passed through

a prepurged heated probe and filter into dilute sulfuric acid and dilute sodium hydroxide solutions which collect the gaseous hydrogen halides and halogens, respectively. The filter collects other particulate matter including halide salts. The hydrogen halides are solubilized in the acidic solution and form chloride ( $\text{Cl}^-$ ), bromide ( $\text{Br}^-$ ), and fluoride ( $\text{F}^-$ ) ions. The halogens have a very low solubility in the acidic solution and pass through to the alkaline solution where they are hydrolyzed to form a proton ( $\text{H}^+$ ), the halide ion, and the hypohalous acid ( $\text{HClO}$  or  $\text{HBrO}$ ). Sodium thiosulfate is added in excess to the alkaline solution to assure reaction with the hypohalous acid to form a second halide ion such that 2 halide ions are formed for each molecule of halogen gas. The halide ions in the separate solutions are measured by ion chromatography (IC).

1.3 Interferences. Volatile materials, such as chlorine dioxide ( $\text{ClO}_2$ ) and ammonium chloride ( $\text{NH}_4\text{Cl}$ ), which produce halide ions upon dissolution during sampling are potential interferences. Interferents for the halide measurements are the halogen gases which disproportionate to a hydrogen halide and a hydrohalous acid upon dissolution in water. However, the use of acidic rather than neutral or basic solutions for collection of the hydrogen halides greatly reduces the dissolution of any halogens passing through this solution. The simultaneous presence of  $\text{HBr}$  and  $\text{Cl}_2$  may cause a positive bias in the  $\text{HCl}$  result with a corresponding negative bias in the  $\text{Cl}_2$  result as well as affecting the  $\text{HBr}/\text{Br}_2$  split. High concentrations of nitrogen oxides ( $\text{NO}_x$ ) may produce sufficient ni-

trate ( $\text{NO}_2^-$ ) to interfere with measurements of very low  $\text{Br}^-$  levels.

1.4 Precision and Bias. The within-laboratory relative standard deviations are 6.2 and 3.2 percent at  $\text{HCl}$  concentrations of 3.9 and 15.3 ppm, respectively. The method does not exhibit a bias to  $\text{Cl}_2$  when sampling at concentrations less than 50 ppm.

1.5 Sample Stability. The collected  $\text{Cl}^-$  samples can be stored for up to 4 weeks.

1.6 Detection Limit. The analytical detection limit for  $\text{Cl}^-$  is 0.1  $\mu\text{g}/\text{ml}$ . Detection limits for the other analyses should be similar.

## 2. Apparatus

2.1 Sampling. The sampling train is shown in Figure 26-1, and component parts are discussed below.

2.1.1 Probe. Borosilicate glass, approximately 3/8-in. (9-mm) I.D. with a heating system to prevent moisture condensation. A Teflon-glass filter in a mat configuration shall be installed behind the probe to remove particulate matter from the gas stream (see section 2.1.5). A glass wool plug should *not* be used to remove particulate matter since a negative bias in the data could result.

2.1.2 Three-Way Stopcock. A borosilicate glass three-way stopcock with a heating system to prevent moisture condensation. The heated stopcock should connect to the outlet of the heated filter and the inlet of the first impinger. The heating system shall be capable of preventing condensation up to the inlet of the first impinger. Silicone grease may be used, if necessary, to prevent leakage.

ER22APB4.011

2.1.3 Impingers. Four 30-ml midget impingers with leak-free glass connectors. Silicone grease may be used, if necessary, to prevent leakage. For sampling at high moisture sources or for sampling times greater than 1 hour, a midget impinger with a shortened stem (such that the gas sample does not bubble through the collected condensate) should be used in front of the first impinger.

2.1.4 Drying Tube or Impinger. Tube or impinger, of Mae West design, filled with 6- to 16-mesh indicating type silica gel, or equivalent, to dry the gas sample and to protect the dry gas meter and pump. If the silica gel has been used previously, dry at 175 °C (350 °F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used.

2.1.5 Filter. When the stack gas temperature exceeds 210 °C (410 °F) and the HCl concentration is greater than 20 ppm, a quartz-fiber filter may be used.

2.1.6 Filter Holder and Support. The filter holder should be made of Teflon or quartz. The filter support shall be made of Teflon. All-Teflon filter holders and supports are available from Savillex Corp., 5325 Hwy 101, Minnetonka, MN 55345.

2.1.7 Sample Line. Leak-free, with compatible fittings to connect the last impinger to the needle valve.

2.1.8 Rate Meter. Rotameter, or equivalent, capable of measuring flow rate to within 2 percent of the selected flow rate of 2 liters/min.

2.1.9 Purge Pump, Purge Line, Drying Tube, Needle Valve, and Rate Meter. Pump capable of purging the sampling probe at 2 liters/min, with drying tube, filled with silica gel or equivalent, to protect pump, and a rate meter capable of measuring 0 to 5 liters/min.

2.1.10 Stopcock Grease, Valve, Pump, Volume Meter, Barometer, and Vacuum Gauge. Same as in Method 6, Sections 2.1.4, 2.1.7, 2.1.8, 2.1.10, 2.1.11, and 2.1.12.

2.1.11 Temperature Measuring Devices. Temperature measuring device to monitor the temperature of the probe and a thermometer or other temperature measuring device to monitor the temperature of the sampling system from the outlet of the probe to the inlet of the first impinger.

2.1.12 Ice Water Bath. To minimize loss of absorbing solution.

2.2 Sample Recovery.

2.2.1 Wash Bottles. Polyethylene or glass, 500-ml or larger, two.

2.2.2 Storage Bottles. 100- or 250-ml, high-density polyethylene bottles with Teflon<sup>®</sup> screw cap liners to store impinger samples.

2.3 Sample Preparation and Analysis. The materials required for volumetric dilution and chromatographic analysis of samples are described below.

2.3.1 Volumetric Flasks. Class A, 100-ml size.

2.3.2 Volumetric Pipets. Class A, assortment. To dilute samples into the calibration range of the instrument.

2.3.3 Ion Chromatograph. Suppressed or nonsuppressed, with a conductivity detector and electronic integrator operating in the peak area mode. Other detectors, strip chart recorders, and peak height measurements may be used.

### 3. Reagents

Unless otherwise indicated, all reagents must conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society (ACS reagent grade). When such specifications are not available, the best available grade shall be used.

#### 3.1 Sampling.

3.1.1 Water. Deionized, distilled water that conforms to ASTM Specification D 1193-77, Type 3.

3.1.2 Acidic Absorbing solution, 0.1 N Sulfuric Acid ( $H_2SO_4$ ). To prepare 100 ml of the absorbing solution for the front impinger pair, slowly add 0.28 ml of concentrated  $H_2SO_4$  to about 90 ml of water while stirring, and adjust the final volume to 100 ml using additional water. Shake well to mix the solution.

3.1.3 Alkaline Absorbing Solution, 0.1 N Sodium Hydroxide (NaOH). To prepare 100 ml of the scrubber solution for the back pair of impingers, dissolve 0.40 g of solid NaOH in about 90 ml of water, and adjust the final solution volume to 100 ml using additional water. Shake well to mix the solution.

3.1.4 Sodium Thiosulfate ( $Na_2S_2O_3 \cdot 5H_2O$ )

3.2 Sample Preparation and Analysis.

3.2.1 Water. Same as in Section 3.1.1.

3.2.2 Absorbing Solution Blanks. A separate blank solution of each absorbing reagent should be prepared for analysis with the field samples. Dilute 30 ml of each absorbing solution to approximately the same final volume as the field samples using the blank sample of rinse water.

3.2.3 Halide Salt Stock Standard Solutions. Prepare concentrated stock solutions from reagent grade sodium chloride (NaCl), sodium bromide (NaBr), and sodium fluoride (NaF). Each must be dried at 110 °C for two or more hours and then cooled to room temperature in a desiccator immediately before weighing. Accurately weigh 1.6 to 1.7 g of the dried NaCl to within 0.1 mg, dissolve in water, and dilute to 1 liter. Calculate the exact Cl<sup>-</sup> concentration using Equation 28-1.

$$\mu\text{g Cl}^-/\text{ml} = \text{g of NaCl} \times 10^3 \times 35.453/58.44$$

Eq. 28-1

In a similar manner, accurately weigh and solubilize 1.2 to 1.3 g of dried NaBr and 2.2 to 2.3 g of NaF to make 1-liter solutions. Use Equations 28-2 and 28-3 to calculate the Br<sup>-</sup> and F<sup>-</sup> concentrations.

$$\mu\text{g Br}^-/\text{ml} = \text{g of NaBr} \times 10^3 \times 79.904/102.90$$

Eq. 28-2

$$\mu\text{g F}^-/\text{ml} = \text{g of NaF} \times 10^3 \times 18.998/41.99$$

#### Eq. 28-3

Alternately, solutions containing a nominal certified concentration of 1000 mg/l NaCl are commercially available as convenient stock solutions from which standards can be made by appropriate volumetric dilution. Refrigerate the stock standard solutions and store no longer than one month.

3.2.4 Chromatographic Eluent. Effective eluents for nonsuppressed IC using a resin- or silica-based weak ion exchange column are a 4 mM potassium hydrogen phthalate solution, adjusted to pH 4.0 using a saturated sodium borate solution, and a 4 mM 4-hydroxy benzoate solution, adjusted to pH 8.6 using 1 N NaOH. An effective eluent for suppressed ion chromatography is a solution containing 3 mM sodium bicarbonate and 2.4 mM sodium carbonate. Other dilute solutions buffered to a similar pH and containing no interfering ions may be used. When using suppressed ion chromatography, if the "water dip" resulting from sample injection interferes with the chloride peak, use a 2 mM NaOH/2.4 mM sodium bicarbonate eluent.

### 4. Procedure

#### 4.1 Sampling.

4.1.1 Preparation of Collection Train. Prepare the sampling train as follows: Pour 15 ml of the acidic absorbing solution into each one of the first pair of impingers, and 15 ml of the alkaline absorbing solution into each one of the second pair of impingers. Connect the impingers in series with the knockout impinger first, if used, followed by the two impingers containing the acidic absorbing solution and the two impingers containing the alkaline absorbing solution. Place a fresh charge of silica gel, or equivalent, in the drying tube or impinger at the end of the impinger train.

4.1.2 Adjust the probe temperature and the temperature of the filter and the stopcock, i.e., the heated area in Figure 28-1 to a temperature sufficient to prevent water condensation. This temperature should be at least 20°C above the source temperature, but not greater than 120 °C. The temperature should be monitored throughout a sampling run to ensure that the desired temperature is maintained.

4.1.3 Leak-Check Procedure. A leak-check prior to the sampling run is optional; however, a leak-check after the sampling run is mandatory. The leak-check procedure is as follows: Temporarily attach a suitable (e.g., 0-40 cc/min) rotameter to the outlet of the dry gas meter and place a vacuum gauge at or near the probe inlet. Plug the probe inlet, pull a vacuum of at least 250 mm Hg (10 in. Hg), and note the flow rate as indicated by the rotameter. A leakage rate not in excess of 2 percent of the average sampling rate is acceptable. (NOTE: Carefully release the probe inlet plug before turning off the

pump.) It is suggested (not mandatory) that the pump be leak-checked separately, either prior to or after the sampling run. If done prior to the sampling run, the pump leak-check shall precede the leak-check of the sampling train described immediately above; if done after the sampling run, the pump leak-check shall follow the train leak-check. To leak-check the pump, proceed as follows: Disconnect the drying tube from the probe-impinger assembly. Place a vacuum gauge at the inlet to either the drying tube or pump, pull a vacuum of 250 mm (10 in.) Hg, plug or pinch off the outlet of the flowmeter, and then turn off the pump. The vacuum should remain stable for at least 30 sec. Other leak-check procedures may be used, subject to the approval of the Administrator, U.S. Environmental Protection Agency.

4.1.4 Purge Procedure. Immediately before sampling, connect the purge line to the stopcock, and turn the stopcock to permit the purge pump to purge the probe (see Figure 1A of Figure 26-1). Turn on the purge pump, and adjust the purge rate to 2 liters/min. Purge for at least 5 minutes before sampling.

4.1.5 Sample Collection. Turn on the sampling pump, pull a slight vacuum of approximately 25 mm Hg (1 in. Hg) on the impinger train, and turn the stopcock to permit stack gas to be pulled through the impinger train (see Figure 1C of Figure 26-1). Adjust the sampling rate to 2 liters/min, as indicated by the rate meter, and maintain this rate to within 10 percent during the entire sampling run. Take readings of the dry gas meter volume and temperature, rate meter, and vacuum gauge at least once every 5 minutes during the run. A sampling time of 1 hour is recommended. Shorter sampling times may introduce a significant negative bias in the HCl concentration. At the conclusion of the sampling run, remove the train from the stack, cool, and perform a leak-check as described in section 4.1.2.

4.2 Sample Recovery. Disconnect the impingers after sampling. Quantitatively transfer the contents of the acid impingers and the knockout impinger, if used, to a leak-free storage bottle. Add the water rinses of each of these impingers and connecting glassware to the storage bottle. Repeat this procedure for the alkaline impingers and connecting glassware using a separate storage bottle. Add 25 mg sodium thiosulfate per the product of ppm of halogen anticipated to be in the stack gas times the dscm stack gas sampled. [Note: This amount of sodium thiosulfate includes a safety factor of approximately 5 to assure complete reaction with the hypohalous acid to form a second  $\text{Cl}^-$  ion in the alkaline solution.] Save portions of the absorbing reagents (0.1 N  $\text{H}_2\text{SO}_4$  and 0.1 N NaOH) equivalent to the

amount used in the sampling train (these are the absorbing solution blanks described in Section 3.2.2); dilute to the approximate volume of the corresponding samples using rinse water directly from the wash bottle being used. Add the same amount of sodium thiosulfate solution to the 0.1 N NaOH absorbing solution blank. Also, save a portion of the rinse water used to rinse the sampling train. Place each in a separate, pre-labeled storage bottle. The sample storage bottles should be sealed, shaken to mix, and labeled. Mark the fluid level.

4.3 Sample Preparation for Analysis. Note the liquid levels in the storage bottles and confirm on the analysis sheet whether or not leakage occurred during transport. If a noticeable leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Quantitatively transfer the sample solutions to 100-ml volumetric flasks, and dilute to 100 ml with water.

#### 4.4 Sample Analysis.

4.4.1 The IC conditions will depend upon analytical column type and whether suppressed or nonsuppressed IC is used. An example chromatogram from a nonsuppressed system using a 150-mm Hamilton PRP-X100 anion column, a 2 ml/min flow rate of 4 mM 4-hydroxy benzoate solution adjusted to a pH of 8.6 using 1 N NaOH, a 50- $\mu$ l sample loop, and a conductivity detector set on 1.0  $\mu$ S full scale is shown in Figure 26-2.

4.4.2 Before sample analysis, establish a stable baseline. Next, inject a sample of water, and determine if any  $\text{Cl}^-$ ,  $\text{Br}^-$ , or  $\text{F}^-$  appears in the chromatogram. If any of these ions are present, repeat the load/injection procedure until they are no longer present. Analysis of the acid and alkaline absorbing solution samples requires separate standard calibration curves; prepare each according to Section 5.2. Ensure adequate baseline separation of the analyses.

4.4.3 Between injections of the appropriate series of calibration standards, inject in duplicate the reagent blanks, quality control sample, and the field samples. Measure the areas or heights of the  $\text{Cl}^-$ ,  $\text{Br}^-$ , and  $\text{F}^-$  peaks. Use the mean response of the duplicate injections to determine the concentrations of the field samples and reagent blanks using the linear calibration curve. The values from duplicate injections should agree within 5 percent of their mean for the analysis to be valid. Dilute any sample and the blank with equal volumes of water if the concentration exceeds that of the highest standard.

4.5 Audit Analysis. An audit sample must be analyzed, subject to availability.

at least four calibration standards for each absorbing reagent containing the appropriate stock solutions such that they are within the linear range of the field samples. Using one of the standards in each series, ensure adequate baseline separation for the peaks of interest. Inject the appropriate series of calibration standards, starting with the lowest concentration standard first both before and after injection of the quality control check sample, reagent blanks, and field samples. This allows compensation for any instrument drift occurring during sample analysis.

Determine the peak areas, or heights, for the standards and plot individual values versus halide ion concentrations in  $\mu\text{g/ml}$ . Draw a smooth curve through the points. Use linear regression to calculate a formula describing the resulting linear curve.

#### 6. Quality Assurance

6.1 Applicability. When the method is used to analyze samples to demonstrate compliance with a source emission regulation, a set of two audit samples must be analyzed.

6.2 Audit Procedure. The audit sample are chloride solutions. Concurrently analyze the two audit samples and a set of compliance samples in the same manner to evaluate the technique of the analyst and the standards preparation. The same analyst, analytical reagents, and analytical system shall be used both for compliance samples and the EPA audit samples. If this condition is met, auditing the subsequent compliance analyses for the same enforcement agency within 30 days is not required. An audit sample set may not be used to validate different sets of compliance samples under the jurisdiction of different enforcement agencies, unless prior arrangements are made with both enforcement agencies.

6.3 Audit Sample Availability. The audit samples may be obtained by writing or calling the EPA Regional Office or the appropriate enforcement agency. The request for the audit samples must be made at least 30 days prior to the scheduled compliance sample analyses.

#### 6.4 Audit Results.

6.4.1 Calculate the concentrations in  $\text{mg/dscm}$  using the specified sample volume in the audit instructions.

NOTE: Indication of acceptable results may be obtained immediately by reporting the audit results in  $\text{mg/dscm}$  and compliance results in total  $\mu\text{g HCl/sample}$  to the responsible enforcement agency. Include the results of both audit samples, their identification numbers, and the analyst's name with the results of the compliance determination samples in appropriate reports to the EPA Regional Office or the appropriate enforcement agency. Include this information with subsequent analyses for the same enforcement agency during the 30-day period.

EC01JN82.267

#### 5. Calibration

5.1 Dry Gas Metering System. Thermometers, Rate Meter, and Barometer. Same as in Method 6, sections 5.1, 5.2, 5.3, and 5.4.

5.2 Ion Chromatograph. To prepare the calibration standards, dilute given amounts (1.0 ml or greater) of the stock standard solutions to convenient volumes, using 0.1 N  $\text{H}_2\text{SO}_4$  or 0.1 N NaOH, as appropriate. Prepare

6.4.2 The concentrations of the audit samples obtained by the analyst shall agree within 10 percent of the actual concentrations. If the 10 percent specification is not met, reanalyze the compliance samples and audit samples, and include initial and reanalysis values in the test report.

6.4.3 Failure to meet the 10 percent specification may require retests until the audit problems are resolved. However, if the audit results do not affect the compliance or non-compliance status of the affected facility, the Administrator may waive the reanalysis requirement, further audits, or retests and accept the results of the compliance test. While steps are being taken to resolve audit analysis problems, the Administrator may also choose to use the data to determine the compliance or noncompliance status of the affected facility.

### 7. Calculations

Retain at least one extra decimal figure beyond those contained in the available data in intermediate calculations, and round off only the final answer appropriately.

7.1 Sample Volume, Dry Basis, Corrected to Standard Conditions. Calculate the sample volume using Eq. 6-1 of Method 6.

7.2 Total  $\mu\text{g}$  HCl, HBr, or HF Per Sample.  
 $m_{HX} = K V_s (S_X^- - B_X^-)$  Eq. 26-4

where:

$B_X^-$  = Mass concentration of applicable absorbing solution blank,  $\mu\text{g}$  halide ion ( $\text{Cl}^-$ ,  $\text{Br}^-$ ,  $\text{F}^-$ )/ml, not to exceed 1  $\mu\text{g}/\text{ml}$  which is 10 times the published analytical detection limit of 0.1  $\mu\text{g}/\text{ml}$ .

$m_{HX}$  = Mass of HCl, HBr, or HF in sample,  $\mu\text{g}$ .

$S_X^-$  = Analysis of sample,  $\mu\text{g}$  halide ion ( $\text{Cl}^-$ ,  $\text{Br}^-$ ,  $\text{F}^-$ )/ml.

$V_s$  = Volume of filtered and diluted sample, ml.

$K_{\text{HCl}} = 1.028$  ( $\mu\text{g}$  HCl/ $\mu\text{g}$ -mole)/( $\mu\text{g}$   $\text{Cl}^-$ / $\mu\text{g}$ -mole).

$K_{\text{HBr}} = 1.013$  ( $\mu\text{g}$  HBr/ $\mu\text{g}$ -mole)/( $\mu\text{g}$   $\text{Br}^-$ / $\mu\text{g}$ -mole).

$K_{\text{HF}} = 1.053$  ( $\mu\text{g}$  HF/ $\mu\text{g}$ -mole)/( $\mu\text{g}$   $\text{F}^-$ / $\mu\text{g}$ -mole).

7.3 Total  $\mu\text{g}$   $\text{Cl}_2$  or  $\text{Br}_2$  Per Sample.

$m_{X_2} = V_s (S_X^- - B_X^-)$  Eq. 26-5

where:

$m_{X_2}$  = Mass of  $\text{Cl}_2$  or  $\text{Br}_2$  in sample,  $\mu\text{g}$ .

7.4 Concentration of Hydrogen Halide or Halogen in Flue Gas.

$C = K m_{HX, X_2} / V_{std}$  Eq. 26-6

where:

$C$  = Concentration of hydrogen halide (HX) or halogen ( $X_2$ ), dry basis, mg/dscm.

$V_{std}$  = Dry gas volume measured by the dry gas meter, corrected to standard conditions, dscm.

$K = 10^{-3}$  mg/ $\mu\text{g}$ .

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Method 26A—Determination of Hydrogen Halide and Halogen Emissions from Stationary Sources—Isokinetic Method

#### 1. Applicability, Principle, Interferences, Precision, Bias, and Stability

1.1 Applicability. This method is applicable for determining emissions of hydrogen halides (HX) [hydrogen chloride (HCl), hydrogen bromide (HBr), and hydrogen fluoride (HF)] and halogens ( $X_2$ ) [chlorine ( $\text{Cl}_2$ ) and bromine ( $\text{Br}_2$ )] from stationary sources. This method collects the emission sample isokinetically and is therefore particularly suited for sampling at sources, such as those controlled by wet scrubbers, emitting acid particulate matter (e.g., hydrogen halides dissolved in water droplets). [Note: Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.]

1.2 Principle. Gaseous and particulate pollutants are withdrawn isokinetically from the source and collected in an optional cyclone, on a filter, and in absorbing solutions. The cyclone collects any liquid droplets and is not necessary if the source emissions do not contain them; however, it is preferable

***METHOD 29 40 CFR PART 60 APPENDIX A***

EC16NC91.235

6.4 Air to Fuel Ratio. Use Equation 28a-3 to calculate the air to fuel ratio on a dry mass basis.

EC16NC91.236

6.5 Burn Rate. Calculate the fuel burn rate as in Method 28, Section 8.3.

### 7. Bibliography

Same as Method 3, Section 7, and Method 5H, Section 7.

## METHOD 29—DETERMINATION OF METALS EMISSIONS FROM STATIONARY SOURCES

### 1. Applicability and Principle

1.1 Applicability. This method is applicable to the determination of antimony (Sb), arsenic (As), barium (Ba), beryllium (Be), cadmium (Cd), chromium (Cr), cobalt (Co), copper (Cu), lead (Pb), manganese (Mn), mercury (Hg), nickel (Ni), phosphorus (P), selenium (Se), silver (Ag), thallium (Tl), and zinc (Zn) emissions from stationary sources. This method may be used to determine particulate emissions in addition to the metals emissions if the prescribed procedures and precautions are followed.

1.1.1 Hg emissions can be measured, alternatively, using EPA Method 101A of Appendix B, 40 CFR Part 81. Method 101-A measures only Hg but it can be of special interest to sources which need to measure both Hg and Mn emissions.

1.2 Principle. A stack sample is withdrawn isokinetically from the source, particulate emissions are collected in the probe and on a heated filter, and gaseous emissions are then collected in an aqueous acidic solution of hydrogen peroxide (analyzed for all metals including Hg) and an aqueous acidic solution of potassium permanganate (analyzed only for Hg). The recovered samples are digested, and appropriate fractions are analyzed for Hg by cold vapor atomic absorption spectroscopy (CVAAS) and for Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, P, Se, Ag, Tl, and Zn by inductively coupled argon plasma emission spectroscopy (ICAP) or atomic absorption spectroscopy (AAS). Graphite furnace atomic absorption spectroscopy (GFAAS) is used for analysis of Sb, As, Cd, Co, Pb, Se, and Tl if these elements require greater analytical sensitivity than can be obtained by ICAP. If one so chooses, AAS may be used for analysis of all listed metals if the resulting in-stack method detection limits meet the goal of the testing program.

Similarly, inductively coupled plasma-mass spectroscopy (ICP-MS) may be used for analysis of Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, As, Tl and Zn.

### 2. Range, Detection Limits, Precision, and Interferences

2.1 Range. For the analysis described and for similar analyses, the ICAP response is linear over several orders of magnitude. Samples containing metal concentrations in the nanograms per ml (ng/ml) to micrograms per ml ( $\mu\text{g/ml}$ ) range in the final analytical solution can be analyzed using this method. Samples containing greater than approximately 50  $\mu\text{g/ml}$  As, Cr, or Pb should be diluted to that level or lower for final analysis. Samples containing greater than approximately 20  $\mu\text{g/ml}$  of Cd should be diluted to that level before analysis.

2.2 Analytical Detection Limits. (NOTE: See section 2.3 for the description of in-stack detection limits.)

2.2.1 ICAP analytical detection limits for the sample solutions (based on Method 6010 in EPA Publication SW-846, Third Edition (November 1988) including updates I, II, IIA, and IIB, as incorporated by reference in §60.17(l)) are approximately as follows: Sb (32 ng/ml), As (53 ng/ml), Ba (2 ng/ml), Be (0.3 ng/ml), Cd (4 ng/ml), Cr (7 ng/ml), Co (7 ng/ml), Cu (6 ng/ml), Pb (42 ng/ml), Mn (2 ng/ml), Ni (15 ng/ml), P (75 ng/ml), Se (75 ng/ml), Ag (7 ng/ml), Tl (40 ng/ml), and Zn (2 ng/ml). ICP-MS analytical detection limits (based on based on Method 6020 in EPA Publication SW-846, Third Edition (November 1988) as incorporated by reference in §60.17(l)) are lower generally by a factor of ten or more. Be is lower by a factor of three. The actual sample analytical detection limits are sample dependent and may vary due to the sample matrix.

2.2.2 The analytical detection limits for analysis by direct aspiration AAS are approximately as follows: Sb (200 ng/ml), As (2 ng/ml), Ba (100 ng/ml), Be (5 ng/ml), Cd (5 ng/ml), Cr (50 ng/ml), Co (50 ng/ml), Cu (20 ng/ml), Pb (100 ng/ml), Mn (10 ng/ml), Ni (40 ng/ml), Se (2 ng/ml), Ag (10 ng/ml), Tl (100 ng/ml), and Zn (5 ng/ml).

2.2.3 The detection limit for Hg by CVAAS (on the resultant volume of the digestion of the aliquots taken for Hg analyses) can be approximately 0.02 to 0.2 ng/ml, depending upon the type of CVAAS analytical instrument used.

2.2.4 The use of GFAAS can enhance the detection limits compared to direct aspiration AAS as follows: Sb (3 ng/ml), As (1 ng/ml), Be (0.2 ng/ml), Cd (0.1 ng/ml), Cr (1 ng/ml), Co (1 ng/ml), Pb (1 ng/ml), Se (2 ng/ml), and Tl (ng/ml).

### 2.3 In-stack Detection Limits.

2.3.1 For test planning purposes in-stack detection limits can be developed by using the following information (1) the procedures

described in this method, (2) the analytical detection limits described in Section 2.2 and in EPA Publication SW-846, Third Edition (November 1986) including updates I, II, IIA and IIB, as incorporated by reference in §60.17(i), (3) the normal volumes of 300 ml (Analytical Fraction 1) for the front-half and 150 ml (Analytical Fraction 2A) for the back-half samples, and (4) a stack gas sample volume of 1.25 m<sup>3</sup>. The resultant in-stack meth-

od detection limits for the above set of conditions are presented in Table 29-1 and were calculated by using Eq. 29-1.

$$A \times B / C = D \quad \text{Eq. 29-1}$$

Where:

- A=Analytical detection limit, µg/ml.
- B=Liquid volume of digested sample prior to aliquotting for analysis, ml.
- C=Stack sample gas volume, dm<sup>3</sup>.
- D=In-stack detection limit, µg/m<sup>3</sup>.

TABLE 29-1.—IN-STACK METHOD DETECTION LIMITS (µg/M<sup>3</sup>) FOR THE FRONT-HALF, THE BACK-HALF, AND THE TOTAL SAMPLING TRAIN USING ICAP AND AAS

Metal	Front-half: Probe and filter	Back-half: Impingers 1-3	Back-half: Impingers (4-6) <sup>a</sup>	Total train:
Antimony	17.7 (0.7)	13.8 (0.4)		111.5 (1.1)
Arsenic	12.7 (0.3)	6.4 (0.1)		119.1 (0.4)
Barium	0.5	0.3		0.8
Beryllium	10.07 (0.05)	10.04 (0.03)		10.11 (0.08)
Cadmium	1.0 (0.02)	0.5 (0.01)		1.5 (0.03)
Chromium	11.7 (0.2)	0.6 (0.1)		12.3 (0.3)
Cobalt	11.7 (0.2)	0.6 (0.1)		12.3 (0.3)
Copper	1.4	0.7		2.1
Lead	110.1 (0.2)	15.0 (0.1)		115.1 (0.3)
Manganese	10.5 (0.2)	0.2 (0.1)		10.7 (0.3)
Mercury	0.06	0.3	0.2	0.66
Nickel	3.6	1.8		5.4
Phosphorus	18	9		27
Selenium	119 (0.5)	18 (0.3)		127 (0.8)
Silver	1.7	0.9		2.6
Thallium	19.6 (0.2)	14.8 (0.1)		14.4 (0.3)
Zinc	0.6	0.3		0.8

<sup>a</sup>Mercury analysis only.

<sup>1</sup>Detection limit when analyzed by GFAAS.

<sup>2</sup>Detection limit when analyzed by CVAAS, estimated for Back-Half and Total Train. See Sections 2.2 and 5.4.3.

Note: Actual method in-stack detection limits may vary from these values, as described in Section 2.3.3.

2.3.2 To ensure optimum precision/resolution in the analyses, the target concentrations of metals in the analytical solutions should be at least ten times their respective analytical detection limits. Under certain conditions, and with greater care in the analytical procedure, these concentrations can be as low as approximately three times the respective analytical detection limits without seriously impairing the precision of the analyses. On at least one sample run in the source test, and for each metal analyzed, perform either repetitive analyses, Method of Standard Additions, serial dilution, or matrix spike addition, etc., to document the quality of the data.

2.3.3 Actual in-stack method detection limits are based on actual source sampling parameters and analytical results as described above. If required, the method in-stack detection limits can be improved over those shown in Table 29-1 for a specific test by either increasing the sampled stack gas volume, reducing the total volume of the digested samples, improving the analytical detection limits, or any combination of the three. For extremely low levels of Hg only, the aliquot size selected for digestion and

analysis can be increased to as much as 10 ml, thus improving the in-stack detection limit by a factor of ten compared to a 1 ml aliquot size.

2.3.3.1 A nominal one hour sampling run will collect a stack gas sampling volume of about 1.25 m<sup>3</sup>. If the sampling time is increased to four hours and 5 m<sup>3</sup> are collected, the in-stack method detection limits would be improved by a factor of four compared to the values shown in Table 29-1.

2.3.3.2 The in-stack detection limits assume that all of the sample is digested and the final liquid volumes for analysis are the normal values of 300 ml for Analytical Fraction 1, and 150 ml for Analytical Fraction 2A. If the volume of Analytical Fraction 1 is reduced from 300 to 30 ml, the in-stack detection limits for that fraction of the sample would be improved by a factor of ten. If the volume of Analytical Fraction 2A is reduced from 150 to 25 ml, the in-stack detection limits for that fraction of the sample would be improved by a factor of six. Matrix effect checks are necessary on sample analyses and typically are of much greater significance for samples that have been concentrated to

less than the normal original sample volume. Reduction of Analytical Fractions 1 and 2A to volumes of less than 30 and 25 ml, respectively, could interfere with the redissolving of the residue and could increase interference by other compounds to an intolerable level.

2.3.3.3 When both of the modifications described in Sections 2.3.3.1 and 2.3.3.2 are used simultaneously on one sample, the resultant improvements are multiplicative. For example, an increase in stack gas volume by a factor of four and a reduction in the total liquid sample digested volume of both Analytical Fractions 1 and 2A by a factor of six would result in an improvement by a factor of twenty-four of the in-stack method detection limit.

2.4 Precision. The precision (relative standard deviation) for each metal detected in a method development test performed at a sewage sludge incinerator were found to be as follows: Sb (12.7 percent), As (13.5 percent), Ba (20.6 percent), Cd (11.5 percent), Cr (11.2 percent), Cu (11.5 percent), Pb (11.6 percent), P (14.6 percent), Se (15.3 percent), Tl (12.3 percent), and Zn (11.8 percent). The precision for Ni was 7.7 percent for another test conducted at a source simulator. Be, Mn, and

Ag were not detected in the tests. However, based on the analytical detection limits of the ICAP for these metals, their precisions could be similar to those for the other metals when detected at similar levels.

2.5 Interferences. Iron (Fe) can be a spectral interference during the analysis of As, Cr, and Cd by ICAP. Aluminum (Al) can be a spectral interference during the analysis of As and Pb by ICAP. Generally, these interferences can be reduced by diluting the analytical sample, but such dilution raises the in-stack detection limits. Background and overlap corrections may be used to adjust for spectral interferences. Refer to Method 6010 in EPA Publication SW-846 Third Edition (November 1986) including updates I, II, IIA and IIB, as incorporated by reference in §60.17(i) the other analytical methods used for details on potential interferences to this method. For all GFAAS analyses, use matrix modifiers to limit interferences, and matrix match all standards.

### 3. Apparatus

3.1 Sampling. A schematic of the sampling train is shown in Figure 29-1. It has general similarities to the Method 5 train.

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3.1.1 Probe Nozzle (Probe Tip) and Borosilicate or Quartz Glass Probe Liner. Same as Method 5, Sections 2.1.1 and 2.1.2, except that glass nozzles are required unless

alternate tips are constructed of materials that are free from contamination and will not interfere with the sample. If a probe tip other than glass is used, no correction to the

sample test results to compensate for the nozzle's effect on the sample is allowed. Probe fittings of plastic such as Teflon, polypropylene, etc. are recommended instead of metal fittings to prevent contamination. If one chooses to do so, a single glass piece consisting of a combined probe tip and probe liner may be used.

3.1.2 Pitot Tube and Differential Pressure Gauge. Same as Method 2, Sections 2.1 and 2.2, respectively.

3.1.3 Filter Holder. Glass, same as Method 5, Section 2.1.5, except use a Teflon filter support or other non-metallic, non-contaminating support in place of the glass frit.

3.1.4 Filter Heating System. Same as Method 5, Section 2.1.6.

3.1.5 Condenser. Use the following system for condensing and collecting gaseous metals and determining the moisture content of the stack gas. The condensing system shall consist of four to seven impingers connected in series with leak-free ground glass fittings or other leak-free, non-contaminating fittings. Use the first impinger as a moisture trap. The second impinger (which is the first  $\text{HNO}_3/\text{H}_2\text{O}_2$  impinger) shall be identical to the first impinger in Method 5. The third impinger (which is the second  $\text{HNO}_3/\text{H}_2\text{O}_2$  impinger) shall be a Greenburg Smith impinger with the standard tip as described for the second impinger in Method 5, Section 2.1.7. The fourth (empty) impinger and the fifth and sixth (both acidified  $\text{KMnO}_4$ ) impingers are the same as the first impinger in Method 5. Place a thermometer capable of measuring to within 1 °C (2 °F) at the outlet of the last impinger. If no Hg analysis is planned, then the fourth, fifth, and sixth impingers are not used.

3.1.6 Metering System, Barometer, and Gas Density Determination Equipment. Same as Method 5, Sections 2.1.6 through 2.1.10, respectively.

3.1.7 Teflon Tape. For capping openings and sealing connections. If necessary, on the sampling train.

3.2. Sample Recovery. Same as Method 5, Sections 2.2.1 through 2.2.8 (Probe-Liner and Probe-Nozzle Brushes or Swabs, Wash Bottles, Sample Storage Containers, Petri Dishes, Glass Graduated Cylinder, Plastic Storage Containers, Funnel and Rubber Policeman, and Glass Funnel), respectively, with the following exceptions and additions:

3.2.1 Non-metallic Probe-Liner and Probe-Nozzle Brushes or Swabs. Use non-metallic probe-liner and probe-nozzle brushes or swabs for quantitative recovery of materials collected in the front-half of the sampling train.

3.2.2 Sample Storage Containers. Use glass bottles (see the *Precaution* in Section 4.3.2 of this Method) with Teflon-lined caps that are non-reactive to the oxidizing solutions, with capacities of 1000- and 500-ml, for storage of acidified  $\text{KMnO}_4$ -containing sam-

ples and blanks. Glass or polyethylene bottles may be used for other sample types.

3.2.3 Graduated Cylinder. Glass or equivalent.

3.2.4 Funnel. Glass or equivalent.

3.2.5 Labels. For identifying samples.

3.2.6 Polypropylene Tweezers and/or Plastic Gloves. For recovery of the filter from the sampling train filter holder.

3.3 Sample Preparation and Analysis.

3.3.1 Volumetric Flasks, 100-ml, 250-ml, and 100-ml. For preparation of standards and sample dilutions.

3.3.2 Graduated Cylinders. For preparation of reagents.

3.3.3 Parr<sup>®</sup> Bombs or Microwave Pressure Relief Vessels with Capping Station (CEM Corporation model or equivalent). For sample digestion.

3.3.4 Beakers and Watch Glasses. 250-ml beakers, with watch glass covers, for sample digestion.

3.3.5 Ring Stands and Clamps. For securing equipment such as filtration apparatus.

3.3.6 Filter Funnels. For holding filter paper.

3.3.7 Disposable Pasteur Pipets and Bulbs.

3.3.8 Volumetric Pipets.

3.3.9 Analytical Balance. Accurate to within .01 mg.

3.3.10 Microwave or Conventional Oven. For heating samples at fixed power levels or temperatures, respectively.

3.3.11 Hot Plates.

3.3.12 Atomic Absorption Spectrometer (AAS). Equipped with a background corrector.

3.3.12.1 Graphite Furnace Attachment. With Sb, As, Cd, Co, Pb, Se, and Tl hollow cathode lamps (HCLs) or electrodeless discharge lamps (EDLs). Same as Methods 7041 (Sb), 7060 (As), 7131 (Cd), 7201 (Co), 7421 (Pb), 7740 (Se), and 7841 (Tl) in EPA publication SW-846 Third Edition (November 1986) including updates I, II, IIA and IIB, as incorporated by reference in §60.17(i).

3.3.12.2 Cold Vapor Mercury Attachment. With a mercury HCL or EDL, an air recirculation pump, a quartz cell, an aerator apparatus, and a heat lamp or desiccator tube. The heat lamp shall be capable of raising the temperature at the quartz cell by 10 °C above ambient, so that no condensation forms on the wall of the quartz cell. Same as Method 6020 in EPA publication SW-846 Third Edition (November 1986) including updates I, II, IIA and IIB, as incorporated by reference in §60.17(i). See NOTE NO. 2: Section 5.4.3 for other acceptable approaches for analysis of Hg in which analytical detection limits of 0.002 ng/ml were obtained.

3.3.13 Inductively Coupled Argon Plasma Spectrometer. With either a direct or sequential reader and an alumina torch. Same as EPA Method 6010 in EPA publication SW-846 Third Edition (November 1986) including

updates I, II, IIA and IIB, as incorporated by reference in §60.17(i).

3.3.14 Inductively Coupled Plasma-Mass Spectrometer. Same as EPA Method 8020 in EPA publication SW-846 Third Edition (November 1986) including updates I, II, IIA and IIB, as incorporated by reference in §60.17(i).

#### 4. Reagents

4.1 Unless otherwise indicated, it is intended that all reagents conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Otherwise, use the best available grade.

##### 4.2 Sampling Reagents.

4.2.1 Sample Filters. Without organic binders. The filters shall contain less than 1.3 µg/in.<sup>2</sup> of each of the metals to be measured. Analytical results provided by filter manufacturers stating metals content of the filters are acceptable. However, if no such results are available, analyze filter blanks for each target metal prior to emission testing. Quartz fiber filters meeting these requirements are recommended. However, if glass fiber filters become available which meet these requirements, they may be used. Filter efficiencies and unreactiveness to sulfur dioxide (SO<sub>2</sub>) or sulfur trioxide (SO<sub>3</sub>) shall be as described in Section 3.1.1 of Method 5.

4.2.2 Water. To conform to ASTM Specification D1193-77, Type II (incorporated by reference—See §60.17). If necessary, analyze the water for all target metals prior to field use. All target metals should be less than 1 ng/ml.

4.2.3 Nitric Acid (HNO<sub>3</sub>). Concentrated. Baker Instra-analyzed or equivalent.

4.2.4 Hydrochloric Acid (HCL). Concentrated. Baker Instra-analyzed or equivalent.

4.2.5 Hydrogen Peroxide (H<sub>2</sub>O<sub>2</sub>), 30 Percent (V/V).

4.2.6 Potassium Permanganate (KMnO<sub>4</sub>).

4.2.7 Sulfuric Acid (H<sub>2</sub>SO<sub>4</sub>). Concentrated.

4.2.8 Silica Gel and Crushed Ice. Same as Method 5, Sections 3.1.2 and 3.1.4, respectively.

4.3 Pretest Preparation of Sampling Reagents.

4.3.1 HNO<sub>3</sub>/H<sub>2</sub>O<sub>2</sub> Absorbing Solution, 5 Percent HNO<sub>3</sub>/10 Percent H<sub>2</sub>O<sub>2</sub>. Add carefully with stirring 50 ml of concentrated HNO<sub>3</sub> to a 1000-ml volumetric flask containing approximately 500 ml of water, and then add carefully with stirring 333 ml of 30 percent H<sub>2</sub>O<sub>2</sub>. Dilute to volume with water. Mix well. This reagent shall contain less than 2 ng/ml of each target metal.

4.3.2 Acidic KMnO<sub>4</sub> Absorbing Solution, 4 Percent KMnO<sub>4</sub> (W/V), 10 Percent H<sub>2</sub>SO<sub>4</sub> (V/V). Prepare fresh daily. Mix carefully, with stirring, 100 ml of concentrated H<sub>2</sub>SO<sub>4</sub> into approximately 800 ml of water, and add water with stirring to make a volume of 1

liter: this solution is 10 percent H<sub>2</sub>SO<sub>4</sub> (V/V). Dissolve, with stirring, 40 g of KMnO<sub>4</sub> into 10 percent H<sub>2</sub>SO<sub>4</sub> (V/V) and add 10 percent H<sub>2</sub>SO<sub>4</sub> (V/V) with stirring to make a volume of 1 liter. Prepare and store in glass bottles to prevent degradation. This reagent shall contain less than 2 ng/ml of Hg.

*Precaution:* To prevent autocatalytic decomposition of the permanganate solution, filter the solution through Whatman 541 filter paper. Also, due to the potential reaction of the potassium permanganate with the acid, there could be pressure buildup in the solution storage bottle. Therefore these bottles shall not be fully filled and shall be vented to relieve excess pressure and prevent explosion potentials. Venting is required, but not in a manner that will allow contamination of the solution. A No. 70-72 hole drilled in the container cap and Teflon liner has been used.

4.3.3 HNO<sub>3</sub>, 0.1 N. Add with stirring 6.3 ml of concentrated HNO<sub>3</sub> (70 percent) to a flask containing approximately 900 ml of water. Dilute to 1000 ml with water. Mix well. This reagent shall contain less than 2 ng/ml of each target metal.

4.3.4 HCL, 8 N. Carefully add with stirring 690 ml of concentrated HCL to a flask containing 250 ml of water. Dilute to 1000 ml with water. Mix well. This reagent shall contain less than 2 ng/ml of Hg.

4.4 Glassware Cleaning Reagents.

4.4.1 HNO<sub>3</sub>, Concentrated. Fisher ACS grade or equivalent.

4.4.2 Water. To conform to ASTM Specification D1193-77, Type II (incorporated by reference—See §60.17).

4.4.3 HNO<sub>3</sub>, 10 Percent (V/V). Add with stirring 500 ml of concentrated HNO<sub>3</sub> to a flask containing approximately 4000 ml of water. Dilute to 5000 ml with water. Mix well. This reagent shall contain less than 2 ng/ml of each target metal.

4.5 Sample Digestion and Analysis Reagents.

The metals standards, except Hg, may also be made from solid chemicals as described in Citation 3 of the Bibliography. Refer to Citations 1, 2, or 5 of the Bibliography for additional information on Hg standards. The 1000 µg/ml Hg stock solution standard may be made according to Section 6.2.5 of Method 101A.

4.5.1 HCL, Concentrated.

4.5.2 Hydrofluoric Acid (HF), Concentrated.

4.5.3 HNO<sub>3</sub>, Concentrated. Baker Instra-analyzed or equivalent.

4.5.4 HNO<sub>3</sub>, 50 Percent (V/V). Add with stirring 125 ml of concentrated HNO<sub>3</sub> to 100 ml of water. Dilute to 250 ml with water. Mix well. This reagent shall contain less than 2 ng/ml of each target metal.

4.5.5 HNO<sub>3</sub>, 5 Percent (V/V). Add with stirring 50 ml of concentrated HNO<sub>3</sub> to 800 ml of water. Dilute to 1000 ml with water. Mix

well. This reagent shall contain less than 2 ng/ml of each target metal.

4.5.6 Water. To conform to ASTM Specification D1193-77, Type II (incorporated by reference—See §60.17).

4.5.7 Hydroxylamine Hydrochloride and Sodium Chloride Solution. See Citation 2 of the Bibliography for preparation.

4.5.8 Stannous Chloride. See Citation 2 of the Bibliography for preparation.

4.5.9  $\text{KMnO}_4$ , 5 Percent (W/V). See Citation 2 of the Bibliography for preparation.

4.5.10  $\text{H}_2\text{SO}_4$ , Concentrated.

4.5.11 Potassium Persulfate, 5 Percent (W/V). See Citation 2 of the Bibliography for preparation.

4.5.12 Nickel Nitrate,  $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ .

4.5.13 Lanthanum Oxide,  $\text{La}_2\text{O}_3$ .

4.5.14 Hg Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

4.5.15 Pb Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

4.5.16 As Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

4.5.17 Cd Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

4.5.18 Cr Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

4.5.19 Sb Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

4.5.20 Ba Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

4.5.21 Be Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

4.5.22 Co Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

4.5.23 Cu Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

4.5.24 Mn Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

4.5.25 Ni Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

4.5.26 P Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

4.5.27 Se Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

4.5.28 Ag Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

4.5.29 Tl Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

4.5.30 Zn Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

4.5.31 Al Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

4.5.32 Fe Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

4.5.33 Hg Standards and Quality Control Samples. Prepare fresh weekly a 10  $\mu\text{g}/\text{ml}$  intermediate Hg standard by adding 5 ml of 1000  $\mu\text{g}/\text{ml}$  Hg stock solution prepared according to Method 101A to a 500-ml volumetric flask; dilute with stirring to 500 ml by first carefully adding 20 ml of 15 percent  $\text{HNO}_3$  and then adding water to the 500-ml volume. Mix well. Prepare a 200 ng/ml working Hg standard solution fresh daily; add 5 ml of the 10  $\mu\text{g}/\text{ml}$  intermediate standard to a 250-ml volumetric flask, and dilute to 250

ml with 5 ml of 4 percent  $\text{KMnO}_4$ , 5 ml of 15 percent  $\text{HNO}_3$ , and then water. Mix well. Use at least five separate aliquots of the working Hg standard solution and a blank to prepare the standard curve. These aliquots and blank shall contain 0.0, 1.0, 2.0, 3.0, 4.0, and 5.0 ml of the working standard solution containing 0, 200, 400, 600, 800, and 1000 ng Hg, respectively. Prepare quality control samples by making a separate 10  $\mu\text{g}/\text{ml}$  standard and diluting until in the calibration range.

4.5.34 ICAP Standards and Quality Control Samples. Calibration standards for ICAP analysis can be combined into four different mixed standard solutions as follows:

#### MIXED STANDARD SOLUTIONS FOR ICAP ANALYSIS

Solution	Elements
I .....	As, Be, Cd, Mn, Pb, Se, Zn.
II .....	Ba, Co, Cu, Fe.
III .....	Al, Cr, Ni.
IV .....	Ag, P, Sb, Tl.

Prepare these standards by combining and diluting the appropriate volumes of the 1000  $\mu\text{g}/\text{ml}$  solutions with 5 percent  $\text{HNO}_3$ . A minimum of one standard and a blank can be used to form each calibration curve. However, prepare a separate quality control sample spiked with known amounts of the target metals in quantities in the mid-range of the calibration curve. Suggested standard levels are 25  $\mu\text{g}/\text{ml}$  for Al, Cr and Pb, 15  $\mu\text{g}/\text{ml}$  for Fe, and 10  $\mu\text{g}/\text{ml}$  for the remaining elements. Prepare any standards containing less than 1  $\mu\text{g}/\text{ml}$  of metal on a daily basis. Standards containing greater than 1  $\mu\text{g}/\text{ml}$  of metal should be stable for a minimum of 1 to 2 weeks. For ICP-MS, follow Method 8020 in EPA Publication SW-846 Third Edition (November 1986) including updates I, II, IIA and IIB, as incorporated by reference in §60.17(i).

4.5.35 GFAAS Standards. Sb, As, Cd, Co, Pb, Se, and Tl. Prepare a 10  $\mu\text{g}/\text{ml}$  standard by adding 1 ml of 1000  $\mu\text{g}/\text{ml}$  standard to a 100-ml volumetric flask. Dilute with stirring to 100 ml with 10 percent  $\text{HNO}_3$ . For GFAAS, matrix match the standards. Prepare a 100 ng/ml standard by adding 1 ml of the 10  $\mu\text{g}/\text{ml}$  standard to a 100-ml volumetric flask, and dilute to 100 ml with the appropriate matrix solution. Prepare other standards by diluting the 100 ng/ml standards. Use at least five standards to make up the standard curve. Suggested levels are 0, 10, 50, 75, and 100 ng/ml. Prepare quality control samples by making a separate 10  $\mu\text{g}/\text{ml}$  standard and diluting until it is in the range of the samples. Prepare any standards containing less than 1  $\mu\text{g}/\text{ml}$  of metal on a daily basis. Standards containing greater than 1  $\mu\text{g}/\text{ml}$  of metal should be stable for a minimum of 1 to 2 weeks.

4.5.36 Matrix Modifiers.

4.5.36.1 Nickel Nitrate, 1 Percent (V/V). Dissolve 4.956 g of  $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  or

other nickel compound suitable for preparation of this matrix modifier in approximately 50 ml of water in a 100-ml volumetric flask. Dilute to 100 ml with water.

4.5.36.2 Nickel Nitrate, 0.1 Percent (V/V). Dilute 10 ml of 1 percent nickel nitrate solution to 100 ml with water. Inject an equal amount of sample and this modifier into the graphite furnace during GFAAS analysis for As.

4.5.36.3 Lanthanum. Carefully dissolve 0.5864 g of  $\text{La}_2\text{O}_3$  in 10 ml of concentrated  $\text{HNO}_3$ , and dilute the solution by adding it with stirring to approximately 50 ml of water. Dilute to 100 ml with water, and mix well. Inject an equal amount of sample and this modifier into the graphite furnace during GFAAS analysis for Pb.

4.5.37 Whatman 40 and 541 Filter Papers (or equivalent). For filtration of digested samples.

### 5. Procedure

5.1 Sampling. The complexity of this method is such that, to obtain reliable results, both testers and analysts must be trained and experienced with the test procedures, including source sampling; reagent preparation and handling; sample handling; safety equipment and procedures; analytical calculations; reporting; and the specific procedural descriptions throughout this method.

5.1.1 Pretest Preparation. Follow the same general procedure given in Method 5, Section 4.1.1, except that, unless particulate emissions are to be determined, the filter need not be desiccated or weighed. First, rinse all sampling train glassware with hot tap water and then wash in hot soapy water. Next, rinse glassware three times with tap water, followed by three additional rinses with water. Then soak all glassware in a 10 percent (V/V) nitric acid solution for a minimum of 4 hours, rinse three times with water, rinse a final time with acetone, and allow to air dry. Cover all glassware openings where contamination can occur until the sampling train is assembled for sampling.

5.1.2 Preliminary Determinations. Same as Method 5, Section 4.1.2.

5.1.3 Preparation of Sampling Train.

5.1.3.1 Set up the sampling train as shown in Figure 29-1. Follow the same general procedures given in Method 5, Section 4.1.3, except place 100 ml of the  $\text{HNO}_3/\text{H}_2\text{O}_2$  solution (Section 4.3.1, of this method) in each of the second and third impingers as shown in Figure 29-1. Place 100 ml of the acidic  $\text{KMnO}_4$  absorbing solution (Section 4.3.2 of this method) in each of the fifth and sixth impingers as shown in Figure 29-1, and transfer approximately 200 to 300 g of pre-weighed silica gel from its container to the last impinger. Alternatively, the silica gel may be

weighed directly in the impinger just prior to final train assembly.

5.1.3.2 Based on the specific source sampling conditions, the use of an empty first impinger can be eliminated if the moisture to be collected in the impingers will be less than approximately 100 ml.

5.1.3.3 If Hg analysis will not be performed, the fourth, fifth, and sixth impingers as shown in Figure 29-1 are not required.

5.1.3.4 To insure leak-free sampling train connections and to prevent possible sample contamination problems, use Teflon tape or other non-contaminating material instead of silicone grease.

*Precaution:* Exercise extreme care to prevent contamination within the train. Prevent the acidic  $\text{KMnO}_4$  from contacting any glassware that contains sample material to be analyzed for Mn. Prevent acidic  $\text{H}_2\text{O}_2$  from mixing with the acidic  $\text{KMnO}_4$ .

5.1.4 Leak-Check Procedures. Follow the leak-check procedures given in Method 5, Section 4.1.4.1 (Pretest Leak-Check), Section 4.1.4.2 (Leak-Checks During the Sample Run), and Section 4.1.4.3 (Post-Test Leak-Checks).

5.1.5 Sampling Train Operation. Follow the procedures given in Method 5, Section 4.1.5. When sampling for Hg, use a procedure analogous to that described in Section 7.1.1 of Method 101A, 40 CFR Part 61, Appendix B, if necessary to maintain the desired color in the last acidified permanganate impinger. For each run, record the data required on a data sheet such as the one shown in Figure 5-2 of Method 5.

5.1.6 Calculation of Percent Isokinetic. Same as Method 5, Section 4.1.6.

5.2 Sample Recovery.

5.2.1 Begin cleanup procedures as soon as the probe is removed from the stack at the end of a sampling period. The probe should be allowed to cool prior to sample recovery. When it can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a rinsed, non-contaminating cap over the probe nozzle to prevent losing or gaining particulate matter. Do not cap the probe tip tightly while the sampling train is cooling; a vacuum can form in the filter holder with the undesired result of drawing liquid from the impingers onto the filter.

5.2.2 Before moving the sampling train to the cleanup site, remove the probe from the sampling train and cap the open outlet. Be careful not to lose any condensate that might be present. Cap the filter inlet where the probe was fastened. Remove the umbilical cord from the last impinger and cap the impinger. Cap the filter holder outlet and impinger inlet. Use non-contaminating caps, whether ground-glass stoppers, plastic caps, serum caps, or Teflon tape to close these openings.

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5.2.3 Alternatively, the following procedure may be used to disassemble the train before the probe and filter holder/oven are completely cooled: Initially disconnect the filter holder outlet/impinger inlet and loosely cap the open ends. Then disconnect the probe from the filter holder or cyclone inlet and loosely cap the open ends. Cap the probe tip and remove the umbilical cord as previously described.

5.2.4 Transfer the probe and filter-impinger assembly to a cleanup area that is clean and protected from the wind and other potential causes of contamination or loss of sample. Inspect the train before and during disassembly and note any abnormal conditions. Take special precautions to assure that all the items necessary for recovery do not contaminate the samples. The sample is recovered and treated as follows (see schematic in Figures 29-2a and 29-2b):

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5.2.5 Container No. 1 (Sample Filter). Carefully remove the filter from the filter holder and place it in its labeled petri dish container. To handle the filter, use either acid-washed polypropylene or Teflon coated tweezers or clean, disposable surgical gloves rinsed with water and dried. If it is necessary

to fold the filter, make certain the particulate cake is inside the fold. Carefully transfer the filter and any particulate matter or filter fibers that adhere to the filter holder gasket to the petri dish by using a dry (acid-cleaned) nylon bristle brush. Do not use any metal-containing materials when recovering this train. Seal the labeled petri dish.

5.2.6 Container No. 2 (Acetone Rinse). Perform this procedure only if a determination of particulate emissions is to be made. Quantitatively recover particulate matter and any condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder by washing these components with a total of 100 ml of acetone, while simultaneously taking great care to see that no dust on the outside of the probe or other surfaces gets in the sample. The use of exactly 100 ml is necessary for the subsequent blank correction procedures. Distilled water may be used instead of acetone when approved by the Administrator and shall be used when specified by the Administrator; in these cases, save a water blank and follow the Administrator's directions on analysis.

5.2.6.1 Carefully remove the probe nozzle, and clean the inside surface by rinsing with acetone from a wash bottle while brushing with a non-metallic brush. Brush until the acetone rinse shows no visible particles, then make a final rinse of the inside surface with acetone.

5.2.6.2 Brush and rinse the sample exposed inside parts of the probe fitting with acetone in a similar way until no visible particles remain. Rinse the probe liner with acetone by tilting and rotating the probe while squirting acetone into its upper end so that all inside surfaces will be wetted with acetone. Allow the acetone to drain from the lower end into the sample container. A funnel may be used to aid in transferring liquid washings to the container. Follow the acetone rinse with a non-metallic probe brush. Hold the probe in an inclined position, squirt acetone into the upper end as the probe brush is being pushed with a twisting action three times through the probe. Hold a sample container underneath the lower end of the probe, and catch any acetone and particulate matter which is brushed through the probe until no visible particulate matter is carried out with the acetone or until none remains in the probe liner on visual inspection. Rinse the brush with acetone, and quantitatively collect these washings in the sample container. After the brushing, make a final acetone rinse of the probe as described above.

5.2.6.3 It is recommended that two people clean the probe to minimize sample losses. Between sampling runs, keep brushes clean and protected from contamination. Clean the inside of the front-half of the filter holder by rubbing the surfaces with a non-metallic brush and rinsing with acetone. Rinse each surface three times or more if needed to re-

move visible particulate. Make a final rinse of the brush and filter holder. After all acetone washings and particulate matter have been collected in the sample container, tighten the lid so that acetone will not leak out when shipped to the laboratory. Mark the height of the fluid level to determine whether or not leakage occurred during transport. Clearly label the container to identify its contents.

5.2.7 Container No. 3 (Probe Rinse). Keep the probe assembly clean and free from contamination during the probe rinse. Rinse the probe nozzle and fitting, probe liner, and front-half of the filter holder thoroughly with a total of 100 ml of 0.1 N HNO<sub>3</sub>, and place the wash into a sample storage container.

(NOTE: The use of a total of exactly 100 ml is necessary for the subsequent blank correction procedures.)

Perform the rinses as applicable and generally as described in Method 12, Section 5.2.2. Record the volume of the rinses. Mark the height of the fluid level on the outside of the storage container and use this mark to determine if leakage occurs during transport. Seal the container, and clearly label the contents. Finally, rinse the nozzle, probe liner, and front-half of the filter holder with water followed by acetone, and discard these rinses.

5.2.8 Container No. 4 (Impingers 1 through 3, Moisture Knockout Impinger, when used, HNO<sub>3</sub>/H<sub>2</sub>O<sub>2</sub> Impingers Contants and Rinses). Due to the potentially large quantity of liquid involved, the tester may place the impinger solutions from impingers 1 through 3 in more than one container, if necessary. Measure the liquid in the first three impingers to within 0.5 ml using a graduated cylinder. Record the volume. This information is required to calculate the moisture content of the sampled flue gas. Clean each of the first three impingers, the filter support, the back half of the filter housing, and connecting glassware by thoroughly rinsing with 100 ml of 0.1 N HNO<sub>3</sub> using the procedure as applicable in Method 12, Section 5.2.4.

(NOTE: The use of exactly 100 ml of 0.1 N HNO<sub>3</sub> rinse is necessary for the subsequent blank correction procedures. Combine the rinses and impinger solutions, measure and record the final total volume. Mark the height of the fluid level, seal the container, and clearly label the contents.)

5.2.9 Container Nos. 5A (0.1 N HNO<sub>3</sub>), 5B (KMnO<sub>4</sub>/H<sub>2</sub>SO<sub>4</sub> absorbing solution), and 5C (8 N HCl rinse and dilution).

5.2.9.1 When sampling for Hg, pour all the liquid from the impinger (normally impinger No. 4) that immediately preceded the two permanganate impingers into a graduated cylinder and measure the volume to within 0.5 ml. This information is required to calculate the moisture content of the sampled

flue gas. Place the liquid in Container No. 5A. Rinse the Impinger with exactly 100 ml of 0.1 N HNO<sub>3</sub> and place this rinse in Container No. 5A.

5.2.9.2 Pour all the liquid from the two permanganate Impingers into a graduated cylinder and measure the volume to within 0.5 ml. This information is required to calculate the moisture content of the sampled flue gas. Place this acidic KMnO<sub>4</sub> solution into Container No. 5B. Using a total of exactly 100 ml of fresh acidified KMnO<sub>4</sub> solution for all rinses (approximately 33 ml per rinse), rinse the two permanganate Impingers and connecting glassware a minimum of three times. Pour the rinses into Container No. 5B, carefully assuring transfer of all loose precipitated materials from the two Impingers. Similarly, using 100 ml total of water, rinse the permanganate Impingers and connecting glass a minimum of three times, and pour the rinses into Container 5B, carefully assuring transfer of any loose precipitated material. Mark the height of the fluid level, and clearly label the contents. Read the Precaution: in Section 4.3.2. *NOTE:* Due to the potential reaction of KMnO<sub>4</sub> with acid, pressure buildup can occur in the sample storage bottles. Do not fill these bottles completely and take precautions to relieve excess pressure. A No. 70-72 hole drilled in the container cap and Teflon liner has been used successfully.

5.2.9.3 If no visible deposits remain after the water rinse, no further rinse is necessary. However, if deposits remain on the Impinger surfaces, wash them with 25 ml of 8 N HCl, and place the wash in a separate sample container labeled No. 5C containing 200 ml of water. First, place 200 ml of water in the container. Then wash the Impinger walls and stem with the HCl by turning the Impinger on its side and rotating it so that the HCl contacts all inside surfaces. Use a total of only 25 ml of 8 N HCl for rinsing both permanganate Impingers combined. Rinse the first Impinger, then pour the actual rinse used for the first Impinger into the second Impinger for its rinse. Finally, pour the 25 ml of 8 N HCl rinse carefully into the container. Mark the height of the fluid level on the outside of the container to determine if leakage occurs during transport.

5.2.10 Container No. 6 (Silica Gel). Note the color of the indicating silica gel to determine whether it has been completely spent and make a notation of its condition. Transfer the silica gel from its Impinger to its original container and seal it. The tester may use a funnel to pour the silica gel and a rubber policeman to remove the silica gel from the Impinger. The small amount of particles that might adhere to the Impinger wall need not be removed. Do not use water or other liquids to transfer the silica gel since weight gained in the silica gel Impinger is used for moisture calculations. Al-

ternatively, if a balance is available in the field, record the weight of the spent silica gel (or silica gel plus Impinger) to the nearest 0.5 g.

5.2.11 Container No. 7 (Acetone Blank). If particulate emissions are to be determined, at least once during each field test, place a 100-ml portion of the acetone used in the sample recovery process into a container labeled No. 7. Seal the container.

5.2.12 Container No. 8A (0.1 N HNO<sub>3</sub> Blank). At least once during each field test, place 300 ml of the 0.1 N HNO<sub>3</sub> solution used in the sample recovery process into a container labeled No. 8A. Seal the container.

5.2.13 Container No. 8B (Water Blank). At least once during each field test, place 100 ml of the water used in the sample recovery process into a container labeled No. 8B. Seal the container.

5.2.14 Container No. 9 (5 Percent HNO<sub>3</sub>/10 Percent H<sub>2</sub>O<sub>2</sub> Blank). At least once during each field test, place 200 ml of the 5 Percent HNO<sub>3</sub>/10 Percent H<sub>2</sub>O<sub>2</sub> solution used as the nitric acid Impinger reagent into a container labeled No. 9. Seal the container.

5.2.15 Container No. 10 (Acidified KMnO<sub>4</sub> Blank). At least once during each field test, place 100 ml of the acidified KMnO<sub>4</sub> solution used as the Impinger solution and in the sample recovery process into a container labeled No. 10. Prepare the container as described in Section 5.2.9.2. Read the Precaution: in Section 4.3.2. and read the *NOTE* in Section 5.2.9.2.

5.2.16 Container No. 11 (8 N HCl Blank). At least once during each field test, place 200 ml of water into a sample container labeled No. 11. Then carefully add with stirring 25 ml of 8 N HCl. Mix well and seal the container.

5.2.17 Container No. 12 (Sample Filter Blank). Once during each field test, place into a petri dish labeled No. 12 three unused blank filters from the same lot as the sampling filters. Seal the petri dish.

5.3 *Sample Preparation.* Note the level of the liquid in each of the containers and determine if any sample was lost during shipment. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. A diagram illustrating sample preparation and analysis procedures for each of the sample train components is shown in Figure 29-3.

#### 5.3.1 *Container No. 1 (Sample Filter).*

5.3.1.1 If particulate emissions are being determined, first desiccate the filter and filter catch without added heat (do not heat the filters to speed the drying) and weigh to a constant weight as described in Section 4.3 of Method 5.

5.3.1.2 Following this procedure, or initially, if particulate emissions are not being determined in addition to metals analysis, divide the filter with its filter catch into portions containing approximately 0.5 g

each. Place the pieces in the analyst's choice of either individual microwave pressure relief vessels or Parr<sup>®</sup> Bombs. Add 6 ml of concentrated HNO<sub>3</sub> and 4 ml of concentrated HF to each vessel. For microwave heating, microwave the samples for approximately 12 to 15 minutes total heating time as follows: heat for 2 to 3 minutes, then turn off the microwave for 2 to 3 minutes, then heat for 2 to 3 minutes, etc., continue this alternation until the 12 to 15 minutes total heating time are completed (this procedure should comprise approximately 24 to 30 minutes at 600 watts). Microwave heating times are approximate and are dependent upon the number of samples being digested simultaneously. Sufficient heating is evidenced by sorbent reflux within the vessel. For conventional heating, heat the Parr<sup>®</sup> Bombs at 140 °C (285 °F) for 6 hours. Then cool the samples to room temperature, and combine with the acid digested probe rinse as required in Section 5.3.3.

5.3.1.3 If the sampling train includes an optional glass cyclone in front of the filter,

prepare and digest the cyclone catch by the procedures described in section 5.3.1.2 and then combine the digestate with the digested filter sample.

5.3.2 Container No. 2 (Acetone Rinse). Note the level of liquid in the container and confirm on the analysis sheet whether or not leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in this container either volumetrically within 1 ml or gravimetrically within 0.5 g. Transfer the contents to an acid-cleaned, tared 250-ml beaker and evaporate to dryness at ambient temperature and pressure. If particulate emissions are being determined, desiccate for 24 hours without added heat, weigh to a constant weight according to the procedures described in Section 4.3 of Method 5, and report the results to the nearest 0.1 mg. Redissolve the residue with 10 ml of concentrated HNO<sub>3</sub>.

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Quantitatively combine the resultant sample, including all liquid and any particulate matter, with Container No. 3 before beginning Section 5.3.3.

5.3.3 Container No. 3 (Probe Rinse). Verify that the pH of this sample is 2 or lower. If it is not, acidify the sample by careful addition with stirring of concentrated HNO<sub>3</sub> to pH 2.

Use water to rinse the sample into a beaker, and cover the beaker with a ribbed watch glass. Reduce the sample volume to approximately 20 ml by heating on a hot plate at a temperature just below boiling. Digest the sample in microwave vessels or Parr<sup>®</sup> Bombs by quantitatively transferring the sample to the vessel or bomb, carefully adding the 6 ml of concentrated HNO<sub>3</sub>, 4 ml of concentrated HF, and then continuing to follow the procedures described in Section 5.3.1.2. Then combine the resultant sample directly with the acid digested portions of the filter prepared previously in Section 5.3.1.2. The resultant combined sample is referred to as "Sample Fraction 1". Filter the combined sample using Whatman 541 filter paper. Dilute to 300 ml (or the appropriate volume for the expected metals concentration) with water. This diluted sample is "Analytical Fraction 1". Measure and record the volume of Analytical Fraction 1 to within 0.1 ml. Quantitatively remove a 50-ml aliquot and label as "Analytical Fraction 1B". Label the remaining 250-ml portion as "Analytical Fraction 1A". Analytical Fraction 1A is used for ICAP or AAS analysis for all desired metals except Hg. Analytical Fraction 1B is used for the determination of front-half Hg.

5.3.4 Container No. 4 (Impingers 1-3). Measure and record the total volume of this sample to within 0.5 ml and label it "Sample Fraction 2". Remove a 75- to 100-ml aliquot for Hg analysis and label the aliquot "Analytical Fraction 2B". Label the remaining portion of Container No. 4 as "Sample Fraction 2A". Sample Fraction 2A defines the volume of Analytical Fraction 2A prior to digestion. All of Sample Fraction 2A is digested to produce "Analytical Fraction 2A". Analytical Fraction 2A defines the volume of Sample Fraction 2A after its digestion and the volume of Analytical Fraction 2A is normally 150 ml. Analytical Fraction 2A is analyzed for all metals except Hg. Verify that the pH of Sample Fraction 2A is 2 or lower. If necessary, use concentrated HNO<sub>3</sub> by careful addition and stirring to lower Sample Fraction 2A to pH 2. Use water to rinse Sample Fraction 2A into a beaker and then cover the beaker with a ribbed watch glass. Reduce Sample Fraction 2A to approximately 20 ml by heating on a hot plate at a temperature just below boiling. Then follow either of the digestion procedures described in Sections 5.3.4.1 or 5.3.4.2.

5.3.4.1 Conventional Digestion Procedure. Add 30 ml of 50 percent HNO<sub>3</sub> and heat for 30 minutes on a hot plate to just below boiling. Add 10 ml of 3 percent H<sub>2</sub>O<sub>2</sub> and heat for 10 more minutes. Add 50 ml of hot water, and heat the sample for an additional 20 minutes. Cool, filter the sample, and dilute to 150 ml (or the appropriate volume for the expected metals concentrations) with water. This dilution produces Analytical Fraction 2A.

Measure and record the volume to within 0.1 ml.

5.3.4.2 Microwave Digestion Procedure. Add 10 ml of 50 percent HNO<sub>3</sub> and heat for 8 minutes total heating time in alternations of 1 to 2 minutes at 600 Watts followed by 1 to 2 minutes with no power, etc., similar to the procedure described in Section 5.3.1. Allow the sample to cool. Add 10 ml of 3 percent H<sub>2</sub>O<sub>2</sub> and heat for 2 more minutes. Add 50 ml of hot water, and heat for an additional 5 minutes. Cool, filter the sample, and dilute to 150 ml (or the appropriate volume for the expected metals concentrations) with water. This dilution produces Analytical Fraction 2A. Measure and record the volume to within 0.1 ml.

(NOTE: All microwave heating times given are approximate and are dependent upon the number of samples being digested at a time. Heating times as given above have been found acceptable for simultaneous digestion of up to 12 individual samples. Sufficient heating is evidenced by solvent reflux within the vessel.)

5.3.5 Container No. 5A (Impinger 4), Container Nos. 5B and 5C (Impingers 5 and 8). Keep the samples in Containers Nos. 5A, 5B, and 5C separate from each other. Measure and record the volume of 5A to within 0.5 ml. Label the contents of Container No. 5A to be Analytical Fraction 3A. To remove any brown MnO<sub>2</sub> precipitate from the contents of Container No. 5B, filter its contents through Whatman 40 filter paper into a 500 ml volumetric flask and dilute to volume with water. Save the filter for digestion of the brown MnO<sub>2</sub> precipitate. Label the 500 ml filtrate from Container No. 5B to be Analytical Fraction 3B. Analyze Analytical Fraction 3B for Hg within 48 hours of the filtration step. Place the saved filter, which was used to remove the brown MnO<sub>2</sub> precipitate, into an appropriately sized vented container, which will allow release of any gases including chlorine formed when the filter is digested. In a laboratory hood which will remove any gas produced by the digestion of the MnO<sub>2</sub>, add 25 ml of 8 N HCl to the filter and allow to digest for a minimum of 24 hours at room temperature. Filter the contents of Container No. 5C through a Whatman 40 filter into a 500-ml volumetric flask. Then filter the result of the digestion of the brown MnO<sub>2</sub> from Container No. 5B through a Whatman 40 filter into the same 500-ml volumetric flask, and dilute and mix well to volume with water. Discard the Whatman 40 filter. Mark this combined 500-ml dilute HCl solution as Analytical Fraction 3C.

5.3.6 Container No. 6 (Silica Gel). Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance.

5.4 Sample Analysis. For each sampling train sample run, seven individual analytical samples are generated; two for all desired

metals except Hg, and five for Hg. A schematic identifying each sample container and the prescribed analytical preparation and analysis scheme is shown in Figure 29-3. The first two analytical samples, labeled Analytical Fractions 1A and 1B, consist of the digested samples from the front-half of the train. Analytical Fraction 1A is for ICAP, ICP-MS or AAS analysis as described in Sections 5.4.1 and 5.4.2, respectively. Analytical Fraction 1B is for front-half Hg analysis as described in Section 5.4.3. The contents of the back-half of the train are used to prepare the third through seventh analytical samples. The third and fourth analytical samples, labeled Analytical Fractions 2A and 2B, contain the samples from the moisture removal impinger No. 1, if used, and HNO<sub>3</sub>, H<sub>2</sub>O<sub>2</sub> impingers Nos. 2 and 3. Analytical Fraction 2A is for ICAP, ICP-MS or AAS analysis for target metals, except Hg. Analytical Fraction 2B is for analysis for Hg. The fifth through seventh analytical samples, labeled Analytical Fractions 3A, 3B, and 3C, consist of the impinger contents and rinses from the empty impinger No. 4 and the H<sub>2</sub>SO<sub>4</sub>/KMnO<sub>4</sub> Impingers Nos. 5 and 6. These analytical samples are for analysis for Hg as described in Section 5.4.3. The total back-half Hg catch is determined from the sum of Analytical Fractions 2B, 3A, 3B, and 3C. Analytical Fractions 1A and 2A can be combined proportionally prior to analysis.

5.4.1 ICAP and ICP-MS Analysis. Analyze Analytical Fractions 1A and 2A by ICAP using Method 6010 or Method 200.7 (40 CFR part 136, appendix C). Calibrate the ICAP, and set up an analysis program as described in Method 6010 or Method 200.7. Follow the quality control procedures described in Section 7.3.1. Recommended wavelengths for analysis are as follows:

Element	Wave-length (nm)
Aluminum .....	308.215
Antimony .....	206.833
Arsenic .....	193.686
Barium .....	455.403
Beryllium .....	313.042

Element	Wave-length (nm)
Cadmium .....	226.502
Chromium .....	267.716
Cobalt .....	228.816
Copper .....	324.754
Iron .....	259.940
Lead .....	220.353
Manganese .....	267.810
Nickel .....	231.604
Phosphorus .....	214.914
Selenium .....	188.028
Silver .....	328.058
Thallium .....	190.864
Zinc .....	213.856

These wavelengths represent the best combination of specificity and potential detection limit. Other wavelengths may be substituted if they can provide the needed specificity and detection limit, and are treated with the same corrective techniques for spectral interference. Initially, analyze all samples for the target metals (except Hg) plus Fe and Al. If Fe and Al are present, the sample might have to be diluted so that each of these elements is at a concentration of less than 50 ppm so as to reduce their spectral interferences on As, Cd, Cr, and Pb. Perform ICP-MS analysis by following Method 6020 in EPA Publication SW-846 Third Edition (November 1986) including updates I, II, IIA, and IIB, as incorporated by reference in §60.17(i).

(NOTE: When analyzing samples in a HF matrix, an alumina torch should be used; since all front-half samples will contain HF, use an alumina torch.)

5.4.2 AAS by Direct Aspiration and/or GFAAS. If analysis of metals in Analytical Fractions 1A and 2A by using GFAAS or direct aspiration AAS is needed, use Table 29-2 to determine which techniques and procedures to apply for each target metal. Use Table 29-2, if necessary, to determine techniques for minimization of interferences. Calibrate the instrument according to Section 6.3 and follow the quality control procedures specified in Section 7.3.2.

TABLE 29-2.—APPLICABLE TECHNIQUES, METHODS AND MINIMIZATION OF INTERFERENCE FOR AAS ANALYSIS

Metal	Technique	SW-846* method No.	Wavelength (nm)	Interferences	
				Cause	Minimization
Fe .....	Aspiration .....	7380	248.3	Contamination .....	Great care taken to avoid contamination. Background correction required.
Pb .....	Aspiration .....	7420	283.3	217.0 nm alternate .....	
Pb .....	Furnace .....	7421	283.3	Poor recoveries .....	
Mn .....	Aspiration .....	7460	279.5	403.1 nm alternate .....	Matrix modifier, add 10 µl of phosphorus acid to 1 ml of prepared sample in sampler cup. Background correction required.

TABLE 29-2.—APPLICABLE TECHNIQUES, METHODS AND MINIMIZATION OF INTERFERENCE FOR AAS ANALYSIS—Continued

Metal	Technique	SW-8461 method No.	Wavelength (nm)	Interferences	
				Cause	Minimization
Ni	Aspiration	7620	232.0	382.4 nm alternate Fe, Co, and Cr.	Background correction required. Matrix matching or nitrous oxide/acetylene flame, sample dilution or use 352.3 nm line.
				Nonlinear response	
Se	Furnace	7740	196.0	Volatility	Spike samples and reference materials and add nickel nitrate to minimize volatilization. Background correction is required and Zeeman background correction can be useful.
				Adsorption & scatter	
Ag	Aspiration	7760	328.1	Adsorption & Scatter AgCl insoluble.	Background correction is required. Avoid Hydrochloric acid unless silver is in solution as a chloride complex Sample and standards monitored for aspiration rate.
Tl	Aspiration	7840	276.8		Background correction is required. Hydrochloric acid should not be used.
Tl	Furnace	7841	276.8	Hydrochloric acid or chloride.	Background correction is required. Verify that losses are not occurring for volatilization by spiked samples or standard addition; Palladium is a suitable matrix modifier.
Zn	Aspiration	7850	213.9	High Si, Cu, & P Contamination.	Strontium removes Cu and phosphate, Great care taken to avoid contamination.
Sb	Aspiration	7040	217.8	1000 mg/ml Pb Ni, Cu, or acid.	Use secondary wavelengths of 231.1 nm; match sample & standards acid concentration or use nitrous oxide/acetylene flame.
Sb	Furnace	7041	217.8	High Pb	Secondary Wavelength or Zeeman correction.
As	Furnace	7060	193.7	Arsenic volatilization Aluminum	Spiked samples and add nickel nitrate solution to digestates prior to analysis. Use Zeeman background correction.
Ba	Aspiration 7080	7080	553.8	Calcium	High hollow cathode current and narrow band set. 2 ml of KCl per 100 ml of sample.
				Barium ionization	
Be	Aspiration	7090	234.9	500 ppm Al High Mg and Si.	Add 0.1% fluoride. Use method of standard additions.
Be	Furnace	7091	234.9	Be in optical path	Optimize parameters to minimize effects.
Cd	Aspiration	7130	228.8	Absorption and light scattering.	Background correction is required.
Cd	Furnace	7131	228.8	As above	As above. Ammonium phosphate used as a matrix modifier. Use cadmium-free tips.
				Excess Chloride Pipet tips	

TABLE 29-2.—APPLICABLE TECHNIQUES, METHODS AND MINIMIZATION OF INTERFERENCE FOR AAS ANALYSIS—Continued

Metal	Technique	SW-846 <sup>1</sup> method No.	Wavelength (nm)	Interferences	
				Cause	Minimization
Cr .....	Aspiration .....	7190	357.9	Alkali metal .....	KCl ionization suppressant in samples and standards—Consult mfgs literature.
Co .....	Furnace .....	7201	240.7	Excess chloride .....	Use Method of Standard Additions.
Cr .....	Furnace .....	7191	357.9	200 mg/L Ca and P .....	All calcium nitrate for a known constant effect and to eliminate effect of phosphate.
Cu .....	Aspiration .....	7210	324.7	Absorption & scatter .....	Consult manufacturer's manual.

<sup>1</sup> Refer to EPA publication SW-846 Third Edition (November 1986) including updates I, II, IIA, and IIB, as incorporated by reference in § 60.17(f).

5.4.3 CVAAS Hg analysis. Analyze Analytical Fractions 1B, 2B, 3A, 3B, and 3C separately for Hg using CVAAS following the method outlined in Method 7470 in EPA Publication SW-848 Third Edition (November 1986) including updates I, II, IIA and IIB, as incorporated by reference in § 60.17(i) or in *Standard Methods for the Examination of Water and Wastewater*, 16th Edition, (1985), Method 303F, as incorporated by reference in § 60.17, or, optionally using *NOTE No. 2* in this section. Set up the calibration curve (zero to 1000 ng) as described in Method 7470 or similar to Method 303F using 300-ml BOD bottles instead of Erlenmeyers. Perform the following for each Hg analysis. From each original sample, select and record an aliquot in the size range from 1 ml to 10 ml. If no prior knowledge of the expected amount of Hg in the sample exists, a 5 ml aliquot is suggested for the first dilution to 100 ml (see *NOTE No. 1* in this Section). The total amount of Hg in the aliquot shall be less than 1 µg and within the range (zero to 1000 ng) of the calibration curve. Place the sample aliquot into a separate 300-ml BOD bottle, and add enough water to make a total volume of 100 ml. Next add to it sequentially the sample digestion solutions and perform the sample preparation described in the procedures of Method 7470 or Method 303F. (See *NOTE No. 2* in this Section). If the maximum readings are off-scale (because Hg in the aliquot exceeded the calibration range; including the situation where only a 1-ml aliquot of the original sample was digested), then dilute the original sample (or a portion of it) with 0.15 percent HNO<sub>3</sub> (1.5 ml concentrated HNO<sub>3</sub> per liter aqueous solution) so that when a 1- to 10-ml aliquot of the "0.15 HNO<sub>3</sub> percent dilution of the original sample" is digested and analyzed by the procedures described above, it will yield an analysis within the range of the calibration curve.

*NOTE NO. 1 TO SECTION 5.4.3.* When Hg levels in the sample fractions are below the in-stack detection limit given in Table 29-1, select a 10 ml aliquot for digestion and analysis as described.

*NOTE NO. 2 TO SECTION 5.4.3.* Optionally, Hg can be analyzed by using the CVAAS analytical procedures given by some instrument manufacturer's directions. These include calibration and quality control procedures for the Leeman Model PS200, the Perkin Elmer FIAS systems, and similar models, if available, of other instrument manufacturers. For digestion and analyses by these instruments, perform the following two steps:

(1) Digest the sample aliquot through the addition of the aqueous hydroxylamine hydrochloride/sodium chloride solution the same as described in this Section 5.4.3.: (*The Leeman, Perkin Elmer, and similar instruments described in this note add automatically the necessary stannous chloride solution during the automated analysis of Hg.*) and

(2) Upon completion of the digestion described in paragraph (1), of this note, analyze the sample according to the instrument manufacturer's directions. This approach allows multiple (including duplicate) automated analyses of a digested sample aliquot.

#### 6. Calibration

Maintain a laboratory log of all calibrations.

6.1 Sampling Train Calibration. Calibrate the sampling train components according to the indicated sections of Method 5: Probe Nozzle (Section 5.1); Pitot Tube (Section 5.2); Metering System (Section 5.3); Probe Heater (Section 5.4); Temperature Gauges (Section 5.5); Leake-Check of the Metering System (Section 5.6); and Barometer (Section 5.7).

6.2 Industrially Coupled Argon Plasma Spectrometer Calibration. Prepare standards

as outlined in Section 4.5. Profile and calibrate the instrument according to the manufacturer's recommended procedures using those standards. Check the calibration once per hour. If the instrument does not reproduce the standard concentrations within 10 percent, perform the complete calibration procedures. Perform ICP-MS analysis by following Method 6020 in EPA Publication SW-846 Third Edition (November 1986) including updates I, II, IIA and IIB, as incorporated by reference in §60.17(l).

6.3 Atomic Absorption Spectrometer—Direct Aspiration AAS, GFAAS, and CVAAS analyses. Prepare the standards as outlined in Section 4.5 and use them to calibrate the spectrometer. Calibration procedures are also outlined in the EPA methods referred to in Table 29-2 and in Method 7470 in EPA Publication SW-846 Third Edition (November 1986) including updates I, II, IIA and IIB, as incorporated by reference in §60.17(l) or in *Standard Methods for the Examination of Water and Wastewater*, 16th Edition, (1985), Method 303F (for Hg) as incorporated by reference in §60.17. Run each standard curve in duplicate and use the mean values to calculate the calibration line. Recalibrate the instrument approximately once every 10 to 12 samples.

#### 7. Quality Control

7.1 Field Reagent Blanks, if analyzed. Perform the digestion and analysis of the blanks in Container Nos. 7 through 12 that were produced in Sections 5.2.11 through 5.2.17, respectively. For Hg field reagent blanks, use a 10 ml aliquot for digestion and analysis.

7.1.1 Digest and analyze one of the filters from Container No. 12 per Section 5.3.1, 100 ml from Container No. 7 per Section 5.3.2, and 100 ml from Container No. 8A per Section 5.3.3. This step produces blanks for Analytical Fractions 1A and 1B.

7.1.2 Combine 100 ml of Container No. 8A with 200 ml from Container No. 9, and digest and analyze the resultant volume per Section 5.3.4. This step produces blanks for Analytical Fractions 2A and 2B.

7.1.3 Digest and analyze a 100-ml portion of Container No. 8A to produce a blank for Analytical Fraction 3A.

7.1.4 Combine 100 ml from Container No. 10 with 33 ml from Container No. 8B to produce a blank for Analytical Fraction 3B. Filter the resultant 133 ml as described for Container No. 5B in Section 5.3.5, except do not dilute the 133ml. Analyze this blank for Hg within 48 hrs. of the filtration step, and use 400 ml as the blank volume when calculating the blank mass value. Use the actual volumes of the other analytical blanks when calculating their mass values.

7.1.5 Digest the filter that was used to remove any brown MnO<sub>2</sub> precipitate from the blank for Analytical Fraction 3B by the same procedure as described in Section 5.3.5

for the similar sample filter. Filter the digestate and the contents of Container No. 11 through Whatman 40 paper into a 500-ml volumetric flask, and dilute to volume with water. These steps produce a blank for Analytical Fraction 3C.

7.1.6 Analyze the blanks for Analytical Fraction Blanks 1A and 2A per Section 5.4.1 and/or Section 5.4.2. Analyze the blanks for Analytical Fractions 1B, 2B, 3A, 3B, and 3C per Section 5.4.3. Analysis of the blank for Analytical Fraction 1A produces the front-half reagent blank correction values for the desired metals except for Hg; Analysis of the blank for Analytical Fraction 1B produces the front-half reagent blank correction value for Hg. Analysis of the blank for Analytical Fraction 2A produces the back-half reagent blank correction values for all of the desired metals except for Hg, while separate analyses of the blanks for Analytical Fractions 2B, 3A, 3B, and 3C produce the back-half reagent blank correction value for Hg.

7.2 Quality Control Samples. Analyze the following quality control samples.

7.2.1 ICAP and ICP-MS Analysis. Follow the respective quality control descriptions in Section 8 of Methods 6010 and 6020 of EPA Publication SW-846 Third Edition (November 1986) including updates I, II, IIA and IIB, as incorporated by reference in §60.17(l). For the purposes of a source test that consists of three sample runs, modify those requirements to include the following: two instrument check standard runs, two calibration blank runs, one interference check sample at the beginning of the analysis (analyze by Method of Standard Additions unless within 25 percent), one quality control sample to check the accuracy of the calibration standards (required to be within 25 percent of calibration), and one duplicate analysis (required to be within 20 percent of average or repeat all analyses).

7.2.2 Direct Aspiration AAS and/or GFAAS Analysis for Sb, As, Ba, Be, Cd, Cu, Cr, Co, Pb, Ni, Mn, Hg, P, Se, Ag, Tl, and Zn. Analyze all samples in duplicate. Perform a matrix spike on at least one front-half sample and one back-half sample, or one combined sample. If recoveries of less than 75 percent or greater than 125 percent are obtained for the matrix spike, analyze each sample by the Method of Standard Additions. Analyze a quality control sample to check the accuracy of the calibration standards. If the results are not within 20 percent, repeat the calibration.

7.2.3 CVAAS Analysis for Hg. Analyze all samples in duplicate. Analyze a quality control sample to check the accuracy of the calibration standards (if not within 15 percent, repeat calibration). Perform a matrix spike on one sample (if not within 25 percent, analyze all samples by the Method of Standard Additions). Additional information on quality control can be obtained from Method

7470 of EPA Publication SW-846 Third Edition (November 1986) including updates I, II, IIA and IIB, as incorporated by reference in §60.17(i) or in *Standard Methods for the Examination of Water and Wastewater*, 16th Edition, (1985), Method 303F as incorporated by reference in §60.17.

### 8. Calculations

8.1 Dry Gas Volume. Using the data from this test, calculate  $V_{\text{std (std)}}$ , the dry gas sample volume at standard conditions as outlined in Section 6.3 of Method 5.

8.2 Volume of Water Vapor and Moisture Content. Using the total volume of condensate collected during the source sampling, calculate the volume of water vapor  $V_w$  (std) and the moisture content  $B_w$  of the stack gas. Use Equations 5-2 and 5-3 of Method 5.

8.3 Stack Gas Velocity. Using the data from this test and Equation 2-9 of Method 2, calculate the average stack gas velocity.

8.4 Metals (Except Hg) in Source Sample.

8.4.1 Analytical Fraction 1A, Front-Half, Metals (except Hg). Calculate separately the amount of each metal collected in Sample Fraction 1 of the sampling train using the following equation:

$$M_{\text{fb}} = C_{\text{a1}} F_d V_{\text{std,1}} \quad \text{Eq. 29-1}$$

where:

$M_{\text{fb}}$  = Total mass of each metal (except Hg) collected in the front half of the sampling train (Sample Fraction 1),  $\mu\text{g}$ .

$C_{\text{a1}}$  = Concentration of metal in Analytical Fraction 1A as read from the standard curve,  $\mu\text{g/ml}$ .

$F_d$  = Dilution factor ( $F_d$  = the inverse of the fractional portion of the concentrated sample in the solution actually used in the instrument to produce the reading  $C_{\text{a1}}$ . For example, if a 2 ml aliquot of Analytical Fraction 1A is diluted to 10 ml to place it in the calibration range,  $F_d = 5$ ).

$V_{\text{std,1}}$  = Total volume of digested sample solution (Analytical Fraction 1), ml.

8.4.1.1 If Analytical Fractions 1A and 2A are combined, use proportional aliquots. Then make appropriate changes in Equations 29-1 through 29-3 to reflect this approach.

8.4.2 Analytical Fraction 2A, Back-Half, Metals (except Hg). Calculate separately the amount of each metal collected in Fraction 2 of the sampling train using the following equation.

$$M_{\text{bb}} = C_{\text{a2}} F_a V_a \quad \text{Eq. 29-2}$$

where:

$M_{\text{bb}}$  = Total mass of each metal (except Hg) collected in the back-half of the sampling train (Sample Fraction 2),  $\mu\text{g}$ .

$C_{\text{a2}}$  = Concentration of metal in Analytical Fraction 2A as read from the standard curve, ( $\mu\text{g/ml}$ ).

$F_a$  = Aliquot factor, volume of Sample Fraction 2 divided by volume of Sample Fraction 2A (see Section 5.3.4.)

$V_a$  = Total volume of digested sample solution (Analytical Fraction 2A), ml (see Section 5.3.4.1 or 5.3.4.2, as applicable).

8.4.3 Total Train, Metals (except Hg). Calculate the total amount of each of the quantified metals collected in the sampling train as follows:

$$M_t = (M_{\text{fb}} - M_{\text{bb}}) + (M_{\text{bb}} - M_{\text{bbb}}) \quad \text{Eq. 29-3}$$

where:

$M_t$  = Total mass of each metal (separately stated for each metal) collected in the sampling train,  $\mu\text{g}$ .

$M_{\text{bb}}$  = Blank correction value for mass of metal detected in front-half field reagent blank,  $\mu\text{g}$ .

$M_{\text{bbb}}$  = Blank correction value for mass of metal detected in back-half field reagent blank,  $\mu\text{g}$ .

8.4.3.1 If the measured blank value for the front half ( $M_{\text{bb}}$ ) is in the range 0.0 to "A"  $\mu\text{g}$  [where "A"  $\mu\text{g}$  equals the value determined by multiplying 1.4  $\mu\text{g}/\text{in.}^2$  times the actual area in  $\text{in.}^2$  of the sample filter], use  $M_{\text{bb}}$  to correct the emission sample value ( $M_{\text{fb}}$ ); if  $M_{\text{bb}}$  exceeds "A"  $\mu\text{g}$ , use the greater of I or II:

- I. "A"  $\mu\text{g}$ .
- II. the lesser of (a)  $M_{\text{fb}}$ , or (b) 5 percent of  $M_{\text{fb}}$ .

If the measured blank value for the back-half ( $M_{\text{bbb}}$ ) is in the range 0.0 to 1  $\mu\text{g}$ , use  $M_{\text{bbb}}$  to correct the emission sample value ( $M_{\text{bb}}$ ); if  $M_{\text{bbb}}$  exceeds 1  $\mu\text{g}$ , use the greater of I or II:

- I. 1  $\mu\text{g}$ .
- II. the lesser of (a)  $M_{\text{bb}}$  or (b) 5 percent of  $M_{\text{bb}}$ .

8.5 Hg in Source Sample.

8.5.1 Analytical Fraction 1B; Front-Half Hg. Calculate the amount of Hg collected in the front-half, Sample Fraction 1, of the sampling train by using Equation 29-4:

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where:

$Hg_{\text{fb}}$  = Total mass of Hg collected in the front-half of the sampling train (Sample Fraction 1),  $\mu\text{g}$ .

$Q_{\text{fb}}$  = Quantity of Hg,  $\mu\text{g}$ , TOTAL in the ALIQUOT of Analytical Fraction 1B selected for digestion and analysis.

8.5.1.1 For example, if a 10 ml aliquot of Analytical Fraction 1B is taken and digested and analyzed (according to Section 5.4.3 and its NOTES Nos. 1 and 2), then calculate and use the total amount of Hg in the 10 ml aliquot for  $Q_{\text{fb}}$ .

$V_{\text{std,1}}$  = Total volume of Analytical Fraction 1, ml.

$V_{\text{FB}}$  = Volume of aliquot of Analytical Fraction 1B analyzed, ml.

8.5.1.2 For example, if a 1 ml aliquot of Analytical Fraction 1B was diluted to 50 ml

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with 0.15 percent HNO<sub>3</sub> as described in Section 5.4.3 to bring it into the proper analytical range, and then 1 ml of that 50-ml wa digested according to Section 5.4.3 and analyzed, V<sub>DB</sub> would be 0.02 ml.

8.5.2 Analytical Fractions 2B, 3A, 3B, and 3C: Back Half Hg.

8.5.2.1 Calculate the amount of Hg collected in Sample Fraction 2 by using Equation 29-5:

ER25AP86.006

where:

Hg<sub>2B2</sub>=Total mass of Hg collected in Sample Fraction 2, µg.

Q<sub>2B2</sub>=Quantity of Hg, µg, TOTAL in the ALIQUOT of Analytical Fraction 2B selected for digestion and analysis.

ER25AP86.007

where:

Hg<sub>3(A,B,C)}</sub>=Total mass of Hg collected separately in Fraction 3A, 3B, or 3C, µg.

Q<sub>3(A,B,C)}</sub>=Quantity of Hg, µg, TOTAL, separately, in the ALIQUOT of Analytical Fraction 3A, 3B, and 3C selected for digestion and analysis, (see previous notes in Sections 8.5.1 and 8.5.2 describing the quantity "Q" and calculate similarly).

V<sub>3(A,B,C)}</sub>=Volume, separately, of Analytical Fraction 3A, 3B, or 3C analyzed, ml (see previous notes in Sections 8.5.1 and 8.5.2, describing the quantity "V" and calculate similarly).

V<sub>2B2(A,B,C)}</sub>=Total volume, separately, of Analytical Fraction 3A, 3B, or 3C, ml.

8.5.2.3 Calculate the total amount of Hg collected in the back-half of the sampling train by using Equation 29-7:

$$Hg_{2B} = Hg_{2B2} + Hg_{2B1A} + Hg_{2B1B} + Hg_{2B1C} \quad \text{Eq. 29-7}$$

where:

Hg<sub>2B</sub>=Total mass of Hg collected in the back-half of the sampling train, µg.

8.5.3 Total Train Hg Catch. Calculate the total amount of Hg collected in the sampling train by using Equation 29-8:

$$Hg_T = (Hg_{2B} - Hg_{2Bb}) + (Hg_{2Bb} - Hg_{2Bbb}) \quad \text{Eq. 29-8}$$

where:

Hg<sub>T</sub>=Total mass of Hg collected in the sampling train, µg.

Hg<sub>2Bb</sub>=Blank correction value for mass of Hg detected in front-half field reagent blank, µg.

8.5.2.1.1 For example, if a 10 ml aliquot of Analytical Fraction 2B is taken and digested and analyzed (according to Section 5.4.3 and its NOTES Nos. 1 and 2), then calculate and use the total amount of Hg in the 10 ml aliquot for Q<sub>2B2</sub>.

V<sub>2B2</sub>=Total volume of Sample Fraction 2, ml.

V<sub>DB</sub>=Volume of Analytical Fraction 2B analyzed, ml.

8.5.2.1.2 For example, if 1 ml of Analytical Fraction 2B was diluted to 10 ml with 0.15 percent HNO<sub>3</sub> as described in Section 5.4.3 to bring it into the proper analytical range, and then 5 ml of that 10-ml was analyzed, V<sub>DB</sub> would be 0.5 ml.

8.5.2.2 Calculate each of the back-half Hg values for Analytical Fractions 3A, 3B, and 3C by using Equation 29-6:

Hg<sub>2Bb</sub>=Blank correction value for mass of Hg detected in back-half field reagent blanks, µg.

8.5.4 If the total of the measured blank values (Hg<sub>2Bb</sub>+Hg<sub>2Bbb</sub>) is in the range of 0.0 to 0.6 µg, then use the total to correct the sample value (Hg<sub>2B</sub>+Hg<sub>2Bb</sub>); if it exceeds 0.6 µg, use the greater of I, or II:

I. 0.6 µg.

II. the lesser of (a) (Hg<sub>2Bb</sub>+Hg<sub>2Bbb</sub>), or (b) 5 percent of the sample value (Hg<sub>2B</sub>+Hg<sub>2Bb</sub>).

8.6 Individual Metal Concentrations in Stack Gas. Calculate the concentration of each metal in the stack gas (dry basis, adjusted to standard conditions) by using Equation 29-9:

ER25AP86.008

C<sub>r</sub>=Concentration of a metal in the stack gas, mg/dscm.

K<sub>r</sub>=10<sup>-3</sup> mg/µg.

M<sub>r</sub>=Total mass of that metal collected in the sampling train, µg; (substitute Hg<sub>r</sub> for M<sub>r</sub> for the Hg calculation).

V<sub>std(dry)}</sub>=Volume of gas sample as measured by the dry gas meter, corrected to dry standard conditions, dscm.

8.7 Isokinetic Variation and Acceptable Results. Same as Method 5, Sections 6.11 and 6.12, respectively.

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9. Bibliography

1. Method 303F in *Standard Methods for the Examination of Water Wastewater*, 16th Edition, 1985. Available from the American Public Health Association, 1015 18th Street NW., Washington, DC 20036.

2. EPA Methods 6010, 6020, 7000, 7041, 7060, 7131, 7421, 7470, 7740, and 7841, *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*, SW-846, Third Edition, September 1986, with updates I, II, IIA and IIB. Office of Solid Waste and Emergency Response, U.S. Environmental Protection Agency, Washington, DC 20460.

3. EPA Method 200.7, *Code of Federal Regulations*, Title 40, Part 136, Appendix C. July 1, 1987.

4. EPA Methods 1 through 5, *Code of Federal Regulations*, Title 40, Part 60, Appendix A. July 1, 1991.

5. EPA Method 101A, *Code of Federal Regulations*, Title 40, Part 61, Appendix B. July 1, 1991.

[36 FR 24877, Dec. 23, 1971]

EDITORIAL NOTE: For FEDERAL REGISTER citations affecting part 60, appendix A see the List of CFR Sections in the Finding Aids section of this volume.

EFFECTIVE DATE NOTE: At 64 FR 26490, May 14, 1999, Appendix A to part 60 was amended by adding Methods 2F, 2G, and 2H and corrected at 64 FR 37186, July 9, 1999, and 64 FR 38241, July 15, 1999, effective July 13, 1999.

***40 CFR PART 60 APPENDIX B***

## APPENDIX B—PERFORMANCE SPECIFICATIONS

- Performance Specification 1—Specifications and test procedures for opacity continuous emission monitoring systems in stationary sources
- Performance Specification 2—Specifications and test procedures for SO<sub>2</sub> and NO<sub>x</sub> continuous emission monitoring systems in stationary sources
- Performance Specification 3—Specifications and test procedures for O<sub>2</sub> and CO<sub>2</sub> continuous emission monitoring systems in stationary sources
- Performance Specification 4—Specifications and test procedures for carbon monoxide continuous emission monitoring systems in stationary sources
- Performance Specification 4A—Specifications and test procedures for carbon monoxide continuous emission monitoring systems in stationary sources
- Performance Specification 5—Specifications and test procedures for TRS continuous emission monitoring systems in stationary sources
- Performance Specification 6—Specifications and test procedures for continuous emission rate monitoring systems in stationary sources
- Performance Specification 7—Specifications and test procedures for hydrogen sulfide continuous emission monitoring systems in stationary sources

### PERFORMANCE SPECIFICATION 1—SPECIFICATIONS AND TEST PROCEDURES FOR OPACITY CONTINUOUS EMISSION MONITORING SYSTEMS IN STATIONARY SOURCES

#### 1. Applicability and Principle

1.1 Applicability. This specification contains requirements for the design, performance, and installation of instruments for opacity continuous emission monitoring systems (CEMS's) and data computation procedures for evaluating the acceptability of a CEMS. Certain design requirements and test procedures established in this specification may not apply to all instrument designs. In such instances, equivalent design requirements and test procedures may be used with prior approval of the Administrator.

Performance Specification 1 (PS 1) applies to opacity monitors installed after March 30, 1983. Opacity monitors installed before March 30, 1983, are required to comply with the provisions and requirements of PS 1 except for the following:

- (a) Section 4. "Installation Specifications."
- (b) Sections 5.1.4, 5.1.6, 5.1.7, and 5.1.8 of Section 5, "Design and Performance Specifications."
- (c) Section 6.4 of Section 6 "Design Specifications Verification Procedure."

An opacity monitor installed before March 30, 1983, need not be tested to demonstrate compliance with PS 1 unless required by regulatory action other than the promulgation of PS 1. If an existing monitor is replaced with a new monitor, PS 1 shall apply except that the new monitor may be located at the old measurement location regardless of whether the location meets the requirements of Section 4. If a new measurement location is to be determined, the new location shall meet the requirements of Section 4.

1.2 Principle. The opacity of particulate matter in stack emissions is continuously monitored by a measurement system based upon the principle of transmissometry. Light having specific spectral characteristics is projected

from a lamp through the effluent in the stack or duct, and the intensity of the projected light is measured by a sensor. The projected light is attenuated because of absorption and scattered by the particulate matter in the effluent; the percentage of visible light attenuated is defined as the opacity of the emission. Transparent stack emissions that do not attenuate light will have a transmittance of 100 percent or an opacity of zero percent. Opaque stack emissions that attenuate all of the visible light will have a transmittance of zero percent or an opacity of 100 percent.

This specification establishes specific design criteria for the transmissometer system. Any opacity CEMS that is expected to meet this specification is first checked to verify that the design specifications are met. Then, the opacity CEMS is calibrated, installed, and operated for a specified length of time. During this specified time period, the system is evaluated to determine conformance with the established performance specifications.

#### 2. Definitions

2.1 Continuous Emission Monitoring System. The total equipment required for the determination of opacity. The system consists of the following major subsystems:

2.1.1 Sample Interface. That portion of CEMS that protects the analyzer from the effects of the stack effluent and aids in keeping the optical surfaces clean.

2.1.2 Analyzer. That portion of the CEMS that senses the pollutant and generates an output that is a function of the opacity.

2.1.3 Data Recorder. That portion of the CEMS that provides a permanent record of the analyzer output in terms of opacity. The data recorder may include automatic data-reduction capabilities.

2.2 Transmissometer. That portion of the CEMS that includes the sample interface and the analyzer.

2.3 Transmittance. The fraction of incident light that is transmitted through an optical medium.

2.4 Opacity. The fraction of incident light that is attenuated by an optical medium. Opacity (Op) and transmittance (Tr) are related by:  $Op = 1 - Tr$ .

2.5 Optical Density. A logarithmic measure of the amount of incident light attenuated. Optical density (D) is related to the transmittance and opacity as follows:  
 $D = -\log_{10} Tr = -\log_{10} (1 - Op)$ .

2.6 Peak Spectral Response. The wavelength of maximum sensitivity of the transmissometer.

2.7 Mean Spectral Response. The wavelength that is the arithmetic mean value of the wavelength distribution for the effective spectral response curve of the transmissometer.

2.8 Angle of View. The angle that contains all of the radiation detected by the photodetector assembly of the analyzer at a level greater than 2.5 percent of the peak detector response.

2.9 Angle of Projection. The angle that contains all of the radiation projected from the lamp assembly of the analyzer at a level of greater than 2.5 percent of the peak illuminance.

2.10 Span Value. The opacity value at which the CEMS is set to produce the maximum data display output as specified in the applicable subpart.

2.11 Upscale Calibration Value. The opacity value at which a calibration check of the CEMS is performed by simulating an upscale opacity condition as viewed by the receiver.

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2.12 Calibration Error. The difference between the opacity values indicated by the CEMS and the known values of a series of calibration attenuators (filters or screens).

2.13 Zero Drift. The difference in the CEMS output readings from the zero calibration value after a stated period of normal continuous operation during which no unscheduled maintenance, repair, or adjustment took place. A calibration value of 10 percent opacity or less may be used in place of the zero calibration value.

2.14 Calibration Drift. The difference in the CEMS output readings from the upscale calibration value after a stated period of normal continuous operation during which no unscheduled maintenance, repair, or adjustment took place.

2.15 Response Time. The amount of time it takes the CEMS to display on the data recorder 95 percent of a step change in opacity.

2.16 Conditioning Period. A period of time (168 hours minimum) during which the CEMS is operated without any unscheduled maintenance, repair, or adjustment prior to initiation of the operational test period.

2.17 Operational Test Period. A period of time (168 hours) during which the CEMS is expected to operate within the established performance specifications without any unscheduled maintenance, repair, or adjustment.

2.18 Path Length. The depth of effluent in the light beam between the receiver and the transmitter of a single-pass transmissometer, or the depth of effluent between the transceiver and reflector of a double-pass transmissometer. Two path lengths are referenced by this specification as follows:

2.18.1 Monitor Path Length. The path length (depth of effluent) at the installed location of the CEMS.

2.18.2 Emission Outlet Path Length. The path length (depth of effluent) at the location where emissions are released to the atmosphere. For noncircular outlets,  $D_o = (2LW) + (L+W)$ , where L is the length of the outlet and W is the width of the outlet. Note that this definition does not apply to pressure baghouse outlets with multiple stacks, side discharge vents, ridge roof monitors, etc.

**3. Apparatus**

3.1 Opacity Continuous Emission Monitoring System. Any opacity CEMS that is expected to meet the design and performance specifications in Section 5 and a suitable data recorder, such as an analog strip chart recorder or other suitable device (e.g., digital computer) with an input signal range compatible with the analyzer output.

3.2 Calibration Attenuators. Minimum of three. These attenuators must be optical filters or screens with neutral spectral characteristics selected and calibrated according to the procedures in Sections 7.1.2 and 7.1.3, and of suffi-

cient size to attenuate the entire light beam received by the detector of the transmissometer.

3.3 Upscale Calibration Value Attenuator. An optical filter with neutral spectral characteristics, a screen, or other device that produces an opacity value (corrected for path length, if necessary) that is greater than or equal to the applicable opacity standard but less than or equal to one-half the applicable instrument span value.

3.4 Calibration Spectrophotometer. A laboratory spectrophotometer meeting the following minimum design specifications:

Parameter	Specification
Wavelength range .....	400-700 nm.
Detector angle of view .....	<10°.
Accuracy .....	<0.5 percent transmittance, NBS traceable calibration.

**4. Installation Specifications**

Install the CEMS at a location where the opacity measurements are representative of the total emissions from the affected facility. These requirements can be met as follows:

4.1 Measurement Location. Select a measurement location that is (a) downstream from all particulate control equipment, (b) where condensed water vapor is not present, (c) free of interference from ambient light (applicable only if transmissometer is responsive to ambient light), and (d) accessible in order to permit routine maintenance. Accessibility is an important criterion because easy access for lens cleaning, alignment checks, calibration checks, and blower maintenance will help assure quality data.

4.2 Measurement Path. The primary concern in locating a transmissometer is determining a location of well-mixed stack gas. Two factors contribute to complete mixing of emission gases: turbulence and sufficient mixing time. The criteria listed below define conditions under which well-mixed emissions can be expected.

Select a measurement path that passes through a centroidal area equal to 25 percent of the cross section. Additional requirements or modifications must be met for certain locations as follows:

4.2.1 If the location is in a straight vertical section of stack or duct and is less than 4 equivalent diameters downstream from a bend, use a path that is in the plane defined by the upstream bend (see Figure 1-1).

4.2.2 If the location is in a straight vertical section of stack or duct and is less than 4 equivalent diameters upstream from a bend, use a path that is in the plane defined by the bend (see Figure 1-2).

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4.2.3 If the location is in a straight vertical section of stack or duct and is less than 4 diameters downstream and is also less than 1 diameter upstream from a bend, use a path in the plane defined by the upstream bend (see Figure 1-3).

4.2.4 If the location is in a horizontal section of duct and is at least 4 diameters downstream from a vertical bend, use a path in the horizontal plane that is between one-third and one-half the distance up the vertical axis from the bottom of the duct (see Figure 1-4).

4.2.5 If the location is in a horizontal section of duct and is less than 4 diameters downstream from a vertical bend, use a path in the horizontal plane that is between one-half and two-thirds the distance up the vertical axis from the bottom of the duct for upward flow in the vertical section, and is between one-third and one-half the distance up the vertical axis from the bottom of the duct for downward flow (Figure 1-3).

4.3 Alternative Locations and Measurement Paths. Other locations and measurement paths may be selected by demonstrating to the Administrator that the average opacity measured at the alternative location or path is equivalent to the opacity as measured at a location meeting the criteria of Sections 4.1 and 4.2. The opacity at the alternative location is considered equivalent if the average value measured at the alternative location is within the range defined by the average measured opacity  $\pm 10$  percent at the location meeting the installation criteria in Section 4.2, or if the difference between the two average opacity values is less than 2 percent opacity. To conduct this demonstration, measure the opacities at the two locations or paths for a minimum period of 2 hours and compare the results. The opacities of the two locations or paths may be measured at different times, but must be measured at the same process operating conditions. Alternative procedures for determining acceptable locations may be used if approved by the Administrator.

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### 5. Design and Performance Specifications

5.1 Design Specifications. The CEMS for opacity shall comply with the following design specifications:

5.1.1 Peak and Mean Spectral Responses. The peak and mean spectral responses must occur between 500 nm and 600 nm. The response at any wavelength below 400 nm or above 700 nm shall be less than 10 percent of the peak spectral response.

5.1.2 Angle of View. The total angle of view shall be no greater than 5 degrees.

5.1.3 Angle of Projection. The total angle of projection shall be no greater than 5 degrees.

5.1.4 Optical Alignment Sight. Each analyzer must provide some method for visually determining that the instrument is optically aligned. The method provided must be capable of indicating that the unit is misaligned when an error of +2 percent opacity occurs due to misalignment at a monitor path length of 8 meters. Instruments that are capable of providing an absolute zero check while in operation on a stack or duct with effluent present, and while maintaining the same optical alignment during measurement and calibration, need not meet this requirement (e.g., some "zero pipe" units).

5.1.5 Simulated Zero and Upscale Calibration System. Each analyzer must include a calibration system for simulating a zero (or no greater than 10 percent) opacity and an upscale opacity value for the purpose of performing periodic checks of the transmissometer calibration while on an operating stack or duct. This calibration system will provide, as a minimum, a system check of the analyzer internal optics and all electronic circuitry including the lamp and photodetector assembly.

5.1.6 Access to External Optics. Each analyzer must provide a means of access to the optical surfaces exposed to the effluent stream in order to permit the surfaces to be cleaned without requiring removal of the unit from the source mounting or without requiring optical realignment of the unit.

5.1.7 Automatic Zero Compensation Indicator. If the CEMS has a feature that provides automatic zero compensation for dirt accumulation on exposed optical surfaces, the system must also provide some means of indicating when a compensation of 4 percent opacity has been exceeded. This indicator shall be at a location accessible to the operator (e.g., the data output terminal). During the operational test period, the system must provide some means (manual or automated) for determining the actual amount of zero compensation at the specified 24-hour intervals so that the actual 24-hour zero drift can be determined (see Section 7.4.1).

5.1.8 Slotted Tube. For transmissometers that use slotted tubes, the length of the slotted portion(s) must be equal to or greater than 90 percent of the effluent path length (distance between duct or stack walls). The slotted tube must be of sufficient size and orientation so as not to interfere with the free flow of effluent through the entire optical volume of the transmissometer photodetector. The manufacturer must also show that the transmissometer minimizes light reflections. As a minimum, this demonstration shall consist of laboratory operation of the transmissometer both with and without the slotted tube in position.

Should the operator desire to use a slotted tube design with a slotted portion equal to or less than 90 percent of the monitor path length, the operator must demonstrate to the Administrator that acceptable results can be obtained. As a minimum demonstration, the effluent opacity shall

be measured using both the slotted tube instrument and another instrument meeting the requirement of this specification but not of the slotted tube design. The measurements must be made at the same location and at the same process operating conditions for a minimum period of 2 hours with each instrument. The shorter slotted tube may be used if the average opacity measured is equivalent to the opacity measured by the non-slotted tube design. The average opacity measured is equivalent if it is within the opacity range defined by the average opacity value  $\pm 10$  percent measured by the non-slotted tube design, or if the difference between the average opacities is less than 2 percent opacity.

5.1.9 External Calibration Filter Access (optional). Provisions in the design of the transmissometer to accommodate an external calibration filter assembly are recommended. An adequate design would permit occasional use of external (i.e., not intrinsic to the instrument) neutral density filters to assess monitor operation.

5.2 Performance Specifications. The opacity CEMS specifications are listed in Table 1-1.

### 6. Design Specifications Verification Procedure

These procedures will not apply to all instrument designs and will require modification in some cases; all procedural modifications are subject to the approval of the Administrator.

Test each analyzer for conformance with the design specifications of Sections 5.1.1-5.1.4, or obtain a certificate of conformance from the analyzer manufacturer as follows:

6.1 Spectral Response. Obtain detector response, lamp emissivity, and filter transmittance data for the components used in the measurement system from their respective manufacturers, and develop the effective spectral response curve of the transmissometer. Then determine and report the peak spectral response wavelength, the mean spectral response wavelength, and the maximum response at any wavelength below 400 nm and above 700 nm expressed as a percentage of the peak response.

Alternatively, conduct a laboratory measurement of the instrument's spectral response curve. The procedures of this laboratory evaluation are subject to approval of the Administrator.

TABLE 1-1—PERFORMANCE SPECIFICATIONS

Parameter	Specifications
1. Calibration error .....	$\leq 3$ percent opacity.
2. Response time .....	$\leq 10$ seconds.
3. Conditioning period <sup>a</sup> .....	$\leq 168$ hours.
4. Operational test period <sup>b</sup> .....	$\leq 168$ hours.
5. Zero drift (24-hour) <sup>c</sup> .....	$\leq 2$ percent opacity.
6. Calibration drift (24-hour) .....	$\leq 2$ percent opacity.
7. Data recorder resolution .....	$\leq 0.5$ percent opacity.

<sup>a</sup>Expressed as the sum of the absolute value of the mean and the absolute value of the confidence coefficient.

<sup>b</sup>During the conditioning and operational test periods, the CEMS must not require any corrective maintenance, repair, replacement, or adjustment other than that clearly specified as routine and required in the operation and maintenance manuals.

6.2 Angle of View. Set up the receiver as specified by the manufacturer's written instructions. Draw an arc with radius of 3 meters in the horizontal direction. Using a small (less than 3 centimeters) nondirectional light source, measure the receiver response at 5-centimeter intervals on the arc for 30 centimeters on either side of the

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detector centerline. Repeat the test in the vertical direction. Then for both the horizontal and vertical directions, calculate the response of the receiver as a function of viewing angle (26 centimeters of arc with a radius of 3 meters equals 5 degrees), report relative angle of view curves, and determine and report the angle of view.

6.3 Angle of Projection. Set up the projector as specified by the manufacturer's written instructions. Draw an arc with a radius of 3 meters in the horizontal direction. Using a small (less than 3 centimeters) photoelectric light detector, measure the light intensity at 5-centimeter intervals on the arc for 30 centimeters on either side of the light source centerline of projection. Repeat the test in the vertical direction. Then for both the horizontal and vertical directions, calculate the response of the photoelectric detector as a function of the projection angle (26 centimeters of arc with a radius of 3 meters equals 5 degrees), report the relative angle of projection curves, and determine and report the angle of projection.

6.4 Optical Alignment Sight. In the laboratory set the instrument up as specified by the manufacturer's written instructions for a monitor path length of 8 meters. Align, zero, and span the instrument. Insert an attenuator of 10 percent (nominal opacity) into the instrument path length. Slowly misalign the projector unit by rotating it until a positive or negative shift of 2 percent opacity is obtained by the data recorder. Then, following the manufacturer's written instructions, check the alignment. The alignment procedure must indicate that the instrument is misaligned. Repeat this test for lateral misalignment of the projector. Realign the instrument and follow the same procedure for checking misalignment of the receiver or retroreflector unit (lateral misalignment only).

6.5 Manufacturer's Certificate of Conformance (alternative to above). Obtain from the manufacturer a certificate of conformance stating that the first analyzer randomly sampled from each month's production was tested according to Sections 6.1 through 6.4 and satisfactorily met all requirements of Section 5 of this specification. If any of the requirements were not met, the certificate must state that the entire month's analyzer production was resampled according to the military standard 105D sampling procedure (MIL-STD-105D) inspection level II; was retested for each of the applicable requirements under Section 5 of this specification; and was determined to be acceptable under MIL-STD-105D procedures, acceptable quality level 1.0. The certificate of conformance must include the results of each test performed for the analyzer(s) sampled during the month the analyzer being installed was produced.

7. Performance Specification Verification Procedure

Test each CEMS that conforms to the design specifications (Section 5.1) using the following procedures to determine conformance with the specifications of Table 1-1. These tests are to be performed using the data recording system to be employed during monitoring. Prior approval from the Administrator is required if different data recording systems are used during the performance test and monitoring.

7.1 Preliminary Adjustments and Tests. Before installing the system on the stack, perform these steps or tests at the affected facility or in the manufacturer's laboratory.

7.1.1 Equipment Preparation. Set up and calibrate the CEMS for the monitor path length to be used in the installation as specified by the manufacturer's written instructions. For this specification, the mounting distance

between the transmitter and receiver/reflector unit at the source must be measured prior to performing the calibrations (do not use distances from engineering drawings). If the CEMS has automatic path length adjustment, follow the manufacturer's instructions to adjust the signal output from the analyzer in order to yield results based on the emission outlet path length. Set the instrument and data recording system ranges so that maximum instrument output is within the span range specified in the applicable subpart.

Align the instrument so that maximum system response is obtained during a zero (or upscale) check performed across the simulated monitor path length. As part of this alignment, include rotating the reflector unit (detector unit for single pass instruments) on its axis until the point of maximum instrument response is obtained.

Follow the manufacturer's instructions to zero and span the instrument. Perform the zero alignment adjustment by balancing the response of the CEMS so that the simulated zero check coincides with the actual zero check performed across the simulated monitor path length. At this time, measure and record the indicated upscale calibration value. The calibration value reading must be within the required opacity range (Section 3.3).

7.1.2 Calibration Attenuator Selection. Based on the span value specified in the applicable subpart, select a minimum of three calibration attenuators (low, mid, and high range) using Table 1-2.

If the system is operating with automatic path length compensation, calculate the attenuator values required to obtain a system response equivalent to the applicable values shown in Table 1-2; use Equation 1-1 for the conversion. A series of filters with nominal optical density (opacity) values of 0.1(20), 0.2(37), 0.3(50), 0.4(60), 0.5(68), 0.6(75), 0.7(80), 0.8(84), 0.9(88), and 1.0(90) are commercially available. Within this limitation of filter availability, select the calibration attenuators having the values given in Table 1-2 or having values closest to those calculated by Equation 1-1.

$$D_1 = D_2 (L_1/L_2)$$

Eq. 1-1

TABLE 1-2—REQUIRED CALIBRATION ATTENUATOR VALUES (NOMINAL)

Span value (percent opacity)	Calibrated attenuator optical density (equivalent opacity in parenthesis)— D <sub>2</sub>		
	Low-range	Mid-range	High-range
40 .....	0.08 (11)	0.1 (20)	0.2 (37)
50 .....	0.1 (20)	0.2 (37)	0.3 (50)
60 .....	0.1 (20)	0.2 (37)	0.3 (50)
70 .....	0.1 (20)	0.3 (50)	0.4 (60)
80 .....	0.1 (20)	0.3 (50)	0.6 (75)
90 .....	0.1 (20)	0.4 (60)	0.7 (80)
100 .....	0.1 (20)	0.4 (60)	0.9 (87.5)

Where:

D<sub>1</sub>—Nominal optical density value of required mid, low, or high range calibration attenuators.

D<sub>2</sub>—Desired attenuator optical density output value from Table 1-2 at the span required by the applicable subpart.

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$L_1$ =Monitor path length.

$L_2$ =Emission outlet path length.

7.1.3 Attenuator Calibration. Select a laboratory calibration spectrophotometer meeting the specifications of Section 3.4. Using this calibration spectrophotometer, calibrate the required filters or screens. Make measurements at wavelength intervals of 20 nm or less. As an alternative procedure, use the calibration spectrophotometer to measure the C.I.E. Daylight  $C$  luminous transmittance of the attenuators. Check the attenuators several times, at different locations on the attenuator.

The attenuator manufacturer must specify the period of time over which the attenuator values can be considered stable, as well as any special handling and storing procedures required to enhance attenuator stability. To assure stability, recheck attenuator values at intervals less than or equal to the period stability guaranteed by the manufacturer. Recheck at least every 3 months. If desired, perform the stability checks with an instrument (secondary) other than the calibration spectrophotometer. This secondary instrument must be a high-quality laboratory transmissometer or spectrophotometer, and the same instrument must always be used for the stability checks. If a secondary instrument is to be used for stability checks, the value of the calibrated attenuator must be measured on this secondary instrument immediately following initial calibration. If over a period of time an attenuator value changes by more than  $\pm 2$  percent opacity, recalibrate the attenuator on the calibration spectrophotometer or replace it with a new attenuator.

If this procedure is conducted by the filter or screen manufacturer or by an independent laboratory, obtain a statement certifying the values and certifying that the specified procedure, or equivalent, is used.

7.1.4 Calibration Error Test. Insert the calibration attenuators (low, mid, and high range) in the transmissometer path at or as near the midpoint of the path as feasible. Place the attenuator in the measurement path at a point where the effluent will be measured; i.e., do not place the calibration attenuator in the instrument housing. If the instrument manufacturer recommends a procedure wherein the attenuators are placed in the instrument housing, the manufacturer must provide data showing this alternative procedure is acceptable. While inserting the attenuator, assure that the entire beam received by the detector will pass through the attenuator and that the attenuator is inserted in a manner which minimizes interference from reflected light. Make a total of five nonconsecutive readings for each filter. Record the monitoring system output readings in percent opacity (see example Figure 1-6). Then, if the path length is not adjusted by the measurement system, subtract the actual calibration attenuator value from the value indicated by the measurement system recorder for each of the 15 readings obtained. If the path length is adjusted by the measurement system, subtract the "path adjusted" calibration attenuator values from the values indicated by the measurement system recorder (the "path adjusted" calibration attenuator values are calculated using Equation 1-6 or 1-7). Calculate the arithmetic mean difference, standard deviation, and confidence coefficient of the five tests at each attenuator value using Equations 1-2, 1-3, and 1-4 (Sections 8.1-8.3). Calculate the sum of the absolute value of the mean difference and the absolute value of the confidence coefficient for each of the three test attenuators; report these three values as the calibration error.

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7.1.5 System Response Test. Insert the high-range calibration attenuator in the transmissometer path five times, and record the time required for the system to respond to 95 percent of final zero and high-range filter

values (see example Figure 1-7). Then calculate the mean time of the 10 upscale and downscale tests and report this value as the system response time.

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7.2 Preliminary Field Adjustments. Install the CEMS on the affected facility according to the manufacturer's written instructions and the specifications in Section 4, and perform the following preliminary adjustments:

7.2.1 Optical and Zero Alignment. When the facility is not in operation, optically align the light beam of the transmissometer upon the optical surface located across the duct or stack (i.e., the retroreflector or photodetector, as applicable) in accordance with the manufacturer's instructions; verify the alignment with the optical alignment sight. Under clear stack conditions, verify the zero alignment (performed in Section 7.1.1) by assuring that the monitoring system response for the simulated zero check coincides with the actual zero measured by the transmissometer across the clear stack. Adjust the zero alignment, if necessary. Then, after the affected facility has been started up and the effluent stream reaches normal operating temperature, recheck the optical alignment. If the opti-

cal alignment has shifted, realign the optics. Note: Careful consideration should be given to whether a "clear stack" condition exists. It is suggested that the stack be monitored and the data output (instantaneous real-time basis) be examined to determine whether fluctuations from zero opacity are occurring before a clear stack condition is assumed to exist.

7.2.2 Optical and Zero Alignment (Alternative Procedure). The procedure given in 7.2.1 is the preferred procedure and should be used whenever possible; however, if the facility is operating and a zero stack condition cannot practicably be obtained, use the zero alignment obtained during the preliminary adjustments (Section 7.1.1) before installing the transmissometer on the stack. After completing all the preliminary adjustments and tests required in Section 7.1, install the system at the source and align the optics, i.e., align the light beam from the transmissometer upon the optical surface located across the duct or stack

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in accordance with the manufacturer's instruction. Verify the alignment with the optical alignment sight. The zero alignment conducted in this manner must be verified and adjusted, if necessary, the first time a clear stack condition is obtained after the operation test period has been completed.

7.3 Conditioning Period. After completing the preliminary field adjustments (Section 7.2), operate the CEMS according to the manufacturer's instructions for an initial conditioning period of not less than 168 hours while the source is operating. Except during times of instrument zero and upscale calibration checks, the CEMS must analyze the effluent gas for opacity and produce a permanent record of the CEMS output. During this conditioning period there must be no unscheduled maintenance, repair, or adjustment. Conduct daily zero calibration and upscale calibration checks; and, when accumulated drift exceeds the daily operating limits, make adjustments and clean the exposed optical surfaces. The data recorder must reflect these checks and adjustments. At the end of the operational test period, verify that the instrument optical alignment is correct. If the conditioning period is interrupted because of source breakdown (record the dates and times of process shutdown), continue the 168-hour period following resumption of source operation. If the conditioning period is interrupted because of monitor failure, restart the 168-hour conditioning period when the monitor becomes operational.

7.4 Operational Test Period. After completing the conditioning period, operate the system for an additional 168-hour period. The 168-hour operational test period need not follow immediately after the 168-hour conditioning period. Except during times of instrument zero and upscale calibration checks, the CEMS must analyze the effluent gas for opacity and must produce a permanent record of the CEMS output. During this period, there will be no unscheduled maintenance, repair, or adjustment. Zero and calibration adjustments, optical surface cleaning, and optical realignment may be performed (optional) only at 24-hour intervals or at such shorter intervals as the manufacturer's written instructions specify. Automatic zero and calibration adjustments made by the CEMS

without operator intervention or initiation are allowable at any time. During the operational test period, record all adjustments, realignments, and lens cleanings. If the operational test period is interrupted because of source breakdown, continue the 168-hour period following resumption of source operation. If the test period is interrupted because of monitor failure, restart the 168-hour period when the monitor becomes operational. During the operational test period, perform the following test procedures:

7.4.1 Zero Drift Test. At the outset of the 168-hour operational test period, record the initial simulated zero (or no greater than 10 percent) and upscale opacity readings (see example Figure 1-8). After each 24-hour interval, check and record the final zero reading before any optional or required cleaning and adjustment. Zero and upscale calibration adjustments, optical surface cleaning, and optical realignment may be performed only at 24-hour intervals (or at such shorter intervals as the manufacturer's written instructions specify), but are optional. However, adjustments and cleaning must be performed when the accumulated zero calibration or upscale calibration drift exceeds the 24-hour drift specification ( $\pm 2$  percent opacity). If no adjustments are made after the zero check, record the final zero reading as the initial zero reading for the next 24-hour period. If adjustments are made, record the zero value after adjustment as the initial zero value for the next 24-hour period. If the instrument has an automatic zero compensation feature for dirt accumulation on exposed lenses and the zero value cannot be measured before compensation is entered, then record the amount of automatic zero compensation (as opacity) for the final zero reading of each 24-hour period. (List the indicated zero values of the CEMS in parenthesis.) From the initial and final zero readings, calculate the zero drift for each 24-hour period. Then calculate the arithmetic mean, standard deviation, and confidence coefficient of the 24-hour zero drift and the 95 percent confidence interval using Equations 1-2, 1-3, and 1-4. Calculate the sum of the absolute value of the mean and the absolute value of the confidence coefficient, and report this value as the 24-hour zero drift.

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7.4.2 Upscale Drift Test. At each 24-hour interval, after the zero calibration value has been checked and any optional or required adjustments have been made, check and record the simulated upscale calibration value. If no further adjustments are made to the calibration system at this time, record the final upscale calibration value as the initial upscale value for the next 24-hour period. If an instrument span adjustment is made, record the upscale value after adjustment as the initial upscale value for the next 24-hour period. From the initial and final upscale readings, calculate the upscale calibration drift for each 24-hour period. Then calculate the arithmetic mean, standard deviation, and confidence coefficient of the 24-hour calibration drift and the 95 percent confidence interval using Equations 1-2, 1-3, and 1-4. Calculate the sum of the absolute value of the mean and the absolute value of the confidence coefficient, and report this value as the 24-hour calibration drift.

8. Equations

8.1 Arithmetic Mean. Calculate the mean,  $\bar{x}$ , of a set of data as follows:

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where:

n=Number of data points.

$\sum_{i=1}^n x_i$ =Algebraic sum of the individual measurements  $x_i$ .

8.2 Standard Deviation. Calculate the standard deviation  $S_d$  as follows:

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8.3 Confidence Coefficient. Calculate the 2.5 percent error confidence coefficient (one-tailed), CC, as follows:

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Where:

0.975=t-value (see Table 1-3).

8.4 Error. Calculate the error (i.e., calibration error, zero drift, and calibration drift), Er, as follows:

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TABLE 1-3—t-VALUES

n*	0.975	n*	0.975	n*	0.975
2	12.708	7	2.447	12	2.201
3	4.303	8	2.365	13	2.179
4	3.182	9	2.306	14	2.160
5	2.776	10	2.262	15	2.145
6	2.571	11	2.228	16	2.131

\*The values in this table are already corrected for n-1 degrees of freedom. Use n equal to the number of individual values.

8.5 Conversion of Opacity Values from Monitor Path Length to Emission Outlet Path Length. When the monitor path length is different than the emission outlet path length, use either of the following equations to convert from one basis to the other (this conversion may be automatically calculated by the monitoring system):

$$\log(1-Op_2)=(L_2/L_1) \log(1-Op_1) \quad (\text{Eq. 1-6})$$

$$D_2=(L_2/L_1) D_1 \quad (\text{Eq. 1-7})$$

Where:

Op<sub>1</sub> = Opacity of the effluent based upon L<sub>1</sub>.

Op<sub>2</sub> = Opacity of the effluent based upon L<sub>2</sub>.

L<sub>1</sub> = Monitor path length.

L<sub>2</sub> = Emission outlet path length.

D<sub>1</sub> = Optical density of the effluent based upon L<sub>1</sub>.

D<sub>2</sub> = Optical density of the effluent based upon L<sub>2</sub>.

9. Reporting

Report the following (summarize in tabular form where appropriate).

9.1 General Information.

- a. Facility being monitored.
- b. Person(s) responsible for operational and conditioning test periods and affiliation.
- c. Instrument manufacturer.
- d. Instrument model number.
- e. Instrument serial number.
- f. Month/year manufactured.
- g. Schematic of monitoring system measurement path location.

- h. Monitor pathlength, meters.
- i. Emission outlet pathlength, meters.
- j. System span value, percent opacity.
- k. Upscale calibration value, percent opacity.
- l. Calibrated Attenuator values (low, mid, and high range), percent opacity.

9.2 Design Specification Test Results.

- a. Peak spectral response, nm.
- b. Mean spectral response, nm.
- c. Response above 700 nm, percent of peak.
- d. Response below 400 nm, percent of peak.
- e. Total angle of view, degrees.
- f. Total angle of projection, degrees.
- g. Results of optical alignment sight test.
- h. Serial number, month/year of manufacturer for unit actually tested to show design conformance.

9.3 Performance Specification Test Results.

- a. Calibration error, high-range, percent opacity.
- b. Calibration error, mid-range, percent opacity.
- c. Calibration error, low-range, percent opacity.
- d. Response time, seconds.
- e. 24-hour zero drift, percent opacity.
- f. 24-hour calibration drift, percent opacity.
- g. Lens cleanings, clock time.
- h. Optical alignment adjustments, clock time.

9.4 Statements. Provide a statement that the conditioning and operational test periods were completed according to the requirements of Sections 7.3 and 7.4. In this statement, include the time periods during which the conditioning and operational test periods were conducted.

9.5 Appendix. Provide the data tabulations and calculations for the above tabulated results.

10. Retest

If the CEMS operates within the specified performance parameters of Table 1-1, the PS tests will be successfully

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concluded. If the CEMS fails one of the preliminary tests, make the necessary corrections and repeat the performance testing for the failed specification prior to conducting the operational test period. If the CEMS fails to meet the specifications for the operational test period, make the necessary corrections and repeat the operational test period; depending on the correction made, it may be necessary to repeat the design and preliminary performance tests.

### 11. Bibliography

1. Experimental Statistics. Department of Commerce. National Bureau of Standards Handbook 91. Paragraph 3-3.1.4 1963. pp. 3-31.
12. Performance Specifications for Stationary-Source Monitoring Systems for Gases and Visible Emissions. U.S. Environmental Protection Agency, Research Triangle Park, NC. EPA-650/2-74-013. January 1974.

### PERFORMANCE SPECIFICATION 2—SPECIFICATIONS AND TEST PROCEDURES FOR SO<sub>2</sub> AND NO<sub>x</sub> CONTINUOUS EMISSION MONITORING SYSTEMS IN STATIONARY SOURCES

#### 1. Applicability and Principle

1.1 Applicability. This specification is to be used for evaluating the acceptability of SO<sub>2</sub> and NO<sub>x</sub> continuous emission monitoring systems (CEMS's) at the time of or soon after installation and whenever specified in the regulations. The CEMS may include, for certain stationary sources, a diluent (O<sub>2</sub> or CO<sub>2</sub>) monitor.

This specification is not designed to evaluate the installed CEMS performance over an extended period of time nor does it identify specific calibration techniques and other auxiliary procedures to assess the CEMS performance. The source owner or operator, however, is responsible to properly calibrate, maintain, and operate the CEMS. To evaluate the CEMS performance, the Administrator may require, under Section 114 of the Act, the operator to conduct CEMS performance evaluations at other times besides the initial test. See § 60.13(c).

1.2 Principle. Installation and measurement location specifications, performance and equipment specifications, test procedures, and data reduction procedures are included in this specification. Reference method tests and calibration drift tests are conducted to determine conformance of the CEMS with the specification.

#### 2. Definitions

2.1 Continuous Emission Monitoring System. The total equipment required for the determination of a gas concentration or emission rate. The system consists of the following major subsystems:

2.1.1 Sample Interface. That portion of the CEMS used for one or more of the following: sample acquisition, sample transportation, and sample conditioning, or protection of the monitor from the effects of the stack effluent.

2.1.2 Pollutant Analyzer. That portion of the CEMS that senses the pollutant gas and generates an output proportional to the gas concentration.

2.1.3 Diluent Analyzer (if applicable). That portion of the CEMS that senses the diluent gas (e.g., CO<sub>2</sub> or O<sub>2</sub>) and generates an output proportional to the gas concentration.

2.1.4 Data Recorder. That portion of the CEMS that provides a permanent record of the analyzer output. The data recorder may include automatic data reduction capabilities.

2.2 Point CEMS. A CEMS that measures the gas concentration either at a single point or along a path equal to or less than 10 percent of the equivalent diameter of the stack or duct cross section.

2.3 Path CEMS. A CEMS that measures the gas concentration along a path greater than 10 percent of the equivalent diameter of the stack or duct cross section.

2.4 Span Value. The upper limit of a gas concentration measurement range specified for affected source categories in the applicable subpart of the regulations.

2.5 Relative Accuracy (RA). The absolute mean difference between the gas concentration or emission rate determined by the CEMS and the value determined by the RM's plus the 2.5 percent error confidence coefficient of a series of tests divided by the mean of the RM tests or the applicable emission limit.

2.6 Calibration Drift (CD). The difference in the CEMS output readings from the established reference value after a stated period of operation during which no unscheduled maintenance, repair, or adjustment took place.

2.7 Centroidal Area. A concentric area that is geometrically similar to the stack or duct cross section and is no greater than 1 percent of the stack or duct cross-sectional area.

2.8 Representative Results. As defined by the RM test procedure outlined in this specification.

#### 3. Installation and Measurement Location Specifications

3.1 The CEMS Installation and Measurement Location. Install the CEMS at an accessible location where the pollutant concentration or emission rate measurements are directly representative or can be corrected so as to be representative of the total emissions from the affected facility or at the measurement location cross section. Then select representative measurement points or paths for monitoring in locations that the CEMS will pass the RA test (see Section 7). If the cause of failure to meet the RA test is determined to be the measurement location and a satisfactory correction technique cannot be established, the Administrator may require the CEMS to be relocated.

Suggested measurement locations and points or paths that are most likely to provide data that will meet the RA requirements are listed below.

3.1.1 Measurement Location. It is suggested that the measurement location be (1) at least two equivalent diameters downstream from the nearest control device, the point of pollutant generation, or other point at which a change in the pollutant concentration or emission rate may occur and (2) at least a half equivalent diameter upstream from the effluent exhaust or control device.

3.1.2 Point CEMS. It is suggested that the measurement point be (1) no less than 1.0 meter from the stack or duct wall or (2) within or centrally located over the centroidal area of the stack or duct cross section.

3.1.3 Path CEMS. It is suggested that the effective measurement path (1) be totally within the inner area bounded by a line 1.0 meter from the stack or duct wall, or (2) have at least 70 percent of the path within the inner 50 percent of the stack or duct cross-sectional area, or (3) be centrally located over any part of the centroidal area.

3.2 Reference Method (RM) Measurement Location and Traverse Points. Select, as appropriate, an accessible RM measurement point at least two equivalent diameters downstream from the nearest control device, the point of pollutant generation, or other point at which a change in the pollutant concentration or emission rate may occur.

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and at least a half equivalent diameter upstream from the effluent exhaust or control device. When pollutant concentration changes are due solely to diluent leakage (e.g., air heater leakages) and pollutants and diluents are simultaneously measured at the same location, a half diameter may be used in lieu of two equivalent diameters. The CEMS and RM locations need not be the same.

Then select traverse points that assure acquisition of representative samples over the stack or duct cross section. The minimum requirements are as follows: Establish a "measurement line" that passes through the centroidal area and in the direction of any expected stratification. If this line interferes with the CEMS measurements, displace the line up to 30 cm (or 5 percent of the equivalent diameter of the cross section, whichever is less) from the centroidal area. Locate three traverse points at 16.7, 50.0, and 83.3 percent of the measurement line. If the measurement line is longer than 2.4 meters and pollutant stratification is not expected, the tester may choose to locate the three traverse points on the line at 0.4, 1.2, and 2.0 meters from the stack or duct wall. This option must not be used after wet scrubbers or at points where two streams with different pollutant concentrations are combined. The tester may select other traverse points, provided that they can be shown to the satisfaction of the Administrator to provide a representative sample over the stack or duct cross section. Conduct all necessary RM tests within 3 cm (but no less than 3 cm from the stack or duct wall) of the traverse points.

### 4. Performance and Equipment Specifications

4.1 Data Recorder Scale. The CEMS data recorder response range must include zero and a high-level value. The high-level value is chosen by the source owner or operator and is defined as follows:

For a CEMS intended to measure an uncontrolled emission (e.g., SO<sub>2</sub> measurements at the inlet of a flue gas desulfurization unit), the high-level value must be between 1.25 and 2 times the average potential emission level, unless otherwise specified in an applicable subpart of the regulations. For a CEMS installed to measure controlled emissions or emissions that are in compliance with an applicable regulation, the high-level value must be between 1.5 times the pollutant concentration corresponding to the emission standard level and the span value. If a lower high-level value is used, the source must have the capability of measuring emissions which exceed the full-scale limit of the CEMS in accordance with the requirements of applicable regulations.

The data recorder output must be established so that the high-level value is read between 90 and 100 percent of the data recorder full scale. (This scale requirement may not be applicable to digital data recorders.) The calibration gas, optical filter, or cell values used to establish the data recorder scale should produce the zero and high-level values. Alternatively, a calibration gas, optical filter, or cell value between 50 and 100 percent of the high-level value may be used in place of the high-level value provided the data recorder full-scale requirements as described above are met.

The CEMS design must also allow the determination of calibration drift at the zero and high-level values. If this is not possible or practical, the design must allow these determinations to be conducted at a low-level value (zero to 20 percent of the high-level value) and at a value between 50 and 100 percent of the high-level value. In spe-

cial cases, if not already approved, the Administrator may approve a single-point calibration-drift determination.

4.2 Calibration Drift. The CEMS calibration must not drift or deviate from the reference value of the gas cylinder, gas cell, or optical filter by more than 2.5 percent of the span value. If the CEMS includes pollutant and diluent monitors, the calibration drift must be determined separately for each in terms of concentrations (see PS 3 for the diluent specifications).

4.3 The CEMS RA. The RA of the CEMS must be no greater than 20 percent of the mean value of the RM test data in terms of the units of the emission standard or 10 percent of the applicable standard, whichever is greater. For SO<sub>2</sub> emission standards between 130 and 86 ng/J (0.30 and 0.20 lb/million Btu), use 15 percent of the applicable standard; below 86 ng/J (0.20 lb/million Btu), use 20 percent of emission standard.

### 5. Performance Specification Test Procedure

5.1 Pretest Preparation. Install the CEMS, prepare the RM test site according to the specifications in Section 3, and prepare the CEMS for operation according to the manufacturer's written instructions.

5.2 Calibration Drift Test Period. While the affected facility is operating at more than 50 percent of normal load, or as specified in an applicable subpart, determine the magnitude of the calibration drift (CD) once each day (at 24-hour intervals) for 7 consecutive days according to the procedure given in Section 6. To meet the requirement of Section 4.2, none of the CD's must exceed the specification.

5.3 RA Test Period. Conduct the RA test according to the procedure given in Section 7 while the affected facility is operating at more than 50 percent or normal load, or as specified in an applicable subpart. To meet the specifications, the RA must be equal to or less than 20 percent of the mean value of the RM test data in terms of the units of the emission standard or 10 percent of the applicable standard, whichever is greater. For instruments that use common components to measure more than one effluent gas constituent, all channels must simultaneously pass the RA requirement, unless it can be demonstrated that any adjustments made to one channel did not affect the others.

The RA test may be conducted during the CD test period.

### 6. The CEMS Calibration Drift Test Procedure

The CD measurement is to verify the ability of the CEMS to conform to the established CEMS calibration used for determining the emission concentration or emission rate. Therefore, if periodic automatic or manual adjustments are made to the CEMS zero and calibration settings, conduct the CD test immediately before these adjustments, or conduct it in such a way that the CD can be determined.

Conduct the CD test at the two points specified in Section 4.1. Introduce to the CEMS the reference gases, gas cells, or optical filters (these need not be certified). Record the CEMS response and subtract this value from the reference value (see example data sheet in Figure 2-1).

### 7. Relative Accuracy Test Procedure

7.1 Sampling Strategy for RM Tests. Conduct the RM tests in such a way that they will yield results representative of the emissions from the source and can be correlated to the CEMS data. Although it is preferable to

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conduct the diluent (if applicable), moisture (if needed), and pollutant measurements simultaneously, the diluent and moisture measurements that are taken within a 30- to 60-minute period, which includes the pollutant measurements, may be used to calculate dry pollutant concentration and emission rate.

In order to correlate the CEMS and RM data properly, mark the beginning and end of each RM test period of each run (including the exact time of the day) on the CEMS chart recordings or other permanent record of output. Use the following strategies for the RM tests:

7.1.1 For integrated samples, e.g., Method 6 and Method 4, make a sample traverse of at least 21 minutes, sampling for 7 minutes at each traverse point.

7.1.2 For grab samples, e.g., Method 7, take one sample at each traverse point, scheduling the grab samples so that they are taken simultaneously (within a 3-minute period) or are an equal interval of time apart over a 21-minute (or less) period. A test run for grab samples must be made up of at least three separate measurements.

**NOTE:** At times, CEMS RA tests are conducted during new source performance standards performance tests. In these cases, RM results obtained during CEMS RA tests may be used to determine compliance as long as the source and test conditions are consistent with the applicable regulations.

7.2 Correlation of RM and CEMS Data. Correlate the CEMS and the RM test data as to the time and duration by first determining from the CEMS final output (the one used for reporting) the integrated average pollutant concentration or emission rate for each pollutant RM test period. Consider system response time, if important, and confirm that the pair of results are on a consistent moisture, temperature, and diluent concentration basis. Then, compare each integrated CEMS value against the corresponding average RM value. Use the following guidelines to make these comparisons.

7.2.1 If the RM has an integrated sampling technique, make a direct comparison of the RM results and CEMS integrated average value.

7.2.2 If the RM has a grab sampling technique, first average the results from all grab samples taken during the test run and then compare this average value against the integrated value obtained from the CEMS chart recording or output during the run. If the pollutant concentration is varying with time over the run, the tester may choose to use the arithmetic average of the CEMS value recorded at the time of each grab sample.

7.3 Number of RM Tests. Conduct a minimum of nine sets of all necessary RM tests. Conduct each set within a period of 30 to 60 minutes.

**NOTE:** The tester may choose to perform more than nine sets of RM tests. If this option is chosen, the tester may, at his discretion, reject a maximum of three sets of the test results so long as the total number of test results used to determine the RA is greater than or equal to nine, but he must report all data including the rejected data.

7.4 Reference Methods. Unless otherwise specified in an applicable subpart of the regulations, Methods 3B, 4, 6, and 7, or their approved alternatives, are the reference methods for diluent (O<sub>2</sub> and CO<sub>2</sub>), moisture, SO<sub>2</sub>, and NO<sub>x</sub>, respectively.

7.5 Calculations. Summarize the results on a data sheet. An example is shown in Figure 2-2. Calculate the mean of the RM values. Calculate the arithmetic differences between the RM and the CEMS output sets.

Then calculate the mean of the difference, standard deviation, confidence coefficient, and CEMS RA, using Equations 2-1, 2-2, 2-3, and 2-4.

**8. Equations**

8.1 Arithmetic Mean. Calculate the arithmetic mean of the difference,  $\bar{d}$ , of a data set as follows:

Insertillus.1A

Where:

$n$ =Number of data points.

Insertillus.2A

When the mean of the differences of pairs of data is calculated, be sure to correct the data for moisture, if applicable.

8.2 Standard Deviation. Calculate the standard deviation,  $S_d$ , as follows:

Insertillus.3A

8.3 Confidence Coefficient. Calculate the 2.5 percent error confidence coefficient (one-tailed), CC, as follows:

Insertillus.4A

Where:

$t_{0.975}$ =t-value (see Table 2-1)

**TABLE 2-1—t-VALUES**

$n^*$	$t_{0.975}$	$n^*$	$t_{0.975}$	$n^*$	$t_{0.975}$
2	12.706	7	2.447	12	2.201
3	4.303	8	2.365	13	2.179
4	3.182	9	2.308	14	2.160
5	2.776	10	2.262	15	2.145
6	2.571	11	2.228	16	2.131

\*The values in this table are already corrected for  $n-1$  degrees of freedom. Use  $n$  equal to the number of individual values.

8.4 Relative Accuracy. Calculate the RA of a set of data as follows:

Insertillus.5A

Where:

$|\bar{d}|$ =Absolute value of the mean of differences (from Equation 2-1).

$|CC|$ =Absolute value of the confidence coefficient (from Equation 2-3).

$\bar{RM}$ =Average RM value or applicable standard.

**9. Reporting**

At a minimum (check with the appropriate regional office, or State, or local agency for additional requirements,

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if any) summarize in tabular form the results of the CD tests and the relative accuracy tests or alternative RA procedure as appropriate. Include all data sheets, calculations, charts (records of CEMS responses), cylinder gas concentration certifications, and calibration cell response certifications (if applicable), necessary to substantiate that the performance of the CEMS met the performance specifications.

10. Alternative Procedures

10.1 Alternative to Relative Accuracy Procedure in section 7. Paragraphs 60.13(j) (1) and (2) contain criteria for which the reference method relative accuracy may be waived and the following procedure substituted.

10.1.1 Conduct a complete CEMS status check following the manufacturer's written instructions. The check should include operation of the light source, signal receiver, timing mechanism functions, data acquisition and data reduction functions, data recorders, mechanically operated functions (mirror movements, zero pipe operation, calibration gas valve operations, etc.), sample filters, sample line heaters, moisture traps, and other related functions of the CEMS, as applicable. All parts of the CEMS shall be functioning properly before proceeding to the alternative RA procedure.

10.1.2 Challenge each monitor (both pollutant and diluent, if applicable) with cylinder gases of known concentrations or calibration cells that produce known responses at two measurement points within the following ranges:

MEASUREMENT RANGE

Measurement point	Pollutant monitor	Diluent monitor for	
		CO <sub>2</sub>	O <sub>2</sub>
1 .....	20-30 percent of span value.	8-8 percent by volume.	4-6 percent by volume.
2 .....	60-80 percent of span value.	10-14 percent by volume.	8-12 percent by volume.

Use a separate cylinder gas or calibration cell for measurement points 1 and 2. Challenge the CEMS and record the responses three times at each measurement point. Do not dilute gas from a cylinder when challenging the CEMS. Use the average of the three responses in determining relative accuracy.

Operate each monitor in its normal sampling mode as nearly as possible. When using cylinder gases, pass the cylinder gas through all filters, scrubbers, conditioners, and other monitor components used during normal sampling and as much of the sampling probe as practical. When using calibration cells, the CEMS components used in the normal sampling mode should not be by-passed

during the RA determination. These include light sources, lenses, detectors, and reference cells. The CEMS should be challenged at each measurement point for a sufficient period of time to assure adsorption-desorption reactions on the CEMS surfaces have stabilized.

Use cylinder gases that have been certified by comparison to National Bureau of Standards (NBS) gaseous standard reference material (SRM) or NBS/EPA-approved gas manufacturer's certified reference material (CRM) (See Citation 2 in the Bibliography) following EPA traceability protocol Number 1 (See Citation 3 in the Bibliography). As an alternative to protocol Number 1 gases, CRM's may be used directly as alternative RA cylinder gases. A list of gas manufacturers that have prepared approved CRM's is available from EPA at the address shown in Citation 2. Procedures for preparation of CRM are described in Citation 2.

Use calibration cells certified by the manufacturer to produce a known response in the CEMS. The cell certification procedure shall include determination of CEMS response produced by the calibration cell in direct comparison with measurement of gases of known concentration. This can be accomplished using SRM or CRM gases in a laboratory source simulator or through extended tests using reference methods at the CEMS location in the exhaust stack. These procedures are discussed in Citation 4 in the Bibliography. The calibration cell certification procedure is subject to approval of the Administrator.

10.1.3 The differences between the known concentrations of the cylinder gases and the concentrations indicated by the CEMS are used to assess the accuracy of the CEMS.

The calculations and limits of acceptable relative accuracy (RA) are as follows:

(a) For pollutant CEMS:

$$RA = \left( \frac{d}{AC} \right) \times 100 \leq 15 \text{ percent}$$

Where:

d=Difference between response and the known concentration/response.

AC=The known concentration/response of the cylinder gas or calibration cell.

(b) For diluent CEMS:

RA=[d] ≤ 0.7 percent O<sub>2</sub> or CO<sub>2</sub>, as applicable.

NOTE: Waiver of the relative accuracy test in favor of the alternative RA procedure does not preclude the requirements to complete the calibration drift (CD) tests nor any other requirements specified in the applicable regulation(s) for reporting CEMS data and performing CEMS drift checks or audits.

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## Pt. 60, App. B, Spec. 3

### 11. Bibliography

1. Department of Commerce. Experimental Statistics. Handbook 91. Washington, DC, p. 3-31, paragraphs 3-3.1.4.
2. "A Procedure for Establishing Traceability of Gas Mixtures to Certain National Bureau of Standards Standard Reference Materials." Joint publication by NBS and EPA. EPA-600/7-81-010. Available from U.S. Environmental Protection Agency, Quality Assurance Division (MD-77), Research Triangle Park, NC 27711.
3. "Traceability Protocol for Establishing True Concentrations of Gases Used for Calibration and Audits of Continuous Source Emission Monitors. (Protocol Number 1)." June 1978. Protocol Number 1 is included in the *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, Stationary Source Specific Methods*. EPA-600/4-77-027b. August 1977. Volume III is available from the U.S. EPA, Office of Research and Development Publications, 26 West St. Clair Street, Cincinnati, OH 45268.
4. "Gaseous Continuous Emission Monitoring Systems—Performance Specification Guidelines for SO<sub>2</sub>, NO<sub>x</sub>, CO<sub>2</sub>, O<sub>2</sub>, and TRS." EPA-450/3-82-026. Available from U.S. Environmental Protection Agency, Emission Standards and Engineering Division (MD-19), Research Triangle Park, NC 27711.

### PERFORMANCE SPECIFICATION 3—SPECIFICATIONS AND TEST PROCEDURES FOR O<sub>2</sub> AND CO<sub>2</sub> CONTINUOUS EMISSION MONITORING SYSTEMS IN STATIONARY SOURCES

#### 1. Applicability and Principle

1.1 Applicability. This specification is to be used for evaluating acceptability of O<sub>2</sub> and CO<sub>2</sub> continuous emission monitoring systems (CEM's) at the time of or soon after installation and whenever specified in an applicable subpart of the regulations. The specification applies to O<sub>2</sub> or CO<sub>2</sub> monitors that are not included under Performance Specification 2 (PS 2).

This specification is not designed to evaluate the installed CEMS performance over an extended period of time, nor does it identify specific calibration techniques and other auxiliary procedures to assess the CEMS performance. The source owner or operator, however, is responsible to calibrate, maintain, and operate the CEMS properly. To evaluate the CEMS performance, the Administrator may require, under Section 114 of the Act, the operator to conduct CEMS performance evaluations in addition to the initial test. See Section 60.13(e).

The definitions, installation and measurement location specifications, test procedures, data reduction procedures, reporting requirements, and bibliography are the same as in PS 2, Sections 2, 3, 5, 6, 8, 9, and 10, and also apply to O<sub>2</sub> and CO<sub>2</sub> CEMS's under this specification. The performance and equipment specifications and the relative accuracy (RA) test procedures for O<sub>2</sub> and CO<sub>2</sub> CEMS do not differ from those for SO<sub>x</sub> and NO<sub>x</sub> CEMS, except as noted below.

1.2 Principle. Reference method (RM) tests and calibration drift tests are conducted to determine conformance of the CEMS with the specification.

#### 2. Performance and Equipment Specifications

2.1 Instrument Zero and Span. This specification is the same as Section 4.1 of PS 2.

2.2 Calibration Drift. The CEMS calibration must not drift by more than 0.5 percent O<sub>2</sub> or CO<sub>2</sub> from the reference value of the gas, gas cell, or optical filter.

2.3 The CEMS RA. The RA of the CEMS must be no greater than 20 percent of the mean value of the RM test data or 1.0 percent O<sub>2</sub> or CO<sub>2</sub>, whichever is greater.

#### 3. Relative Accuracy Test Procedure

3.1 Sampling Strategy for RM Tests, Correlation of RM and CEMS Data, Number of RM Tests, and Calculations. This is the same as PS 2, Sections 7.1, 7.2, 7.3, and 7.5, respectively.

3.2 Reference Method. Unless otherwise specified in an applicable subpart of the regulations, Method 3B of appendix A or any approved alternative is the RM for O<sub>2</sub> or CO<sub>2</sub>.

### PERFORMANCE SPECIFICATION 4—SPECIFICATIONS AND TEST PROCEDURES FOR CARBON MONOXIDE CONTINUOUS EMISSION MONITORING SYSTEMS IN STATIONARY SOURCES

#### 1. Applicability and Principle

1.1 Applicability. This specification is to be used for evaluating the acceptability of carbon monoxide (CO) continuous emission monitoring systems (CEMS) at the time of or soon after installation and whenever specified in an applicable subpart of the regulations.

This specification is not designed to evaluate the installed CEMS performance over an extended period of time nor does it identify specific calibration techniques and other auxiliary procedures to assess CEMS performance. The source owner or operator, however, is responsible to calibrate, maintain, and operate the CEMS. To evaluate CEMS performance, the Administrator may require, under section 114 of the Act, the source owner or operator to conduct CEMS performance evaluations at other times besides the initial test. See § 60.13(e).

The definitions, installation specifications, test procedures, data reduction procedures for determining calibration drifts (CD) and relative accuracy (RA), and reporting of Performance Specification 2 (PS 2), Sections 2, 3, 5, 6, 8, and 9 apply to this specification.

1.2 Principle. Reference method (RM), CD, and RA tests are conducted to determine that the CEMS conforms to the specification.

#### 2. Performance and Equipment Specifications

2.1 Instrument Zero and Span. This specification is the same as Section 4.1 of PS 2.

2.2 Calibration Drift. The CEMS calibration must not drift or deviate from the reference value of the calibration gas, gas cell, or optical filter by more than 5 percent of the established span value for 6 out of 7 test days (e.g., the established span value is 1000 ppm for subpart J affected facilities).

2.3 Relative Accuracy. The RA of the CEMS shall be no greater than 10 percent of the mean value of the RM test data in terms of the units of the emission standard or 5 percent of the applicable standard, whichever is greater.

#### 3. Relative Accuracy Test Procedure

3.1 Sampling Strategy for RM Tests, Correlation of RM and CEMS Data, Number of RM Tests, and Calculations. These are the same as PS 2, Sections 7.1, 7.2, 7.3, and 7.5, respectively.

3.2 Reference Methods. Unless otherwise specified in an applicable subpart of the regulation, Method 10 is the RM for this PS. When evaluating nondispersive infrared

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continuous emission analyzers, Method 10 shall use the alternative interference trap specified in section 10.1 of the method. Method 10A or 10B is an acceptable alternative to method 10.

### 4. Bibliography

1. Ferguson, B.B., R.E. Lester, and W.J. Mitchell. Field Evaluation of Carbon Monoxide and Hydrogen Sulfide Continuous Emission Monitors at an Oil Refinery. U.S. Environmental Protection Agency, Research Triangle Park, NC. Publication No. EPA-600/4-82-054. August 1982. 100 p.
2. Repp, M. Evaluation of Continuous Monitors for Carbon Monoxide in Stationary Sources. U.S. Environmental Protection Agency, Research Triangle Park, NC. Publication No. EPA-600/2-77-063. March 1977/ 135 p.
3. Smith, F., D.E. Wagoner, and R.P. Donovan. Guidelines for Development of a Quality Assurance Program: Volume VIII—Determination of CO Emissions from Stationary Sources by NDIR Spectrometry. U.S. Environmental Protection Agency, Research Triangle Park, NC. Publication No. EPA-650/4-74-005-h. February 1975. 96 p.

### PERFORMANCE SPECIFICATION 4A—SPECIFICATIONS AND TEST PROCEDURES FOR CARBON MONOXIDE CONTINUOUS EMISSION MONITORING SYSTEMS IN STATIONARY SOURCES

#### 1. Applicability and Principle

##### 1.1 Applicability.

1.1.1 This specification is to be used for evaluating the acceptability of carbon monoxide (CO) continuous emission monitoring systems (CEMS's) at the time of or soon after installation and whenever specified in an applicable subpart of the regulations.

1.1.2 This specification is not designed to evaluate the installed CEMS performance over an extended period of time nor does it identify specific calibration techniques and other auxiliary procedures to assess CEMS performance. The source owner or operator, however, is responsible to calibrate, maintain, and operate the CEMS. To evaluate CEMS performance, the Administrator may require, under section 114 of the Act, the source owner or operator to conduct CEMS performance evaluations at other times besides the initial test. See § 60.13(c).

1.1.3 The definition, installation specifications, test procedures, data reduction procedures for determining calibration drifts (CD) and relative accuracy (RA), and reporting of Performance Specification 2 (PS 2), sections 2, 3, 5, 6, 8, and 9 apply to this specification.

1.2 Principle. Reference method (RM), CD and RA tests are conducted to determine that the CEMS conforms to the specification.

#### 2. Performance and Equipment Specifications

2.1 Data Recorder Scale. This specification is the same as section 4.1 of PS 2. The CEMS shall be capable of measuring emission levels under normal conditions and under periods of short-duration peaks of high concentrations. This dual-range capability may be met using two separate analyzers, one for each range, or by using dual-range units which have the capability of measuring both levels with a single unit. In the latter case, when the reading goes above the full-scale measurement value of the lower range, the higher-range operation shall be started

automatically. The CEMS recorder range must include zero and a high-level value.

For the low-range scale, the high-level value shall be between 1.5 times the pollutant concentration corresponding to the emission standard level and the span value. For the high-range scale, the high-level value shall be set at 2000 ppm, as a minimum, and the range shall include the level of the span value. There shall be no concentration gap between the low- and high-range scales.

2.2 Interference Check. The CEMS must be shown to be free from the effects of any interferences.

2.3 Response Time. The CEMS response time shall not exceed 1.5 min to achieve 95 percent of the final stable value.

2.4 Calibration Drift. The CEMS calibration must not drift or deviate from the reference value of the calibration gas, gas cell, or optical filter by more than 5 percent of the established span value for 6 out of 7 test days.

2.5 Relative Accuracy. The RA of the CEMS shall be no greater than 10 percent of the mean value of the RM test data in terms of the units of the emission standard or 5 ppm, whichever is greater. Under conditions where the average CO emissions are less than 10 percent of the standard, a cylinder gas audit may be performed in place of the RA test to determine compliance with these limits. In this case, the cylinder gas shall contain CO in 12 percent carbon dioxide as an interference check. If this option is exercised, Method 10 must be used to verify that emission levels are less than 10 percent of the standard.

#### 3. Response Time Test Procedure

The response time test applies to all types of CEMS's, but will generally have significance only for extractive systems. The entire system is checked with this procedure including applicable sample extraction and transport, sample conditioning, gas analyses, and data recording.

Introduce zero gas into the system. For extractive systems, the calibration gases should be introduced at the probe as near to the sample location as possible. For in-situ systems, introduce the zero gas at the sample interface so that all components active in the analysis are tested. When the system output has stabilized (no change greater than 1 percent of full scale for 30 sec), switch to monitor stack effluent and wait for a stable value. Record the time (upscale response time) required to reach 95 percent of the final stable value. Next, introduce a high-level calibration gas and repeat the procedure (stabilize, switch the sample, stabilize, record). Repeat the entire procedure three times and determine the mean upscale and downscale response times. The slower or longer of the two means is the system response time.

#### 4. Relative Accuracy Test Procedure

4.1 Sampling Strategy for RM Tests, Correlation of RM and CEMS Data, Number of RM Tests, and Calculations. These are the same as PS 2, sections 7.1, 7.2, 7.3, and 7.5, respectively.

4.2 Reference Methods. Unless otherwise specified in an applicable subpart of the regulation, Method 10 is the RM for this PS. When evaluating nondispersive infrared continuous emission analyzers, Method 10 shall use the alternative interference trap specified in section 10.1 of the method. Method 10A or 10B is an acceptable alternative to Method 10.

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### 5. Bibliography

1. Same as in Performance Specification 4, section 4.
2. "Gaseous Continuous Emission Monitoring Systems—Performance Specification Guidelines for SO<sub>2</sub>, NO<sub>x</sub>, CO<sub>2</sub>, O<sub>2</sub>, and TRS." EPA-450/3-82-026. U.S. Environmental Protection Agency, Technical Support Division (MD-19), Research Triangle Park, NC 27711.

### PERFORMANCE SPECIFICATION 5—SPECIFICATIONS AND TEST PROCEDURES FOR TRS CONTINUOUS EMISSION MONITORING SYSTEMS IN STATIONARY SOURCES

#### 1. Applicability and Principle

1.1 Applicability. This specification is to be used for evaluating the acceptability of total reduced sulfur (TRS) and whenever specified in an applicable subpart of the regulations. (At present, these performance specifications do not apply to petroleum refineries, subpart J.) Sources affected by the promulgation of the specification shall be allowed 1 year beyond the promulgation date to install, operate, and test the CEMS. The CEMS's may include O<sub>2</sub> monitors which are subject to Performance Specification 3 (PS 3).

The definitions, installation specifications, test procedures, and data reduction procedures for determining calibration drifts (CD's) and relative accuracy (RA), and reporting of PS 2, Sections 2, 3, 4, 5, 6, 8, and 9 also apply to this specification and must be consulted. The performance and equipment specifications do not differ from PS 2 except as listed below and are included in this specification.

1.2 Principle. The CD and RA tests are conducted to determine conformance of the CEMS with the specification.

#### 2. Performance and Equipment Specifications

2.1 Instrument Zero and Span. The CEMS recorder span must be set at 90 to 100 percent of recorder full-scale using a span level between 1.5 times the pollutant concentration corresponding to the emission standard level and the span value. The CEMS design shall also allow the determination of calibration at the zero level of the calibration curve. If zero calibration is not possible or is impractical, this determination may be conducted at a low level (up to 20 percent of span value) point. The components of an acceptable permeation tube system are listed on pages 87-94 of Citation 4.2 of the Bibliography.

2.2 Calibration Drift. The CEMS detector calibration must not drift or deviate from the reference value of the calibration gas by more than 5 percent (1.5 ppm) of the established span value of 30 ppm for 6 out of 7 test days. If the CEMS includes pollutant and diluent monitors, the CD must be determined separately for each in terms of concentrations (see PS 3 for the diluent specifications).

2.3 The CEMS Relative Accuracy. The RA of the CEMS shall be no greater than 20 percent of the mean value of the reference method (RM) test data in terms of the units of the emission standard or 10 percent of the applicable standard, whichever is greater.

#### 3. Relative Accuracy Test Procedure

3.1 Sampling Strategy for RM Tests, Correlation of RM and CEMS Data, Number of RM Tests, and Calculations. This is the same as PS 2, Sections 7.1, 7.2, 7.3, and 7.5, respectively. Note: For Method 16, a sample is made up of at least three separate injects equally spaced over time. For Method 16A, a sample is collected for at least 1 hour.

3.2 Reference Methods. Unless otherwise specified in an applicable subpart of the regulations, Method 16, Method 16A, or other approved alternative, shall be the RM for TRS.

#### 4. Bibliography

1. Department of Commerce. Experimental Statistics. National Bureau of Standards. Handbook 91. 1963. Paragraphs 3-3.1.4, p. 3-31.
2. A Guide to the Design, Maintenance and Operation of TRS Monitoring Systems. National Council for Air and Stream Improvement Technical Bulletin No. 89. September 1977.
3. Observation of Field Performance of TRS Monitors on a Kraft Recovery Furnace. National Council for Air and Stream Improvement Technical Bulletin No. 91. January 1978.

### PERFORMANCE SPECIFICATION 6—SPECIFICATIONS AND TEST PROCEDURES FOR CONTINUOUS EMISSION RATE MONITORING SYSTEMS IN STATIONARY SOURCES

#### 1. Applicability and Principle

1.1 Applicability. The applicability for this specification is the same as Section 1.1 of Performance Specification 2 (PS 2), except this specification is to be used for evaluating the acceptability of continuous emission rate monitoring systems (CERMS's). The installation and measurement location specifications, performance specification test procedure, data reduction procedures, and reporting requirements of PS 2, Section 3, 5, 8, and 9, apply to this specification.

1.2 Principle. Reference method (RM), calibration drift (CD), and relative accuracy (RA) tests are conducted to determine that the CERMS conforms to the specification.

#### 2. Defaults

The definitions are the same as in Section 2 of PS 2, except that this specification refers to the continuous emission rate monitoring system rather than the continuous emission monitoring system. The following definitions are added:

2.1 Continuous Emission Rate Monitoring System (CERMS). The total equipment required for the determination and recording of the pollutant mass emission rate (in terms of mass per unit of time).

2.2 Flow Rate Sensor. That portion of the CERMS that senses the volumetric flow rate and generates an output proportional to flow rate. The flow rate sensor shall have provisions to check the CD for each flow rate parameter that it measures individually (e.g., velocity pressure).

#### 3. Performance and Equipment Specifications

3.1 Data Recorder Scale. Same as Section 4.1 of PS 2.

3.2 CD. Since the CERMS includes analyzers for several measurements, the CD shall be determined separately for each analyzer in terms of its specific measurement. The calibration for each analyzer used for the measurement of flow rate except a temperature analyzer shall not drift or deviate from either of its reference values by more than 3 percent of 1.25 times the average potential absolute value for that measurement. For a temperature analyzer, the specification is 1.5 percent of 1.25 times the

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average potential absolute temperature. The CD specification for each analyzer for which other PS's have been established (e.g., PS 2 for SO<sub>2</sub> and NO<sub>x</sub>), shall be the same as in the applicable PS.

3.3 CERMS RA. The RA of the CERMS shall be no greater than 20 percent of the mean value of the RM's test data in terms of the units of the emission standard, or 10 percent of the applicable standard, whichever is greater.

### 4. CD Test Procedure

The CD measurements are to verify the ability of the CERMS to conform to the established CERMS calibrations used for determining the emission rate. Therefore, if periodic automatic or manual adjustments are made to the CERMS test data and calibration settings, conduct the CD tests immediately before these adjustments, or conduct them in such a way what CD can be determined.

Conduct the CD tests for pollutant concentration at the two values specified in Section 4.1 of PS 2. For each of the other parameters that are selectively measured by the CERMS (e.g., velocity pressure), use two analogous values: one that represents zero to 20 percent of the high-level value (a value that is between 1.25 and 2 times the average potential value) for that parameter, and one that represents 50 to 100 percent of the high-level value. Introduce, or activate internally, the reference signals to the CERMS (these need not be certified). Record the CERMS response to each, and subtract this value from the respective reference value (see example data sheet in Figure 6-1).

### 5. RA Test Procedure

5.1 Sampling Strategy for RM's Tests, Correlation of RM and CERMS Data, Number of RM's Tests, and Calculations. These are the same as PS 2, Sections 7.1, 7.2, 7.3, and 7.5, respectively. Summarize the results on a data sheet. An example is shown in Figure 6-2. The RA test may be conducted during the CD test period.

5.2 Reference Methods (RM's). Unless otherwise specified in the applicable subpart of the regulations, the RM for the pollutant gas is the appendix A method that is cited for compliance test purposes, or its approved alternatives. Methods 2, 2A, 2B, 2C, or 2D, as applicable are the RM's for the determination of volumetric flow rate.

### 6. Bibliography

1. Brooks, E.F., E.C. Beder, C.A. Flegal, D.J. Luciani, and R. Williams. Continuous Measurement of Total Gas Flow Rate from Stationary Sources. U.S. Environmental Protection Agency, Research Triangle Park, NC. Publication No. EPA-650/2-75-020. February 1975. 248 p.

PERFORMANCE SPECIFICATION 7—SPECIFICATIONS AND TEST PROCEDURES FOR HYDROGEN SULFIDE CONTINUOUS EMISSION MONITORING SYSTEMS IN STATIONARY SOURCES

#### 1. Applicability and Principle

1.1 *Applicability.* 1.1.1 This specification is to be used for evaluating the acceptability of hydrogen sulfide (H<sub>2</sub>S) continuous emission monitoring systems (CEMS's) at the time of or soon after installation and whenever specified in an applicable subpart of the regulations.

1.1.2 This specification is not designed to evaluate the installed CEMS performance over an extended period of time nor does it identify specific calibration techniques and other auxiliary procedures to assess CEMS performance. The source owner or operator, however, is responsible to calibrate, maintain, and operate the CEMS. To evaluate CEMS performance, the Administrator may require, under Section 114 of the Act, the source owner or operator to conduct CEMS performance evaluations at other times besides the initial test. See § 60.13(c).

1.1.3 The definitions, installation specifications, test procedures, data reduction procedures for determining calibration drifts (CD) and relative accuracy (RA), and reporting of Performance Specification 2 (PS 2), Sections 2, 3, 5, 6, 8, and 9 apply to this specification.

1.2 Principle. Reference method (RM), CD, and RA tests are conducted to determine that the CEMS conforms to the specification.

### 2. Performance and Equipment Specifications

2.1 *Instrument zero and span.* This specification is the same as Section 4.1 of PS 2.

2.2 *Calibration drift.* The CEMS calibration must not drift or deviate from the reference value of the calibration gas or reference source by more than 5 percent of the established span value for 6 out of 7 test days (e.g., the established span value is 300 ppm for subpart J fuel gas combustion devices).

2.3 *Relative accuracy.* The RA of the CEMS shall be no greater than 20 percent of the mean value of the RM test data in terms of the units of the emission standard or 10 percent of the applicable standard, whichever is greater.

### 3. Relative Accuracy Test Procedure

3.1 Sampling Strategy for RM Tests, Correlation of RM and CEMS Data Number of RM Tests, and Calculations. These are the same as that in PS 2, § 7.1, 7.2, 7.3, and 7.5, respectively.

3.2 Reference Methods. Unless otherwise specified in an applicable subpart of the regulation, Method 11 is the RM for this PS.

### 4. Bibliography

1. U.S. Environmental Protection Agency. Standards of Performance for New Stationary Sources; Appendix B; Performance Specifications 2 and 3 for SO<sub>2</sub>, NO<sub>x</sub>, CO<sub>2</sub>, and O<sub>2</sub> Continuous Emission Monitoring Systems; Final Rule. 48 CFR 23608. Washington, DC, U.S. Government Printing Office. May 25, 1983.

2. U.S. Government Printing Office. Gaseous Continuous Emission Monitoring Systems—Performance Specification Guidelines for SO<sub>2</sub>, NO<sub>x</sub>, CO<sub>2</sub>, O<sub>2</sub>, and TRS. U.S. Environmental Protection Agency. Washington, DC, EPA-450/3-82-026. October 1982. 26p.

3. Maines, G.D., W.C. Kelly (Scott Environmental Technology, Inc.), and J.B. Homolya. Evaluation of Monitors for Measuring H<sub>2</sub>S in Refinery Gas. Prepared for the U.S. Environmental Protection Agency. Research Triangle Park, NC. Contract No. 68-02-2707. 1978. 60 p.

4. Ferguson, B.B., R.E. Lester (Harmon Engineering and Testing), and W.J. Mitchell. Field Evaluation of Carbon Monoxide and Hydrogen Sulfide Continuous Emission Monitors at an Oil Refinery. Prepared for the U.S. Environmental Protection Agency. Research Triangle

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Park, NC. Publication No. EPA-600/4-82-054. August 1982. 100 p.

### PERFORMANCE SPECIFICATION 8—PERFORMANCE SPECIFICATIONS FOR VOLATILE ORGANIC COMPOUND CONTINUOUS EMISSION MONITORING SYSTEMS IN STATIONARY SOURCES

#### 1. Applicability and Principle

##### 1.1 Applicability.

1.1.1 This specification is to be used for evaluating a continuous emission monitoring system (CEMS) that measures a mixture of volatile organic compounds (VOC's) and generates a single combined response value. The VOC detection principle may be flame ionization (FI), photoionization (PI), nondispersive infrared absorption (NDIR), or any other detection principle that is appropriate for the VOC species present in the emission gases and that meets this performance specification. The performance specification includes procedures to evaluate the acceptability of the CEMS at the time of or soon after its installation and whenever specified in emission regulations or permits. This specification is not designed to evaluate the installed CEMS performance over an extended period of time, nor does it identify specific calibration techniques and other auxiliary procedures to assess the CEMS performance. However, it is the responsibility of the source owner or operator, to calibrate, maintain, and operate the CEMS properly. Under section 114 of the Act, the Administrator may require the operator to evaluate the CEMS performance by conducting CEMS performance evaluations in addition to the initial test. See section 60.13(c).

The definitions, installation and measurement location specifications, test procedures, data reduction procedures, reporting requirements, and bibliography are the same as in PS 2, sections 2, 3, 5, 6, 8, 9, and 10, and also apply to VOC CEMS's under this specification. The performance and equipment specifications and the relative accuracy (RA) test procedures for VOC CEMS do not differ from those for SO<sub>2</sub> and NO<sub>x</sub> CEMS, except as noted below.

1.1.2 In most emission circumstances, most VOC monitors can provide only a relative measure of the total mass or volume concentration of a mixture of organic gases, rather than an accurate quantification. This problem is removed when an emission standard is based on a total VOC measurement as obtained with a particular detection principle. In those situations where a true mass or volume VOC concentration is needed, the problem can be mitigated by using the VOC CEMS as a relative indicator of total VOC concentration if statistical analysis indicates that a sufficient margin of compliance exists for this approach to be acceptable. Otherwise, consideration can be given to calibrating the CEMS with a mixture of the same VOC's in the same proportions as they actually occur in the measured source. In those circumstances where only one organic species is present in the source, or where equal incremental amounts of each of the organic species present generate equal CEMS responses, the latter choice can be more easily achieved.

1.2 Principle. Calibration drift and relative accuracy tests are conducted to determine the adherence of the CEMS to specifications given for those items. The performance specifications include criteria for installation and measurement location, equipment and performance, and procedures for testing and data reduction.

#### 2. Performance and Equipment Specifications

2.1 VOC CEMS Selection. When possible, select a VOC CEMS with the detection principle of the reference method specified in the regulation or permit (usually either FI, NDIR, or PI). Otherwise, use knowledge of the source process chemistry, previous emission studies, or gas chromatographic analysis of the source gas to select an appropriate VOC CEMS. Exercise extreme caution in choosing and installing any CEMS in an area with the potential for explosive hazards.

2.2 Data Recorder Scale. Same as section 4.1 of PS 2.

2.3 Calibration Drift. The CEMS calibration must not drift by more than 2.5 percent of the span value.

2.4 CEMS Relative Accuracy. Unless stated otherwise in the regulation or permit, the RA of the CEMS must be no greater than 20 percent of the mean value of the reference method (RM) test data in terms of the units of the emission standard, or 10 percent of the applicable standard, whichever is greater.

#### 3. Relative Accuracy Test Procedure

3.1 Sampling Strategy for RM Tests, Correlation of RM and CEMS Data, Number of RM Tests, and Calculations. Follow PS 2, sections 7.1, 7.2, 7.3, and 7.5, respectively.

3.2 Reference Method. Use the method specified in the applicable regulation or permit, or any approved alternative, as the RM.

### PERFORMANCE SPECIFICATION 9—SPECIFICATIONS AND TEST PROCEDURES FOR GAS CHROMATOGRAPHIC CONTINUOUS EMISSION MONITORING SYSTEMS IN STATIONARY SOURCES

#### 1. Applicability and Principle

1.1 Applicability. These requirements apply to continuous emission monitoring systems (CEMS) that use gas chromatography (GC) to measure gaseous organic compound emissions. The requirements include procedures intended to evaluate the acceptability of the CEMS at the time of its installation and whenever specified in regulations or permits. Quality assurance procedures for calibrating, maintaining, and operating the CEMS properly at all times are also given in this procedure.

1.2 Principle. Calibration precision, calibration error, and performance audit tests are conducted to determine conformance of the CEMS with these specifications. Daily calibration and maintenance requirements are also specified.

#### 2. Definitions

2.1 Gas Chromatograph (GC). That portion of the system that separates and detects organic analytes and generates an output proportional to the gas concentration. The GC must be temperature controlled.

NOTE: The term "temperature controlled" refers to the ability to maintain a certain temperature around the column. Temperature-programmable GC is not required for this performance specification, as long as all other requirements for precision, linearity, and accuracy listed in this performance specification are met. It should be noted that temperature programming a GC will speed up peak elution, thus allowing increased sampling frequency.

2.1.1 Column. An analytical column capable of separating the analytes of interest.

2.1.2 Detector. A detection system capable of detecting and quantifying all analytes of interest.

2.1.3 Integrator. That portion of the system that quantifies the area under a particular sample peak generated by the GC.

2.1.4 Data Recorder. A strip chart recorder, computer, or digital recorder capable of recording all readings within the instrument's calibration range.

2.2 Calibration Precision. The error between triplicate injections of each calibration standard.

### 3. Installation and Measurement Location Specifications

Install the CEMS in a location where the measurements are representative of the source emissions. Consider other factors, such as ease of access for calibration and maintenance purposes. The location should not be close to air in-leakages. The sampling location should be at least two equivalent duct diameters downstream from the nearest control device, point of pollutant generation, or other point at which a change in the pollutant concentration or emission rate occurs. The location should be at least 0.5 diameter upstream from the exhaust or control device. To calculate equivalent duct diameter, see section 2.1 of Method 1 (40 CFR part 60, appendix A). Sampling locations not conforming to the requirements in this section may be used if necessary upon approval of the Administrator.

### 4. CEMS Performance and Equipment Specifications

4.1 Presurvey Sample Analysis and GC Selection. Determine the pollutants to be monitored from the applicable regulation or permit and determine the approximate concentration of each pollutant (this information can be based on past compliance test results). Select an appropriate GC configuration to measure the organic compounds. The GC components should include a heated sample injection loop (or other sample introduction systems), separatory column, temperature-controlled oven, and detector. If the source chooses dual column and/or dual detector configurations, each column/detector is considered a separate instrument for the purpose of this performance specification and thus the procedures in this performance specification shall be carried out on each system. If this method is applied in highly explosive areas, caution should be exercised in selecting the equipment and method of installation.

4.2 Sampling System. The sampling system shall be heat traced and maintained at a minimum of 120 °C with no cold spots. All system components shall be heated, including the probe, calibration valve, sample lines, sampling loop (or sample introduction system), GC oven, and the detector block (when appropriate for the type of detector being utilized, e.g., flame ionization detector).

4.3 Calibration Gases. Obtain three concentrations of calibration gases certified by the manufacturer to be accurate to within 2 percent of the value on the label. A gas dilution system may be used to prepare the calibration gases from a high concentration certified standard if the gas dilution system meets the requirements specified in Test Method 205, 40 CFR part 51, appendix M. The performance test specified in Test Method 205 shall be repeated quarterly, and the results of the Method 205 test shall be included in the report. The calibration gas concentration of each target analyte shall be as follows

(measured concentration is based on the presurvey concentration determined in section 4.1).

NOTE: If the low level calibration gas concentration falls at or below the limit of detection for the instrument for any target pollutant, a calibration gas with a concentration at 4 to 5 times the limit of detection for the instrument may be substituted for the low-level calibration gas listed in section 4.3.1

4.3.1 Low-level. 40-60 percent of measured concentration.

4.3.2 Mid-level. 90-110 percent of measured concentration.

4.3.3 High-level. 140-160 percent of measured concentration, or select highest expected concentration.

4.4 Performance Audit Gas. A certified EPA audit gas shall be used, when possible. A Protocol 1 gas mixture containing all the target compounds within the calibration range may be used when EPA performance audit materials are not available. The instrument relative error shall be  $\leq 10$  percent of the certified value of the audit gas.

4.5 Calibration Error (CE). The CEMS must allow the determination of CE at all three calibration levels. The average CEMS calibration response must not differ by more than 10 percent of calibration gas value at each level after each 24-hour period of the initial test.

4.6 Calibration Precision and Linearity. For each triplicate injection at each concentration level for each target analyte, any one injection shall not deviate more than 5 percent from the average concentration measured at that level. The linear regression curve for each organic compound at all three levels shall have an  $r^2 \geq 0.995$  (using Equation 1).

4.7 Measurement Frequency. The sample to be analyzed shall flow continuously through the sampling system. The sampling system time constant (T) shall be  $\leq 5$  minutes or the sampling frequency specified in the applicable regulation, whichever is less. Use Equation 3 to determine T. The analytical system shall be capable of measuring the effluent stream at the frequency specified in the appropriate regulation or permit.

### 5. Performance Specification Test (PST) Periods

5.1 Pretest Preparation Period. Using the procedures described in Method 18 (40 CFR part 60, appendix A), perform initial tests to determine GC conditions that provide good resolution and minimum analysis time for compounds of interest. Resolution interferences that may occur can be eliminated by appropriate GC column and detector choice or by shifting the retention times through changes in the column flow rate and the use of temperature programming.

5.2 7-Day CE Test Period. At the beginning of each 24-hour period, set the initial instrument setpoints by conducting a multipoint calibration for each compound. The multipoint calibration shall meet the requirements in section 4.7. Throughout the 24-hour period, sample and analyze the stack gas at the sampling intervals prescribed in the regulation or permit. At the end of the 24-hour period, inject the three calibration gases for each compound in triplicate and determine the average instrument response. Determine the CE for each pollutant at each level using the equation in section 6.2. Each CE shall be  $\leq 10$  percent. Repeat this procedure six more times for a total of 7 consecutive days.

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5.3 Performance Audit Test Periods. Conduct the performance audit once during the initial 7-day CE test and quarterly thereafter. Sample and analyze the EPA audit gas(es) (or the Protocol 1 gas mixture if an EPA audit gas is not available) three times. Calculate the average instrument response. Report the audit results as part of the reporting requirements in the appropriate regulation or per-

mit (if using a Protocol 1 gas mixture, report the certified cylinder concentration of each pollutant).

### 6. Equations

6.1 Coefficient of Determination. Calculate  $r^2$  using linear regression analysis and the average concentrations obtained at three calibration points as shown in Equation 1.

ER15DE94.000

Where:

$r^2$ =Coefficient of determination.  
 $n$ =Number of measurement points.  
 $x$ =CEMS response.  
 $y$ =Actual value of calibration standard.

6.2 Calibration Error Determination. Determine the percent calibration error (CE) at each concentration for each pollutant using the following equation.

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where:

$C_m$ =average instrument response, ppm.  
 $C_c$ =cylinder gas value, ppm.

6.3 Sampling System Time Constant (T).

ER15DE94.002

where:

$F$ =Flow rate of stack gas through sampling system, in liters/min.

$V$ =Sample system volume, in Liters, which is the volume inside the sample probe and tubing leading from the stack to the sampling loop.

### 7. Daily Calibration

7.1 Initial Multipoint Calibration. After initial startup of the GC, after routine maintenance or repair, or at least once per month, conduct a multipoint calibration of the GC for each target analyte. The multipoint calibration for each analyte shall meet the requirements in section 4.7.

7.2 Daily Calibration. Once every 24 hours, analyze the mid-level calibration standard for each analyte in triplicate. Calculate the average instrument response for each analyte. The average instrument response shall not vary more than 10 percent from the certified concentration value of the cylinder for each analyte. If the difference between the analyzer response and the cylinder concentration for any target compound is greater than 10 percent, immediately take corrective action on the instrument if necessary, and conduct an initial multipoint calibration as described in section 7.1.

*8. Reporting*

Follow the reporting requirements of the applicable regulation or permit. If the reporting requirements include the results of this performance specification, summarize in tabular form the results of the CE tests. Include all data sheets, calculations, CEMS data records, performance audit results, and calibration gas concentrations and certifications.

[48 FR 13327, Mar. 30, 1983 and 48 FR 23611, May 25, 1983, as amended at 48 FR 32986, July 20, 1983; 51 FR 31701, Aug. 5, 1985; 52 FR 17556, May 11, 1987; 52 FR 30675, Aug. 18, 1987; 52 FR 34650, Sept. 14, 1987; 53 FR 7515, Mar. 9, 1988; 53 FR 41335, Oct. 21, 1988; 55 FR 18876, May 7, 1990; 55 FR 40178, Oct. 2, 1990; 55 FR 47474, Nov. 14, 1990; 56 FR 5526, Feb. 11, 1991; 59 FR 64593, Dec. 15, 1994]

***40 CFR PART 60 APPENDIX F***

APPENDIX F TO PART 60—QUALITY ASSURANCE PROCEDURES

PROCEDURE 1. QUALITY ASSURANCE REQUIREMENTS FOR GAS CONTINUOUS EMISSION MONITORING SYSTEMS USED FOR COMPLIANCE DETERMINATION

1. *Applicability and Principle*

1.1 *Applicability.* Procedure 1 is used to evaluate the effectiveness of quality control (QC) and quality assurance (QA) procedures and the quality of data produced by any continuous emission monitoring system (CEMS) that is used for determining compliance with the emission standards on a continuous basis as specified in the applicable regulation. The CEMS may include pollutant (e.g., SO<sub>2</sub> and NO<sub>x</sub>) and diluent (e.g., O<sub>2</sub> or CO<sub>2</sub>) monitors.

This procedure specifies the minimum QA requirements necessary for the control and assessment of the quality of CEMS data submitted to the Environmental Protection Agency (EPA). Source owners and operators responsible for one or more CEMS's used for compliance monitoring must meet these minimum requirements and are encouraged to develop and implement a more extensive QA program or to continue such programs where they already exist.

Data collected as a result of QA and QC measures required in this procedure are to be submitted to the Agency. These data are to be used by both the Agency and the CEMS operator in assessing the effectiveness of the CEMS QC and QA procedures in the maintenance of acceptable CEMS operation and valid emission data.

Appendix F, Procedure 1 is applicable December 4, 1987. The first CEMS accuracy assessment shall be a relative accuracy test audit (RATA) (see section 5) and shall be completed by March 4, 1988 or the date of the initial performance test required by the applicable regulation, whichever is later.

1.2 *Principle.* The QA procedures consist of two distinct and equally important functions. One function is the assessment of the quality of the CEMS data by estimating accuracy. The other function is the control and improvement of the quality of the CEMS data by implementing QC policies and corrective actions. These two functions form a control loop: When the assessment function indicates that the data quality is inadequate, the control effort must be increased until the data quality is acceptable. In order to provide uniformity in the assessment and reporting of data quality, this procedure explicitly specifies the assessment methods for response drift and accuracy. The methods are based on procedures included in the applicable performance specifications (PS's) in appendix B of 40 CFR part 60. Procedure 1 also requires the analysis of the EPA audit samples concurrent with certain reference method (RM) analyses as specified in the applicable RM's.

Because the control and corrective action function encompasses a variety of policies, specifications, standards, and corrective measures, this procedure treats QC requirements in general terms to allow each source owner or operator to develop a QC system that is most effective and efficient for the circumstances.

2. *Definitions*

2.1 *Continuous Emission Monitoring System.* The total equipment required for the determination of a gas concentration or emission rate.

2.2 *Diluent Gas.* A major gaseous constituent in a gaseous pollutant mixture. For combustion sources, CO<sub>2</sub> and O<sub>2</sub> are the major gaseous constituents of interest.

2.3 *Span Value.* The upper limit of a gas concentration measurement range that is specified for affected source categories in the applicable subpart of the regulation.

2.4 *Zero, Low-Level, and High-Level Values.* The CEMS response values related to the source specific span value. Determination of zero, low-level, and high-level values is defined in the appropriate PS in appendix B of this part.

2.5 *Calibration Drift (CD).* The difference in the CEMS output reading from a reference value after a period of operation during which no unscheduled maintenance, repair or adjustment took place. The reference value may be supplied by a cylinder gas, gas cell, or optical filter and need not be certified.

2.6 *Relative Accuracy (RA).* The absolute mean difference between the gas concentration or emission rate determined by the CEMS and the value determined by the RM's plus the 2.5 percent error confidence coefficient of a series of tests divided by the mean of the RM tests or the applicable emission limit.

3. *QC Requirements*

Each source owner or operator must develop and implement a QC program. As a minimum, each QC program must include written procedures which should describe in detail, complete, step-by-step procedures and operations for each of the following activities:

1. Calibration of CEMS.
2. CD determination and adjustment of CEMS.
3. Preventive maintenance of CEMS (including spare parts inventory).
4. Data recording, calculations, and reporting.
5. Accuracy audit procedures including sampling and analysis methods.
6. Program of corrective action for malfunctioning CEMS.

As described in Section 5.2, whenever excessive inaccuracies occur for two consecutive quarters, the source owner or operator must revise the current written procedures or modify or replace the CEMS to correct the deficiency causing the excessive inaccuracies.

These written procedures must be kept on record and available for inspection by the enforcement agency.

4. *CD Assessment*

4.1 *CD Requirement.* As described in 40 CFR 60.13(d), source owners and operators of CEMS must check, record, and quantify the CD at two concentration values at least once daily (approximately 24 hours) in accordance with the method prescribed by the manufacturer. The CEMS calibration must, as minimum, be adjusted whenever the daily zero (or low-level) CD or the daily high-level CD exceeds two times the limits of the applicable PS's in appendix B of this regulation.

4.2 *Recording Requirement for Automatic CD Adjusting Monitors.* Monitors that automatically adjust the data to the corrected calibration values (e.g., microprocessor control) must be programmed to record the unadjusted concentration measured in the CD prior to resetting the calibration, if performed, or record the amount of adjustment.

4.3 *Criteria for Excessive CD.* If either the zero (or low-level) or high-level CD result exceeds twice the applicable drift specification in appendix B for five, consecutive, daily periods, the CEMS is out-of-control. If ei-

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ther the zero (or low-level) or high-level CD result exceeds four times the applicable drift specification in appendix B during any CD check, the CEMS is out-of-control. If the CEMS is out-of-control, take necessary corrective action. Following corrective action, repeat the CD checks.

**4.3.1 Out-Of-Control Period Definition.** The beginning of the out-of-control period is the time corresponding to the completion of the fifth, consecutive, daily CD check with a CD in excess of two times the allowable limit, or the time corresponding to the completion of the daily CD check preceding the daily CD check that results in a CD in excess of four times the allowable limit. The end of the out-of-control period is the time corresponding to the completion of the CD check following corrective action that results in the CD's at both the zero (or low-level) and high-level measurement points being within the corresponding allowable CD limit (i.e., either two times or four times the allowable limit in appendix B).

**4.3.2 CEMS Data Status During Out-of-Control Period.** During the period the CEMS is out-of-control, the CEMS data may not be used in calculating emission compliance nor be counted towards meeting minimum data availability as required and described in the applicable subpart [e.g., § 60.47a(f)].

**4.4 Data Recording and Reporting.** As required in § 60.7(d) of this regulation (40 CFR part 60), all measurements from the CEMS must be retained on file by the source owner for at least 2 years. However, emission data obtained on each successive day while the CEMS is out-of-control may not be included as part of the minimum daily data requirement of the applicable subpart [e.g., § 60.47a(f)] nor be used in the calculation of reported emissions for that period.

### 5. Data Accuracy Assessment

**5.1 Auditing Requirements.** Each CEMS must be audited at least once each calendar quarter. Successive quarterly audits shall occur no closer than 2 months. The audits shall be conducted as follows:

**5.1.1 Relative Accuracy Test Audit (RATA).** The RATA must be conducted at least once every four calendar quarters. Conduct the RATA as described for the RA test procedure in the applicable PS in appendix B (e.g., PS 2 for SO<sub>2</sub> and NO<sub>x</sub>). In addition, analyze the appropriate performance audit samples received from EPA as described in the applicable sampling methods (e.g., Methods 6 and 7).

**5.1.2 Cylinder Gas Audit (CGA).** If applicable, a CGA may be conducted in three of four calendar quarters, but in no more than three quarters in succession.

To conduct a CGA: (1) Challenge the CEMS (both pollutant and diluent portions of the CEMS, if applicable) with an audit gas of known concentration at two points within the following ranges:

Audit point	Audit range		
	Pollutant monitors	Diluent monitors for—	
		CO <sub>2</sub>	O <sub>2</sub>
1 .....	20 to 30% of span value.	5 to 8% by volume.	4 to 6% by volume.
2 .....	50 to 60% of span value.	10 to 14% by volume.	8 to 12% by volume.

Challenge the CEMS three times at each audit point, and use the average of the three responses in determining accuracy.

Use of separate audit gas cylinder for audit points 1 and 2. Do not dilute gas from audit cylinder when challenging the CEMS.

The monitor should be challenged at each audit point for a sufficient period of time to assure adsorption-desorption of the CEMS sample transport surfaces has stabilized.

(2) Operate each monitor in its normal sampling mode, i.e., pass the audit gas through all filters, scrubbers, conditioners, and other monitor components used during normal sampling, and as much of the sampling probe as is practical. At a minimum, the audit gas should be introduced at the connection between the probe and the sample line.

(3) Use audit gases that have been certified by comparison to National Bureau of Standards (NBS) gaseous Standard Reference Materials (SRM's) or NBS/EPA approved gas manufacturer's Certified Reference Materials (CRM's) (See Citation 1) following EPA Traceability Protocol No. 1 (See Citation 2). As an alternative to Protocol No. 1 audit gases, CRM's may be used directly as audit gases. A list of gas manufacturers that have prepared approved CRM's is available from EPA at the address shown in Citation 1. Procedures for preparation of CRM's are described in Citation 1. Procedures for preparation of EPA Traceability Protocol 1 materials are described in Citation 2.

The difference between the actual concentration of the audit gas and the concentration indicated by the monitor is used to assess the accuracy of the CEMS.

**5.1.3 Relative Accuracy Audit (RAA).** The RAA may be conducted three of four calendar quarters, but in no more than three quarters in succession. To conduct a RAA, follow the procedure described in the applicable PS in appendix B for the relative accuracy test, except that only three sets of measurement data are required. Analyses of EPA performance audit samples are also required.

The relative difference between the mean of the RM values and the mean of the CEMS responses will be used to assess the accuracy of the CEMS.

**5.1.4 Other Alternative Audits.** Other alternative audit procedures may be used as approved by the Administrator for three of four calendar quarters. One RATA is required at least once every four calendar quarters.

**5.2 Excessive Audit Inaccuracy.** If the RA, using the RATA, CGA, or RAA exceeds the criteria in section 5.2.3, the CEMS is out-of-control. If the CEMS is out-of-control, take necessary corrective action to eliminate the problem. Following corrective action, the source owner or operator must audit the CEMS with a RATA, CGA, or RAA to determine if the CEMS is operating within the specifications. A RATA must always be used following an out-of-control period resulting from a RATA. The audit following corrective action does not require analysis of EPA performance audit samples. If audit results show the CEMS to be out-of-control, the CEMS operator shall report both the audit showing the CEMS to be out-of-control and the results of the audit following corrective action showing the CEMS to be operating within specifications.

**5.2.1 Out-Of-Control Period Definition.** The beginning of the out-of-control period is the time corresponding to the completion of the sampling for the RATA, RAA, or CGA. The end of the out-of-control period is the time

corresponding to the completion of the sampling of the subsequent successful audit.

5.2.2 CEMS Data Status During Out-Of-Control Period. During the period the monitor is out-of-control, the CEMS data may not be used in calculating emission compliance nor be counted towards meeting minimum data availability as required and described in the applicable subpart [e.g., § 60.47a(f)].

5.2.3 Criteria for Excessive Audit Inaccuracy. Unless specified otherwise in the applicable subpart, the criteria for excessive inaccuracy are:

(1) For the RATA, the allowable RA in the applicable PS in appendix B.

(2) For the CGA, ±15 percent of the average audit value or ±5 ppm, whichever is greater.

(3) For the RAA, ±15 percent of the three run average or ±7.5 percent of the applicable standard, whichever is greater.

5.3 Criteria for Acceptable QC Procedure. Repeated excessive inaccuracies (i.e., out-of-control conditions resulting from the quarterly audits) indicates the QC procedures are inadequate or that the CEMS is incapable of providing quality data. Therefore, whenever excessive inaccuracies occur for two consecutive quarters, the source owner or operator must revise the QC procedures (see Section 3) or modify or replace the CEMS.

6. Calculations for CEMS Data Accuracy

6.1 RATA RA Calculation. Follow the equations described in Section 8 of appendix B, PS 2 to calculate the RA for the RATA. The RATA must be calculated in units of the applicable emission standard (e.g., ng/l).

6.2 RAA Accuracy Calculation. Use Equation 1-1 to calculate the accuracy for the RAA. The RAA must be calculated in units of the applicable emission standard (e.g., ng/l).

6.3 CGA Accuracy Calculation. Use Equation 1-1 to calculate the accuracy for the CGA, which is calculated in units of the appropriate concentration (e.g., ppm SO<sub>2</sub> or percent O<sub>2</sub>). Each component of the CEMS must meet the acceptable accuracy requirement.

$$A = \frac{C_m - C_a}{C_a} \times 100 \quad \text{Eq. 1-1}$$

where:

A = Accuracy of the CEMS, percent.

C<sub>m</sub> = Average CEMS response during audit in units of applicable standard or appropriate concentration.

C<sub>a</sub> = Average audit value (CGA certified value or three-run average for RAA) in units of applicable standard or appropriate concentration.

6.4 Example Accuracy Calculations. Example calculations for the RATA, RAA, and CGA are available in Citation 3.

7. Reporting Requirements

At the reporting interval specified in the applicable regulation, report for each CEMS the accuracy results from Section 6 and the CD assessment results from Section 4. Report the drift and accuracy information as a Data Assessment Report (DAR), and include one copy of this DAR for each quarterly audit with the report of emissions required under the applicable subparts of this part.

As a minimum, the DAR must contain the following information:

1. Source owner or operator name and address.

2. Identification and location of monitors in the CEMS.
3. Manufacturer and model number of each monitor in the CEMS.

4. Assessment of CEMS data accuracy and date of assessment as determined by a RATA, RAA, or CGA described in Section 5 including the RA for the RATA, the A for the RAA or CGA, the RM results, the cylinder gases certified values, the CEMS responses, and the calculations results as defined in Section 6. If the accuracy audit results show the CEMS to be out-of-control, the CEMS operator shall report both the audit results showing the CEMS to be out-of-control and the results of the audit following corrective action showing the CEMS to be operating within specifications.

5. Results from EPA performance audit samples described in Section 5 and the applicable RM's.

6. Summary of all corrective actions taken when CEMS was determined out-of-control, as described in Sections 4 and 5.

An example of a DAR format is shown in Figure 1.

8. Bibliography

1. "A Procedure for Establishing Traceability of Gas Mixtures to Certain National Bureau of Standards Standard Reference Materials." Joint publication by NBS and EPA-600/7-81-010. Available from the U.S. Environmental Protection Agency, Quality Assurance Division (MD-77), Research Triangle Park, NC 27711.

2. "Traceability Protocol for Establishing True Concentrations of Gases Used for Calibration and Audits of Continuous Source Emission Monitors (Protocol Number 1)" June 1978. Section 3.0.4 of the Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, Stationary Source Specific Methods, EPA-600/4-77-027b, August 1977. U.S. Environmental Protection Agency, Office of Research and Development Publications, 26 West St. Clair Street, Cincinnati, OH 45268.

3. Calculation and Interpretation of Accuracy for Continuous Emission Monitoring Systems (CEMS). Section 3.0.7 of the Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, Stationary Source Specific Methods, EPA-600/4-77-027b, August 1977. U.S. Environmental Protection Agency, Office of Research and Development Publications, 26 West St. Clair Street, Cincinnati, OH 45268.

FIGURE 1—EXAMPLE FORMAT FOR DATA ASSESSMENT REPORT

Period ending date: \_\_\_\_\_  
 Year: \_\_\_\_\_  
 Company name: \_\_\_\_\_  
 Plant name: \_\_\_\_\_  
 Source unit no.: \_\_\_\_\_  
 CEMS manufacturer: \_\_\_\_\_  
 Model no.: \_\_\_\_\_  
 CEMS serial no.: \_\_\_\_\_  
 CEMS type (e.g., in situ): \_\_\_\_\_  
 CEMS sampling location (e.g., control device outlet): \_\_\_\_\_  
 CEMS span values as per the applicable regulation:  
 \_\_\_\_\_ (e.g., SO<sub>2</sub> \_\_\_\_\_ ppm, NO<sub>x</sub> \_\_\_\_\_ ppm).

1. Accuracy assessment results (Complete A, B, or C below for each CEMS or for each pollutant and diluent analyzer, as applicable.) If the quarterly audit results show the CEMS to be out-of-control, report the results of both the quarterly audit and the audit following corrective action showing the CEMS to be operating properly.

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A. Relative accuracy test audit (RATA) for \_\_\_\_\_ (e.g., SO<sub>2</sub> in ng/l).

1. Date of audit \_\_\_\_\_.
2. Reference methods (RM's) used \_\_\_\_\_ (e.g., Methods 3 and 6).
3. Average RM value \_\_\_\_\_ (e.g., ng/l, mg/dsm<sup>3</sup>, or percent volume).
4. Average CEMS value \_\_\_\_\_.
5. Absolute value of mean difference [d] \_\_\_\_\_.
6. Confidence coefficient [CC] \_\_\_\_\_.
7. Percent relative accuracy (RA) \_\_\_\_\_ percent.
8. EPA performance audit results:
  - a. Audit lot number (1) \_\_\_\_\_ (2) \_\_\_\_\_
  - b. Audit sample number (1) \_\_\_\_\_ (2) \_\_\_\_\_
  - c. Results (mg/dsm<sup>3</sup>) (1) \_\_\_\_\_ (2) \_\_\_\_\_
  - d. Actual value (mg/dsm<sup>3</sup>) \* (1) \_\_\_\_\_ (2) \_\_\_\_\_
  - e. Relative error\* (1) \_\_\_\_\_ (2) \_\_\_\_\_

B. Cylinder gas audit (CGA) for \_\_\_\_\_ (e.g., SO<sub>2</sub> in ppm).

	Audit point 1	Audit point 2	
1. Date of audit .....	.....	.....	
2. Cylinder ID number .....	.....	.....	
3. Date of certification .....	.....	.....	
4. Type of certification .....	.....	.....	(e.g., EPA Protocol 1 or CRM).
5. Certified audit value .....	.....	.....	(e.g., ppm).
6. CEMS response value .....	.....	.....	(e.g., ppm).
7. Accuracy .....	.....	.....	percent.

C. Relative accuracy audit (RAA) for \_\_\_\_\_ (e.g., SO<sub>2</sub> in ng/l).

1. Date of audit \_\_\_\_\_.
2. Reference methods (RM's) used \_\_\_\_\_ (e.g., Methods 3 and 6).
3. Average RM value \_\_\_\_\_ (e.g., ng/l).
4. Average CEMS value \_\_\_\_\_.
5. Accuracy \_\_\_\_\_ percent.
6. EPA performance audit results:
  - a. Audit lot number (1) \_\_\_\_\_ (2) \_\_\_\_\_
  - b. Audit sample number (1) \_\_\_\_\_ (2) \_\_\_\_\_
  - c. Results (mg/dsm<sup>3</sup>) (1) \_\_\_\_\_ (2) \_\_\_\_\_
  - d. Actual value (mg/dsm<sup>3</sup>) \* (1) \_\_\_\_\_ (2) \_\_\_\_\_
  - e. Relative error\* (1) \_\_\_\_\_ (2) \_\_\_\_\_
- D. Corrective action for excessive inaccuracy.
  1. Out-of-control periods.
    - a. Date(s) \_\_\_\_\_.
    - b. Number of days \_\_\_\_\_.
  2. Corrective action taken \_\_\_\_\_.

3. Results of audit following corrective action. (Use format of A, B, or C above, as applicable.)

II. Calibration drift assessment.

A. Out-of-control periods.

1. Date(s) \_\_\_\_\_.
2. Number of days \_\_\_\_\_.

B. Corrective action taken \_\_\_\_\_.

[52 FR 21008, June 4, 1987; 52 FR 27612, July 22, 1987, as amended at 56 FR 5527, Feb. 11, 1991]

\* To be completed by the Agency.