



AIR HYGIENE

Testing Methods

The following is a list of the testing methods with which Air Hygiene is most familiar. Following each method is a brief description of the principles and applicability of each method. This list is by no means all inclusive, but serves as a summary of the more common testing procedures Air Hygiene performs.

APPENDIX A TO PART 60 – TEST METHODS

METHOD 1 – SAMPLE AND VELOCITY TRAVERSES FOR STATIONARY SOURCES

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

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^2 (113 in.^2) cross-sectional area
- (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.

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

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 m^2 (113 in.^2) in cross-sectional area, but equal to or greater than about 0.10 meter (4 in.) in diameter or 0.0081 m^2 (12.57 in.^2) in cross-sectional area.

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.

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 throughout the method.

METHOD 2 – DETERMINATION OF STACK GAS VELOCITY AND VOLUMETRIC FLOW RATE (TYPE S PITOT TUBE)

The average gas velocity in a stack is determined from the gas density and from measurement of the average velocity head with a Type S (Stausscheibe or reverse type) pitot tube.

This method is applicable for measurement of the average velocity of a gas stream and for quantifying gas flow. This procedure is not applicable at measurement sites that fail to meet the criteria of Method 1, Section 2.1. Also, the method cannot be used for direct measurement in cyclonic or swirling gas streams; Section 2.4 of Method 1 shows how to determine cyclonic or swirling flow conditions. When unacceptable conditions exist, alternative procedures, subject to the approval of the Administrator, U.S. Environmental Protection Agency, must be employed to make accurate flow rate determinations; examples of such alternative procedures are:

- (1) to install straightening vanes
- (2) to calculate the total volumetric flow rate stoichiometrically
- (3) to move to another measurement site at which the flow is acceptable

METHOD 2A – DIRECT MEASUREMENT OF GAS VOLUME THROUGH PIPES AND SMALL DUCTS

This method applies to the measurement of gas flow rates in pipes and small ducts, either inline or at exhaust positions, within the temperature range of 0 to 50°C.

A gas volume meter is used to measure gas volume directly. Temperature and pressure measurements are made to correct the volume to standard conditions.

METHOD 2B – DETERMINATION OF EXHAUST GAS VOLUME FLOW RATE FROM GASOLINE VAPOR INCINERATORS

This method applies to the measurement of exhaust volume flow rate from incinerators that process gasoline vapors consisting primarily of alkanes, alkenes, and/or arenes (aromatic hydrocarbons). It is assumed that the amount of auxiliary fuel is negligible.

The incinerator exhaust flow rate is determined by carbon balance. Organic carbon concentration and volume flow rate are measured at the incinerator inlet. Organic carbon, carbon dioxide (CO₂), and carbon monoxide (CO) concentrations are measured at the outlet. Then the ratio of total carbon at the incinerator inlet and outlet is multiplied by the inlet volume to determine the exhaust volume and volume flow rate.

METHOD 2C – DETERMINATION OF STACK GAS VELOCITY AND VOLUMETRIC FLOW RATE IN SMALL STACKS OR DUCTS (STANDARD PITOT TUBE)

The applicability of this method is identical to Method 2, except this method is limited to stationary source stacks or ducts less than about 0.30 meter (12 in.) in diameter or 0.071 m² (113 in.²) in cross-sectional area, but equal to or greater than about 0.10 meter (4 in.) in diameter or 0.0081 m² (12.57 in.²) in cross-sectional area.

The apparatus, procedure, calibration, calculations, and bibliography are the same as in Method 2, Sections 2, 3, 4, 5, and 6, except as noted in the method.

The average gas velocity in a stack or duct is determined from the gas density and from measurement of velocity heads with a standard pitot tube.

METHOD 2D – MEASUREMENT OF GAS VOLUMETRIC FLOW RATES IN SMALL PIPES AND DUCTS

This method applies to the measurement of gas flow rates in small pipes and ducts, either before or after emission control devices.

To measure flow rate or pressure drop, all the stack gas is directed through a rotameter, orifice plate or similar flow rate-measuring device. The measuring device has been previously calibrated in a manner that insures its proper calibration for the gas or gas mixture being measured. Absolute temperature and pressure measurements are also made to calculate volumetric flow rates at standard conditions.

METHOD 2E – DETERMINATION OF LANDFILL GAS; GAS PRODUCTION FLOW RATE

This method applies to the measurement of landfill gas (LFG) production flow rate from municipal solid waste (MSW) landfills and is used to calculate the flow rate of non-methane organic compounds (NMOC) from landfills. This method also applies to calculating a site-specific k value as provided in § 60.754(a)(4). It is unlikely that a site-specific k value obtained through Method 2E testing will lower the annual emission estimate below 50 Mg/yr NMOC unless the Tier 2 emission estimate is only slightly higher than 50 Mg/yr NMOC. Dry, arid regions may show a more significant difference between the default and calculated k values than wet regions.

Extraction wells are installed either in a cluster of three or at five locations dispersed throughout the landfill. A blower is used to extract LFG from the landfill. LFG composition, landfill pressures near the extraction well, and volumetric flow rate of LFG extracted from the wells are measured and the landfill gas production flow rate is calculated.

METHOD 2F – DETERMINATION OF STACK GAS VELOCITY AND VOLUMETRIC FLOW RATE WITH THREE DIMENSIONAL PROBES

This method does not include all of the specifications (e.g., equipment and supplies) and procedures (e.g., sampling) essential to its performance. Some material has been incorporated from other methods in this

part. Therefore, to obtain reliable results, those using this method should have a thorough knowledge of at least the following additional test methods: Methods 1, 2, 3 or 3A, and 4.

This method is applicable for the determination of yaw angle, pitch angle, axial velocity and the volumetric flow rate of a gas stream in a stack or duct using a three-dimensional (3-D) probe. This method may be used only when the average stack or duct gas velocity is greater than or equal to 20 ft/sec. When the above condition cannot be met, alternative procedures, approved by the Administrator, U.S. Environmental Protection Agency, shall be used to make accurate flow rate determinations.

A 3-D probe is used to determine the velocity pressure and the yaw and pitch angles of the flow velocity vector in a stack or duct. The method determines the yaw angle directly by rotating the probe to null the pressure across a pair of symmetrically placed ports on the probe head. The pitch angle is calculated using probe-specific calibration curves. From these values and a determination of the stack gas density, the average axial velocity of the stack gas is calculated. The average gas volumetric flow rate in the stack or duct is then determined from the average axial velocity.

METHOD 2G – DETERMINATION OF STACK GAS VELOCITY AND VOLUMETRIC FLOW RATE WITH TWO DIMENSIONAL PROBES

This method does not include all of the specifications (e.g., equipment and supplies) and procedures (e.g., sampling) essential to its performance. Some material has been incorporated from other methods in this part. Therefore, to obtain reliable results, those using this method should have a thorough knowledge of at least the following additional test methods: Methods 1, 2, 3 or 3A, and 4.

This method is applicable for the determination of yaw angle, near-axial velocity, and the volumetric flow rate of a gas stream in a stack or duct using a two-dimensional (2-D) probe.

A 2-D probe is used to measure the velocity pressure and the yaw angle of the flow velocity vector in a stack or duct. Alternatively, these measurements may be made by operating one of the three-dimensional (3-D) probes described in Method 2F, in yaw determination mode only. From these measurements and a determination of the stack gas density, the average near-axial velocity of the stack gas is calculated. The near-axial velocity accounts for the yaw, but not the pitch, component of flow. The average gas volumetric flow rate in the stack or duct is then determined from the average near-axial velocity.

METHOD 2H – DETERMINATION OF STACK GAS VELOCITY TAKING INTO ACCOUNT VELOCITY DECAY NEAR THE STACK WALL

This method is applicable in conjunction with Methods 2, 2F, and 2G (40 CFR Part 60, Appendix A) to account for velocity decay near the wall in circular stacks and ducts.

This method is not applicable for testing stacks and ducts less than 3.3 ft (1.0 m) in diameter. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

A wall effects adjustment factor is determined. It is used to adjust the average stack gas velocity obtained under Method 2, 2F, or 2G of this appendix to take into account velocity decay near the stack or duct wall. The method contains two possible procedures: a calculational approach which derives an

adjustment factor from velocity measurements and a default procedure which assigns a generic adjustment factor based on the construction of the stack or duct.

The calculational procedure derives a wall effects adjustment factor from velocity measurements taken using Method 2, 2F, or 2G at 16 (or more) traverse points specified under Method 1 of this appendix and a total of eight (or more) wall effects traverse points specified under this method. The calculational procedure based on velocity measurements is not applicable for horizontal circular ducts where buildup of particulate matter or other material in the bottom of the duct is present.

A default wall effects adjustment factor of 0.9900 for brick and mortar stacks and 0.9950 for all other types of stacks and ducts may be used without taking wall effects measurements in a stack or duct. When the calculational procedure is conducted as part of a relative accuracy test audit (RATA) or other multiple-run test procedure, the wall effects adjustment factor derived from a single traverse (i.e., single RATA run) may be applied to all runs of the same RATA without repeating the wall effects measurements. Alternatively, wall effects adjustment factors may be derived for several traverses and an average wall effects adjustment factor applied to all runs of the same RATA.

METHOD 3 – GAS ANALYSIS FOR THE DETERMINATION OF DRY MOLECULAR WEIGHT

This method is applicable for determining carbon dioxide (CO₂) and oxygen (O₂) 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 CO₂, O₂, carbon monoxide (CO), and nitrogen (N₂) are not present in concentrations sufficient to affect the results.

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 multipoint sampling method using an Orsat analyzer to analyze individual grab samples obtained at each point
- (2) a method using CO₂ or O₂ and stoichiometric calculations to determine dry molecular weight
- (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).

A gas sample is extracted from a stack by one of the following methods:

- (1) Single point, grab sampling
- (2) single point, integrated sampling
- (3) multipoint, integrated sampling

The gas sample is analyzed for percent CO₂, percent O₂, and if necessary, for percent CO. For dry molecular weight determination, either an Orsat or a Fyrite analyzer may be used for the analysis.

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

This method is applicable to the determination of oxygen (O₂) and carbon dioxide (CO₂) concentrations in emissions from stationary sources only when specified within the regulations.

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 O₂ and CO₂ concentration(s). Performance specifications and test procedures are provided to ensure reliable data.

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

This method is applicable for determining carbon dioxide (CO₂), oxygen (O₂), 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.

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 multipoint sampling method using an Orsat analyzer to analyze individual grab samples obtained at each point
- (2) a method using CO₂ or O₂ 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).

A gas sample is extracted from a stack by one of the following methods:

- (1) Singlepoint, grab sampling
- (2) single point, integrated sampling
- (3) multipoint, integrated sampling

The gas sample is analyzed for percent CO₂ percent O₂, and, if necessary, percent CO. An Orsat analyzer must be used for excess air or emission rate correction factor determinations.

METHOD 3C – DETERMINATION OF CARBON DIOXIDE, METHANE, NITROGEN, AND OXYGEN FROM STATIONARY SOURCES

This method applies to the analysis of carbon dioxide (CO₂), methane (CH₄), nitrogen (N₂), and oxygen (O₂) in samples from municipal solid waste landfills and other sources when specified in an applicable subpart.

A portion of the sample is injected into a gas chromatograph (GC) and the CO₂, CH₄, N₂, and O₂ concentrations are determined by using a thermal conductivity detector (TCD) and integrator.

METHOD 4 – DETERMINATION OF MOISTURE CONTENT IN STACK GASES

A gas sample is extracted at a constant rate from the source; moisture is removed from the sample stream and determined either volumetrically or gravimetrically.

This method is applicable for determining the moisture content of stack gas. Two procedures are given. The first is a reference method, for accurate determinations of moisture content (such as are needed to calculate emission data). The second is an approximation method, which provides estimates of percent moisture to aid in setting isokinetic sampling rates prior to a pollutant emission measurement run. The approximation method described herein is only a suggested approach; alternative means for approximating the moisture content, e.g., drying tubes, wet bulb-dry bulb techniques, condensation techniques, stoichiometric calculations, previous experience, etc., are also acceptable. The reference method is often conducted simultaneously with a pollutant emission measurement run; when it is, calculation of percent isokinetic, pollutant emission rate, etc., for the run shall be based upon the results of the reference method or its equivalent; these calculations shall not be based upon the results of the approximation method, unless the approximation method is shown, to the satisfaction of the Administrator, U.S. Environmental Protection Agency, to be capable of yielding results within one percent H₂O of the reference method.

The reference method may yield questionable results when applied to saturated gas streams or to streams that contain water droplets. Therefore, when these conditions exist or are suspected, a second determination of the moisture content shall be made simultaneously with the reference method, as follows: Assume that the gas stream is saturated. Attach a temperature sensor [capable of measuring to $\pm 1^\circ\text{C}$ (2°F)] to the reference method probe. Measure the stack gas temperature at each traverse point (see Section 2.2.1) during the reference method traverse; calculate the average stack gas temperature. Next, determine the moisture percentage, either by:

- (1) using a psychrometric chart and making appropriate corrections if stack pressure is different from that of the chart
- (2) using saturation vapor pressure tables

In cases where the psychrometric chart or the saturation vapor pressure tables are not applicable (based on evaluation of the process), alternative methods, subject to the approval of the Administrator, shall be used.

METHOD 5 – DETERMINATION OF PARTICULATE EMISSIONS FROM STATIONARY SOURCES

Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter, maintained at a temperature in the range of $120\pm 14^\circ\text{C}$ ($248\pm 25^\circ\text{F}$) 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 determined gravimetrically after removal of uncombined water.

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

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

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.

Particulate matter is withdrawn isokinetically from the source and collected on a glass filter fiber maintained at a temperature of $42^{\circ}\pm 10^{\circ}\text{C}$ ($108^{\circ}\pm 18^{\circ}\text{F}$). The particulate mass, which includes any material that condenses at or above the filtration temperature, is determined gravimetrically after removal of uncombined water.

METHOD 5B – DETERMINATION OF NON-SULFURIC ACID PARTICULATE MATTER FROM STATIONARY SOURCES

This method is to be used for determining non-sulfuric acid particulate matter from stationary sources. Use of this method must be specified by an applicable subpart, or approved by the Administrator, U.S. Environmental Protection Agency, for a particular application.

Particulate matter is withdrawn isokinetically from the source using the Method 5 train at 160°C (320°F). The collected sample is then heated in the oven at 160°C (320°F) for 6 hours to volatilize any condensed sulfuric acid that may have been collected, and the non-sulfuric acid particulate mass is determined gravimetrically.

METHOD 5D – DETERMINATION OF PARTICULATE MATTER EMISSIONS FROM POSITIVE PRESSURE FABRIC FILTERS

This method applies to the determination of particulate matter emissions from positive pressure fabric filters. Emissions are determined in terms of concentration (mg/m^3) and emission rate (kg/h). The General Provisions of 40 CFR Part 60, § 60.8(e), require that the owner or operator of an affected facility shall provide performance testing facilities. Such performance testing facilities include sampling ports, safe sampling platforms, safe access to sampling sites, and utilities for testing. It is intended that affected facilities also provide sampling locations that meet the specification for adequate stack length and minimal flow disturbances as described in Method 1. Provisions for testing are often overlooked factors in designing fabric filters or are extremely costly. The purpose of this procedure is to identify appropriate alternative locations and procedures for sampling the emissions from positive pressure fabric filters. The requirements that the affected facility owner or operator provide adequate access to performance testing facilities remain in effect.

Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at a temperature at or above the exhaust gas temperature up to a nominal 120°C ($120\pm 14^{\circ}\text{C}$ or $248\pm 25^{\circ}\text{F}$). The particulate mass, which includes any material that condenses at or above the filtration temperature, is determined gravimetrically after removal of uncombined water.

METHOD 5E – DETERMINATION OF PARTICULATE EMISSIONS FROM THE WOOL FIBERGLASS INSULATION MANUFACTURING INDUSTRY

This method is applicable for the determination of particulate emissions from wool fiberglass insulation manufacturing sources.

Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at a temperature in the range of $120^{\circ}\pm 14^{\circ}\text{C}$ ($248^{\circ}\pm 25^{\circ}\text{F}$) and in solutions of 0.1 N NaOH. The filtered particulate mass, which includes any material that condenses at or above the filtration

temperature, is determined gravimetrically after removal of uncombined water. The condensed particulate material collected in the impinger solutions is determined as total organic carbon (TOC) using a nondispersive infrared type of analyzer. The sum of the filtered particulate mass and the condensed particulate matter is reported as the total particulate mass.

METHOD 5F – DETERMINATION OF NON-SULFATE PARTICULATE MATTER FROM STATIONARY SOURCES

This method is to be used for determining nonsulfate particulate matter from stationary sources. Use of this method must be specified by an applicable subpart of the standards, or approved by the Administrator, U.S. Environmental Protection Agency, for a particular application.

Particulate matter is withdrawn isokinetically from the source using the Method 5 train at 160°C (320°F). The collected sample is then extracted with water. A portion of the extract is analyzed for sulfate content. The remainder is neutralized with ammonium hydroxide before it is dried and weighed.

METHOD 5G – DETERMINATION OF PARTICULATE EMISSIONS FROM WOOD HEATERS FROM A DILUTION TUNNEL SAMPLING LOCATION

This method is applicable for the determination of particulate matter emissions from wood heaters.

Particulate matter is withdrawn proportionally at a single point from a total collection hood and sampling tunnel that combines the wood heater exhaust with ambient dilution air. The particulate matter is collected on two glass fiber filters in series. The filters are maintained at a temperature of no greater than 32°C (90°F). The particulate mass is determined gravimetrically after removal of uncombined water. There are three sampling train approaches described in this method:

- (1) one dual-filter dry sampling train operated at about 0.015 m³/min
- (2) one dual-filter plus impingers sampling train operated at about 0.015 m³/min
- (3) two dual-filter dry sampling trains operated simultaneously at any flow rate.

Options (2) and (3) are referenced in Section 7 of this method. The dual-filter sampling train equipment and operation, option (1), are described in detail in this method.

METHOD 5H – DETERMINATION OF PARTICULATE EMISSIONS FROM WOOD HEATERS FROM A STACK LOCATION

This method is applicable for the determination of particulate matter and condensible emissions from wood heaters.

Particulate matter is withdrawn proportionally from the wood heater exhaust and is collected on two glass fiber filters separated by impingers immersed in an ice bath. The first filter is maintained at a temperature of no greater than 120°C (248°F). The second filter and the impinger system are cooled such that the exiting temperature of the gas is no greater than 20°C (68°F). The particulate mass collected in the probe, on the filters, and in the impingers is determined gravimetrically after removal of uncombined water.

METHOD 6 – DETERMINATION OF SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES

A gas sample is extracted from the sampling point in the stack. The sulfuric acid mist (including sulfur trioxide) and the sulfur dioxide are separated. The sulfur dioxide fraction is measured by the barium thiorin titration method.

This method is applicable for the determination of sulfur dioxide emissions from stationary sources. The minimum detectable limit of the method has been determined to be 3.4 milligrams (mg) of SO_2/m^3 (2.12×10^{-7} lb/ft³). Although no upper limit has been established, tests have shown that concentrations as high as 80,000 mg/m³ of SO_2 can be collected efficiently in two midjet impingers, each containing 15 milliliters of 3 percent hydrogen peroxide, at a rate of 1.0 lpm for 20 minutes. Based on theoretical calculations, the upper concentration limit in a 20-liter sample is about 93,300 mg/m³.

Possible interferents are free ammonia, water-soluble cations, and fluorides. The cations and fluorides are removed by glass wool filters and an isopropanol bubbler, and hence do not affect the SO_2 analysis. When samples are being taken from a gas stream with high concentrations of very fine metallic fumes (such as in inlets to control devices), a high-efficiency glass fiber filter must be used in place of the glass wool plug (i.e., the one in the probe) to remove the cation interferents. Free ammonia interferes by reacting with SO_2 to form particulate sulfite and by reacting with the indicator. If free ammonia is present (this can be determined by knowledge of the process and the presence of white particulate matter in the probe and isopropanol bubbler), the alternative procedures in Section 7.2 shall be used.

METHOD 6A – DETERMINATION OF SULFUR DIOXIDE, MOISTURE, AND CARBON DIOXIDE EMISSIONS FROM FOSSIL FUEL COMBUSTION SOURCES

This method applies to the determination of sulfur dioxide (SO_2) emissions from fossil fuel combustion sources in terms of concentration (mg/m³) and in terms of emission rate (ng/J) and to the determination of carbon dioxide (CO_2) concentration (percent). Moisture, if desired, may also be determined by this method. The minimum detectable limit, the upper limit, and the interferences of the method for the measurement of SO_2 are the same as for Method 6. For a 20-liter sample, the method has a precision of 0.5 percent CO_2 for concentrations between 2.5 and 25 percent CO_2 and 1.0 percent moisture for moisture concentrations greater than 5 percent.

The principle of sample collection is the same as for Method 6 except that moisture and CO_2 are collected in addition to SO_2 in the same sampling train. Moisture and CO_2 fractions are determined gravimetrically.

METHOD 6B – DETERMINATION OF SULFUR DIOXIDE AND CARBON DIOXIDE DAILY AVERAGE EMISSIONS FROM FOSSIL FUEL COMBUSTION SOURCES

This method applies to the determination of sulfur dioxide (SO_2) emissions from combustion sources in terms of concentration (ng/m³) and emission rate (ng/J), and for the determination of carbon dioxide (CO_2) concentration (percent) on a daily (24 hours) basis. The minimum detectable limits, upper limit, and the interferences for SO_2 measurements are the same as for Method 6. EPA-sponsored collaborative studies were undertaken to determine the magnitude of repeatability and reproducibility achievable by qualified testers following the procedures in this method. The results of the studies evolve from 145 field tests including comparisons with Methods 3 and 6. For measurements of emission rates from wet, flue

gas desulfurization units in (ng/J), the repeatability (within laboratory precision) is 8.0 percent and the reproducibility (between laboratory precision) is 11.1 percent.

A gas sample is extracted from the sampling point in the stack intermittently over a 24-hour or other specified time period. Sampling may also be conducted continuously if the apparatus and procedures are appropriately modified (see Note in Section 4.1.1). The SO₂ and CO₂ are separated and collected in the sampling train. The SO₂ fraction is measured by the bariumthorin titration method, and CO₂ is determined gravimetrically.

METHOD 6C – DETERMINATION OF SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES (INSTRUMENTAL ANALYZER PROCEDURE)

This method is applicable to the determination of sulfur dioxide (SO₂) concentrations in controlled and uncontrolled emissions from stationary sources only when specified within the regulations.

A gas sample is continuously extracted from a stack, and a portion of the sample is conveyed to an instrumental analyzer for determination of SO₂ gas concentration using an ultraviolet (UV), nondispersive infrared (NDIR), or fluorescence analyzer. Performance specifications and test procedures are provided to ensure reliable data.

METHOD 7E – DETERMINATION OF NITROGEN OXIDES EMISSIONS FROM STATIONARY SOURCES (INSTRUMENTAL ANALYZER PROCEDURE)

This method is applicable to the determination of nitrogen oxides (NO_x) concentrations in emissions from stationary sources only when specified within the regulations.

A gas sample is continuously extracted from a stack, and a portion of the sample is conveyed to an instrumental chemiluminescent analyzer for determination of NO_x concentration. Performance specifications and test procedures are provided to ensure reliable data.

METHOD 8 – DETERMINATION OF SULFURIC ACID MIST AND SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES

A gas sample is extracted isokinetically from the stack. The sulfuric acid mist (including sulfur trioxide) and the sulfur dioxide are separated, and both fractions are measured separately by the barium thorin titration method.

This method is applicable for the determination of sulfuric acid mist (including sulfur trioxide, and in the absence of other particulate matter) and sulfur dioxide emissions from stationary sources. Collaborative tests have shown that the minimum detectable limits of the method are 0.05 milligrams/cubic meter (0.03x10⁻⁷ pounds/cubic foot) for sulfur trioxide and 1.2 mg/m³ (0.74x10⁻⁷ lb/ft³) for sulfur dioxide. No upper limits have been established. Based on theoretical calculations for 200 milliliters of 3 percent hydrogen peroxide solution, the upper concentration limit for sulfur dioxide in a 1.0 m³ (35.3 ft³) gas sample is about 12,500 mg/m³ (7.7x10⁻⁴ lb/ft³). The upper limit can be extended by increasing the quantity of peroxide solution in the impingers. Possible interfering agents of this method are fluorides, free ammonia, and dimethyl aniline. If any of these interfering agents are present (this can be determined by knowledge of the process), alternative methods, subject to the approval of the Administrator, U. S. E. P.A., are required. Filterable particulate matter may be determined along with SO₃ and SO₂ (subject to

the approval of the Administrator) by inserting a heated glass fiber filter between the probe and isopropanol impinger (see Section 2.1 of Method 6). If this option is chosen, particulate analysis is gravimetric only; H₂SO₄ acid mist is not determined separately.

METHOD 9 – VISUAL DETERMINATION OF THE OPACITY OF EMISSIONS FROM STATIONARY SOURCES (ALTERNATE METHOD 1 – DETERMINATION OF THE OPACITY OF EMISSIONS FROM STATIONARY SOURCES REMOTELY BY LIDAR)

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:

- (1) angle of the observer with respect to the plume
- (2) angle of the observer with respect to the sun
- (3) point of observation of attached and detached steam plume
- (4) 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 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, 298 sets at a sulfuric acid plant), 99 percent of the sets were read with a positive error of less than 7.5 percent opacity; 95 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.

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.

METHOD 10 – DETERMINATION OF CARBON MONOXIDE EMISSIONS FROM STATIONARY SOURCES

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

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.

METHOD 10A – DETERMINATION OF CARBON MONOXIDE EMISSIONS IN CERTIFYING CONTINUOUS EMISSION MONITORING SYSTEMS AT PETROLEUM REFINERIES

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)].

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 psulfaminobenzoic acid.

METHOD 10B – DETERMINATION OF CARBON MONOXIDE EMISSIONS FROM STATIONARY SOURCES

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.

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 (CH₄) 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.

Carbon dioxide (CO₂) and organics potentially can interfere with the analysis. Carbon dioxide is primarily removed from the sample by the alkaline permanganate conditioning system; any residual CO₂ and organics are separated from the CO by GC.

METHOD 11 – DETERMINATION OF HYDROGEN SULFIDE CONTENT OF FUEL GAS STREAMS IN PETROLEUM REFINERIES

Hydrogen sulfide (H_2S) is collected from a source in a series of midjet impingers and absorbed in pH 3.0 cadmium sulfate ($CdSO_4$) solution to form cadmium sulfide (CdS). The latter compound is then measured iodometrically. An impinger containing hydrogen peroxide is included to remove SO_2 as an interfering species. This method is a revision of the H_2S method originally published in the FEDERAL REGISTER, Volume 39, No. 47, dated Friday, March 8, 1974.

This method is applicable for the determination of the hydrogen sulfide content of fuel gas streams at petroleum refineries.

METHOD 12 – DETERMINATION OF INORGANIC LEAD EMISSIONS FROM STATIONARY SOURCES

This method applies to the determination of inorganic lead (Pb) emissions from specified stationary sources only.

Particulate and gaseous Pb emissions are withdrawn isokinetically from the source and collected on a filter and in dilute nitric acid. The collected samples are digested in acid solution and analyzed by atomic absorption spectrometry using an air acetylene flame.

METHOD 13A – DETERMINATION OF TOTAL FLUORIDE EMISSIONS FROM STATIONARY SOURCES (SPADNS ZIRCONIUM LAKE METHOD)

This method applies to the determination of fluoride (F) emissions from sources as specified in the regulations. It does not measure fluorocarbons, such as freons.

Gaseous and particulate F are withdrawn isokinetically from the source and collected in water and on a filter. The total F is then determined by the SPADNS Zirconium Lake Colorimetric Method.

METHOD 13B – DETERMINATION OF TOTAL FLUORIDE EMISSIONS FROM STATIONARY SOURCES (SPECIFIC ION ELECTRODE METHOD)

This method applies to the determination of fluoride (F) emissions from stationary sources as specified in the regulations. It does not measure fluorocarbons, such as freons.

Gaseous and particulate F are withdrawn isokinetically from the source and collected in water and on a filter. The total F is then determined by the specific ion electrode method.

METHOD 14 – DETERMINATION OF FLUORIDE EMISSIONS FROM POTROOM ROOF MONITORS FOR PRIMARY ALUMINUM PLANTS

This method is applicable for the determination of fluoride emissions from stationary sources only when specified by the test procedures for determining compliance with new source performance standards.

Gaseous and particulate fluoride roof monitor emissions are drawn into a permanent sampling manifold through several large nozzles. The sample is transported from the sampling manifold to ground level through a duct. The gas in the duct is sampled using Method 13A or 13B (Determination of Total Fluoride Emissions from Stationary Sources). Effluent velocity and volumetric flow rate are determined with anemometers located in the roof monitor.

METHOD 14A – DETERMINATION OF TOTAL FLUORIDE EMISSIONS FROM SELECTED SOURCES AT PRIMARY ALUMINUM PRODUCTION FACILITIES

This method does not include all the specifications (e.g., equipment and supplies) and procedures (e.g., sampling) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods:

- (1) Method 5
- (2) Method 13A
- (3) Method 13B
- (4) Method 14

This method is applicable for the determination of total fluorides (TF) emissions from sources specified in the applicable regulation. This method was developed by consensus with the Aluminum Association and the U.S. Environmental Protection Agency (EPA).

Total fluorides, in the form of solid and gaseous fluorides, are withdrawn from the ascending air stream inside of an aluminum reduction potroom and, prior to exiting the potroom roof monitor, into a specific cassette arrangement. The cassettes are connected by tubing to flowmeters and a manifold system that allows for the equal distribution of volume pulled through each cassette, and finally to a dry gas meter. The cassettes have a specific internal arrangement of one unaltered cellulose filter and support pad in the first section of the cassette for solid fluoride retention and two cellulose filters with support pads that are impregnated with sodium formate for the chemical absorption of gaseous fluorides in the following two sections of the cassette. A minimum of eight cassettes shall be used for a potline and shall be strategically located at equal intervals across the potroom roof so as to encompass a minimum of 8 percent of the total length of the potroom. A greater number of cassettes may be used should the regulated facility choose to do so. The mass flow rate of pollutants is determined with anemometers and temperature-sensing devices located immediately below the opening of the roof monitor and spaced evenly within the cassette group.

METHOD 17 – DETERMINATION OF PARTICULATE EMISSIONS FROM STATIONARY SOURCES (INSTACK FILTRATION METHOD)

Particulate matter is not an absolute quantity; rather, it is a function of temperature and pressure. Therefore, to prevent variability in particulate matter emission regulations and/or associated test methods, the temperature and pressure at which particulate matter is to be measured must be carefully defined. Of the two variables (i.e., temperature and pressure), temperature has the greater effect upon the amount of particulate matter in an effluent gas stream; in most stationary source categories, the effect of pressure appears to be negligible. In Method 5, 250°F is established as a nominal reference temperature. Thus, where Method 5 is specified in an applicable subpart of the standards, particulate matter is defined with respect to temperature. In order to maintain a collection temperature of 250°F, Method 5 employs a heated glass sample probe and a heated filter holder. This equipment is somewhat cumbersome and

requires care in its operation. Therefore, where particulate matter concentrations (over the normal range of temperature associated with a specified source category) are known to be independent of temperature, it is desirable to eliminate the glass probe and heating systems, and sample at stack temperature.

This method describes an instack sampling system and sampling procedures for use in such cases. It is intended to be used only when specified by an applicable subpart of the standards, and only within the applicable temperature limits (if specified), or when otherwise approved by the Administrator.

Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at stack temperature. The particulate mass is determined gravimetrically after removal of uncombined water.

This method applies to the determination of particulate emissions from stationary sources for determining compliance with new source performance standards, only when specifically provided for in an applicable subpart of the standards. This method is not applicable to stacks that contain liquid droplets or are saturated with water vapor. In addition, this method shall not be used as written if the projected cross-sectional area of the probe extension-filter holder assembly covers more than 5 percent of the stack cross-sectional area (see Section 4.1.2).

METHOD 19 – DETERMINATION OF SULFUR DIOXIDE REMOVAL EFFICIENCY AND PARTICULATE, SULFUR DIOXIDE, AND NITROGEN OXIDES EMISSION RATES

This method is applicable for:

- (a) determining particulate matter (PM), sulfur dioxide (SO₂), and nitrogen oxides (NO_x) emission rates
- (b) determining sulfur removal efficiencies of fuel pretreatment and SO₂ control devices
- (c) determining overall reduction of potential SO₂ emissions from steam generating units or other sources as specified in applicable regulations
- (d) determining SO₂ rates based on fuel sampling and analysis procedures.

Pollutant emission rates are determined from concentrations of PM, SO₂, or NO_x, and oxygen (O₂) or carbon dioxide (CO₂) along with F factors (ratios of combustion gas volumes to heat inputs). An overall SO₂ emission reduction efficiency is computed from the efficiency of fuel pretreatment systems (optional) and the efficiency of SO₂ control devices. Fuel sampling and analysis of the sulfur and heat contents of the fuel before and after the pretreatment system determine the sulfur removal efficiency of a fuel pretreatment system. The SO₂ removal efficiency of a control device is determined by measuring the SO₂ rates before and after the control device. The inlet rates to SO₂ control systems and when SO₂ control systems are not used, SO₂ emission rates to the atmosphere may be determined by fuel sampling and analysis (optional).

METHOD 20 – DETERMINATION OF NITROGEN OXIDES, SULFUR DIOXIDE, AND DILUENT EMISSIONS FROM STATIONARY GAS TURBINES

This method is applicable for the determination of nitrogen oxides (NO_x), sulfur dioxide (SO₂), and a diluent gas, either oxygen (O₂) or carbon dioxide (CO₂), emissions from stationary gas turbines. For the NO_x and diluent concentration determinations, this method includes:

- (1) measurement system design criteria
- (2) analyzer performance specifications and performance test procedures
- (3) procedures for emission testing

A gas sample is continuously extracted from the exhaust stream of a stationary gas turbine; a portion of the sample stream is conveyed to instrumental analyzers for determination of NO_x and diluent content. During each NO_x and diluent determination, a separate measurement of SO₂ emissions is made, using Method 6, or its equivalent. The diluent determination is used to adjust the NO_x and SO₂ concentrations to a reference condition.

METHOD 22 – VISUAL DETERMINATION OF FUGITIVE EMISSIONS FROM MATERIAL SOURCES AND SMOKE EMISSIONS FROM FLARES

This method involves the visual determination of fugitive emissions, i.e., emissions not emitted directly from a process stack or duct. Fugitive emissions include emissions that:

- (1) escape capture by process equipment exhaust hoods
- (2) are emitted during material transfer
- (3) are emitted from buildings housing material processing or handling equipment
- (4) are emitted directly from process equipment.

This method is used also to determine visible smoke emissions from flares used for combustion of waste process materials. This method determines the amount of time that any visible emissions occur during the observation period, i.e., the accumulated emission time. This method does not require that the opacity of emissions be determined. Since this procedure requires only the determination of whether a visible emission occurs and does not require the determination of opacity levels, observer certification according to the procedures of Method 9 are not required. However, it is necessary that the observer is educated on the general procedures for determining the presence of visible emissions. As a minimum, the observer must be trained and knowledgeable regarding the effects on the visibility of emissions caused by background contrast, ambient lighting, observer position relative to lighting, wind, and the presence of uncombined water (condensing water vapor). This training is to be obtained from written materials found in Citations 1 and 2 of the bibliography section of this method or from the lecture portion of the Method 9 certification course.

This method applies to the determination of the frequency of fugitive emissions from stationary sources (located indoors or outdoors) when specified as the test method for determining compliance with new source performance standards. This method also is applicable for the determination of the frequency of visible smoke emissions from flares.

Fugitive emissions produced during material processing, handling, and transfer operations or smoke emissions from flares are visually determined by an observer without the aid of instruments.

METHOD 23 – DETERMINATION OF POLYCHLORINATED DIBENZOPDIOXINS AND POLYCHLORINATED DIBENZOFURANS FROM STATIONARY SOURCES

This method is applicable to the determination of polychlorinated dibenzopdioxins (PCDD's) and polychlorinated dibenzofurans (PCDF's) from stationary sources.

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 material. The sample cannot be separated into a particle vapor fraction. The PCDD's and PCDF's are extracted from the sample, separated by high resolution gas chromatography, and measured by high resolution mass spectrometry.

METHOD 24 – DETERMINATION OF VOLATILE MATTER CONTENT, WATER CONTENT, DENSITY, VOLUME SOLIDS, AND WEIGHT SOLIDS OF SURFACE COATINGS

This method applies to the determination of volatile matter content, water content, density, volume solids, and weight solids of paint, varnish, lacquer, or related surface coatings.

Standard methods are used to determine the volatile matter content, water content, density, volume solids, and weight solids of the paint, varnish, lacquer, or related surface coatings.

Use the apparatus, reagents, and procedures specified in the standard methods below:

- (1) ASTM D1475 – 60 (Reapproved 1980), Standard Test Method for Density of Paint, Varnish, Lacquer, and Related Products (incorporated by reference, see § 60.17)
- (2) ASTM D2369 – 81, Standard Test Method for Volatile Content of Coatings (incorporated by reference, see § 60.17)
- (3) ASTM D3792 – 79, Standard Test Method for Water Content of Water-Reducible Paints by Direct Injection into a Gas Chromatograph (incorporated by reference, see § 60.17)
- (4) ASTM D4017 – 81, Standard Test Method for Water in Paints and Paint Materials by the Karl Fischer Titration Method (incorporated by reference, see § 60.17)
- (5) ASTM D4457 – 85 Standard Test Method for Determination of Dichloromethane and 1,1,1-Trichloroethane in Paints and Coatings by Direct Injection into a Gas Chromatograph (incorporated by reference, see § 60.17)
- (6) ASTM D 5403 – 93 Standard Test Methods for Volatile Content of Radiation Curable Materials (incorporated by reference, see § 60.17)

METHOD 24A – DETERMINATION OF VOLATILE MATTER CONTENT AND DENSITY OF PRINTING INKS AND RELATED COATINGS

This method applies to the determination of the volatile organic compound (VOC) content and density of solvent borne (solvent reducible) printing inks or related coatings.

Separate procedures are used to determine the VOC weight fraction and density of the coating and the density of the solvent in the coating. The VOC weight fraction is determined by measuring the weight loss of a known sample quantity, which has been heated for a specified length of time at a specified temperature. The density of both the coating and solvent are measured by a standard procedure. From this information, the VOC volume fraction is calculated.

METHOD 25 – DETERMINATION OF TOTAL GASEOUS NON-METHANE ORGANIC EMISSIONS AS CARBON

This method applies to the measurement of volatile organic compounds (VOC) as total gaseous nonmethane organics (TGNMO) as carbon in source emissions. Organic particulate matter will interfere with the analysis and, therefore, a particulate filter is required. The minimum detectable for the method is 50 ppm as carbon. When carbon dioxide (CO₂) and water vapor are present together in the stack, they can produce a positive bias in the sample. The magnitude of the bias depends on the concentrations of CO₂ and water vapor. As a guideline, multiply the CO₂ concentration, expressed as volume percent, times the water vapor concentration. If this product does not exceed 100, the bias can be considered insignificant.

For example, the bias is not significant for a source having 10 percent CO₂ and 10 percent water vapor, but it would be significant for a source near the detection limit having 10 percent CO₂ and 20 percent water vapor. This method is not the only method that applies to the measurement of TGNMO. Costs, logistics, and other practicalities of source testing may make other test methods more desirable for measuring VOC contents of certain effluent streams. Proper judgment is required in determining the most applicable VOC test method. For example, depending upon the molecular weight of the organics in the effluent stream, a totally automated semicontinuous nonmethane organics (NMO) analyzer interfaced directly to the source may yield accurate results. This approach has the advantage of providing emission data semicontinuously over an extended time period. Direct measurement of an effluent with a flame ionization detector (FID) analyzer may be appropriate with prior characterization of the gas stream and knowledge that the detector responds predictably to the organic compounds in the stream.

If present, methane (CH₄) will, of course, also be measured. The FID can be applied to the determination of the mass concentration of the total molecular structure of the organic emissions under any of the following limited conditions:

- (1) Where only one compound is known to exist
- (2) when the organic compounds consist of only hydrogen and carbon
- (3) where the relative percentages of the compounds are known or can be determined, and the FID responses to the compounds are known
- (4) where a consistent mixture of the compounds exists before and after emission control and only the relative concentrations are to be assessed
- (5) where the FID can be calibrated against mass standards of the compounds emitted (solvent emissions, for example).

Another example of the use of a direct FID is as a screening method. If there is enough information available to provide a rough estimate of the analyzer accuracy, the FID analyzer can be used to determine the VOC content of an uncharacterized gas stream. With a sufficient buffer to account for possible inaccuracies, the direct FID can be a useful tool to obtain the desired results without costly exact determination. In situations where a qualitative/quantitative analysis of an effluent stream is desired or required, a gas chromatographic FID system may apply. However, for sources emitting numerous organics, the time and expense of this approach will be formidable.

An emission sample is withdrawn from the stack at a constant rate through a heated filter and a chilled condensate trap by means of an evacuated sample tank. After sampling is completed, the TGNMO are determined by independently analyzing the condensate trap and sample tank fractions and combining the analytical results. The organic content of the condensate trap fraction is determined by oxidizing the NMO to CO₂ and quantitatively collecting the effluent in an evacuated vessel; then a portion of the CO₂ is reduced to CH₄ and measured by an FID. The organic content of the sample tank fraction is measured by injecting a portion of the sample into a gas chromatographic column to separate the NMO from carbon

monoxide (CO), CO₂, and CH₄; the NMO are oxidized to CO₂, reduced to CH₄, and measured by an FID. In this manner, the variable response of the FID associated with different types of organics is eliminated.

METHOD 25A – DETERMINATION OF TOTAL GASEOUS ORGANIC CONCENTRATION USING A FLAME IONIZATION ANALYZER

This method applies to the measurement of total gaseous organic concentration of vapors consisting primarily of alkanes, alkenes, and/or arenes (aromatic hydrocarbons). The concentration is expressed in terms of propane (or other appropriate organic calibration gas) or in terms of carbon.

A gas sample is extracted from the source through a heated sample line, if necessary, and glass fiber filter to a flame ionization analyzer (FIA). Results are reported as volume concentration equivalents of the calibration gas or as carbon equivalents.

METHOD 25B – DETERMINATION OF TOTAL GASEOUS ORGANIC CONCENTRATION USING A NONDISPERSIVE INFRARED ANALYZER

This method applies to the measurement of total gaseous organic concentration of vapors consisting primarily of alkanes (Other organic materials may be measured using the general procedure in this method, the appropriate calibration gas, and an analyzer set to the appropriate absorption band). The concentration is expressed in terms of propane (or other appropriate organic calibration gas) or in terms of carbon.

A gas sample is extracted from the source through a heated sample line, if necessary, and glass fiber filter to a nondispersive infrared analyzer (NDIR). Results are reported as volume concentration equivalents of the calibration gas or as carbon equivalents.

METHOD 25C – DETERMINATION OF NON-METHANE ORGANIC COMPOUNDS (NMOC) IN MSW LANDFILL GASES

This method is applicable to the sampling and measurement of nonmethane organic compounds (NMOC) as carbon in MSW landfill gases.

A sample probe that has been perforated at one end is driven or augered to a depth of 1.0 meter below the bottom of the landfill cover. A sample of the landfill gas is extracted with an evacuated cylinder. The NMOC content of the gas is determined by injecting a portion of the gas into a gas chromatographic column to separate the NMOC from carbon monoxide (CO), carbon dioxide (CO₂), and methane (CH₄); the NMOC are oxidized to CO₂, reduced to CH₄, and measured by a flame ionization detector (FID). In this manner, the variable response of the FID associated with different types of organics is eliminated.

METHOD 25D – DETERMINATION OF THE VOLATILE ORGANIC CONCENTRATION OF WASTE SAMPLES

Performance of this method should not be attempted by persons unfamiliar with the operation of a flame ionization detector (FID) or an electrolytic conductivity detector (ELCD) because knowledge beyond the scope of this presentation is required.

This method is applicable for determining the volatile organic (VO) concentration of a waste sample.

A sample of waste is obtained at a point that is most representative of the unexposed waste (where the waste has had minimum opportunity to volatilize to the atmosphere). The sample is suspended in an organic/aqueous matrix, then heated and purged with nitrogen for 30 min in order to separate certain organic compounds. Part of the sample is analyzed for carbon concentration, as methane, with an FID, and part of the sample is analyzed for chlorine concentration, as chloride, with an ELCD. The VO concentration is the sum of the carbon and chlorine content of the sample.

METHOD 25E – DETERMINATION OF VAPOR PHASE ORGANIC CONCENTRATION IN WASTE SAMPLES

Performance of this method should not be attempted by persons unfamiliar with the operation of a flame ionization detector (FID) nor by those who are unfamiliar with source sampling because knowledge beyond the scope of this presentation is required.

This method is applicable for determining the vapor pressure of waste samples, which represent waste, which is or will be managed in tanks.

The headspace vapor of the sample is analyzed for carbon content by a headspace analyzer, which uses an FID. The analyst shall select the operating parameters best suited to the requirements for a particular analysis. The analyst shall produce confirming data through an adequate supplemental analytical technique and have the data available for review by the Administrator.

METHOD 26 – DETERMINATION OF HYDROGEN CHLORIDE EMISSIONS FROM STATIONARY SOURCES

This method is applicable for determining emissions of hydrogen halides (HX) [hydrogen chloride (HCl), hydrogen bromide (HBr), and hydrogen fluoride (HF)] and halogens (X₂) [chlorine (Cl₂) and bromine (Br₂)] from stationary sources. Sources, such as those controlled by wet scrubbers, that emit acid particulate matter must be sampled using Method 26A.

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 (Cl⁻), bromide (Br⁻), and fluoride (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 (H⁺), the halide ion, and the hypohalous acid (HClO or 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).

METHOD 26A – DETERMINATION OF HYDROGEN HALIDE AND HALOGEN EMISSIONS FROM STATIONARY SOURCES (ISOKINETIC METHOD)

This method is applicable for determining emissions of hydrogen halides (HX) [hydrogen chloride (HCl), hydrogen bromide (HBr), and hydrogen fluoride (HF)] and halogens (X₂) [chlorine (Cl₂) and bromine (Br₂)] 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).

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 to include the cyclone in the sampling train to protect the filter from any moisture present. The filter collects other particulate matter including halide salts. Acidic and alkaline absorbing solutions collect the gaseous hydrogen halides and halogens, respectively. Following sampling of emissions containing liquid droplets, any halides/halogens dissolved in the liquid in the cyclone and on the filter are vaporized to gas and collected in the impingers by pulling conditioned ambient air through the sampling train. The hydrogen halides are solubilized in the acidic solution and form chloride (Cl⁻), bromide (Br⁻), and fluoride (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 (H⁺), the halide ion, and the hypohalous acid (HClO or HBrO). Sodium thiosulfate is added 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). If desired, the particulate matter recovered from the filter and the probe is analyzed following the procedures in Method 5.

If the tester intends to use this sampling arrangement to sample concurrently for particulate matter, the alternative Teflon R probe liner, cyclone, and filter holder should not be used. The Teflon R filter support must be used. The tester must also meet the probe and filter temperature requirements of both sampling trains.

METHOD 28 – CERTIFICATION AND AUDITING OF WOOD HEATERS

This method is applicable for the certification and auditing of wood heaters. This method describes the test facility, test fuel charge, and wood heater operation as well as procedures for determining burn rates and particulate emission rates and for reducing data.

Particulate matter emissions are measured from a wood heater burning a prepared test fuel crib in a test facility maintained at a set of prescribed conditions.

METHOD 28A – MEASUREMENT OF AIR TO FUEL RATIO AND MINIMUM ACHIEVABLE BURN RATES FOR WOOD FIRED APPLIANCES

This method is applicable for the measurement of air to fuel ratios and minimum achievable burn rates, for determining whether a wood fired appliance is an affected facility, as specified in 40 CFR 60.530.

A gas sample is extracted from a location in the stack of a wood fired appliance while the appliance is operating at a prescribed set of conditions. The gas sample is analyzed for percent carbon dioxide (CO₂), percent oxygen (O₂), and percent carbon monoxide (CO). These stack gas components are measured for determining dry molecular weight of exhaust gas. Total moles of exhaust gas are determined stoichiometrically. Air to fuel ratio is determined by relating the mass of dry combustion air to the mass of dry fuel consumed.

METHOD 29 – DETERMINATION OF METALS EMISSIONS FROM STATIONARY SOURCES

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.

Hg emissions can be measured, alternatively, using EPA Method 101A of Appendix B, 40 CFR Part 61. Method 101A measures only Hg but it can be of special interest to sources which need to measure both Hg and Mn emissions.

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.

APPENDIX M TO PART 51 – TEST METHODS

METHOD 201 – DETERMINATION OF PM₁₀ EMISSIONS (EXHAUST GAS RECYCLE PROCEDURE)

This method applies to the in-stack measurement of particulate matter (PM) emissions equal to or less than an aerodynamic diameter of nominally 10 μm (PM₁₀) from stationary sources. The EPA recognizes that condensable emissions not collected by an in-stack method are also PM₁₀, and that emissions that contribute to ambient PM₁₀ levels are the sum of condensable emissions and emissions measured by an in-stack PM₁₀ method, such as this method or Method 201A. Therefore, for establishing source contributions to ambient levels of PM₁₀, such as for emission inventory purposes, EPA suggests that source PM₁₀ measurement include both in-stack PM₁₀ and condensable emissions. Condensable emissions may be measured by an impinger analysis in combination with this method.

A gas sample is isokinetically extracted from the source. An in-stack cyclone is used to separate PM greater than PM₁₀, and an in-stack glass fiber filter is used to collect the PM₁₀. To maintain isokinetic flow rate conditions at the tip of the probe and a constant flow rate through the cyclone, a clean, dried portion of the sample gas at stack temperature is recycled into the nozzle. The particulate mass is determined gravimetrically after removal of uncombined water.

METHOD 201A – DETERMINATION OF PM₁₀ EMISSIONS (CONSTANT SAMPLING RATE PROCEDURE)

This method applies to the in-stack measurement of particulate matter (PM) emissions equal to or less than an aerodynamic diameter of nominally 10 (PM10) from stationary sources. The EPA recognizes that condensable emissions not collected by an in-stack method are also PM10, and that emissions that contribute to ambient, PM10 levels are the sum of condensable emissions and emissions measured by an in-stack PM10 method, such as this method or Method 201. Therefore, for establishing source contributions to ambient levels of PM10, such as for emission inventory purposes, EPA suggests that source PM10 measurement include both in-stack PM10 and condensable emissions. Condensable emissions may be measured by an impinger analysis in combination with this method.

A gas sample is extracted at a constant flow rate through an in-stack sizing device, which separates PM greater than PM10. Variations from isokinetic sampling conditions are maintained within well-defined limits. The particulate mass is determined gravimetrically after removal of uncombined water.

APPENDIX A TO PART 63 – TEST METHODS

METHOD 306 – DETERMINATION OF CHROMIUM EMISSIONS FROM DECORATIVE AND HARD CHROMIUM ELECTROPLATING AND ANODIZING OPERATIONS

This method applies to the determination of chromium (Cr) in emissions from decorative and hard chrome electroplating facilities and anodizing operations.

A sample is extracted isokinetically from the source using an unheated Method 5 sampling train (40 CFR part 60, appendix A), with a glass nozzle and probe liner, but with the filter omitted. The sample time has to be at least 2 hours. The Cr emissions are collected in an alkaline solution: 0.1 N sodium hydroxide (NaOH) or 0.1 N sodium bicarbonate (NaHCO₃). The collected samples remain in the alkaline solution until analysis. Samples with high Cr concentrations may be analyzed using inductively coupled plasma emission spectrometry (ICP) at 267.72 nm. Alternatively, if improved detection limits are required, a portion of the alkaline impinger solution is digested with nitric acid and analyzed by graphite furnace atomic absorption spectroscopy (GFAAS) at 357.9 nm.

If it is desirable to determine hexavalent chromium (Cr⁺⁶) emissions, the samples may be analyzed using an ion chromatograph equipped with a post-column reactor (IC/PCR) and a visible wavelength detector. To increase sensitivity for trace levels of Cr⁺⁶, a pre-concentration system can be used in conjunction with the IC/PCR.