

**APPENDIX "D"**

11/29/94

Test Methods and Compliance Procedures:

Emission Capture and Destruction or  
Removal Efficiency and Monitoring Requirements.

a. Determining the efficiency of volatile organic compound (VOC) capture systems.

1. For purposes of this paragraph, the following definitions and abbreviations apply:

"Gas/gas method" means either of two methods for determining capture that rely only on gas phase measurements. One method requires construction of a temporary total enclosure (TTE) to ensure all potential fugitive emissions are measured while the other method uses the room or building that houses the source as an enclosure.

"Hood" means a partial enclosure or canopy for capturing and exhausting, by means of a draft, the organic vapors or other fumes rising from a coating process or other source.

"Liquid/gas Method" means either of two methods for determining capture that require both gas phase and liquid phase measurements and analysis. One liquid/gas method requires construction of a temporary enclosure, and the other uses the building or room that houses the facility as an enclosure.

"Process line" means any coating unit, coating line, coating operation, or printing press.

"PTE" is a permanent total enclosure, which contains a process that emits VOC and meets the specifications given in this **Appendix "D"**, Method 30.

"TTE" is a temporary total enclosure that is built around a process that emits VOC and meets the specifications given in this **Appendix "D"**, Method 30.

"BE" is a building or room enclosure that contains a process that emits VOCs. If a BE is to substitute for a PTE or TTE, the appropriate requirements given in this **Appendix "D"** shall be met.

2. Applicability.

i. The requirements of paragraph (a)(3) shall apply to all regulated VOC emitting processes using a control system except as provided below.

ii. If a source owner or operator installs a PTE that meets EPA

specifications, and that directs all VOC to a control device, the capture efficiency is assumed to be 100 percent, and the source is exempted from the requirements described in paragraph (a)(3). The method given in this **Appendix "D"** shall be used to determine whether a structure is a PTE. This does not exempt a source from performing any control device efficiency testing required under this regulation. In addition, a source shall demonstrate that all criteria for a PTE are met during the testing for capture efficiency.

- iii. If a source owner or operator uses a control device designed to collect and recover VOC (e.g., carbon adsorber), an explicit measurement of capture efficiency is not necessary if the conditions given below are met. The overall emission reduction efficiency of the control system shall be determined each day by directly comparing the input liquid VOC (L) to the recovered liquid VOC. The procedure for use in this situation is specified in 40 CFR 60.433 (July 1, 1992) with the following modifications:
  - A. The source owner or operator shall obtain data each day for the solvent usage and solvent recovery and determine the solvent recovery efficiency of the system each day using a 7-day rolling period. The recovery efficiency for each day is computed as the ratio of the total recovered solvent for that day and the prior 6 consecutive operating days to the total solvent usage for the same 7-day period used for the recovered solvent, rather than a 30-day weighted average as given in 40 CFR 60.433 (July 1, 1992). This ratio shall be expressed as a percentage. This shall be done within 72 hours following each 24-hour period. A source that believes that the 7-day rolling period is not appropriate may use the method provided in **Appendix "I"** of this regulation to determine an alternative multiday rolling period. In no event shall the rolling period determined under this method exceed a 30-day rolling period.
  - B. If the solvent recovery system controls multiple process lines, the source owner or operator shall demonstrate that the overall control (i.e., the total recovered solvent VOC divided by the sum of liquid VOC input to all process lines venting to the control system) meets or exceeds the most stringent standard applicable for any process line

venting to the control system.

3. Specific Requirements.

- i. The capture efficiency shall be measured using one of the four protocols given in paragraphs (a)(3)(iii)(A) through (a)(3)(iii)(E) of this Section.
- ii. Any error margin associated with a test protocol may not be incorporated into the results of a capture efficiency test.
- iii. Any source required to comply with this Section shall use one of the following protocols to measure capture efficiency, unless a suitable alternative protocol is approved by the U.S. EPA as part of a SIP or FIP revision:
  - A. Gas/gas method using TTE. Method 30, given in this **Appendix "D"**, shall be used to determine whether a temporary enclosure is a TTE. The capture efficiency equation to be used for this protocol is:

$$CE = \frac{G}{G + F}$$

where:

- CE = Capture efficiency, decimal fraction.  
G = Mass of VOC captured and delivered to control device using a TTE.  
F = Mass of fugitive VOC that escapes from a TTE.

Either Method 30B or Method 30C of this **Appendix "D"** is used to obtain G. Method 30D of this **Appendix "D"** is used to obtain F.

- B. Liquid/gas method using TTE. Method 30, given in this **Appendix "D"**, shall be used to determine whether a temporary enclosure is a TTE. The capture efficiency equation to be used for this protocol is:

$$CE = \frac{L - F}{L}$$

where:

CE = Capture efficiency, decimal fraction.  
 L = Mass of liquid VOC input to process.  
 F = Mass of fugitive VOC that escapes from a TTE.

Method 30A of this **Appendix "D"**, is used to obtain L. Method 30D of this **Appendix "D"** is used to obtain F.

- C. Gas/gas method using the building or room (BE) in which the source is located as the enclosure and in which G and F are measured while operating only the source to be tested. All fans and blowers in the building or room shall be operated as they would be under normal production. The capture efficiency equation to be used for this protocol is:

$$CE = \frac{G}{G + F_B}$$

where:

CE = Capture efficiency, decimal fraction.  
 G = Mass of VOC captured and delivered to a control device.  
 F<sub>B</sub> = Mass of fugitive VOC that escapes from building enclosure.

Either Method 30B or Method 30C of this **Appendix "D"** is used to obtain G. Method 30E of this **Appendix "D"** is used to obtain F<sub>B</sub>.

- D. Liquid/gas method using the building or room (BE) in which the source is located as the enclosure and in which L and F are measured while operating only the source to be tested. All fans and blowers in the building or room shall be operated as they would under normal production. The capture efficiency equation to be used for this protocol is:

$$CE = \frac{L - F_B}{L}$$

where:

- CE = Capture efficiency, decimal fraction.  
 L = Mass of liquid VOC input to process.  
 F<sub>B</sub> = Mass of fugitive VOC that escapes from building enclosure.

Method 30A of this **Appendix "D"** is used to obtain L. Method 30E of this **Appendix "D"** is used to obtain F<sub>B</sub>.

- E. Liquid/gas method using the collection device to determine the mass of gas collected. The Capture Efficiency equation to be used for this process is:

where:

$$CE = \frac{C}{L}$$

- CE = Capture Efficiency, decimal fraction  
 C = Mass rate of VOC captured by collection system  
 L = Mass rate of liquid VOC input to process

4. Recordkeeping and Reporting.

- i. All sources complying with this Section shall maintain on file a copy of the capture efficiency protocol submitted to the Department. All results of appropriate test methods and CE protocols shall be reported to the Department within 60 days of the test date. A copy of the results shall be kept on file with the

source.

- ii. If any changes are made to capture or control equipment, the source is required to notify the Department within 30 days of these changes and a new capture efficiency and/or control device destruction or removal efficiency test may be required.

b. Determining the destruction or removal efficiency of incinerators and carbon adsorbers.

1. Testing.

- i. The control device destruction or removal efficiency shall be determined from data obtained by simultaneously measuring the inlet and outlet gas-phase VOC concentrations and gas volumetric flow rates in accordance with the gas-phase test methods specified in **Appendix "E"** of this regulation. The control device destruction or removal efficiency shall be calculated using the following equation:

$$E = \frac{\sum_{i=1}^n Q_i C_i - \sum_{j=1}^m Q_j C_j}{\sum_{i=1}^n Q_i C_i}$$

where:

- E = VOC destruction efficiency of the control device.
- $Q_i$  = Volumetric flow rate of the effluent gas flowing through stack i entering the control device, dry standard cubic meters per hour (dscm/hr).
- $C_i$  = Concentration of VOC (as carbon) in the effluent gas flowing through stack i entering the control device, parts per million by volume (ppmv).
- $Q_j$  = Volumetric flow rate of the effluent gas flowing through stack j leaving the control device, dscmh.
- $C_j$  = Concentration of VOC (as carbon) in the effluent gas flowing through stack j leaving the control device, ppmv.
- n = The number of vents to the control device.
- m = The number of vents after the control device.

- ii. A source using a PTE (or a BE as a PTE) shall demonstrate that this enclosure meets the requirements given in Method 30 of this **Appendix "D"** for a PTE during any testing of a control device.
- iii. A source using a TTE (or a BE as a TTE) shall demonstrate that this enclosure meets the requirements given in Method 30 of this **Appendix "D"** for a TTE during testing of a control device. The source shall also provide documentation that the quality assurance criteria for a TTE have been achieved.

2. Monitoring.

- i. Any owner or operator who uses an incinerator or regenerative carbon adsorber to comply with any part of this regulation shall install, calibrate, certify to the Department, operate, and maintain continuous monitoring equipment. The continuous monitoring equipment shall monitor the following parameters:
    - A. Combustion chamber temperature of each thermal incinerator or afterburner.
    - B. Temperature rise immediately before the catalyst bed and across each catalytic incinerator bed.
    - C. The VOC concentration of the outlet from each carbon adsorption bed.
  - ii. The continuous temperature monitoring equipment must be equipped with a continuous recorder and have an accuracy of  $\pm 1$  percent of the combustion temperature being measured expressed in degrees Celsius ( $^{\circ}\text{C}$ ) or  $\pm 0.5^{\circ}\text{C}$ , whichever is greater.
  - iii. The owner or operator shall ensure that the quality assurance measures in **Appendix "G" (j)** and the quality control procedures in **Appendix "H"** of this regulation are met.
- c. Determining the overall emission reduction efficiency. The overall emission reduction efficiency of the emission control system shall be determined each day as the product of the capture efficiency, as determined using the capture efficiency test method in this **Appendix "D"**, and the control device destruction or removal efficiency; or for each solvent recovery system, by the test protocol described in paragraph (a)(2)(iii)(A) of this Section for comparing liquid input to liquid VOC recovery. The results of the capture efficiency test and control device destruction or removal efficiency test remain valid for each day until a subsequent test is performed. The results of any

valid test may be used for each day until superseded by the results of a valid test subsequently performed.

## METHOD 30 - CRITERIA FOR AND VERIFICATION OF A PERMANENT OR TEMPORARY TOTAL ENCLOSURE

### 1. Introduction

- 1.1 Applicability. This procedure is used to determine whether a permanent or temporary enclosure meets the criteria for a total enclosure.
- 1.2 Principle. An enclosure is evaluated against a set of criteria. If the criteria are met and if all the exhaust gases from the enclosure are ducted to a control device, then the VOC CE is assumed to be 100 percent, and CE need not be measured. However, if part of the exhaust gas stream is not ducted to a control device, CE must be determined.
- 1.3 Note. An evaluation of the proposed building materials is recommended to minimize any potential hazards.

### 2. Definitions

- 2.1 Natural Draft Opening (NDO). Any permanent opening in the enclosure that remains open during operation of the facility and is not connected to a duct in which a fan is installed.
- 2.2 Permanent Total Enclosure (PTE). A permanently installed enclosure that completely surrounds a source of emissions such that all VOC emissions are captured and contained for discharge to a control device.
- 2.3 Temporary Total Enclosure (TTE). A temporarily installed enclosure that completely surrounds a source of emissions such that all fugitive VOC emissions are captured and contained for discharge through ducts that allow for the accurate measurement of fugitive VOC emissions.

### 3. Criteria for Temporary Total Enclosure

- 3.1 Any NDO shall be at least four equivalent opening diameters from each VOC emitting point unless otherwise specified by the Department.
- 3.2 Any exhaust point from the enclosure shall be at least four equivalent duct or hood diameters from each NDO.
- 3.3 The total area of all NDO's shall not exceed 5 percent of the surface area of the

enclosure's four walls, floor, and ceiling.

- 3.4 The average facial velocity (FV) of air through all NDO's shall be at least 3,600 m/hr (200 fpm). The direction of air flow through all NDO's shall be into the enclosure.
  - 3.5 All access doors and windows whose areas are not included in Section 3.3 and are not included in the calculation in Section 3.4 shall be closed during routine operation of the process.
4. Criteria for a Permanent Total Enclosure
    - 4.1 Same as Sections 3.1 and 3.3 through 3.5.
    - 4.2 All VOC emissions must be captured and contained for discharge through a control device.
5. Procedure
    - 5.1 Determine the equivalent diameters of the NDO's and determine the distances from each VOC emitting point to all NDO's. Determine the equivalent diameter of each exhaust duct or hood and its distance to all NDO's. Calculate the distances in terms of equivalent diameters. The number of equivalent diameters shall be at least four.
    - 5.2 Measure the total area ( $A_T$ ) of the enclosure and the total area ( $A_N$ ) of all NDO's in the enclosure. Calculate the NDO to enclosure area ratio (NEAR) as follows:

$$NEAR = \frac{A_N}{A_T} \quad \text{Eq. 30-1}$$

The NEAR must be  $\leq 0.05$ .

- 5.3 Measure the volumetric flow rate, corrected to standard conditions, of each gas stream exiting the enclosure through an exhaust duct or hood using EPA Method 2. In some cases (e.g., when the building is the enclosure), it may be necessary to measure the volumetric flow rate, corrected to standard conditions, of each gas stream entering the enclosure through a forced makeup air duct using Method 2. Calculate FV using the following equation:

$$FV = \frac{Q_D - Q_I}{A_N} \quad \text{Eq. 30-2}$$

where:

$Q_O$  = the sum of the volumetric flow from all gas streams exiting the enclosure through an exhaust duct or hood.

$Q_I$  = the sum of the volumetric flow from all gas streams into the enclosure through a forced makeup air duct; zero, if there is no forced makeup air into the enclosure.

$A_N$  = total area of all NDO's in enclosure.

The FV shall be at least 3,600 m/hr (200 fpm). Alternatively, measure the pressure differential across the enclosure. A pressure drop of 0.0075 mm Hg (0.004 in. H<sub>2</sub>O) corresponds to an FV of 3,600 m/hr (200 fpm).

5.4 Verify that the direction of air flow through all NDO's is inward. Streamers, smoke tubes, or tracer gases may be used. Strips of plastic wrapping film have also been found to be effective. Monitor the direction of air flow for at least 1 hour, with checks made no more than 10 minutes apart.

## 6. Quality Assurance

6.1 The success of this method lies in designing the TTE to simulate the conditions that exist without the TTE (i.e., the effect of the TTE on the normal flow patterns around the affected facility or the amount of fugitive VOC emissions should be minimal). The TTE must enclose the application stations, coating reservoirs, and all areas from the application station to the oven. The oven does not have to be enclosed if it is under negative pressure. The NDO's of the temporary enclosure and a fugitive exhaust fan must be properly sized and placed.

6.2 Estimate the ventilation rate of the TTE that best simulates the conditions that exist without the TTE (i.e., the effect of the TTE on the normal flow patterns around the affected facility or the amount of fugitive VOC emissions should be minimal). Measure the concentration ( $C_G$ ) and flow rate ( $Q_G$ ) of the captured gas stream, specify a safe concentration ( $C_F$ ) for the fugitive gas stream, estimate the CE, and then use a plot available from the Department to determine the volumetric flow rate of the fugitive gas stream ( $Q_F$ ). A fugitive VOC emission exhaust fan that has a variable flow control is desirable.

6.3 Monitor the concentration of VOC into the capture device without the TTE. To minimize the effect of temporal variation on the captured emissions, the baseline measurement should be made over as long a time period as practical. However, the process conditions must be the same for the measurement in Section 6.5 as they are for this baseline measurement. This may require short measuring times for this quality control check before and after the

construction of the TTE.

- 6.4 After the TTE is constructed, monitor the VOC concentration inside the TTE. This concentration shall not continue to increase, and must not exceed the safe level according to Occupational Safety and Health Administration requirements for permissible exposure limits. An increase in VOC concentration indicates poor TTE design or poor capture efficiency.
- 6.5 Monitor the concentration of VOC into the capture device with the TTE. To limit the effect of the TTE on the process, the VOC measurement with and without the TTE must be within  $\pm 10$  percent. If the measurements do not agree, adjust the ventilation rate from the TTE until they agree within 10 percent.

## METHOD 30A - VOLATILE ORGANIC COMPOUNDS CONTENT IN LIQUID INPUT STREAM

### 1. Introduction

- 1.1 **Applicability.** This procedure is applicable for determining the input of VOC. It is intended to be used in the development of liquid/gas protocols for determining VOC CE for surface coating and printing operations.
- 1.2 **Principle.** The amount of VOC introduced to the process (L) is the sum of the products of the weight (W) of each VOC containing liquid (ink, paint, solvent, etc.) used and its VOC content (V). A sample of each VOC containing liquid is analyzed with a flame ionization analyzer (FIA) to determine V.
- 1.3 **Estimated Measurement Uncertainty.** The measurement uncertainties are estimated for each VOC containing liquid as follows:  $W = \pm 2.0$  percent and  $V = \pm 12.0$  percent. Based on these numbers, the probable uncertainty for L is estimated at about  $\pm 12.2$  percent for each VOC containing liquid.
- 1.4 **Sampling Requirements.** A CE test shall consist of at least three sampling runs. Each run shall cover at least one complete production cycle, but shall be at least 3 hours long. The sampling time for each run need not exceed 8 hours, even if the production cycle has not been completed. Alternative sampling times may be used with the approval of the Department.
- 1.5 **Notes.** Because this procedure is often applied in highly explosive areas, caution and care should be exercised in choosing, installing, and using the appropriate equipment. Mention of trade names or company products does not constitute endorsement. All gas concentrations (percent, ppm) are by volume, unless otherwise noted.

## 2. Apparatus and Reagents

### 2.1 Liquid Weight.

2.1.1 Balances/Digital Scales. To weigh drums of VOC containing liquids to within 0.2 lb.

2.1.2 Volume Measurement Apparatus (Alternative). Volume meters, flow meters, density measurement equipment, etc., as needed to achieve the same accuracy as direct weight measurements.

### 2.2 VOC Content (FIA Technique). The following equipment is required:

2.2.1 Sample Collection Can. An appropriately-sized metal can to be used to collect VOC containing materials. The can must be constructed in such a way that it can be grounded to the coating container.

2.2.2 Needle Valves. To control gas flow.

2.2.3 Regulators. For carrier gas and calibration gas cylinders.

2.2.4 Tubing. Teflon or stainless steel tubing with diameters and lengths determined by connection requirements of equipment. The tubing between the sample oven outlet and the FIA shall be heated to maintain a temperature of  $120 \pm 5^{\circ}\text{C}$ .

2.2.5 Atmospheric Vent. A tee and 0- to 0.5-liter/min rotameter placed in the sampling line between the carrier gas cylinder and the VOC sample vessel to release the excess carrier gas. A toggle valve placed between the tee and the rotameter facilitates leak tests of the analysis system.

2.2.6 Thermometer. Capable of measuring the temperature of the hot water bath to within  $1^{\circ}\text{C}$ .

2.2.7 Sample Oven. Heated enclosure, containing calibration gas coil heaters, critical orifice, aspirator, and other liquid sample analysis components, capable of maintaining a temperature of  $120 \pm 5^{\circ}\text{C}$ .

2.2.8 Gas Coil Heaters. Sufficient lengths of stainless steel or Teflon tubing to allow zero and calibration gases to be heated to the sample oven temperature before entering the critical orifice or aspirator.

2.2.9 Water Bath. Capable of heating and maintaining a sample vessel temperature of  $100 \pm 5^{\circ}\text{C}$ .

2.2.10 Analytical Balance. To measure  $\pm 0.001$  g.

- 2.2.11 Disposable Syringes. 2-cc or 5-cc.
- 2.2.12 Sample Vessel. Glass, 40-ml septum vial. A separate vessel is needed for each sample.
- 2.2.13 Rubber Stopper. Two-hole stopper to accommodate 3.2-mm (1/8-in.) Teflon tubing, appropriately sized to fit the opening of the sample vessel. The rubber stopper should be wrapped in Teflon tape to provide a tighter seal and to prevent any reaction of the sample with the rubber stopper. Alternatively, any leak-free closure fabricated of nonreactive materials and accommodating the necessary tubing fittings may be used.
- 2.2.14 Critical Orifices. Calibrated critical orifices capable of providing constant flow rates from 50 to 250 ml/min at known pressure drops. Sapphire orifice assemblies (available from O'Keefe Controls Company) and glass capillary tubing have been found to be adequate for this application.
- 2.2.15 Vacuum Gauge. Zero to 760-mm (0- to 30-in.) Hg U-Tube manometer or vacuum gauge.
- 2.2.16 Pressure Gauge. Bourdon gauge capable of measuring the maximum air pressure at the aspirator inlet (e.g., 100 psig, 690 kiloPascals).
- 2.2.17 Aspirator. A device capable of generating sufficient vacuum at the sample vessel to create critical flow through the calibrated orifice when sufficient air pressure is present at the aspirator inlet. The aspirator must also provide sufficient sample pressure to operate the FIA. The sample is also mixed with the dilution gas within the aspirator.
- 2.2.18 Soap Bubble Meter. Of an appropriate size to calibrate the critical orifices in the system.
- 2.2.19 Organic Concentration Analyzer. An FIA with a span value of 1.5 times the expected concentration as propane; however, other span values may be used if it can be demonstrated that they would provide more accurate measurements. The FIA instrument should be the same instrument used in the gaseous analyses adjusted with the same fuel, combustion air, and sample back-pressure (flow rate) settings. The system shall be capable of meeting or exceeding the following specifications:
- 2.2.19.1 Zero Drift. Less than  $\pm 3.0$  percent of the span value.
- 2.2.19.2 Calibration Drift. Less than  $\pm 3.0$  percent of the span value.
- 2.2.19.3 Calibration Error. Less than  $\pm 5.0$  percent of the calibration gas value.
- 2.2.20 Integrator/Data Acquisition System. An analog or digital device or computerized data acquisition system used to integrate the FIA response or compute the average response and record measurement data. The minimum data sampling frequency for computing

average or integrated values is one measurement value every 5 seconds. The device shall be capable of recording average values at least once per minute.

2.2.21 Chart Recorder (Optional). A chart recorder or similar device is recommended to provide a continuous analog display of the measurement results during the liquid sample analysis.

2.2.22 Calibration and Other Gases. Gases used for calibration, fuel, and combustion air (if required) are contained in compressed gas cylinders. All calibration gases shall be traceable to National Institute of Standards and Technology standards and shall be certified by the manufacturer to  $\pm 1$  percent of the tag value. Additionally, the manufacturer of the cylinder should provide a recommended shelf life for each calibration gas cylinder over which the concentration does not change more than  $\pm 2$  percent from the certified value. For calibration gas values not generally available, alternative methods for preparing calibration gas mixtures, such as dilution systems, may be used with the approval of the Department.

2.2.22.1 Fuel. A 40 percent H<sub>2</sub>/60 percent He or 40 percent H<sub>2</sub>/60 percent N<sub>2</sub> gas mixture is recommended to avoid an oxygen synergism effect that reportedly occurs when oxygen concentration varies significantly from a mean value.

2.2.22.2 Carrier Gas. High purity air with less than 1 ppm of organic material (as propane) or less than 0.1 percent of the span value, whichever is greater.

2.2.22.3 FIA Linearity Calibration Gases. Low-, mid- and high-range gas mixture standards with nominal propane concentrations of 20-30, 45-55, and 70-80 percent of the span value in air, respectively. Other calibration values and other span values may be used if it can be shown to the Department's satisfaction that more accurate measurements would be achieved.

2.2.22.4 System Calibration Gas. Gas mixture standard containing propane in air, approximating the undiluted VOC concentration expected for the liquid samples.

### 3. Determination of Liquid Input Weight

3.1 Weight Difference. Determine the amount of material introduced to the process as the weight difference of the feed material before and after each sampling run. In determining the total VOC containing liquid usage, account for:

- (a) The initial (beginning) VOC containing liquid mixture.
- (b) Any solvent added during the test run.
- (c) Any coating added during the test run.

(d) Any residual VOC containing liquid mixture remaining at the end of the sample run.

3.1.1 Identify all points where VOC containing liquids are introduced to the process. To obtain an accurate measurement of VOC containing liquids, start with an empty fountain (if applicable). After completing the run, drain the liquid in the fountain back into the liquid drum (if possible) and weigh the drum again. Weigh the VOC containing liquids to  $\pm 0.5$  percent of the total weight (full) or  $\pm 0.1$  percent of the total weight of VOC containing liquid used during the sample run, whichever is less. If the residual liquid cannot be returned to the drum, drain the fountain into a preweighed empty drum to determine the final weight of the liquid.

3.1.2 If it is not possible to measure a single representative mixture, then weigh the various components separately (e.g., if solvent is added during the sampling run, weigh the solvent before it is added to the mixture). If a fresh drum of VOC containing liquid is needed during the run, then weigh both the empty drum and fresh drum.

3.2 Volume Measurement (Alternative). If direct weight measurements are not feasible, the tester may use volume meters and flow rate meters (and density measurements) to determine the weight of liquids used if it can be demonstrated that the technique produces results equivalent to the direct weight measurements. If a single representative mixture cannot be measured, measure the components separately.

#### 4. Determination of VOC Content in Input Liquids

##### 4.1 Collection of Liquid Samples.

4.1.1 Collect a 100-ml or larger sample of the VOC containing liquid mixture at each application location at the beginning and end of each test run. A separate sample should be taken of each VOC containing liquid added to the application mixture during the test run. If a fresh drum is needed during the sampling run, then obtain a sample from the fresh drum.

4.1.2 When collecting the sample, ground the sample container to the coating drum. Fill the sample container as close to the rim as possible to minimize the amount of headspace.

4.1.3 After the sample is collected, seal the container so the sample cannot leak out or evaporate.

4.1.4 Label the container to clearly identify the contents.

##### 4.2 Liquid Sample VOC Content.

4.2.1 Assemble the liquid VOC content analysis system.

- 4.2.2 Permanently identify all of the critical orifices that may be used. Calibrate each critical orifice under the expected operating conditions (i.e., sample vacuum and temperature) against a volume meter as described in Section 5.3.
- 4.2.3 Label and tare the sample vessels (including the stoppers and caps) and the syringes.
- 4.2.4 Install an empty sample vessel and perform a leak test of the system. Close the carrier gas valve and atmospheric vent and evacuate the sample vessel to 250 mm (10 in.) Hg absolute or less using the aspirator. Close the toggle valve at the inlet to the aspirator and observe the vacuum for at least 1 minute. If there is any change in the sample pressure, release the vacuum, adjust or repair the apparatus as necessary, and repeat the leak test.
- 4.2.5 Perform the analyzer calibration and linearity checks according to the procedure in Section 5.1. Record the responses to each of the calibration gases and the back-pressure setting of the FIA.
- 4.2.6 Establish the appropriate dilution ratio by adjusting the aspirator air supply or substituting critical orifices. Operate the aspirator at a vacuum of at least 25 mm (1 in.) Hg greater than the vacuum necessary to achieve critical flow. Select the dilution ratio so that the maximum response of the FIA to the sample does not exceed the high-range calibration gas.
- 4.2.7 Perform system calibration checks at two levels by introducing compressed gases at the inlet to the sample vessel while the aspirator and dilution devices are operating. Perform these checks using the carrier gas (zero concentration) and the system calibration gas. If the response to the carrier gas exceeds  $\pm 0.5$  percent of span, clean or repair the apparatus and repeat the check. Adjust the dilution ratio as necessary to achieve the correct response to the upscale check, but do not adjust the analyzer calibration. Record the identification of the orifice, aspirator air supply pressure, FIA back-pressure, and the responses of the FIA to the carrier and system calibration gases.
- 4.2.8 After completing the above checks, inject the system calibration gas for approximately 10 minutes. Time the exact duration of the gas injection using a stopwatch. Determine the area under the FIA response curve and calculate the system response factor based on the sample gas flow rate, gas concentration, and the duration of the injection as compared to the integrated response using Equations 30A-2 and 30A-3.
- 4.2.9 Verify that the sample oven and sample line temperatures are  $120 \pm 5^\circ\text{C}$  and that the water bath temperature is  $100 \pm 5^\circ\text{C}$ .
- 4.2.10 Fill a tared syringe with approximately 1 g of the VOC containing liquid and weigh it. Transfer the liquid to a tared sample vessel. Plug the sample vessel to minimize sample loss. Weigh the sample vessel containing the liquid to determine the amount of sample

actually received. Also, as a quality control check, weigh the empty syringe to determine the amount of material delivered. The two coating sample weights should agree within 0.02 g. If not, repeat the procedure until an acceptable sample is obtained.

- 4.2.11 Connect the vessel to the analysis system. Adjust the aspirator supply pressure to the correct value. Open the valve on the carrier gas supply to the sample vessel and adjust it to provide a slight excess flow to the atmospheric vent. As soon as the initial response of the FIA begins to decrease, immerse the sample vessel in the water bath. (Applying heat to the sample vessel too soon may cause the FIA response to exceed the calibrated range of the instrument and, thus, invalidate the analysis.)
- 4.2.12 Continuously measure and record the response of the FIA until all of the volatile material has been evaporated from the sample and the instrument response has returned to the baseline (i.e., response less than 0.5 percent of the span value). Observe the aspirator supply pressure, FIA back-pressure, atmospheric vent, and other system operating parameters during the run; repeat the analysis procedure if any of these parameters deviate from the values established during the system calibration checks in Section 4.2.7. After each sample, perform the drift check described in Section 5.2. If the drift check results are acceptable, calculate the VOC content of the sample using the equations in Section 7. Integrate the area under the FIA response curve, or determine the average concentration response and the duration of sample analysis.

## 5. Calibration and Quality Assurance

- 5.1 FIA Calibration and Linearity Check. Make necessary adjustments to the air and fuel supplies for the FIA and ignite the burner. Allow the FIA to warm up for the period recommended by the manufacturer. Inject a calibration gas into the measurement system and adjust the back-pressure regulator to the value required to achieve the flow rates specified by the manufacturer. Inject the zero- and the high-range calibration gases and adjust the analyzer calibration to provide the proper responses. Inject the low- and mid-range gases and record the responses of the measurement system. The calibration and linearity of the system are acceptable if the responses for all four gases are within 5 percent of the respective gas values. If the performance of the system is not acceptable, repair or adjust the system and repeat the linearity check. Conduct a calibration and linearity check after assembling the analysis system and after a major change is made to the system.
- 5.2 Systems Drift Checks. After each sample, repeat the system calibration checks in Section 4.2.7 before any adjustments to the FIA or measurement system are made. If the zero or calibration drift exceeds  $\pm 3$  percent of the span value, discard the result and repeat the analysis.
- 5.3 Critical Orifice Calibration.

- 5.3.1 Each critical orifice must be calibrated at the specific operating conditions under which it will be used. Therefore, assemble all components of the liquid sample analysis system as they are to be used. A stopwatch is also required.
- 5.3.2 Turn on the sample oven, sample line, and water bath heaters, and allow the system to reach the proper operating temperature. Adjust the aspirator to a vacuum of 380 mm (15 in.) Hg vacuum. Measure the time required for one soap bubble to move a known distance and record barometric pressure.
- 5.3.3 Repeat the calibration procedure at a vacuum of 406 mm (16 in.) Hg and at 25-mm (1-in.) Hg intervals until three consecutive determinations provide the same flow rate. Calculate the critical flow rate for the orifice in ml/min at standard conditions. Record the vacuum necessary to achieve critical flow.

## 6. Nomenclature

$A_L$	=	area under the response curve of the liquid sample, area count.
$A_S$	=	area under the response curve of the calibration gas, area count.
$C_S$	=	actual concentration of system calibration gas, ppm propane.
$K$	=	$1.830 \times 10^{-9}$ g/(ml-ppm).
$L$	=	total VOC content of liquid input, kg.
$M_L$	=	mass of liquid sample delivered to the sample vessel, g.
$q$	=	flow rate through critical orifice, ml/min.
$RF$	=	liquid analysis system response factor, g/area count.
$\theta_S$	=	total gas injection time for system calibration gas during integrator calibration, min.
$V_{Fj}$	=	final VOC fraction of VOC containing liquid j.
$V_{Ij}$	=	initial VOC fraction of VOC containing liquid j.
$V_{Aj}$	=	VOC fraction of VOC containing liquid j added during the run.
$V$	=	VOC fraction of liquid sample.
$W_{Fj}$	=	weight of VOC containing liquid j remaining at end of the run, kg.
$W_{Ij}$	=	weight of VOC containing liquid j at beginning of the run, kg.
$W_{Aj}$	=	weight of VOC containing liquid j added during the run, kg.

## 7. Calculations

### 7.1 Total VOC Content of the Input VOC Containing Liquid.

$$L = \sum_{j=1}^n V_{Ij} W_{Ij} - \sum_{j=1}^n V_{Fj} W_{Fj} + \sum_{j=1}^n V_{Aj} W_{Aj} \quad \text{Eq. 30A-1}$$

7.2 Liquid Sample Analysis System Response Factor for Systems Using Integrators, Grams/Area Count.

$$RF = \frac{C_s q \theta_s K}{A_s}$$

Eq. 30A-2

7.3 VOC Content of the Liquid Sample.

$$V = \frac{A_L RF}{M_L}$$

Eq. 30A-3

## METHOD 30B - VOLATILE ORGANIC COMPOUNDS EMISSIONS IN CAPTURED STREAM

### 1. Introduction

- 1.1 **Applicability.** This procedure is applicable for determining the VOC content of captured gas streams. It is intended to be used in the development of liquid/gas or gas/gas protocols for determining VOC CE for surface coating and printing operations. The procedure may not be acceptable in certain site-specific situations [e.g., when: (1) direct-fired heaters or other circumstances affect the quantity of VOC at the control device inlet; and (2) particulate organic aerosols are formed in the process and are present in the captured emissions].
- 1.2 **Principle.** The amount of VOC captured ( $G$ ) is calculated as the sum of the products of the VOC content ( $C_{Gj}$ ), the flow rate ( $Q_{Gj}$ ), and the sample time ( $\theta_c$ ) from each captured emissions point.
- 1.3 **Estimated Measurement Uncertainty.** The measurement uncertainties are estimated for each captured or fugitive emissions point as follows:  $Q_{Gj} = \pm 5.5$  percent and  $C_{Gj} = \pm 5.0$  percent. Based on these numbers, the probable uncertainty for  $G$  is estimated at about  $\pm 7.4$  percent.
- 1.4 **Sampling Requirements.** A CE test shall consist of at least three sampling runs. Each run shall cover at least one complete production cycle, but shall be at least 3 hours long. The sampling time for each run need not exceed 8 hours, even if the production cycle has not been completed. Alternative sampling times may be used with the approval of the Department.
- 1.5 **Notes.** Because this procedure is often applied in highly explosive areas, caution and care should be exercised in choosing, installing, and using the appropriate equipment. Mention of trade names or company products does not constitute endorsement. All gas concentrations (percent, ppm) are by volume, unless otherwise noted.

### 2. Apparatus and Reagents

- 2.1 **Gas VOC Concentration.** The main components are as follows:
  - 2.1.1 **Sample Probe.** Stainless steel or equivalent. The probe shall be heated to prevent VOC condensation.
  - 2.1.2 **Calibration Valve Assembly.** Three-way valve assembly at the outlet of the sample probe to direct the zero and calibration gases to the analyzer. Other methods, such as quick-connect lines, to route calibration gases to the outlet of the sample probe are acceptable.

- 2.1.3 Sample Line. Stainless steel or Teflon tubing to transport the sample gas to the analyzer. The sample line must be heated to prevent condensation.
- 2.1.4 Sample Pump. A leak-free pump, to pull the sample gas through the system at a flow rate sufficient to minimize the response time of the measurement system. The components of the pump that contact the gas stream shall be constructed of stainless steel or Teflon. The sample pump must be heated to prevent condensation.
- 2.1.5 Sample Flow Rate Control. A sample flow rate control valve and rotameter, or equivalent, to maintain a constant sampling rate within 10 percent. The flow rate control valve and rotameter must be heated to prevent condensation. A control valve may also be located on the sample pump bypass loop to assist in controlling the sample pressure and flow rate.
- 2.1.6 Sample Gas Manifold. Capable of diverting a portion of the sample gas stream to the FIA, and the remainder to the bypass discharge vent. The manifold components shall be constructed of stainless steel or Teflon. If captured or fugitive emissions are to be measured at multiple locations, the measurement system shall be designed to use separate sampling probes, lines, and pumps for each measurement location and a common sample gas manifold and FIA. The sample gas manifold and connecting lines to the FIA must be heated to prevent condensation.
- 2.1.7 Organic Concentration Analyzer. An FIA with a span value of 1.5 times the expected concentration as propane; however, other span values may be used if it can be demonstrated to the Department's satisfaction that they would provide more accurate measurements. The system shall be capable of meeting or exceeding the following specifications:
- 2.1.7.1 Zero Drift. Less than  $\pm 3.0$  percent of the span value.
- 2.1.7.2 Calibration Drift. Less than  $\pm 3.0$  percent of the span value.
- 2.1.7.3 Calibration Error. Less than  $\pm 5.0$  percent of the calibration gas value.
- 2.1.7.4 Response Time. Less than 30 seconds.
- 2.1.8 Integrator/Data Acquisition System. An analog or digital device, or computerized data acquisition system used to integrate the FIA response or compute the average response and record measurement data. The minimum data sampling frequency for computing average or integrated values is one measurement value every 5 seconds. The device shall be capable of recording average values at least once per minute.
- 2.1.9 Calibration and Other Gases. Gases used for calibration, fuel, and combustion air (if required) are contained in compressed gas cylinders. All calibration gases shall be

traceable to National Institute of Standards and Technology standards and shall be certified by the manufacturer to  $\pm 1$  percent of the tag value. Additionally, the manufacturer of the cylinder should provide a recommended shelf life for each calibration gas cylinder over which the concentration does not change more than  $\pm 2$  percent from the certified value. For calibration gas values not generally available, alternative methods for preparing calibration gas mixtures, such as dilution systems, may be used with the approval of the Department.

2.1.9.1 Fuel. A 40 percent H<sub>2</sub>/60 percent He or 40 percent H<sub>2</sub>/60 percent N<sub>2</sub> gas mixture is recommended to avoid an oxygen synergism effect that reportedly occurs when oxygen concentration varies significantly from a mean value.

2.1.9.2 Carrier Gas. High purity air with less than 1 ppm of organic material (as propane or carbon equivalent) or less than 0.1 percent of the span value, whichever is greater.

2.1.9.3 FIA Linearity Calibration Gases. Low-, mid-, and high-range gas mixture standards with nominal propane concentrations of 20-30, 45-55, and 70-80 percent of the span value in air, respectively. Other calibration values and other span values may be used if it can be shown to the Department's satisfaction that more accurate measurements would be achieved.

2.1.9.4 Dilution Check Gas. Gas mixture standard containing propane in air, approximately half the span value after dilution.

2.1.10 Particulate Filter. An in-stack or an out-of-stack glass fiber filter is recommended if exhaust gas particulate loading is significant. An out-of-stack filter must be heated to prevent any condensation unless it can be demonstrated that no condensation occurs.

## 2.2 Captured Emissions Volumetric Flow Rate.

2.2.1 Method 2 or 2A Apparatus. For determining volumetric flow rate.

2.2.2 Method 3 Apparatus and Reagents. For determining molecular weight of the gas stream. An estimate of the molecular weight of the gas stream may be used if approved by the Department.

2.2.3 Method 4 Apparatus and Reagents. For determining moisture content, if necessary.

## 3. Determination of Volumetric Flow Rate of Captured Emissions

3.1 Locate all points where emissions are captured from the affected facility. Using Method 1, determine the sampling points. Be sure to check each site for cyclonic or swirling flow.

3.2 Measure the velocity at each sampling site at least once every hour during each sampling run using Method 2 or 2A.

#### 4. Determination of VOC Content of Captured Emissions

4.1 Analysis Duration. Measure the VOC responses at each captured emissions point during the entire test run or, if applicable, while the process is operating. If there are multiple captured emission locations, design a sampling system to allow a single FIA to be used to determine the VOC responses at all sampling locations.

4.2 Gas VOC Concentration.

4.2.1 Assemble the sample train. Calibrate the FIA according to the procedure in Section 5.1.

4.2.2 Conduct a system check according to the procedure in Section 5.3.

4.2.3 Install the sample probe so that the probe is centrally located in the stack, pipe, or duct, and is sealed tightly at the stack port connection.

4.2.4 Inject zero gas at the calibration valve assembly. Allow the measurement system response to reach zero. Measure the system response time as the time required for the system to reach the effluent concentration after the calibration valve has been returned to the effluent sampling position.

4.2.5 Conduct a system check before, and a system drift check after, each sampling run according to the procedures in Sections 5.2 and 5.3. If the drift check following a run indicates unacceptable performance (see Section 5.3), the run is not valid. The tester may elect to perform system drift checks during the run not to exceed one drift check per hour.

4.2.6 Verify that the sample lines, filter, and pump temperatures are  $120 \pm 5^\circ\text{C}$ .

4.2.7 Begin sampling at the start of the test period and continue to sample during the entire run. Record the starting and ending times and any required process information as appropriate. If multiple captured emission locations are sampled using a single FIA, sample at each location for the same amount of time (e.g., 2 minutes) and continue to switch from one location to another for the entire test run. Be sure that total sampling time at each location is the same at the end of the test run. Collect at least four separate measurements from each sample point during each hour of testing. Disregard the measurements at each sampling location until two times the response time of the measurement system has elapsed. Continue sampling for at least 1 minute and record the concentration measurements.

4.3 Background Concentration. NOTE: Not applicable when the building is used as the TTE.

- 4.3.1 Locate all NDO's of the TTE. A sampling point shall be at the center of each NDO, unless otherwise specified by the Department. If there are more than six NDO's, choose six sampling points evenly spaced among the NDO's.
- 4.3.2 Assemble the sample train. Calibrate the FIA and conduct a system check according to the procedures in Sections 5.1 and 5.3. NOTE: This sample train shall be separate from the sample train used to measure the captured emissions.
- 4.3.3 Position the probe at the sampling location.
- 4.3.4 Determine the response time, conduct the system check, and sample according to the procedures described in Sections 4.2.4 through 4.2.7.
- 4.4 Alternative Procedure. The direct interface sampling and analysis procedure described in Section 7.2 of Method 18 may be used to determine the gas VOC concentration. The system must be designed to collect and analyze at least one sample every 10 minutes.

## 5. Calibration and Quality Assurance

- 5.1 FIA Calibration and Linearity Check. Make necessary adjustments to the air and fuel supplies for the FIA and ignite the burner. Allow the FIA to warm up for the period recommended by the manufacturer. Inject a calibration gas into the measurement system and adjust the back-pressure regulator to the value required to achieve the flow rates specified by the manufacturer. Inject the zero- and the high-range calibration gases and adjust the analyzer calibration to provide the proper responses. Inject the low- and mid-range gases and record the responses of the measurement system. The calibration and linearity of the system are acceptable if the responses for all four gases are within 5 percent of the respective gas values. If the performance of the system is not acceptable, repair or adjust the system and repeat the linearity check. Conduct a calibration and linearity check after assembling the analysis system and after a major change is made to the system.
- 5.2 Systems Drift Checks. Select the calibration gas that most closely approximates the concentration of the captured emissions for conducting the drift checks. Introduce the zero and calibration gases at the calibration valve assembly and verify that the appropriate gas flow rate and pressure are present at the FIA. Record the measurement system responses to the zero and calibration gases. The performance of the system is acceptable if the difference between the drift check measurement and the value obtained in Section 5.1 is less than 3 percent of the span value. Conduct the system drift checks at the end of each run.
- 5.3 System Check. Inject the high-range calibration gas at the inlet of the sampling probe and record the response. The performance of the system is acceptable if the measurement system response is within 5 percent of the value obtained in Section 5.1 for the high-range

calibration gas. Conduct a system check before and after each test run.

- 5.4 Analysis Audit Procedure. Immediately before each test, analyze an audit cylinder as described in Section 5.2. The analysis audit must agree with the audit cylinder concentration within 10 percent.

## 6. Nomenclature

$A_i$	=	area of NDO $i$ , $\text{ft}^2$ .
$A_N$	=	total area of all NDO's in the enclosure, $\text{ft}^2$ .
$C_{Bi}$	=	corrected average VOC concentration of background emissions at point $i$ , ppm propane.
$C_B$	=	average background concentration, ppm propane.
$C_{Gj}$	=	corrected average VOC concentration of captured emissions at point $j$ , ppm propane.
$C_{DH}$	=	average measured concentration for the drift check calibration gas, ppm propane.
$C_{D0}$	=	average system drift check concentration for zero concentration gas, ppm propane.
$C_H$	=	actual concentration of the drift check calibration gas, ppm propane.
$C_i$	=	uncorrected average background VOC concentration measured at point $i$ , ppm propane.
$C_j$	=	uncorrected average VOC concentration measured at point $j$ , ppm propane.
$G$	=	total VOC content of captured emissions, kg.
$K_1$	=	$1.830 \times 10^{-6} \text{ kg}/(\text{m}^3\text{-ppm})$ .
$n$	=	number of measurement points.
$Q_{Gj}$	=	average effluent volumetric flow rate corrected to standard conditions at captured emissions point $j$ , $\text{m}^3/\text{min}$ .
$\theta_C$	=	total duration of captured emissions.

## 7. Calculations

### 7.1 Total VOC Captured Emissions.

$$G = \sum_{j=1}^n (C_{Gj} - C_B) Q_{Gj} \theta_c K_1 \quad \text{Eq. 30B-1}$$

### 7.2 VOC Concentration of the Captured Emissions at Point j.

$$C_{Gj} = (C_j - C_{DO}) \frac{C_H}{C_{DH} - C_{DO}} \quad \text{Eq. 30B-2}$$

### 7.3 Background VOC Concentration at Point i.

$$C_{Bi} = (C_i - C_{DO}) \frac{C_H}{C_{DH} - C_{DO}} \quad \text{Eq. 30B-3}$$

### 7.4 Average Background Concentration.

$$C_B = \frac{\sum_{i=1}^n C_{Bi} A_i}{A_N} \quad \text{Eq. 30B-4}$$

NOTE: If the concentration at each point is within 20 percent of the average concentration of all points, then use the arithmetic average.

## IN CAPTURED STREAM (DILUTION TECHNIQUE)

### 1. Introduction

- 1.1 **Applicability.** This procedure is applicable for determining the VOC content of captured gas streams. It is intended to be used in the development of a gas/gas protocol in which fugitive emissions are measured for determining VOC CE for surface coating and printing operations. A dilution system is used to reduce the VOC concentration of the captured emissions to about the same concentration as the fugitive emissions. The procedure may not be acceptable in certain site-specific situations [e.g., when: (1) direct-fired heaters or other circumstances affect the quantity of VOC at the control device inlet; and (2) particulate organic aerosols are formed in the process and are present in the captured emissions].
- 1.2 **Principle.** The amount of VOC captured ( $G$ ) is calculated as the sum of the products of the VOC content ( $C_{Gj}$ ), the flowrate ( $Q_{Gj}$ ), and the sampling time ( $\theta_C$ ) from each captured emissions point.
- 1.3 **Estimated Measurement Uncertainty.** The measurement uncertainties are estimated for each captured or fugitive emissions point as follows:  $Q_{Gj} = \pm 5.5$  percent and  $C_{Gj} = \pm 5$  percent. Based on these numbers, the probable uncertainty for  $G$  is estimated at about  $\pm 7.4$  percent.
- 1.4 **Sampling Requirements.** A CE test shall consist of at least three sampling runs. Each run shall cover at least one complete production cycle, but shall be at least 3 hours long. The sampling time for each run need not exceed 8 hours, even if the production cycle has not been completed. Alternative sampling times may be used with the approval of the Department.
- 1.5 **Notes.** Because this procedure is often applied in highly explosive areas, caution and care should be exercised in choosing, installing, and using the appropriate equipment. Mention of trade names or company products does not constitute endorsement. All gas concentrations (percent, ppm) are by volume, unless otherwise noted.

### 2. Apparatus and Reagents

- 2.1 **Gas VOC Concentration.** The main components are as follows:
  - 2.1.1 **Dilution System.** A Kipp in-stack dilution probe and controller or similar device may be used. The dilution rate may be changed by substituting different critical orifices or adjustments of the aspirator supply pressure. The dilution system shall be heated to prevent VOC condensation. Note: An out-of-stack dilution device may be used.

- 2.1.2 Calibration Valve Assembly. Three-way valve assembly at the outlet of the sample probe to direct the zero and calibration gases to the analyzer. Other methods, such as quick-connect lines, to route calibration gases to the outlet of the sample probe are acceptable.
- 2.1.3 Sample Line. Stainless steel or Teflon tubing to transport the sample gas to the analyzer. The sample line must be heated to prevent condensation.
- 2.1.4 Sample Pump. A leak-free pump, to pull the sample gas through the system at a flow rate sufficient to minimize the response time of the measurement system. The components of the pump that contact the gas stream shall be constructed of stainless steel or Teflon. The sample pump must be heated to prevent condensation.
- 2.1.5 Sample FlowRate Control. A sample flow rate control valve and rotameter, or equivalent, to maintain a constant sampling rate within 10 percent. The flow control valve and rotameter must be heated to prevent condensation. A control valve may also be located on the sample pump bypass loop to assist in controlling the sample pressure and flow rate.
- 2.1.6 Sample Gas Manifold. Capable of diverting a portion of the sample gas stream to the FIA, and the remainder to the bypass discharge vent. The manifold components shall be constructed of stainless steel or Teflon. If captured or fugitive emissions are to be measured at multiple locations, the measurement system shall be designed to use separate sampling probes, lines, and pumps for each measurement location and a common sample gas manifold and FIA. The sample gas manifold and connecting lines to the FIA must be heated to prevent condensation.
- 2.1.7 Organic Concentration Analyzer. An FIA with a span value of 1.5 times the expected concentration as propane; however, other span values may be used if it can be demonstrated to the Department's satisfaction that they would provide more accurate measurements. The system shall be capable of meeting or exceeding the following specifications:
  - 2.1.7.1 Zero Drift. Less than  $\pm 3.0$  percent of the span value.
  - 2.1.7.2 Calibration Drift. Less than  $\pm 3.0$  percent of the span value.
  - 2.1.7.3 Calibration Error. Less than  $\pm 5.0$  percent of the calibration gas value.
  - 2.1.7.4 Response Time. Less than 30 seconds.
- 2.1.8 Integrator/Data Acquisition System. An analog or digital device or computerized data acquisition system used to integrate the FIA response or compute the average response and record measurement data. The minimum data sampling frequency for computing average or integrated values is one measurement value every 5 seconds. The device shall

be capable of recording average values at least once per minute.

- 2.1.9 Calibration and Other Gases. Gases used for calibration, fuel, and combustion air (if required) are contained in compressed gas cylinders. All calibration gases shall be traceable to National Institute of Standards and Technology standards and shall be certified by the manufacturer to  $\pm 1$  percent of the tag value. Additionally, the manufacturer of the cylinder should provide a recommended shelf life for each calibration gas cylinder over which the concentration does not change more than  $\pm 2$  percent from the certified value. For calibration gas values not generally available, alternative methods for preparing calibration gas mixtures, such as dilution systems, may be used with the approval of the Department.
  - 2.1.9.1 Fuel. A 40 percent H<sub>2</sub>/60 percent He or 40 percent H<sub>2</sub>/60 percent N<sub>2</sub> gas mixture is recommended to avoid an oxygen synergism effect that reportedly occurs when oxygen concentration varies significantly from a mean value.
  - 2.1.9.2 Carrier Gas and Dilution Air Supply. High purity air with less than 1 ppm of organic material (as propane or carbon equivalent), or less than 0.1 percent of the span value, whichever is greater.
  - 2.1.9.3 FIA Linearity Calibration Gases. Low-, mid-, and high-range gas mixture standards with nominal propane concentrations of 20-30, 45-55, and 70-80 percent of the span value in air, respectively. Other calibration values and other span values may be used if it can be shown to the Department's satisfaction that more accurate measurements would be achieved.
  - 2.1.9.4 Dilution Check Gas. Gas mixture standard containing propane in air, approximately half the span value after dilution.
- 2.1.10 Particulate Filter. An in-stack or an out-of-stack glass fiber filter is recommended if exhaust gas particulate loading is significant. An out-of-stack filter must be heated to prevent any condensation unless it can be demonstrated that no condensation occurs.
- 2.2 Captured Emissions Volumetric Flow Rate.
  - 2.2.1 Method 2 or 2A Apparatus. For determining volumetric flow rate.
  - 2.2.2 Method 3 Apparatus and Reagents. For determining molecular weight of the gas stream. An estimate of the molecular weight of the gas stream may be used if approved by the Department.
  - 2.2.3 Method 4 Apparatus and Reagents. For determining moisture content, if necessary.

### 3. Determination of Volumetric Flow Rate of Captured Emissions

- 3.1 Locate all points where emissions are captured from the affected facility. Using Method 1, determine the sampling points. Be sure to check each site for cyclonic or swirling flow.
- 3.2 Measure the velocity at each sampling site at least once every hour during each sampling run using Method 2 or 2A.

### 4. Determination of VOC Content of Captured Emissions

- 4.1 **Analysis Duration.** Measure the VOC responses at each captured emissions point during the entire test run or, if applicable, while the process is operating. If there are multiple captured emissions locations, design a sampling system to allow a single FIA to be used to determine the VOC responses at all sampling locations.
- 4.2 **Gas VOC Concentration.**
  - 4.2.1 Assemble the sample train. Calibrate the FIA according to the procedure in Section 5.1.
  - 4.2.2 Set the dilution ratio and determine the dilution factor according to the procedure in Section 5.3.
  - 4.2.3 Conduct a system check according to the procedure in Section 5.4.
  - 4.2.4 Install the sample probe so that the probe is centrally located in the stack, pipe, or duct, and is sealed tightly at the stack port connection.
  - 4.2.5 Inject zero gas at the calibration valve assembly. Measure the system response time as the time required for the system to reach the effluent concentration after the calibration valve has been returned to the effluent sampling position.
  - 4.2.6 Conduct a system check before, and a system drift check after, each sampling run according to the procedures in Sections 5.2 and 5.4. If the drift check following a run indicates unacceptable performance (see Section 5.4), the run is not valid. The tester may elect to perform system drift checks during the run not to exceed one drift check per hour.
  - 4.2.7 Verify that the sample lines, filter, and pump temperatures are  $120 \pm 5^{\circ}\text{C}$ .
  - 4.2.8 Begin sampling at the start of the test period and continue to sample during the entire run. Record the starting and ending times and any required process information as appropriate. If multiple captured emission locations are sampled using a single FIA, sample at each location for the same amount of time (e.g., 2 min.) and continue to switch from one location to another for the entire test run. Be sure that total sampling time at each location is the

same at the end of the test run. Collect at least four separate measurements from each sample point during each hour of testing. Disregard the measurements at each sampling location until two times the response time of the measurement system has elapsed. Continue sampling for at least 1 minute and record the concentration measurements.

- 4.3 Background Concentration. NOTE: Not applicable when the building is used as the TTE.
  - 4.3.1 Locate all NDO's of the TTE. A sampling point shall be at the center of each NDO, unless otherwise approved by the Department. If there are more than six NDO's, choose six sampling points evenly spaced among the NDO's.
  - 4.3.2 Assemble the sample train. Calibrate the FIA and conduct a system check according to the procedures in Sections 5.1 and 5.4.
  - 4.3.3 Position the probe at the sampling location.
  - 4.3.4 Determine the response time, conduct the system check, and sample according to the procedures described in Sections 4.2.4 through 4.2.8.
- 4.4 Alternative Procedure. The direct interface sampling and analysis procedure described in Section 7.2 of Method 18 may be used to determine the gas VOC concentration. The system must be designed to collect and analyze at least one sample every 10 minutes.

## 5. Calibration and Quality Assurance

- 5.1 FIA Calibration and Linearity Check. Make necessary adjustments to the air and fuel supplies for the FIA and ignite the burner. Allow the FIA to warm up for the period recommended by the manufacturer. Inject a calibration gas into the measurement system after the dilution system and adjust the back-pressure regulator to the value required to achieve the flow rates specified by the manufacturer. Inject the zero- and the high-range calibration gases and adjust the analyzer calibration to provide the proper responses. Inject the low- and mid-range gases and record the responses of the measurement system. The calibration and linearity of the system are acceptable if the responses for all four gases are within 5 percent of the respective gas values. If the performance of the system is not acceptable, repair or adjust the system and repeat the linearity check. Conduct a calibration and linearity check after assembling the analysis system and after a major change is made to the system.
- 5.2 Systems Drift Checks. Select the calibration gas that most closely approximates the concentration of the diluted captured emissions for conducting the drift checks. Introduce the zero and calibration gases at the calibration valve assembly, and verify that the appropriate gas flow rate and pressure are present at the FIA. Record the measurement system responses to the zero and calibration gases. The performance of the system is

acceptable if the difference between the drift check measurement and the value obtained in Section 5.1 is less than 3 percent of the span value. Conduct the system drift check at the end of each run.

- 5.3 Determination of Dilution Factor. Inject the dilution check gas into the measurement system before the dilution system and record the response. Calculate the dilution factor using Equation 30C-3.
- 5.4 System Check. Inject the high-range calibration gas at the inlet to the sampling probe while the dilution air is turned off. Record the response. The performance of the system is acceptable if the measurement system response is within 5 percent of the value obtained in Section 5.1 for the high-range calibration gas. Conduct a system check before and after each test run.
- 5.5 Analysis Audit Procedure. Immediately before each test, analyze an audit cylinder as described in Section 5.2. The analysis audit must agree with the audit cylinder concentration within 10 percent.

## 6. Nomenclature

$A_i$  = area of NDO  $i$ ,  $\text{ft}^2$ .

$A_N$  = total area of all NDO's in the enclosure,  $\text{ft}^2$ .

$C_A$  = actual concentration of the dilution check gas, ppm propane.

$C_{Bi}$  = corrected average VOC concentration of background emissions at point  $i$ , ppm propane.

$C_B$  = average background concentration, ppm propane.

$C_{DH}$  = average measured concentration for the drift check calibration gas, ppm propane.

$C_{D0}$  = average system drift check concentration for zero concentration gas, ppm propane.

$C_H$  = actual concentration of the drift check calibration gas, ppm propane.

$C_i$  = uncorrected average background VOC concentration measured at point  $i$ , ppm propane.

$C_j$  = uncorrected average VOC concentration measured at point  $j$ , ppm propane.

$C_M$	=	measured concentration of the dilution check gas, ppm propane.
DF	=	dilution factor.
G	=	total VOC content of captured emissions, kg.
$K_1$	=	$1.830 \times 10^{-6}$ kg/(m <sup>3</sup> -ppm).
n	=	number of measurement points.
$Q_{Gj}$	=	average effluent volumetric flow rate corrected to standard conditions at captured emissions point j, m <sup>3</sup> /min.
$\theta_c$	=	total duration of CE sampling run, min.

## 7. Calculations

### 7.1 Total VOC Captured Emissions.

$$G = \sum_{j=1}^n (C_{Gj} - C_B) Q_{Gj} \theta_c K_1 \quad \text{Eq. 30C-1}$$

### 7.2 VOC Concentration of the Captured Emissions at Point j.

$$C_{Gj} = DF (C_j - C_{DO}) \frac{C_H}{C_{DH} - C_{DO}} \quad \text{Eq. 30C-2}$$

### 7.3 Dilution Factor.

$$DF = \frac{C_A}{C_H} \quad \text{Eq. 30C-3}$$

### 7.4 Background VOC Concentration at Point i.

$$C_{Bi} = (C_i - C_{DO}) \frac{C_H}{C_{DH} - C_{DO}} \quad \text{Eq. 30C-4}$$

## 7.5 Average Background Concentration.

$$C_B = \frac{\sum_{i=1}^n C_{B_i} A_i}{A_N}$$

Eq. 30C-5

NOTE: If the concentration at each point is within 20 percent of the average concentration of all points, then use the arithmetic average.

## METHOD 30D - VOLATILE ORGANIC COMPOUNDS EMISSIONS IN FUGITIVE STREAM FROM TEMPORARY TOTAL ENCLOSURE

### 1. Introduction

- 1.1 **Applicability.** This procedure is applicable for determining the fugitive VOC emissions from a TTE. It is intended to be used as a segment in the development of liquid/gas or gas/gas protocols for determining VOC CE for surface coating and printing operations.
- 1.2 **Principle.** The amount of fugitive VOC emissions (F) from the TTE is calculated as the sum of the products of the VOC content ( $C_{Fj}$ ), the flow rate ( $Q_{Fj}$ ), and the sampling time ( $\theta_F$ ) from each fugitive emissions point.
- 1.3 **Estimated Measurement Uncertainty.** The measurement uncertainties are estimated for each fugitive emission point as follows:  $Q_{Fj} = \pm 5.5$  percent and  $C_{Fj} = \pm 5.0$  percent. Based on these numbers, the probable uncertainty for F is estimated at about  $\pm 7.4$  percent.
- 1.4 **Sampling Requirements.** A CE test shall consist of at least three sampling runs. Each run shall cover at least one complete production cycle, but shall be at least 3 hours long. The sampling time for each run need not exceed 8 hours, even if the production cycle has not been completed. Alternative sampling times may be used with the approval of the Department.
- 1.5 **Notes.** Because this procedure is often applied in highly explosive areas, caution and care should be exercised in choosing, installing, and using the appropriate equipment. Mention of trade names or company products does not constitute endorsement. All gas concentrations (percent, ppm) are by volume, unless otherwise noted.

### 2. Apparatus and Reagents

- 2.1 Gas VOC Concentration. The main components are as follows:
  - 2.1.1 Sample Probe. Stainless steel or equivalent. The probe shall be heated to prevent VOC condensation.
  - 2.1.2 Calibration Valve Assembly. Three-way valve assembly at the outlet of the sample probe to direct the zero and calibration gases to the analyzer. Other methods, such as quick-connect lines, to route calibration gases to the outlet of the sample probe are acceptable.
  - 2.1.3 Sample Line. Stainless steel or Teflon tubing to transport the sample gas to the analyzer. The sample line must be heated to prevent condensation.
  - 2.1.4 Sample Pump. A leak-free pump, to pull the sample gas through the system at a flow rate sufficient to minimize the response time of the measurement system. The components of the pump that contact the gas stream shall be constructed of stainless steel or Teflon. The sample pump must be heated to prevent condensation.
  - 2.1.5 Sample Flow Rate Control. A sample flow rate control valve and rotameter, or equivalent, to maintain a constant sampling rate within 10 percent. The flow control valve and rotameter must be heated to prevent condensation. A control valve may also be located on the sample pump bypass loop to assist in controlling the sample pressure and flow rate.
  - 2.1.6 Sample Gas Manifold. Capable of diverting a portion of the sample gas stream to the FIA, and the remainder to the bypass discharge vent. The manifold components shall be constructed of stainless steel or Teflon. If emissions are to be measured at multiple locations, the measurement system shall be designed to use separate sampling probes, lines, and pumps for each measurement location and a common sample gas manifold and FIA. The sample gas manifold and connecting lines to the FIA must be heated to prevent condensation.
  - 2.1.7 Organic Concentration Analyzer. An FIA with a span value of 1.5 times the expected concentration as propane; however, other span values may be used if it can be demonstrated to the Department's satisfaction that they would provide more accurate measurements. The system shall be capable of meeting or exceeding the following specifications:
    - 2.1.7.1 Zero Drift. Less than  $\pm 3.0$  percent of the span value.
    - 2.1.7.2 Calibration Drift. Less than  $\pm 3.0$  percent of the span value.
    - 2.1.7.3 Calibration Error. Less than  $\pm 5.0$  percent of the calibration gas value.
    - 2.1.7.4 Response Time. Less than 30 seconds.

- 2.1.8 Integrator/Data Acquisition System. An analog or digital device or computerized data acquisition system used to integrate the FIA response or compute the average response and record measurement data. The minimum data sampling frequency for computing average or integrated values is one measurement value every 5 seconds. The device shall be capable of recording average values at least once per minute.
- 2.1.9 Calibration and Other Gases. Gases used for calibration, fuel, and combustion air (if required) are contained in compressed gas cylinders. All calibration gases shall be traceable to National Institute of Standards and Technology standards and shall be certified by the manufacturer to  $\pm 1$  percent of the tag value. Additionally, the manufacturer of the cylinder should provide a recommended shelf life for each calibration gas cylinder over which the concentration does not change more than  $\pm 2$  percent from the certified value. For calibration gas values not generally available, alternative methods for preparing calibration gas mixtures, such as dilution systems, may be used with the approval of the Department.
- 2.1.9.1 Fuel. A 40 percent  $H_2$ /60 percent He or 40 percent  $H_2$ /60 percent  $N_2$  gas mixture is recommended to avoid an oxygen synergism effect that reportedly occurs when oxygen concentration varies significantly from a mean value.
- 2.1.9.2 Carrier Gas. High purity air with less than 1 ppm of organic material (as propane or carbon equivalent) or less than 0.1 percent of the span value, whichever is greater.
- 2.1.9.3 FIA Linearity Calibration Gases. Low-, mid-, and high-range gas mixture standards with nominal propane concentrations of 20-30, 45-55, and 70-80 percent of the span value in air, respectively. Other calibration values and other span values may be used if it can be shown to the Department's satisfaction that more accurate measurements would be achieved.
- 2.1.10 Particulate Filter. An in-stack or an out-of-stack glass fiber filter is recommended if exhaust gas particulate loading is significant. An out-of-stack filter must be heated to prevent any condensation unless it can be demonstrated that no condensation occurs.
- 2.2 Fugitive Emissions Volumetric Flow Rate.
- 2.2.1 Method 2 or 2A Apparatus. For determining volumetric flow rate.
- 2.2.2 Method 3 Apparatus and Reagents. For determining molecular weight of the gas stream. An estimate of the molecular weight of the gas stream may be used if approved by the Department.
- 2.2.3 Method 4 Apparatus and Reagents. For determining moisture content, if necessary.

- 2.3 Temporary Total Enclosure. The criteria for designing an acceptable TTE are specified in Method 30.
3. Determination of Volumetric Flow Rate of Fugitive Emissions
    - 3.1 Locate all points where emissions are exhausted from the TTE. Using Method 1, determine the sampling points. Be sure to check each site for cyclonic or swirling flow.
    - 3.2 Measure the velocity at each sampling site at least once every hour during each sampling run using Method 2 or 2A.
4. Determination of VOC Content of Fugitive Emissions
    - 4.1 Analysis Duration. Measure the VOC responses at each fugitive emission point during the entire test run or, if applicable, while the process is operating. If there are multiple emission locations, design a sampling system to allow a single FIA to be used to determine the VOC responses at all sampling locations.
    - 4.2 Gas VOC Concentration.
      - 4.2.1 Assemble the sample train. Calibrate the FIA and conduct a system check according to the procedures in Sections 5.1 and 5.3, respectively.
      - 4.2.2 Install the sample probe so that the probe is centrally located in the stack, pipe, or duct, and is sealed tightly at the stack port connection.
      - 4.2.3 Inject zero gas at the calibration valve assembly. Allow the measurement system response to reach zero. Measure the system response time as the time required for the system to reach the effluent concentration after the calibration valve has been returned to the effluent sampling position.
      - 4.2.4 Conduct a system check before, and a system drift check after, each sampling run according to the procedures in Sections 5.2 and 5.3. If the drift check following a run indicates unacceptable performance (see Section 5.3), the run is not valid. The tester may elect to perform system drift checks during the run not to exceed one drift check per hour.
      - 4.2.5 Verify that the sample lines, filter, and pump temperatures are  $120 \pm 5^{\circ}\text{C}$ .
      - 4.2.6 Begin sampling at the start of the test period and continue to sample during the entire run. Record the starting and ending times and any required process information, as appropriate. If multiple emission locations are sampled using a single FIA, sample at each location for

the same amount of time (e.g., 2 min.) and continue to switch from one location to another for the entire test run. Be sure that total sampling time at each location is the same at the end of the test run. Collect at least four separate measurements from each sample point during each hour of testing. Disregard the response measurements at each sampling location until 2 times the response time of the measurement system has elapsed. Continue sampling for at least 1 minute and record the concentration measurements.

#### 4.3 Background Concentration.

4.3.1 Locate all NDO's of the TTE. A sampling point shall be at the center of each NDO, unless otherwise approved by the Department. If there are more than six NDO's, choose six sampling points evenly spaced among the NDO's.

4.3.2 Assemble the sample train. Calibrate the FIA and conduct a system check according to the procedures in Sections 5.1 and 5.3.

4.3.3 Position the probe at the sampling location.

4.3.4 Determine the response time, conduct the system check, and sample according to the procedures described in Sections 4.2.3 through 4.2.6.

4.4 Alternative Procedure. The direct interface sampling and analysis procedure described in Section 7.2 of Method 18 may be used to determine the gas VOC concentration. The system must be designed to collect and analyze at least one sample every 10 minutes.

### 5. Calibration and Quality Assurance

5.1 FIA Calibration and Linearity Check. Make necessary adjustments to the air and fuel supplies for the FIA and ignite the burner. Allow the FIA to warm up for the period recommended by the manufacturer. Inject a calibration gas into the measurement system and adjust the back-pressure regulator to the value required to achieve the flow rates specified by the manufacturer. Inject the zero- and the high-range calibration gases and adjust the analyzer calibration to provide the proper responses. Inject the low- and mid-range gases and record the responses of the measurement system. The calibration and linearity of the system are acceptable if the responses for all four gases are within 5 percent of the respective gas values. If the performance of the system is not acceptable, repair or adjust the system and repeat the linearity check. Conduct a calibration and linearity check after assembling the analysis system and after a major change is made to the system.

5.2 Systems Drift Checks. Select the calibration gas concentration that most closely approximates that of the fugitive gas emissions to conduct the drift checks. Introduce the zero and calibration gases at the calibration valve assembly and verify that the appropriate gas flow rate and pressure are present at the FIA. Record the measurement system

responses to the zero and calibration gases. The performance of the system is acceptable if the difference between the drift check measurement and the value obtained in Section 5.1 is less than 3 percent of the span value. Conduct a system drift check at the end of each run.

- 5.3 System Check. Inject the high-range calibration gas at the inlet of the sampling probe and record the response. The performance of the system is acceptable if the measurement system response is within 5 percent of the value obtained in Section 5.1 for the high-range calibration gas. Conduct a system check before each test run.
- 5.4 Analysis Audit. Immediately before each test, analyze an audit cylinder as described in Section 5.2. The analysis audit must agree with the audit cylinder concentration within 10 percent.

## 6. Nomenclature

$A_i$  = area of NDO  $i$ ,  $\text{ft}^2$ .

$A_N$  = total area of all NDO's in the enclosure,  $\text{ft}^2$ .

$C_{Bi}$  = corrected average VOC concentration of background emissions at point  $i$ , ppm propane.

$C_B$  = average background concentration, ppm propane.

- $C_{DH}$  = average measured concentration for the drift check calibration gas, ppm propane.
- $C_{D0}$  = average system drift check concentration for zero concentration gas, ppm propane.
- $C_{Fj}$  = corrected average VOC concentration of fugitive emissions at point j, ppm propane.
- $C_H$  = actual concentration of the drift check calibration gas, ppm propane.
- $C_i$  = uncorrected average background VOC concentration at point i, ppm propane.
- $C_j$  = uncorrected average VOC concentration measured at point j, ppm propane.
- $F$  = total VOC content of fugitive emissions, kg.
- $K_1$  =  $1.830 \times 10^{-6}$  kg/(m<sup>3</sup>-ppm).
- $n$  = number of measurement points.
- $Q_{Fj}$  = average effluent volumetric flow rate corrected to standard conditions at fugitive emissions point j, m<sup>3</sup>/min.
- $\theta_F$  = total duration of fugitive emissions sampling run, min.

## 7. Calculations

### 7.1 Total VOC Fugitive Emissions.

$$F = \sum_{j=1}^n (C_{Fj} - C_D) Q_{Fj} \theta_F K_1 \quad \text{Eq. 30D-1}$$

### 7.2 VOC Concentration of the Fugitive Emissions at Point j.

$$C_{Fj} = (C_j - C_{D0}) \frac{C_H}{C_{DH} - C_{D0}} \quad \text{Eq. 30D-2}$$

7.3 Background VOC Concentration at Point i.

$$C_{BI} = (C_i - C_{DO}) \frac{C_H}{C_{DE} - C_{DO}} \quad \text{Eq. 30D-3}$$

7.4 Average Background Concentration.

$$C_B = \frac{\sum_{i=1}^n C_{BI} A_i}{A_N} \quad \text{Eq. 30D-4}$$

NOTE: If the concentration at each point is within 20 percent of the average concentration of all points, use the arithmetic average.

## METHOD 30E - VOLATILE ORGANIC COMPOUNDS EMISSIONS IN FUGITIVE STREAM FROM BUILDING ENCLOSURE

### 1. Introduction

- 1.1 **Applicability.** This procedure is applicable for determining the fugitive VOC emissions from a BE. It is intended to be used in the development of liquid/gas or gas/gas protocols for determining VOC CE for surface coating and printing operations.
- 1.2 **Principle.** The total amount of fugitive VOC emissions ( $F_B$ ) from the BE is calculated as the sum of the products of the VOC content ( $C_{Fj}$ ) of each fugitive emissions point, the flow rate ( $Q_{Fj}$ ) at each fugitive emissions point, and time ( $\theta_F$ ).
- 1.3 **Measurement Uncertainty.** The measurement uncertainties are estimated for each fugitive emissions point as follows:  $Q_{Fj} = \pm 10.0$  percent and  $C_{Fj} = \pm 5.0$  percent. Based on these numbers, the probable uncertainty for  $F_B$  is estimated at about  $\pm 11.2$  percent.
- 1.4 **Sampling Requirements.** A CE test shall consist of at least three sampling runs. Each run shall cover at least one complete production cycle, but shall be at least 3 hours long. The sampling time for each run need not exceed 8 hours, even if the production cycle has not been completed. Alternative sampling times may be used with the approval of the Department.

1.5 Notes. Because this procedure is often applied in highly explosive areas, caution and care should be exercised in choosing, installing, and using the appropriate equipment. Mention of trade names or company products does not constitute endorsement. All gas concentrations (percent, ppm) are by volume, unless otherwise noted.

## 2. Apparatus and Reagents

2.1 Gas VOC Concentration. The main components are as follows:

2.1.1 Sample Probe. Stainless steel or equivalent. The probe shall be heated to prevent VOC condensation.

2.1.2 Calibration Valve Assembly. Three-way valve assembly at the outlet of the sample probe to direct the zero and calibration gases to the analyzer. Other methods, such as quick-connect lines, to route calibration gases to the outlet of the sample probe are acceptable.

2.1.3 Sample Line. Stainless steel or Teflon tubing to transport the sample gas to the analyzer. The sample line must be heated to prevent condensation.

2.1.4 Sample Pump. A leak-free pump, to pull the sample gas through the system at a flow rate sufficient to minimize the response time of the measurement system. The components of the pump that contact the gas stream shall be constructed of stainless steel or Teflon. The sample pump must be heated to prevent condensation.

2.1.5 Sample Flow Rate Control. A sample flow rate control valve and rotameter, or equivalent, to maintain a constant sampling rate within 10 percent. The flow rate control valve and rotameter must be heated to prevent condensation. A control valve may also be located on the sample pump bypass loop to assist in controlling the sample pressure and flow rate.

2.1.6 Sample Gas Manifold. Capable of diverting a portion of the sample gas stream to the FIA, and the remainder to the bypass discharge vent. The manifold components shall be constructed of stainless steel or Teflon. If emissions are to be measured at multiple locations, the measurement system shall be designed to use separate sampling probes, lines, and pumps for each measurement location, and a common sample gas manifold and FIA. The sample gas manifold must be heated to prevent condensation.

2.1.7 Organic Concentration Analyzer. An FIA with a span value of 1.5 times the expected concentration as propane; however, other span values may be used if it can be demonstrated to the Department's satisfaction that they would provide more accurate measurements. The system shall be capable of meeting or exceeding the following specifications:

2.1.7.1 Zero Drift. Less than  $\pm 3.0$  percent of the span value.

- 2.1.7.2 Calibration Drift. Less than  $\pm 3.0$  percent of the span value.
- 2.1.7.3 Calibration Error. Less than  $\pm 5.0$  percent of the calibration gas value.
- 2.1.7.4 Response Time. Less than 30 seconds.
- 2.1.8 Integrator/Data Acquisition System. An analog or digital device or computerized data acquisition system used to integrate the FIA response or compute the average response and record measurement data. The minimum data sampling frequency for computing average or integrated values is one measurement value every 5 seconds. The device shall be capable of recording average values at least once per minute.
- 2.1.9 Calibration and Other Gases. Gases used for calibration, fuel, and combustion air (if required) are contained in compressed gas cylinders. All calibration gases shall be traceable to National Institute of Standards and Technology standards and shall be certified by the manufacturer to  $\pm 1$  percent of the tag value. Additionally, the manufacturer of the cylinder should provide a recommended shelf life for each calibration gas cylinder over which the concentration does not change more than  $\pm 2$  percent from the certified value. For calibration gas values not generally available, alternative methods for preparing calibration gas mixtures, such as dilution systems, may be used with the approval of the Department.
- 2.1.9.1 Fuel. A 40 percent  $H_2$ /60 percent He or 40 percent  $H_2$ /60 percent  $N_2$  gas mixture is recommended to avoid an oxygen synergism effect that reportedly occurs when oxygen concentration varies significantly from a mean value.
- 2.1.9.2 Carrier Gas. High purity air with less than 1 ppm of organic material (propane or carbon equivalent) or less than 0.1 percent of the span value, whichever is greater.
- 2.1.9.3 FIA Linearity Calibration Gases. Low-, mid-, and high-range gas mixture standards with nominal propane concentrations of 20-30, 45-55, and 70-80 percent of the span value in air, respectively. Other calibration values and other span values may be used if it can be shown to the Department's satisfaction that more accurate measurements would be achieved.
- 2.1.10 Particulate Filter. An in-stack or an out-of-stack glass fiber filter is recommended if exhaust gas particulate loading is significant. An out-of-stack filter must be heated to prevent any condensation unless it can be demonstrated that no condensation occurs.
- 2.2 Fugitive Emissions Volumetric Flow Rate.
- 2.2.1 Flow Direction Indicators. Any means of indicating inward or outward flow, such as light plastic film or paper streamers, smoke tubes, filaments, and sensory perception.

- 2.2.2 Method 2 or 2A Apparatus. For determining volumetric flow rate. Anemometers or similar devices calibrated according to the manufacturer's instructions may be used when low velocities are present. Vane anemometers (Young-maximum response propeller), specialized pitots with electronic manometers (e.g., Shortridge Instruments Inc., Airdata Multimeter 860) are commercially available with measurement thresholds of 15 and 8 mpm (50 and 25 fpm), respectively.
  - 2.2.3 Method 3 Apparatus and Reagents. For determining molecular weight of the gas stream. An estimate of the molecular weight of the gas stream may be used if approved by the Department.
  - 2.2.4 Method 4 Apparatus and Reagents. For determining moisture content, if necessary.
  - 2.3 Total Temporary Enclosure. The criteria for an acceptable TTE are specified in Method 30.
3. Determination of Volumetric Flow Rate of Fugitive Emissions
- 3.1 Preliminary Determinations. To determine which exhaust points should be measured for volumetric flow rates and VOC concentrations, the following procedure shall be used:
    - 3.1.1 Forced Draft Openings. Identify all forced draft openings. Determine the volumetric flow rate according to Method 2.
    - 3.1.2 The NDO's Exhaust Points. The NDO's in the roof of a facility are considered to be exhaust points. Determine volumetric flow rate from these NDO's. Divide the cross-sectional area according to Method 1 using 12 equal areas. Use the appropriate velocity measurement devices (e.g., propeller anemometers).
    - 3.1.3 Other NDO's.
      - 3.1.3.1 This step is optional. Determine the exhaust flow rate, including that of the control device, from the enclosure and the intake air flow rate. If the exhaust flow rate divided by the intake air flow rate is greater than 1.1, then all other NDO's are not considered to be significant exhaust points. It is not necessary to measure the volumetric flow rate and VOC concentration from these insignificant exhaust points during the CE test.
      - 3.1.3.2 If the option above is not taken, identify all other NDO's and other potential points through which fugitive emissions may escape the enclosure; then use the following criteria to determine whether flow rates and VOC concentrations need to be measured.
        - 3.1.3.2.1 Using the appropriate flow direction indicator, determine the flow

direction. An NDO with zero or inward flow is not an exhaust point.

- 3.1.3.2.2 Measure the outward volumetric flow rate from the remainder of the NDO's. If the collective flow rate is 2 percent or less of the flow rate from Sections 3.1.1 and 3.1.2, then these NDO's, except those within two equivalent diameters (based on NDO opening) from VOC sources, may be considered to be non-exhaust points.
- 3.1.3.2.3 If the percentage calculated in Section 3.1.3.2.2 is greater than 2 percent, those NDO's (except those within two equivalent diameters from VOC sources) whose volumetric flowrates total 2 percent of the flow rate from Sections 3.1.1 and 3.1.2 may be considered as non-exhaust points. All remaining NDO's shall be measured for volumetric flow rate and VOC concentrations during the CE test.
- 3.1.3.2.4 The tester may choose to measure VOC concentrations at the forced exhaust points and the NDO's. If the total VOC emissions from the non-roof NDO's are less than 2 percent of the emissions from the forced draft and roof NDO's, then it is not necessary to measure the VOC concentration at the non-roof NDO's during the CE test.

## 3.2 Determination of Flow Rates.

- 3.2.1 Measure the volumetric flow rate at all locations identified as exhaust points in Section 3.1. Divide each exhaust opening into nine equal areas for rectangular openings and into eight equal areas for circular openings.
- 3.2.2 Measure the velocity at each site at least once every hour during each sampling run using Method 2 or 2A, if applicable, or using the low velocity instruments in Section 2.2.2.

## 4. Determination of VOC Content of Fugitive Emissions

- 4.1 Analysis Duration. Measure the VOC responses at each fugitive emissions point during the entire test run or, if applicable, while the process is operating. If there are multiple emissions locations, design a sampling system to allow a single FIA to be used to determine the VOC responses at all sampling locations.
- 4.2 Gas VOC Concentration.
  - 4.2.1 Assemble the sample train. Calibrate the FIA and conduct a system check according to the procedures in Sections 5.1 and 5.3, respectively.
  - 4.2.2 Install the sample probe so that the probe is centrally located in the stack, pipe, or duct, and is sealed tightly at the stack port connection.

- 4.2.3 Inject zero gas at the calibration valve assembly. Allow the measurement system response to reach zero. Measure the system response time as the time required for the system to reach the effluent concentration after the calibration valve has been returned to the effluent sampling position.
- 4.2.4 Conduct a system check before, and a system drift check after, each sampling run according to the procedures in Sections 5.2 and 5.3. If the drift check following a run indicates unacceptable performance (see Section 5.3), the run is not valid. The tester may elect to perform drift checks during the run, not to exceed one drift check per hour.
- 4.2.5 Verify that the sample lines, filter, and pump temperatures are  $120 \pm 5^{\circ}\text{C}$ .
- 4.2.6 Begin sampling at the start of the test period and continue to sample during the entire run. Record the starting and ending times, and any required process information, as appropriate. If multiple emission locations are sampled using a single FIA, sample at each location for the same amount of time (e.g., 2 minutes) and continue to switch from one location to another for the entire test run. Be sure that total sampling time at each location is the same at the end of the test run. Collect at least four separate measurements from each sample point during each hour of testing. Disregard the response measurements at each sampling location until 2 times the response time of the measurement system has elapsed. Continue sampling for at least 1 minute, and record the concentration measurements.
- 4.3 Alternative Procedure. The direct interface sampling and analysis procedure described in Section 7.2 of Method 18 may be used to determine the gas VOC concentration. The system must be designed to collect and analyze at least one sample every 10 minutes.

## 5. Calibration and Quality Assurance

- 5.1 FIA Calibration and Linearity Check. Make necessary adjustments to the air and fuel supplies for the FIA and ignite the burner. Allow the FIA to warm up for the period recommended by the manufacturer. Inject a calibration gas into the measurement system and adjust the back-pressure regulator to the value required to achieve the flow rates specified by the manufacturer. Inject the zero- and the high-range calibration gases, and adjust the analyzer calibration to provide the proper responses. Inject the low- and mid-range gases and record the responses of the measurement system. The calibration and linearity of the system are acceptable if the responses for all four gases are within 5 percent of the respective gas values. If the performance of the system is not acceptable, repair or adjust the system and repeat the linearity check. Conduct a calibration and linearity check after assembling the analysis system and after a major change is made to the system.
- 5.2 Systems Drift Checks. Select the calibration gas that most closely approximates the concentration of the captured emissions for conducting the drift checks. Introduce the zero

and calibration gases at the calibration valve assembly and verify that the appropriate gas flow rate and pressure are present at the FIA. Record the measurement system responses to the zero and calibration gases. The performance of the system is acceptable if the difference between the drift check measurement and the value obtained in Section 5.1 is less than 3 percent of the span value. Conduct a system drift check at the end of each run.

5.3 System Check. Inject the high-range calibration gas at the inlet of the sampling probe and record the response. The performance of the system is acceptable if the measurement system response is within 5 percent of the value obtained in Section 5.1 for the high-range calibration gas. Conduct a system check before each test run.

5.4 Analysis Audit. Immediately before each test, analyze an audit cylinder as described in Section 5.2. The analysis audit must agree with the audit cylinder concentration within 10 percent.

## 6. Nomenclature

$C_{DH}$  = average measured concentration for the drift check calibration gas, ppm propane.

$C_{D0}$  = average system drift check concentration for zero concentration gas, ppm propane.

$C_{Fj}$  = corrected average VOC concentration of fugitive emissions at point j, ppm propane.

$C_H$  = actual concentration of the drift check calibration gas, ppm propane.

$C_j$  = uncorrected average VOC concentration measured at point j, ppm propane.

$F_B$  = total VOC content of fugitive emissions from the building, kg.

$K_1$  =  $1.830 \times 10^{-6}$  kg/(m<sup>3</sup>-ppm).

$n$  = number of measurement points.

$Q_{Fj}$  = average effluent volumetric flow rate corrected to standard conditions at fugitive emissions point j, m<sup>3</sup>/min.

$\theta_F$  = total duration of CE sampling run, min.

## 7. Calculations

7.1 Total VOC Fugitive Emissions from the Building.

$$F_B = \sum_{j=1}^n C_{Fj} Q_{Fj} \theta_F K_1$$

Eq. 30E-1

7.2 VOC Concentration of the Fugitive Emissions at Point j.

$$C_{Fj} = (C_j - C_{DO}) \frac{C_H}{C_{DH} - C_{DO}}$$

Eq. 30E-2