ASTM INTERNATIONAL

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Standard Test Method for
Sampling of Process Vents with a Portable Gas Chromatograph[1]

This standard is issued under the fixed designation D6060; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes a method for direct sampling and analysis of process vents for volatile organic compound (VOC) vapors and permanent gases using a portable gas chromatograph (GC).

1.2 This test method is applicable to analysis of permanent gases such as oxygen (O_2), carbon dioxide (CO_2) and nitrogen (N_2), as well as vapors from organic compounds with boiling points up to 125°C.

1.3 The detection limits obtained will depend on the portable gas chromatograph and detector used. Detectors available include but are not limited to thermal conductivity, photoionization, argon ionization, and electron capture. For instruments equipped with thermal conductivity detectors, typical detection limits are one to two parts per million by volume (ppm(v)) with an applicable concentration range to high percent by volume levels. For instruments with photoionization detectors detection limit of one to ten parts per billion by volume (ppb(v)) are obtainable with a concentration range from 1000 to 2000 ppm(v). The argon ionization detector has an achievable detection limit of one (ppb(v)), while the electron capture detector has an achievable detection limit of one part per trillion by volume (ppt(v)) for chlorinated compounds.

1.4 The applicability of this test method should be evaluated for each VOC by determining stability, reproducibility, and linearity.

1.5 The appropriate concentration range must also be determined for each VOC, as the range will depend on the vapor pressure of the particular VOC.

1.6 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Refer to Section 8 on Hazards for additional safety precautions.

1.8 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]
D1356 Terminology Relating to Sampling and Analysis of Atmospheres
D3154 Test Method for Average Velocity in a Duct (Pitot Tube Method)
D3464 Test Method for Average Velocity in a Duct Using a Thermal Anemometer
E355 Practice for Gas Chromatography Terms and Relationships

2.2 *Other Document:*
NFPA 70 National Electrical Code[3]
NFPA 496 Standard for Purged and Pressurized Enclosures for Electrical Equipment[3]

3. Terminology

3.1 *Definitions*—For the definition of terms used in this test method, refer to Terminology D1356 and Practice E355.

3.2 *Definitions of Terms Specific to This Standard:*
3.2.1 *portable, n*—refers to gas chromatograph with internal battery, internal sample pump, and internal/rechargeable carrier gas supply cylinder.

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4. Summary of Test Method

4.1 One end of a sampling line (typically 6 mm outside diameter TFE-fluorocarbon tubing) is connected to a tee in a process vent and the other end to a condensation trap (see 6.1), which is connected to a gas sampling bulb. The outlet of the gas sampling bulb is connected to a sampling pump set at a flow rate of 0.5 to 2 L/min. The sample line from the portable gas chromatograph is inserted through the septum port of the gas sampling bulb. At user selected intervals, the internal pump of the portable gas chromatograph is activated and process vapors drawn through the injection valve of the gas chromatograph and analyzed.

5. Significance and Use

5.1 This test method has been widely used to obtain mass balance data for process scrubbers, to determine the efficiency of VOC emission control equipment, and to obtain data to support air permit applications.

5.2 This test method will have applications to the Maximum Achievable Control Technology (MACT) Rule and may have applications to Compliance Assurance Monitoring verification required by the 1990 Clean Air Act Title III Amendments.

5.3 This test method, when used with Test Methods D3464 or D3154 or on-line process flow meter data, can be used to calculate detailed emission rate profiles for VOCs from process vents.

5.4 This test method provides nearly real time results that can detect process changes or upsets that may be missed using conventional sorbent tube or integrated gas sampling bag sampling.

6. Interferences

6.1 Water or liquid in the process line will plug the sample line of the gas chromatograph, since the injection valve of most portable GCs is not heated. The condensation trap is designed to protect the portable gas chromatograph if liquids are present or occur during process upset.

6.2 Interferences sometimes result from analytes having similar retention times during gas chromatography.

6.3 General approaches which can be followed to resolve such interferences are given below:

6.3.1 Change the type of column, length of column, or operating conditions.

6.3.2 Analyze using a nonpolar methyl silicone column which separates according to boiling point of the compounds and a polar column whose separations are influenced by the polarity of the compounds.[4]

6.3.3 Use a mass spectrometer to verify the identity of peaks.

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[4] The columns in most portable gas chromatographs are easily interchanged. One manufacturer has an instrument that simultaneously injects onto two user selected column modules.
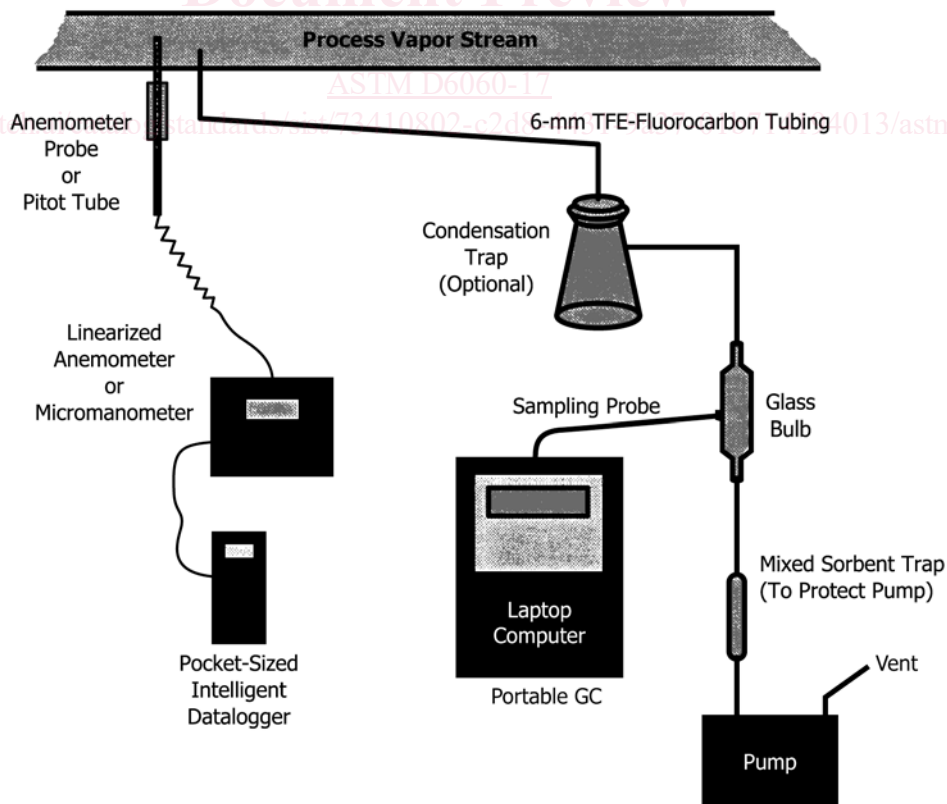

FIG. 1   Schematic of Process Sampling Equipment

7. Apparatus

7.1 A schematic drawing of a typical sampling setup is shown in Fig. 1. The laptop computer may be physically located near the gas chromatograph as shown in Fig. 1, or located remotely. In addition, some portable gas chromatographs have an integral computer. Use a short piece of 1.5 mm outside diameter by 1 mm inside diameter stainless steel tubing as the sampling probe line from the gas sampling bulb to the GC inlet.

7.2 *Portable Gas Chromatograph (GC),* with a thermal conductivity, photoionization, argon ionization, electron capture or other appropriate detector, internal/rechargeable carrier gas supply, and internal sampling pump.

7.2.1 Portable gas chromatographs are typically equipped with particulate filters which should be replaced periodically.

7.3 *Data Logger,* device used for automated storage of output from a flow measurement device.

7.4 *Gas Sampling Bulb,* 125 mL capacity with septum port.

7.5 *Personal Sampling Pump.*

7.6 *Gas-Tight Syringe,* 1, 10, 100, 500 mL capacity or other convenient sizes for preparing standards.

7.7 *Microlitre Syringes,* 10, 25, 50, 100 µL or other convenient sizes for preparing standards.

7.8 *Gas Sampling Bags,* for preparation of gas standards. Bags constructed of various polymer films, such as polyvinylidene fluoride, fluorinated ethylenepropylene, (tetrafluoroethylene)-fluorocarbon, polyvinylidene chloride, polyethylene and mixed polymer multilayers, with a variety of fittings and capacities (typically 1 to 200 L) are available.

7.9 *Thermal Anemometer, Vane Anemometer, Mass Flowmeter or Pitot Tube,* for measurement of vent velocity.

7.10 *Condensation Trap, Filtering Flask,* 250 or 500 mL polypropylene fitted with a stopper.

7.11 *TFE-Fluorocarbon Tubing,* 6 mm outside diameter by 5 mm inside diameter.

7.12 *Data System,* an integral or external computer used for control of operation of a portable gas chromatograph, data reduction, and storage of results.

8. Hazards

8.1 See NFPA 496 for use of electrical equipment in areas classified as hazardous by Article 500 of NFPA 70. A purged and pressurized enclosure is required.

9. Calibration

9.1 Suitable analytes at known concentrations may be prepared by the filling of a gas sampling bag with a known volume of air. Inject a known volume of gas or liquid containing the analyte(s) of interest into the bag and knead the bag to mix. Permeation tubes or rigid chambers may also be used for preparation of gas standards. Reference standards in compressed gas cylinders certified as to concentration and analytic uncertainty by the manufacturer are also available. Refer to *Methods of Air Sampling and Analysis*[5] for applicable guidelines for all of these gas standard preparation techniques.

9.2 Although standards of some compounds prepared in gas bags are very stable, others show sample loss during storage due to permeation, reactivity, or surface adsorption. As a general guideline prepare standards fresh daily.

9.3 Prepare at least two reference standards containing varying concentrations of each component. Bracket the expected concentrations of each component in the testing of the process vent, if known.

9.3.1 Connect the gas sampling bag to the inlet or the calibration port of the GC and initiate the analysis. Perform at least triplicate injections of each standard.

9.3.2 The quantitative response (calibration) of a GC detector may be determined by the measurement of the peak height or peak area using the Data System or electronic integrator.[6]

9.3.3 Following the calibration, analyze a gas sampling bag containing air only (blank). If carryover is >1 % increase the sampling period (internal GC pump time). Typical sampling periods are 20 to 45 s, however, this parameter must be optimized for each VOC analyzed.

10. Procedure

10.1 *Preparation of the Gas Chromatograph:*

10.1.1 Fill the internal carrier gas reservoir as described by the manufacturer.

10.1.2 Select a carrier gas flow or column pressure and column temperature compatible with the column selected for the separation.

10.1.3 Calibrate the chromatographic system to determine the relative retention times and response of the various compounds of interest.

10.2 *Preparation of the Sampling Train:*

10.2.1 Assemble the sampling train as shown in Fig. 1. Stainless steel or glass may be substituted for the TFE-fluorocarbon transfer line.[7]

10.2.2 For process vents containing high concentrations of higher boiling (>125°C) low vapor pressure (<2 kPa) VOCS a heated transfer line may be necessary. A portable GC with a heated injector is also required.

10.2.3 Recheck the calibration after assembly of the sampling train by connecting an appropriate standard to the inlet of the sample line. If results obtained for the analysis of the standard are not within 15 % relative standard deviation of the expected concentration, check the sampling train for leaks. Flow rate through sampling train is typically 0.5 to 2 L/min.

10.3 *Analysis:*

10.3.1 Set the gas chromatograph at a desired automated sampling interval and sampling period. At the desired time interval, activate the internal vacuum pump of the GC and pull the sample vapors from the sampling bulb. Inject and analyze.

[5] Lodge, James P., ed., *Methods of Air Sampling and Analysis*, Intersociety Committee, Lewis Publishers, Inc., 3rd ed., 1988, pp. 15–26.
[6] Most portable GCS are equipped with data systems that automatically generate a calibration curve.
[7] Heated steel lines will degrade or destroy chlorinated organic compounds.