Designation: D6348 – 12

StandardTest Method for
Determination of Gaseous Compounds by Extractive Direct Interface Fourier Transform Infrared (FTIR) Spectroscopy[1]

This standard is issued under the fixed designation D6348; 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.

INTRODUCTION

This extractive FTIR based field test method is used to quantify gas phase concentrations of multiple target analytes from stationary source effluent. Because an FTIR analyzer is potentially capable of analyzing hundreds of compounds, this test method is not analyte or source specific. The analytes, detection levels, and data quality objectives are expected to change for any particular testing situation. It is the responsibility of the tester to define the target analytes, the associated detection limits for those analytes in the particular source effluent, and the required data quality objectives for each specific test program. Provisions are included in this test method that require the tester to determine critical sampling system and instrument operational parameters, and for the conduct of QA/QC procedures. Testers following this test method will generate data that will allow an independent observer to verify the valid collection, identification, and quantification of the subject target analytes.

1. Scope

1.1 This field test method employs an extractive sampling system to direct stationary source effluent to an FTIR spectrometer for the identification and quantification of gaseous compounds. Concentration results are provided. This test method is potentially applicable for the determination of compounds that (*1*) have sufficient vapor pressure to be transported to the FTIR spectrometer and (*2*) absorb a sufficient amount of infrared radiation to be detected.

1.2 This field test method provides near real time analysis of extracted gas samples from stationary sources. Gas streams with high moisture content may require conditioning to minimize the excessive spectral absorption features imposed by water vapor.

1.3 This field test method requires the preparation of a source specific field test plan. The test plan must include the following: (*1*) the identification of the specific target analytes (*2*) the known analytical interferents specific to the test facility source effluent (*3*) the test data quality necessary to meet the specific test requirements and (*4*) the results obtained from the laboratory testing (see Annex A1 for test plan requirements).

1.4 The FTIR instrument range should be sufficient to measure from high ppm(v) to ppb(v) and may be extended to higher or lower concentrations using any or all of the following procedures:

1.4.1 The gas absorption cell path length may be either increased or decreased,

1.4.2 The sample conditioning system may be modified to reduce the water vapor, CO_2, and other interfering compounds to levels that allow for quantification of the target compound(s), and

1.4.3 The analytical algorithm may be modified such that interfering absorbance bands are minimized or stronger/weaker absorbance bands are employed for the target analytes.

1.5 The practical minimum detectable concentration is instrument, compound, and interference specific (see Annex A2 for procedures to estimate the achievable minimum detectable concentrations (MDCs)). The actual sensitivity of the FTIR measurement system for the individual target analytes depends upon the following:

1.5.1 The specific infrared absorptivity (signal) and wavelength analysis region for each target analyte,

1.5.2 The amount of instrument noise (see Annex A6), and

1.5.3 The concentration of interfering compounds in the sample gas (in particular, percent moisture and CO_2), and the amount of spectral overlap imparted by these compounds in the wavelength region(s) used for the quantification of the target analytes.

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[1] This test method is under the jurisdiction of Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.03 on Ambient Atmospheres and Source Emissions.

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1.5.4 Any sampling system interferences such as adsorption or outgassing.

1.6 Practices E168 and E1252 are suggested for additional reading.

1.7 *This standard does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.* Additional safety precautions are described in Section 9.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D3195 Practice for Rotameter Calibration

E168 Practices for General Techniques of Infrared Quantitative Analysis

E1252 Practice for General Techniques for Obtaining Infrared Spectra for Qualitative Analysis

2.2 *EPA Methods (40 CFR Part 60 Appendix A)*[3]

Method 1 Sample and Velocity Traverses for Stationary Sources

Method 2 Series Determination of Stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube)

Method 3 Series Gas Analysis for Carbon Dioxide, Oxygen, Excess Air, and Dry Molecular Weight

Method 4 Series Determination of Moisture Content in Stack Gases

3. Terminology

3.1 See Terminology D1356 for definition of terms related to sampling and analysis of atmospheres.

3.2 *This section contains the terms and definitions used in this test method and those that are relevant to extractive FTIR based sampling and analysis of stationary source effluent. When possible, definitions of terms have been drawn from authoritative texts or manuscripts in the fields of air pollution monitoring, spectroscopy, optics, and analytical chemistry.*

3.2.1 *absorbance, n*—the negative logarithm of the transmission, $A = -\log (I/I_0)$, where I is the transmitted intensity of the light and I_0 is the incident intensity.

3.2.2 *absorptivity, adj*—the amount of infrared radiation that is absorbed by each molecule.

3.2.3 *analyte spiking, n*—the process of quantitatively co-adding calibration standards with source effluent to determine the effectiveness of the FTIR measurement system to quantify the target analytes.

3.2.4 *analytical algorithm, n*—the method used to quantify the concentration of the target analytes and interferences in each FTIR Spectrum. The analytical algorithm should account for the analytical interferences by conducting the analysis in a portion of the infrared spectrum that is the most unique for that particular compound.

3.2.5 *analytical interference, n*—the physical effects of superimposing two or more light waves. Analytical interferences occur when two or more compounds have overlapping absorbance bands in their infrared spectra.

3.2.6 *apodization, v*—a mathematical transformation carried out on data received from an interferometer to reduce the side lobes of the measured peaks. This procedure alters the instrument's response function. There are various types of transformation; the most common forms are boxcar, triangular, Happ-Genzel, and Beer-Norton functions.

3.2.7 *background spectrum, n*—the spectrum taken in the absence of absorbing species or sample gas, typically conducted using dry nitrogen or zero air in the gas cell.

3.2.8 *bandwidth, adj*—the width of a spectral feature as recorded by a spectroscopic instrument. This width is listed as the full width at the half maximum of the feature or as the half width at the half maximum of the spectral feature. This is also referred to as the line width (**1**).[4]

3.2.9 *beam splitter, n*—a device located in the interferometer that splits the incoming infrared radiation into two separate beams that travel two separate paths before recombination.

3.2.10 *Beer's law, n*—the principal by which FTIR spectra are quantified. Beer's law states that the intensity of a monochromatic plane wave incident on an absorbing medium of constant thickness diminishes exponentially with the number of absorbers in the beam. Strictly speaking, Beer's law holds only if the following conditions are met: (*1*) perfectly monochromatic radiation (*2*) no scattering (*3*) a beam that is strictly collimated (*4*) negligible pressure-broadening effects (**2, 3**). For an excellent discussion of the derivation of Beer's law, see (**4**).

3.2.11 *calibration transfer standard, n*—a certified calibration standard that is used to verify the instrument stability on a daily basis when conducting sampling.

3.2.12 *classical least squares, n*—a common method of analyzing multicomponent infrared spectra by scaled absorbance subtraction.

3.2.13 *condenser system,(dryer), n*—a moisture removal system that condenses water vapor from the source effluent to provide a dry sample to the FTIR gas cell. Part of the sample conditioning system.

3.2.14 *cooler, n*—a device into which a quantum detector is placed for maintaining it at a low temperature in an IR system. At a low temperature, the detector provides the high sensitivity that is required for the IR system. The two primary types of coolers are a liquid nitrogen Dewar and a closed-cycle Stirling cycle refrigerator.

3.2.15 *electromagnetic spectrum, n*—the total set of all possible frequencies of electromagnetic radiation. Different

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from Superintendent of Documents, U. G. Government Printing Office, Washington, DC 20402.

[4] The boldface numbers in parentheses refer to the list of references at the end of the standard.