Designation: E 1865 – 97 (Reapproved 2007)

Standard Guide for Open-Path Fourier Transform Infrared (OP/FT-IR) Monitoring of Gases and Vapors in Air[1]

This standard is issued under the fixed designation E 1865; 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 guide covers active open-path Fourier transform infrared (OP/FT-IR) monitors and provides guidelines for using active OP/FT-IR monitors to obtain concentrations of gases and vapors in air.

1.2 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

E 131 Terminology Relating to Molecular Spectroscopy

E 168 Practices for General Techniques of Infrared Quantitative Analysis

E 1421 Practice for Describing and Measuring Performance of Fourier Transform Mid-Infrared (FT-MIR) Spectrometers: Level Zero and Level One Tests

E 1655 Practices for Infrared Multivariate Quantitative Analysis

3. Terminology

3.1 For definitions of terms relating to general molecular spectroscopy used in this guide refer to Terminology E 131. A complete glossary of terms relating to optical remote sensing is given in Ref (**1**).[3]

3.2 *Definitions:*

3.2.1 *background spectrum*, *n*—a single-beam spectrum that does not contain the spectral features of the analyte(s) of interest.

3.2.2 *bistatic system*, *n*—a system in which the IR source is some distance from the detector. For OP/FT-IR monitoring, this implies that the IR source and the detector are at opposite ends of the monitoring path.

3.2.3 *monitoring path*, *n*—the location in space over which concentrations of gases and vapors are measured and averaged.

3.2.4 *monitoring pathlength*, *n*—the distance the optical beam traverses through the monitoring path.

3.2.5 *monostatic or unistatic system*, *n*—a system with the IR source and the detector at the same end of the monitoring path. For OP/FT-IR systems, the beam is generally returned by a retroreflector.

3.2.6 *open-path monitoring*, *n*—monitoring over a path that is completely open to the atmosphere.

3.2.7 *parts per million meters*, *n*—the units associated with the quantity path-integrated concentration and a possible unit of choice for reporting data from OP/FT-IR monitors because it is independent of the monitoring pathlength.

3.2.8 *path-averaged concentration*, *n*—the result of dividing the path-integrated concentration by the pathlength.

3.2.8.1 *Discussion*—Path-averaged concentration gives the average value of the concentration along the path, and typically is expressed in units of parts per million (ppm), parts per billion (ppb), or micrograms per cubic meter (μgm^{-3}).

3.2.9 *path-integrated concentration*, *n*—the quantity measured by an OP/FT-IR monitor over the monitoring path. It has units of concentration times length, for example, ppm·m.

3.2.10 *plume*, *n*—the gaseous and aerosol effluents emitted from a stack or other pollutant source and the volume of space they occupy.

3.2.11 *retroreflector*, *n*—an optical device that returns radiation in directions close to the direction from which it came.

3.2.11.1 *Discussion*—Retroreflectors come in a variety of forms. The retroreflector commonly used in OP/FT-IR monitoring uses reflection from three mutually perpendicular surfaces. This kind of retroreflector is usually called a cube-corner retroreflector.

3.2.12 *single-beam spectrum*, *n*—the radiant power measured by the instrument detector as a function of frequency.

3.2.12.1 *Discussion*—In FT-IR absorption spectrometry the single-beam spectrum is obtained after a fast Fourier transform of the interferogram.

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[1] This guide is under the jurisdiction of ASTM Committee E13 on Molecular Spectroscopy and Separation Science and is the direct responsibility of Subcommittee E13.03 on Infrared and Near Infrared Spectroscopy.

Current edition approved Dec. 1, 2007. Published December 2007. Originally approved in 1997. Last previous edition approved in 2002 as E 1865 - 97 (2002).

[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] The boldface numbers in parentheses refer to a list of references at the end of this guide.

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3.2.13 *synthetic background spectrum*, *n*—a background spectrum made by choosing points along the envelope of a single-beam spectrum and fitting a series of short, straight lines or a polynomial function to the chosen data points to simulate the instrument response in the absence of absorbing gases or vapors.

4. Significance and Use

4.1 This guide is intended for users of OP/FT-IR monitors. Applications of OP/FT-IR systems include monitoring for hazardous air pollutants in ambient air, along the perimeter of an industrial facility, at hazardous waste sites and landfills, in response to accidental chemical spills or releases, and in workplace environments.

5. Principles of OP/FT-IR Monitoring

5.1 Long-path IR spectrometry has been used since the mid-1950s to characterize hazardous air pollutants (**2**). For the most part, this earlier work involved the use of multiple-pass, long-path IR cells to collect and analyze air samples. In the late 1970s a mobile FT-IR system capable of detecting pollutants along an open path was developed (**3**). The 1990 amendments to the Clean Air Act, which may require that as many as 189 compounds be monitored in the atmosphere, have led to a renewed interest in OP/FT-IR monitoring (**4**). The OP/FT-IR monitor is a spectrometric instrument that uses the mid-IR spectral region to identify and quantify atmospheric gases. These instruments can be either transportable or permanently installed. An open-path monitor contains many of the same components as those in a laboratory FT-IR system, for example the same types of interferometers and detectors are used, except that the sample volume consists of the open atmosphere. In contrast to more conventional point monitors, the OP/FT-IR monitor provides path-integrated concentration data. Unlike many other air monitoring methods, such as those that use canisters or sorbent cartridges, the OP/FT-IR monitor measures pollutants in situ. Therefore, no samples need be collected, extracted, or returned to the laboratory for analysis. Detection limits in OP/FT-IR depend on several factors, such as the monitoring pathlength, the absorptivity of the analyte, and the presence of interfering species. For most analytes of interest, detection limits typically range between path-integrated concentrations of 1.5 and 50 ppm·m.

NOTE 1—The OP/FT-IR monitor can be configured to operate in two modes: active or passive. In the active mode, a collimated beam of radiation from an IR source that is a component of the system is transmitted along the open-air path. In the passive mode, radiation emitted from objects in the field of view of the instrument is used as the source of IR energy. Passive FT-IR monitors have been used for environmental applications, such as characterizing the plumes of smoke stacks. More recently these systems have been developed to detect chemical warfare agents in military applications. However, to date, the active mode has been used for most environmental applications of OP/FT-IR monitoring. In addition to open-air measurements, extractive measurements can be made by interfacing a closed cell to an FT-IR system. This type of system can be used as a point monitor or to measure the effluent in stacks or pipelines.

6. Description of OP/FT-IR Systems

6.1 There are two primary geometrical configurations available for transmitting the IR beam along the path in active OP/FT-IR systems. One configuration is referred to as bistatic, while the other is referred to as monostatic, or unistatic.

6.1.1 *Bistatic Configuration*—In this configuration, the detector and the IR source are at opposite ends of the monitoring path. In this case, the optical pathlength is equal to the monitoring pathlength. Two configurations can be used for bistatic systems. One configuration places the IR source, interferometer, and transmitting optics at one end of the path and the receiving optics and detector at the other end (Fig. 1(A)). Typically a Cassegrain or Newtonian telescope is used to transmit and collect the IR beam. The advantage of the configuration depicted in Fig. 1(A) is that the IR beam is modulated along the path, which enables the unmodulated ambient radiation to be rejected by the system's electronics. The maximum distance that the interferometer and the detector can be separated in this configuration is limited because communication between these two components is required for timing purposes. For example, a bistatic system with this configuration developed for monitoring workplace environments had a maximum monitoring pathlength of 40 m (**5**). The other bistatic configuration places the IR source and transmitting optics at one end of the path and the receiving optics, interferometer, and detector at the other end of the path (Fig. 1(B)). This is the most common configuration of bistatic systems in current use. In this configuration the beam from the IR source is collimated by a mirror shaped as a paraboloid. The configuration shown in Fig. 1(B) allows the maximum monitoring path, in principle, to be doubled compared to that of the monostatic configuration. The main drawback to this bistatic configuration is that the IR radiation is not modulated before it is transmitted along the path. Therefore, radiation from the active IR source and the ambient background cannot be distinguished by electronic processing.

6.1.2 *Monostatic Configuration*—In monostatic configurations, the IR source and the detector are at the same end of the monitoring path. A retroreflector of some sort is required at the midpoint of the optical path to return the beam to the detector. Thus, the optical pathlength is twice the distance between the source and the retroreflector. Two techniques are currently in use for returning the beam along the optical path in the monostatic configuration. One technique uses an arrangement of mirrors, such as a single cube-corner retroreflector, at one end of the path that translates the beam slightly so that it does not fold back on itself (Fig. 2(A)). The other end of the path then has a second telescope slightly removed from the transmitter to collect the returned beam. Initial alignment with this configuration can be difficult, and this type of monostatic system is normally used in permanent installations rather than as a transportable unit. Another configuration of the monostatic monitoring mode uses the same telescope to transmit and receive the IR beam. A cube-corner retroreflector array is placed at the end of the monitoring path to return the beam (Fig. 2(B)). To transmit and receive with the same optics, a beamsplitter must be placed in the optical path to divert part of the returned beam to the detector. A disadvantage to this configuration is that the IR energy must traverse this beamsplitter twice. The most efficient beamsplitter transmits 50 % of the light and rejects the other 50 %. Thus, in two passes, the

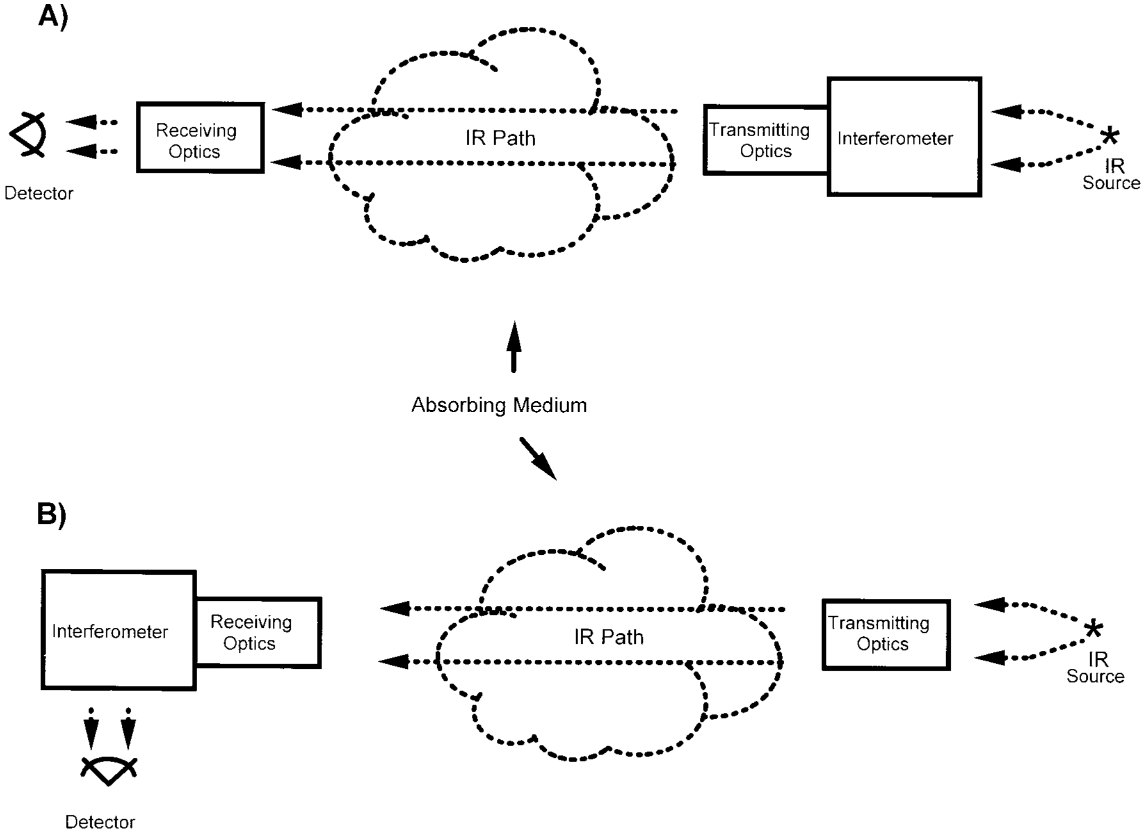

FIG. 1 Schematic Diagram of the Bistatic OP/FT-IR Configuration Showing (*A*) a System with the IR Source and Interferometer at One End of the Path and the Detector at the Opposite End, and (*B*) a System with the IR Source at One End of the Path and the Interferometer and Detector at the Opposite End

transmission is only 25 % of the original beam. Because this loss of energy decreases the signal-to-noise ratio (*S/N*), it can potentially be a significant drawback of this configuration.

7. Selection of Instrumental Parameters

7.1 *Introduction and Overview*—One important issue regarding the operation of OP/FT-IR systems is the appropriate instrumental parameters, such as measurement time, resolution, apodization, and degree of zero filling, to be used during data acquisition and processing. The choice of some of these parameters is governed by the trading rules in FT-IR spectrometry and by specific data quality objectives of the study.

7.2 *Trading Rules in FT-IR Spectrometry*—The quantitative relationships between the *S/N*, resolution, and measurement time in FT-IR spectrometry are called “trading rules.” The factors that affect the S/N and dictate the trading rules are expressed in Eq 1, which gives the *S/N* of a spectrum measured with a rapid-scanning Michelson interferometer (6):

$$\frac{S}{N} = \frac{U_v(T)\cdot\theta\cdot\Delta v\cdot t^{1/2}\cdot\xi\cdot D^*}{(A_D)^{1/2}} \tag{1}$$

where:

$U_v(T)$ = spectral energy density at wavenumber v from a blackbody source at a temperature T,
θ = optical throughput of the spectrometric system,
Δv = resolution of the interferometer,
t = measurement time in seconds,
ξ = efficiency of the interferometer,
D^* = specific detectivity, a measure of the sensitivity of the detector, and
A_D = area of the detector element.

NOTE 2—This equation is correct but assumes that the system is detector noise limited, which is not always true. For example, source fluctuations, the analog-to-digital converter, or mechanical vibrations can contribute to the system noise.

7.3 *Measurement Time*—As shown in Eq 1, the *S/N* is proportional to the square root of the measurement time ($t^{1/2}$). For measurements made with a rapid scanning interferometer operating at a constant mirror velocity and a given resolution, the *S/N*increases with the square root of the number of co-added scans. The choice of measurement time for signal