



Standard Practice for General Techniques of Infrared Microanalysis¹

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1. Scope

1.1 This practice covers techniques that are of general use in securing and analyzing microgram quantities of samples by infrared spectrophotometric techniques. This practice makes repetition of description of specific techniques unnecessary in individual infrared methods.

1.2 These recommendations are supplementary to Practices E 168, E 573, and E 1252, which should be referred to for theory, general techniques of sample preparation, and calculations.

2. Referenced Documents

2.1 ASTM Standards:

- E 131 Terminology Relating to Molecular Spectroscopy²
- E 168 Practices for General Techniques of Infrared Quantitative Analysis²
- E 573 Practices for Internal Reflection Spectroscopy²
- E 1252 Practice for General Techniques for Obtaining Infrared Spectra for Qualitative Analysis²
- E 1642 Practice for General Techniques of Gas Chromatography Infrared (GC/IR) Analysis²
- E 2105 Practice for General Techniques of Thermogravimetric Analysis (TGA) Coupled with Infrared Analysis (TGA/IR)²
- E 2106 Practice for General Techniques of Liquid Chromatography-Infrared (LC/IR) and Size Exclusion Chromatography (SEC/IR) Analysis²

3. Terminology

3.1 *Definitions and Symbols*—For definitions of terms and symbols, refer to Terminology E 131.

3.2 *Beam Condenser*—A specialized accessory designed for analysis of samples of a microgram or less, comprising an analyte area or volume of 2.0 mm diameter or less.

4. Contamination

4.1 Although the presence of contaminants is a general problem in any type of analysis, contamination can be particularly severe in micro work. For example, minor impurities in a

solvent can become major components of a residue remaining after solvent evaporation. Materials extracted from thin-layer chromatographic materials, from the paper used in paper chromatography, and from solid adsorbents in general, may include particular contaminants of concern. It should also be noted that the gas-chromatographic stationary phase may lead to significant contamination. Consideration of these and other sources of contamination must always enter interpretation of results in microanalysis. Erroneous results can be minimized by the use of pure reagents, extreme care in sample handling, and the frequent use of “blanks” in the course of separation and subsequent recording of spectra.

5. General Microspectroscopic Techniques

5.1 Spectroscopic techniques used for the examination of microsamples are usually adaptations of comparable macro techniques, and many have been described in the literature (1, 2).³

5.2 In computerized dispersive spectrometers or Fourier transform-infrared (FT-IR) instruments, computer routines for multiple scanning, signal averaging, absorbance subtraction, and scale expansion can be used very effectively to enhance the observed signal-to-noise ratio of weak bands and increase sensitivity (3, 4). Absorbance subtraction is also commonly used to eliminate interfering bands from the sample matrix and thus lower the limits of detection (see Practice E 168).

5.3 *Use of Masking Apertures*—The aperture of sample holders used for microspectroscopic study (without the use of an infrared microscope) are usually significantly smaller than the beam at the sample position of the instrument. As a consequence of these small apertures, steps need to be taken to ensure that the best quality spectra be obtained, and the techniques used will depend on the type of spectrometer being used. In general, the use of a beam condensing accessory will greatly improve the results obtained (see 5.4).

5.3.1 When a double-beam dispersive spectrometer that is not equipped for control by minicomputer is used, the reference beam should be masked to a corresponding aperture. This can be accomplished by using an opaque sheet of stiff material punched with an appropriate opening, with reference screens, or with commercially available optical attenuators. Attenuation of the reference beam affects instrument performance, and

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² *Annual Book of ASTM Standards*, Vol. 03.06.

³ The boldface numbers in parentheses refer to a list of references at the end of this practice.

