



Standard Practices for Internal Reflection Spectroscopy¹

This standard is issued under the fixed designation E573; 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 These practices provide general recommendations covering the various techniques commonly used in obtaining internal reflection spectra.^{2,3} Discussion is limited to the infrared region of the electromagnetic spectrum and includes a summary of fundamental theory, a description of parameters that determine the results obtained, instrumentation most widely used, practical guidelines for sampling and obtaining useful spectra, and interpretation features specific for internal reflection.

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

2. Referenced Documents

2.1 ASTM Standards:⁴

E131 Terminology Relating to Molecular Spectroscopy

E168 Practices for General Techniques of Infrared Quantitative Analysis (Withdrawn 2015)⁵

E284 Terminology of Appearance

3. Terminology

3.1 *Definitions of Terms and Symbols*—For definitions of terms and symbols, refer to Terminologies E131 and E284, and to Appendix X1.

¹ These practices are under the jurisdiction of ASTM Committee E13 on Molecular Spectroscopy and Separation Science and are the direct responsibility of Subcommittee E13.03 on Infrared and Near Infrared Spectroscopy.

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² Internal Reflection Spectroscopy, IRS, is the accepted nomenclature for the technique described in these practices. Other terms are sometimes used which include: Attenuated Total Reflection, ATR; Frustrated Total Reflection, FTR; Multiple Internal Reflection, MIR; and other less commonly used terms. In older literature, one may find references to Frustrated Total Internal Reflection, FTIR. This should not be confused with Fourier Transform Infrared Spectroscopy FT-IR.

³ Other terms sometimes used for referring to the internal reflection element are: ATR crystal, MIR plate, or sample plate.

⁴ 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.

⁵ The last approved version of this historical standard is referenced on www.astm.org.

4. Significance and Use

4.1 These practices provide general guidelines for the good practice of internal reflection infrared spectroscopy.

5. Theory

5.1 In his studies of total reflection at the interface between two media of different refractive indices, Newton (1)⁶ discovered that light extends into the rarer medium beyond the reflecting surface (see Fig. 1). In internal reflection spectroscopy, IRS, this phenomenon is applied to obtain absorption spectra by measuring the interaction of the penetrating radiation with an external medium, which will be called the sample (2,3). Theoretical explanation for the interaction mechanisms for both absorbing and nonabsorbing samples is provided by Snell's law, the Fresnel equations (4), and the Maxwell relationships (5).

NOTE 1—To provide a basic understanding of internal reflection phenomena applied to spectroscopy, a brief description of the theory appears in Appendix X2. For a detailed theoretical discussion of the subject, see (4).

6. Parameters of Reflectance Measurements

6.1 Practical application of IRS depends on many precisely controlled variables. Since an understanding of these variables is necessary for proper utilization of the technique, descriptions of essential parameters are presented.

6.2 *Angle of Incidence, θ* —When θ is greater than the critical angle, θ_c , total internal reflection occurs at the interface between the sample and the internal reflection element, IRE. When θ is appreciably greater than θ_c , the reflection spectra most closely resemble transmission spectra. When θ is less than θ_c , radiation is both refracted and internally reflected, generally leading to spectral distortions. θ should be selected far enough away from the average critical angle of the sample—IRE combination that the change of θ_c through the region of changing index (which is related to the presence of the absorption band of the sample) has a minimal effect on the shape of the internal reflection band. Increasing θ decreases the number of reflections, and reduces penetration. In practice,

⁶ The boldface numbers in parentheses refer to the list of references at the end of these practices.