



# Standard Practice for Describing and Measuring Performance of Dispersive Infrared Spectrometers<sup>1</sup>

This standard is issued under the fixed designation E932; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This practice covers the necessary information to qualify dispersive infrared instruments for specific analytical applications, and especially for methods developed by ASTM International.

1.2 This practice is not to be used as a rigorous test of performance of instrumentation.

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

1.4 *This standard does not purport to address all of the safety problems, 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:*<sup>2</sup>

[E131 Terminology Relating to Molecular Spectroscopy](#)

[E168 Practices for General Techniques of Infrared Quantitative Analysis](#)

[E387 Test Method for Estimating Stray Radiant Power Ratio of Dispersive Spectrophotometers by the Opaque Filter Method](#)

[E1252 Practice for General Techniques for Obtaining Infrared Spectra for Qualitative Analysis](#)

## 3. Terminology

3.1 *Definitions and Symbols*—For definitions of terms and symbols, refer to Terminology [E131](#) and *Compilation of ASTM*

<sup>1</sup> This practice 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.

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

*Standard Definitions.*<sup>3</sup>

## 4. Significance and Use

4.1 This practice is intended for all infrared spectroscopists who are using dispersive instruments for qualitative or quantitative areas of analysis.

4.2 The purpose of this practice is to set forth performance guidelines for testing instruments used in developing an analytical method. These guidelines can be used to compare an instrument in a specific application with the instrument(s) used in developing the method.

4.3 An infrared procedure must include a description of the instrumentation and of the performance needed to duplicate the precision and accuracy of the method.

## 5. Apparatus

5.1 For the purposes of this practice, dispersive instruments include those employing prisms, gratings, or filters to separate infrared radiation into its component wavelengths.

5.2 For each new method, describe the apparatus and instrumentation both physically and mechanically, and also in terms of performance as taught in this practice. That is, the description should give numerical values showing the frequency accuracy and the frequency and the photometric precision. State the spectral slit width maximum or slit width program if one is used. Where possible, state the maximum and minimum resolution if those data are a part of the instrument display. Show typical component spectra as produced by the instrument to establish the needed resolution.

5.3 If a computer program is used, describe the program. Include the programming language and availability, or whether the program is proprietary to a manufacturer.

## 6. Reference to this Practice in Standards

6.1 Reference to this practice should be included in all ASTM infrared methods. The reference should appear in the section on apparatus where the particular spectrometer is described.

<sup>3</sup> Available from ASTM International Headquarters, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428.

## 7. Parameters in Spectroscopy

7.1 Dispersive infrared spectrometers have a source of quasi-monochromatic radiation together with a photometer for measuring relative radiant power. Accurate spectrometry involves a large number of interrelated factors that determine the quality of the radiant power passing through a sample and the sensitivity and linearity with which this radiant power can be measured. Assuming proper instrumentation and its use, the instrumental factors responsible for inaccuracies in spectrometry are resolution, linearity (Practices **E168**), stray radiant power (Test Method **E387**), and cell constants (Practice **E1252**). Rigorous measurement of these factors is beyond the scope of this practice, and a more practical approach is described for the accessible factors.

## 8. Instrument Operation

8.1 The analyst selects the proper instrumental operating conditions in order to get satisfactory performance (**1-3**).<sup>4</sup> Because instrument design varies, the manufacturer's recommendations are usually best. A record of operating conditions should be kept so that data can be duplicated by future users.

8.2 In addition to operating conditions, the following should be checked and recorded:

8.2.1 *Ambient temperature,*

8.2.2 *Pen response time,*

8.2.3 *Scanning speed,*

NOTE 1—In some instruments these functions are integrated in the scan modes.

8.2.4 *Noise level, and*

8.2.5 *Mechanical repeatability.*

8.3 Each of the above factors is important in the measurement of analytical wavenumber and photometric data. There is usually some lag between the recorded reading and the correct reading. Proper selection of operating conditions and good, reproducible, sample handling techniques minimize these effects or make the effects repeatable. For example:

8.3.1 Variation in temperature of the monochromator or sample may cause changes in wavenumber precision and accuracy.

8.3.2 Scanning too fast will displace the apparent wavenumber towards the direction scanned and will decrease the peak absorbance reading for each band.

NOTE 2—Some instruments provide for automatic monitoring and correction of this effect.

8.4 Mechanical repeatability of the monochromator and recording system as well as positioning of chart paper are important in wavenumber measurement.

8.4.1 Chart paper should be checked for uniformity of the printed scale length as received and rechecked at time of use, particularly if the paper has been subjected to pronounced humidity changes. Instructions on obtaining proper mechanical repeatability may be given in the manufacturer's literature.

<sup>4</sup> The boldface numbers in parentheses refer to a list of references at the end of this standard.

8.5 In the case of computerized dispersive instruments, any spectrum printed from a computer file must be obtained as prescribed by the manufacturer and should be identical to the original data.

## PRECISION AND ACCURACY

### 9. Definitions

9.1 *wavenumber precision*—a measure of the capability of a spectrometer to return to the same spectral position as measured by a well-defined absorption or emission band when the instrument is reset or rescanned. The index used in this practice is the standard deviation.

9.2 *wavenumber accuracy*—the deviation of the average wavenumber reading of an absorption band or emission band from the known wavenumber of that band.

### 10. Nature of Test

10.1 For the purpose of calibration, most methods employ pure compounds and known mixtures at specified analytical wavenumbers. The wavenumbers are either read from a dial, optical display, chart paper, or a computer file.

### 11. Reference Wavenumbers in the Infrared Region (2)

11.1 The recommended wavenumber calibration points are the absorption maxima of a standard (98.4/0.8/0.8 by weight) indene/camphor/cyclohexanone mixture listed in **Table 1**. Suitable path lengths are 0.2 mm for the range from 3800 to 1580  $\text{cm}^{-1}$  and 0.03 mm for the wavenumber range from 1600 to 600  $\text{cm}^{-1}$ . A mixture containing equal parts by weight of indene, camphor, and cyclohexanone (1/1/1 by weight) at a path length

**TABLE 1 Indene-Camphor-Cyclohexanone (98.4/0.8/0.8) Mixture—Recommended Calibration Bands**

NOTE 1—**Table 1** and **Table 2** contain wavenumber values for bands in **Fig. 1**.

Band No.	Wavenumber, $\text{cm}^{-1}$	Band No.	Wavenumber, $\text{cm}^{-1}$
1	3927.2 ± 1.0	44 $\alpha$	1741.9
2	3901.6	44 $\beta$	1713.4
3	3798.9	47	1661.8
5	3660.6 ± 1.0	48	1609.8
8	3297.8 ± 1.0	49	1587.5
9	3139.5	51	1553.2
10	3110.2	53	1457.3 ± 1.0
12	3025.4	54	1393.5
15	2887.6	55	1361.1
17	2770.9	57	1312.4
19	2673.3	58	1288.0
20	2622.3	60	1226.2
21	2598.4 ± 1.0	61	1205.1
23	2525.5	62	1166.1
28	2305.1	64	1122.4
29	2271.4	66	1067.7 ± 1.0
30	2258.7	67	1018.5
33	2172.8	69	947.2
34	2135.8 ± 1.0	70	942.4
35	2113.2	71	914.7
36	2090.2	72	861.3
39	1943.1	73	830.5
40	1915.3	74	765.3
41	1885.1	76	718.1
42	1856.9	77	692.6 ± 1.0
44	1797.7 ± 1.0		