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Standard Test Method for Infrared Identification of Vehicle Solids From Solvent-Reducible Paints¹

This standard is issued under the fixed designation D 2621; 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.

 ϵ^1 Note—Keywords were added editorially in May 1995.

1. Scope

- 1.1 This test method covers the qualitative characterization or identification of separated paint vehicle solids by infrared spectroscopy within the limitations of infrared spectroscopy.
- 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:
- D 1467 Guide for Testing Fatty Acids Used in Protective Coatings²
- D 1962 Test Method for Saponification Value of Drying Oils, Fatty Acids, and Polymerized Fatty Acids²
- D 2372 Practice for Separation of Vehicle from Solvent-Reducible Paints³
- E 131 Terminology Relating to Molecular Spectroscopy⁴
- E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near Infared Spectrophotometers⁴

3. Terminology

- 3.1 Definitions:
- 3.1.1 For definitions of terms of symbols, refer to Terminology E 131.

4. Summary of Test Method

4.1 Infrared spectra are prepared from dried films of isolated paint vehicles. Vehicle types are identified by comparing the spectra to a collection of reference infrared spectra.

5. Significance and Use

5.1 The ability to qualitatively identify paint vehicles is

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- ² Annual Book of ASTM Standards, Vol 06.03.
- ³ Annual Book of ASTM Standards, Vol 06.01.
- ⁴ Annual Book of ASTM Standards, Vol 03.06.

useful for characterizing unknown or competitive coatings, for complaint investigations, and for in-process control.

6. Apparatus

- 6.1 Spectrophotometer—A recording double-beam infrared spectrophotometer with a wavelength range from at least 2.5 to 15 μ m and a spectral resolution of at least 0.04 μ m over that range. See Practice E 275.
 - 6.2 Demountable Cell Mount with NaCl window.
- 6.3 *Vacuum Drying Oven* thermostatically controlled to operate at $60 \pm 2^{\circ}$ C. A water aspirator vacuum source is satisfactory.
- 6.4 Oven, Gravity or Forced Draft, capable of maintaining temperature from 105 to 110°C.

7. Procedure

7.1 Place the vehicle, separated from the paint in accordance with Practice D 2372, on a NaCl window and spread to form a uniform film. Make sure that the thickness of the film is such that when the infrared spectrum is recorded the transmittance of the strongest band falls between 5 and 15 % (Note). Dry the film in an oven at 105 to 110°C for 15 min and cool in a desiccator. Inspect the film visually for defects such as bubbles, wrinkles, contamination, etc. If defects are present, cast another film. If easily oxidizable substances are present such as tung, oiticica, or linseed oils, make sure that the film is dried at 60 ± 2 °C in a vacuum oven for 1 h. If solvents of low volatility such as cyclohexanone or isophorone are present, the film may need to be dried for several hours in a 60°C vacuum oven.

Note 1—Numerous procedures and variations may be used to obtain a film on which to prepare a suitable spectrum. These include liquid mounting between two NaCl plates, transmission through free films, and reflectance from highly polished surfaces.

- 7.2 Immediately record the infrared spectrum from 2.5 to 15 μm so that a spectral resolution of 0.04 μm is maintained throughout that range (methods for achieving this resolution will vary according to the directions of the manufacturer of the instrument used).
- 7.3 Compare the spectrum obtained with reference spectra prepared from nonvolatile vehicles of known composition (see Annex A1) or consult other published spectra available in the literature (Annex A3). Interpret the spectrum on the basis of



available information, recognizing certain limitations of infrared spectroscopy, and qualifying the interpretation accordingly (Annex A2). Table 1

8. Keywords

8.1 infrared spectra; paint binders; solvent reducible paint

TABLE 1 Correlation of Absorption Bands in Alkyd Spectra

Wavelength, µm	Wavenumbers, cm ⁻¹	Group Vibration
2.9	3448	O–H stretch
3.4 to 3.5	2941 to 2857	alkane C-H stretch
5.8	1724	ester, C=O stretch
6.2, 6.3, 6.6, 6.7	1613, 1587, 1515, 1493	skeletal in-plane aromatic C=C
6.9, 7.3	1449, 1369	aliphatic C–H bending
7.5 to 9.4	1333 to 1063	ester, C-O-C stretch (o-phthalate ester)
8.6	1163	ester, C-O-C stretch (fatty acid ester)
9.6, 13.5, 14.3	1042, 741, 699	out-of-plane aromatic C-H bending denoting o-disubstituted benzene ring.

ANNEXES

(Mandatory Information)

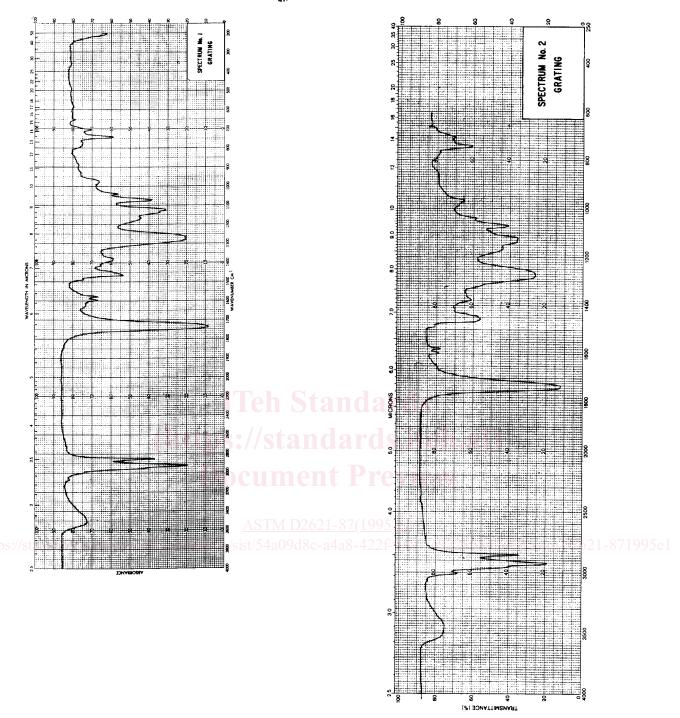
A1. INFRARED SPECTRA OF NONVOLATILE VEHICLES OF KNOWN COMPOSITION

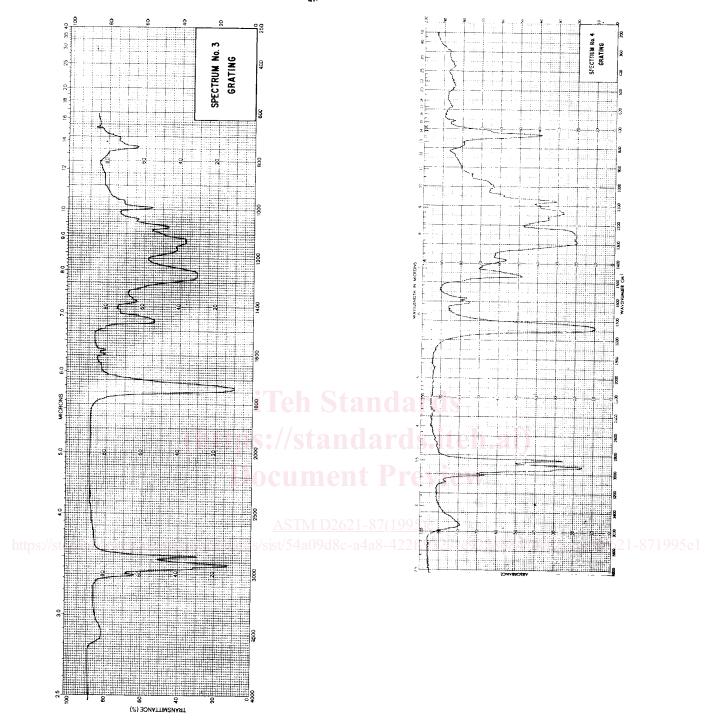
A1.1 A set of reference infrared spectra on grating and prism is reproduced on the following pages.

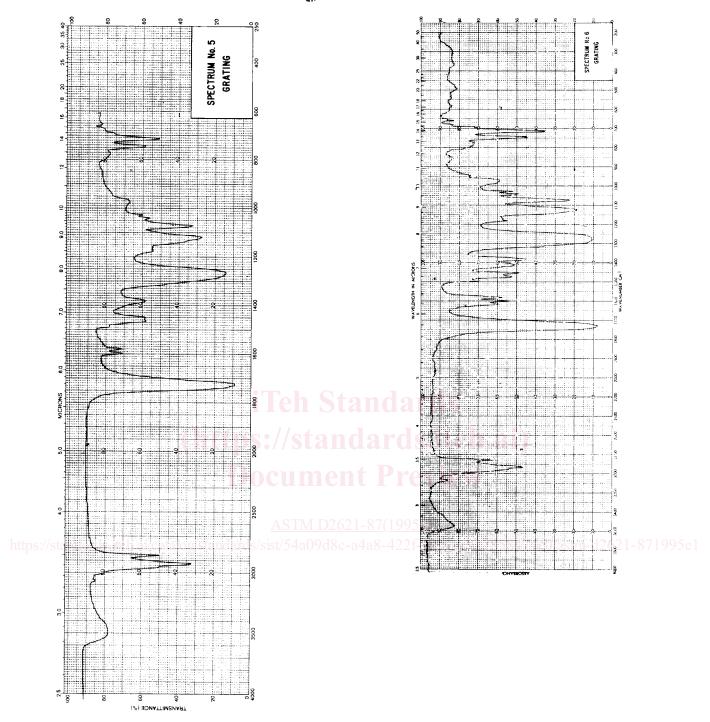
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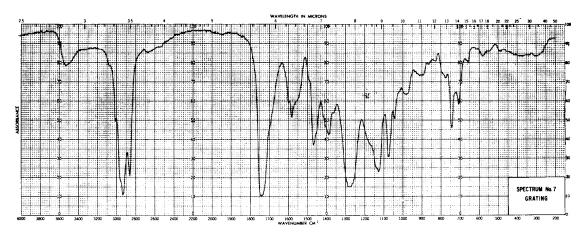
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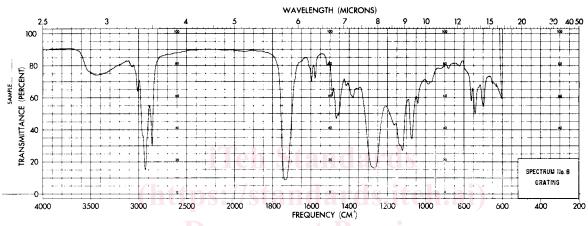


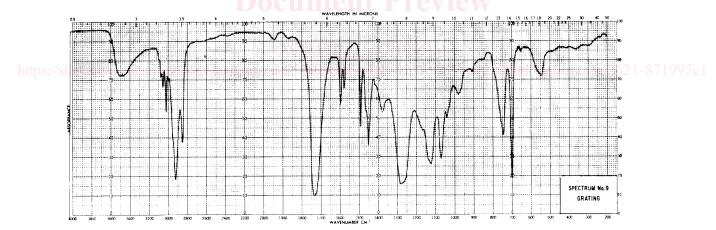




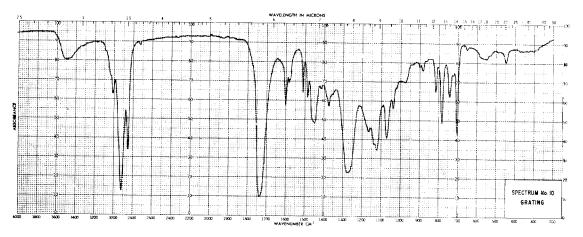








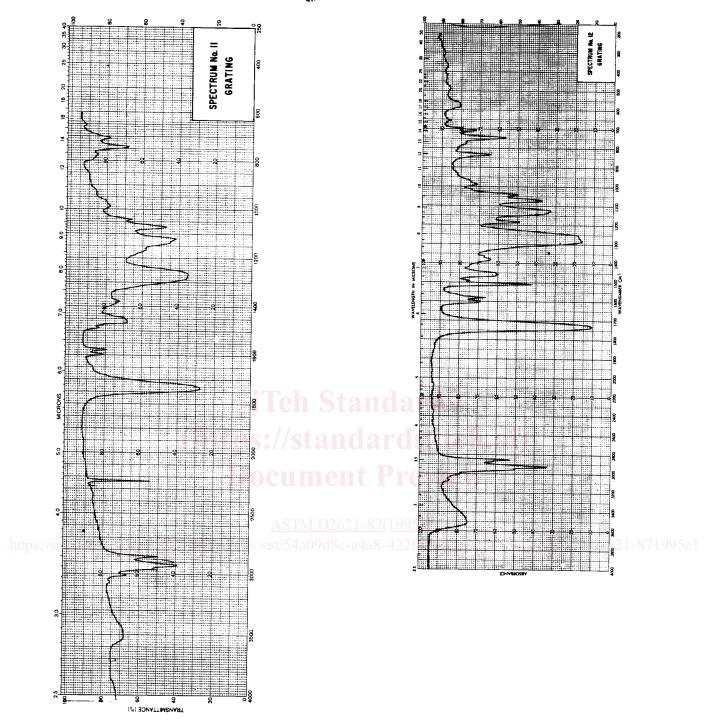




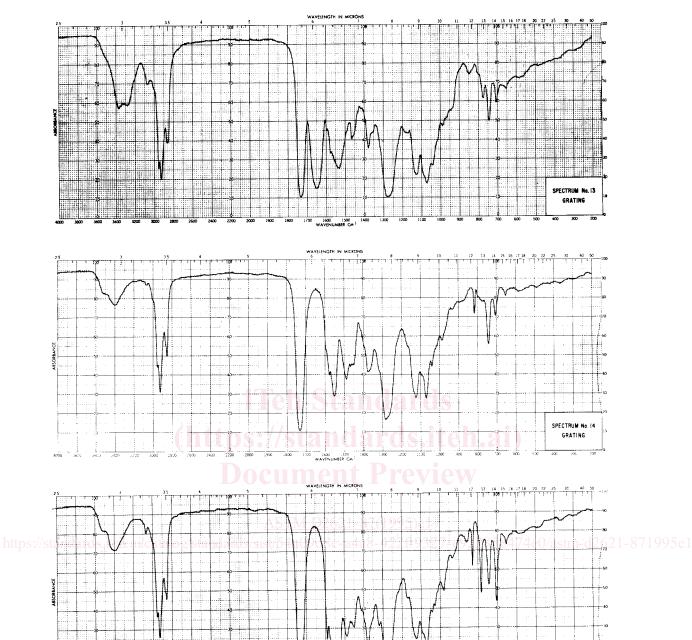
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SPECTRUM No. 15 GRATING



