



Designation: ~~D2357–74 (Reapproved 2003)~~ Designation: D2357 – 11

## Standard for Qualitative Classification of Surfactants by Infrared Absorption<sup>1</sup>

This standard is issued under the fixed designation D2357; 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 standard covers the qualitative classification of synthetic detergent products or mixtures of synthetic detergents. It is applicable to built detergent formulations as well as individual surfactant compositions.

NOTE 1—The organic active ingredient must be isolated from built syndet compositions in accordance with Test Method D2358.

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:*<sup>2</sup>

D2358 [Test Method for Separation of Active Ingredient from Surfactant and Syndet Compositions](#)

### 3. Summary of Classification

3.1 A portion of the active ingredient is scanned in the infrared region of the spectrum from at least 2.5 to 15  $\mu\text{m}$  (4000 to 667  $\text{cm}^{-1}$ ). Qualitative identification of surfactant type is based on the presence of infrared absorption bands attributable to specific functional groups.

3.2 A listing of ~~absorption~~ absorbance bands corresponding to the characteristic functional groups of some of the more common types of commercial surfactants is included in this method. Use of available detergent reference spectra may provide additional information.

### 4. Apparatus

4.1 ~~Spectrophotometer, recording, infrared,~~ recording, infrared, or Fourier transform Infrared (FTIR if available).

4.2 *Dies and Press*, for preparation of KBr disks.

4.3 *Detergent Reference Spectra*.

4.4 *Appropriate Sampling Accessories*—ATR, Diffuse reflectance and others as appropriate.

### 5. Reagents

5.1 ~~Mineral Oil, USP.~~

5.2 *Potassium Bromide* (KBr), infrared quality, powdered.

5.3

5.2 *Additional Reagents*, as specified in Test Method D2358.

### 6. Preparation of Sample

6.1 *Sample in Pure Form*—If the sample is in pure form or contains only volatile solvents, dry the sample in accordance with 5.8 of Test Method D2358, and obtain the infrared spectrum of the dried sample.

6.2 *For Built Surfactants and Synthetic Detergent Compositions*—Obtain the infrared spectrum of the active ingredient(s) separated in accordance with Test Method D2358.

<sup>1</sup> This classification is under the jurisdiction of ASTM Committee D12 on Soaps and Other Detergents and is the direct responsibility of Subcommittee D12.12 on Analysis and Specifications of Soaps, Synthetics, Detergents and their Components.

Current edition approved Feb. 10, 2003. Published March 2003. Originally published as D2357–65T. Last previous edition D2357–74. DOI: 10.1520/D2357-74R03.

Current edition approved Sept. 1, 2011. Published October 2011. Originally approved in 1965 as D2357–65 T. Last previous edition approved in 2003 as D2357–74(2003). DOI: 10.1520/D2357-11.

<sup>2</sup> 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.

## 7. Procedure

7.1 Whenever possible, the infrared spectrum should be obtained directly from the organic material, by formation of a film between salt blocks, and by recording the spectrum between 2.5 and 15  $\mu\text{m}$  (4000 and 667  $\text{cm}^{-1}$ ). The film is satisfactory if the spectrum shows 10 to 30 % transmittance in the strongest absorption/absorbance region.

7.2 If the physical properties of the sample prevent use of the film technique, the spectrum may be made from a mineral oil mull. Form the mull by adding 2 to 3 drops of mineral oil to a small quantity of sample contained in a polished grinding surface (agate) mortar, and grinding for a minimum of 5 min. This should produce a very fine syrupy dispersion. Use the mull as a film between salt blocks and obtain the spectrum as in 7.1.

7.3 If the sample can be obtained in dry, powdered form, the KBr pressed-disk method may be used. Grind the sample sufficiently fine to prevent radiation scatter, and add approximately 1 part sample to 19 parts of dry powdered KBr. Using suitable equipment, form the disk at room temperature in vacuum, under pressure of 10 to 30 tons/in.

7.2 Alternatively, the sample may be applied to an ATR crystal and the spectrum collected according to the manufacturer's instructions. Care should be taken to use an appropriate ATR crystal material as different crystals will produce interference in different regions of the spectrum. Diamond crystals are generally preferred for durability but they will produce interference in the 3.85 to 5.5  $\mu\text{m}$  (2600 to 1800  $\text{cm}^{-1}$ ) region of the spectrum. A correction factor may need to be applied to the spectrum to change the absorbance values to what would be expected in a transmission spectrum. Most software packages have algorithms to perform this correction.

7.3 If the sample can be obtained in dry, powdered form, the diffuse reflectance or KBr pressed-disk method may be used. Grind the sample sufficiently fine to prevent radiation scatter, and add approximately 1 part sample to 19 parts of dry powdered KBr. For diffuse reflectance load this mixture into the accessory according to the manufacturer's instructions. To prepare a KBr disk, using suitable equipment, form the disk at room temperature in vacuum, under pressure of 10 to 30 tons/in.<sup>2</sup> Obtain the spectrum of the disk, sample between 2 and 15  $\mu\text{m}$  as in (150 to 450 MPa). Obtain the spectrum of the sample between 2.5 and 15  $\mu\text{m}$  (4000 and 667  $\text{cm}^{-1}$ ) as in 7.1.<sup>3</sup>

7.4 Other sample accessories may be used to collect spectra provided they work in the appropriate wavelength/frequency range and any interferences are known and accounted for in the interpretation.

## 8. Identification of Components

8.1 Note the wavelength/frequency position of each specific absorption band.

8.2 Identify the absorption bands in terms of functional groups by the use of data in Table 1 or other appropriate reference source.

**TABLE 1 Infrared Absorption Bands of Typical Commercial Detergents**

Detergent	Wavelength, $\mu\text{m}$	Frequency, $\text{cm}^{-1}$	Band Shape <sup>A</sup>	Band Intensity <sup>B</sup>	Identity
Alkylbenzene sulfonate	6.7	1493	shoulder	weak	aromatic bands
	8.1 to 8.5	1235 to 1176	broad <sup>C</sup>	strong	sulfonate group
	8.8	1136	sharp	moderate	sulfonate group
	9.6	1042	sharp	strong	ABS bands
	9.9	1010	sharp	strong	ABS bands
	12.0	833	broad	moderate	para substitution
Fatty alcohol sulfate	8.0	1250	sharp <sup>D</sup>	strong	organic sulfate group
	8.3	1205	sharp <sup>D</sup>	strong	organic sulfate group
	9.2	1087	sharp	moderate	most characteristic alcohol sulfate band
	10.3	971	broad	weak	characteristic alcohol sulfate bands
	10.8	926	broad	weak	characteristic alcohol sulfate bands
Sulfonated amide (taurate)	6.1	1639	sharp	strong	amide carbonyl
	6.4	1563	shoulder	weak	secondary amide
	8.1 to 8.5	1235 to 1176	broad	strong	sulfonate group
	9.4	1064	sharp	strong	ClCzN or alkyl sulfonate
Sulfonated ester (isethionate)	5.8	1724	sharp	strong	ester carbonyl
	6.4	1563	broad	weak	soap
	8.1 to 8.5	1235 to 1176	broad	strong	sulfonate group
	8.5	1176	broad	strong	ester ClCzO
	9.4	1064	sharp	strong	alkyl sulfonate
Sulfated monoglyceride	3.0	3333	sharp	strong	hindered OH
	5.8	1724	sharp	strong	ester carbonyl
	7.9	1266	sharp <sup>E</sup>	strong	organic sulfate

<sup>3</sup> For details of infrared experimental techniques, see Jones, R. N. and Sandorfy, C., "The Application of Infrared and Raman Spectrometry to the Elucidation of Molecular Structure." *Technique of Organic Chemistry*, Vol IX, 1956. Interscience Publishers, Inc., New York, NY.