Designation: D2357 – 11 (Reapproved 2016)

Standard for
Qualitative Classification of Surfactants by Infrared Absorption [1]

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 (ε) 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:*[2]

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 µm (4000 to 667 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 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, 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 *Potassium Bromide* (KBr), infrared quality, powdered.

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.

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 µm (4000 and 667 cm^{-1}). The film is satisfactory if the spectrum shows 10 to 30 % transmittance in the strongest absorbance region.

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 µm (2600 to 1800 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,

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[1] 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.

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[2] 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.

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.^{2} (150 to 450 MPa). Obtain the spectrum of the sample between 2.5 and 15 µm (4000 and 667 cm^{-1}) as in 7.1.[3]

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, µm	Frequency, cm^{-1}	Band Shape[A]	Band Intensity[B]	Identity
Alkylbenzene sulfonate	6.7	1493	shoulder	weak	aromatic bands
	8.1 to 8.5	1235 to 1176	broad[C]	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[D]	strong	organic sulfate group
	8.3	1205	sharp[D]	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	C|CzN 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 C|CzO
	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[E]	strong	organic sulfate
	8.1	1235	sharp[E]	strong	organic sulfate
	8.5	1176	broad	moderate	ester C|CzO
	9.0	1111	broad	weak	H|CzC|CzOH secondary
	9.4	1064	sharp	moderate	H|CzC|CzOH secondary
Sulfated phenoxy ether	6.2	1613	sharp	weak	aromatic bands
	6.6	1515	sharp	moderate	aromatic bands
	7.4	1351	broad	weak	polyethylene oxide
	8.0 to 8.2	1250 to 1220	broad	strong	organic sulfate
	8.7 to 9.2	1149 to 1087	broad	strong	polyethylene oxide
	10.5 to 10.9	952 to 917	broad	moderate	polyethylene oxide
	12.0	833	broad	moderate	para substitution
Ethoxylated fatty acid	2.9	3448	sharp	moderate	OH
	5.8	1724	sharp	strong	ester carbonyl
	7.4	1351	sharp	moderate	polyethylene oxide
	8.7 to 9.2	1149 to 1087	broad	strong	polyethylene oxide
	10.5 to 10.9	952 to 917	broad	strong	polyethylene oxide
	8.5	1176	shoulder	moderate	ester C|CzO
Ethoxylated fatty alcohol	2.9	3448	sharp	moderate	OH
	7.4	1351	sharp	strong	polyethylene oxide
	8.7 to 9.2	1149 to 1087	broad	strong	polyethylene oxide
	9.4	1064	sharp	moderate	C|CzOH
	10.5 to 10.9	952 to 917	broad	strong	polyethylene oxide
Ethoxylated alkyl phenol	2.9	3448	sharp	moderate	OH
	6.2	1613	sharp	moderate	aromatic bands
	6.6	1515	sharp	strong	aromatic bands
	7.4	1351	sharp	strong	polyethylene oxide
	8.0	1250	sharp	strong	characteristic alkylphenol polyether band
	8.4	1190	sharp	strong	characteristic alkylphenol polyether band

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[3] 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.

TABLE 1 *Continued*

Detergent	Wavelength, µm	Frequency, cm^{-1}	Band Shape[A]	Band Intensity[B]	Identity
	8.7 to 9.2	1149 to 1087	broad	strong	polyethylene oxide
	12.0	833	broad	moderate	para substitution
Glyceryl monostearate	3.0	3333	broad	strong	OH
	5.8	1724	sharp	strong	ester carbonyl
	8.5	1176	broad	weak	ester ClCzO
	9.0	1111	broad	moderate	ClCzOH secondary
	9.4	1064	broad	moderate	ClCzOH secondary
	9.6	1042	broad	moderate	ClCzOH primary
Mono-alkylol amide	3.0	3333	sharp	strong	OH
	3.2	3125	shoulder	weak	NH
	6.1	1639	sharp	strong	amide carbonyl
	6.4	1563	sharp	strong	secondary amide
	9.4	1064	sharp	moderate	ClCzOH
	9.6	1042	sharp	moderate	ClCzOH
	10.8	926	broad	weak	ethanol
Soap	6.4	1563	sharp	strong	carboxylate (sodium)
	10.4	962	broad	weak	organic acid salt band
	10.8	926	broad	weak	organic acid salt band
	13.8	725	sharp	medium	characteristic for soap
	14.4	694	sharp	medium	characteristic for soap
Aliphatic quaternary ammonium chloride	2.9	3448	sharp	strong	typical quaternary ammonium compound bands
	6.1	1639	broad	weak	typical quaternary ammonium compound bands
	10.2 to 10.6	980 to 943	sharp	moderate	typical quaternary ammonium compound bands
	10.9	917	sharp	weak	typical quaternary ammonium compound bands
Alcohol polyether sulfate	7.4	1351	broad	weak	polyethylene oxide
	7.9 to 8.2	1266 to 1220	broad[F]	strong	organic sulfate
	8.7 to 9.2	1149 to 1087	broad	strong	polyethylene oxide
	10.5 to 10.9	952 to 917	broad	strong	polyethylene oxide
Alpha olefin sulfonate	2.9	3448	broad	weak	lCzOH
	3.4	2940	sharp	moderate	lCzCH$_2$
	3.5	2950	sharp	moderate	lCzCH$_2$
	6.8	1470	sharp	moderate	lCzCH$_2$
	8.4	1190	broad	strong	sulfonate
	9.4	1070	sharp	strong	sulfonate
	10.3	970	broad	weak	trans olefin

[A] Band shape descriptions are based on spectra scanned linearly with respect to wavelength.
[B] Some bands marked "weak" in the attached table may not appear in all materials.
[C] May appear as two separate broad bands.
[D] May appear as a broad 8.0 to 8.3-µm band.
[E] May appear as a broad 7.9 to 8.2-µm band.
[F] May appear as separate 8.0 to 8.3-µm bands.

8.3 From reference spectra of known standards obtained in the same manner as the unknown samples, identify the types of surfactants present from their characteristic functional groups.

NOTE 2—Application of chemical tests to a portion of the active ingredient may assist in the identification of components, particularly in the case of surfactant mixtures.

8.4 Table 1 lists some of the more common types of commercial surfactants and the wavelengths/frequencies of their characteristic absorbance bands. This table is intended only as a guide to spectral interpretation and qualitative classification of surfactants. Reference should also be made to *ASTM STP 358*, Serial Number List of Compound Names and References to Published Infrared Spectra.[4]

9. Precision and Bias

9.1 Since this standard is for qualitative analysis only, precision and bias do not apply. The accuracy of the identification, however, may be affected by impurities in the sample. New surfactant molecules not listed in Table 1 and other molecules with similar functional groups may also lead to erroneous identifications. Care should be taken to reference appropriate standard spectra whenever possible to avoid errors.

10. Keywords

10.1 infrared absorbance; surfactant functional groups

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[4] Available as a separate publication.

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