



Designation: D3712 – 05 (Reapproved 2011)

Designation: 369/(95)

Standard Test Method of Analysis of Oil-Soluble Petroleum Sulfonates by Liquid Chromatography¹

This standard is issued under the fixed designation D3712; 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 test method covers the analysis of refined and crude natural and synthetic oil-soluble sulfonate products. Resins, if present, are recovered with the oil phase and carboxylates are recovered as sulfonates.

1.2 This test method covers the determination of mineral oil, sodium sulfonate, inorganic salts, water, basicity or acidity, average molecular weight, and relative density of sodium sulfonate products.

1.3 This test method covers the determination of mineral oil, sulfonate, water, base number, average molecular weight, and relative density of calcium, barium, magnesium, and ammonium sulfonate products.

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.5 *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

- D95 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation
- D2896 Test Method for Base Number of Petroleum Products by Potentiometric Perchloric Acid Titration

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.04.0C on Liquid Chromatography.

Current edition approved Jan. 1, 2011. Published February 2011. Originally approved in 1978. Last previous edition approved in 2005 as D3712 – 05. DOI: 10.1520/D3712-05R11.

This test method was adopted as a joint ASTM-IP standard.

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.

3. Terminology

3.1 Symbols:

3.1.1 Following are definitions of the symbols used in Section 17, and as noted in the sections in parentheses.

A	=	grams of sample of calcium, barium, magnesium, or ammonium sulfonate (8.1.1).
B	=	volume of chloroform solution, mL (10.1).
C	=	grams of sample of sodium sulfonate (10.1.1).
D	=	grams of oil recovered (10.4).
E	=	grams of sodium sulfonate recovered (10.5).
F	=	grams of residue from chloroform blank (10.6).
G	=	grams of residue from alcohol blank (10.6).
H	=	grams of sodium sulfonate (11.1).
I	=	grams of sodium sulfate ash from sodium sulfonate (11.2).
J	=	T/KS.
K	=	valence of cation.
S	=	average equivalent weight of sodium sulfonate (17.1.4).
T	=	average molecular weight of calcium, barium, magnesium, or ammonium sulfonate (17.1.5).
U	=	percentage of sodium sulfonate (17.1.2).
V	=	percentage of calcium, barium, magnesium, or ammonium sulfonate (17.1.3).
W _c	=	grams of water contained in pycnometer at 25°C (6.9).
W _s	=	grams of sample contained in pycnometer at 25°C (15.1).
X	=	grams of sodium sulfonate sample for basicity (12.1).
Y	=	volume of standard H ₂ SO ₄ or NaOH solution used to determine basicity or acidity (12.1).
Z	=	normality of standard H ₂ SO ₄ or NaOH solution to determine free basicity or acidity (12.1).
AA	=	grams of sodium sulfonate product ashed (13.1).
BB	=	grams of sodium sulfate from inorganic salt determination (13.1).
CC _A	=	percentage of free acidity of sodium sulfonate product as H ₂ SO ₄ (17.1.6).
CC _B	=	percentage of free basicity of sodium sulfonate product as NaOH (17.1.6).
DD	=	percentage of inorganic salts as sodium sulfate (17.1.7).

4. Summary of Test Method

4.1 The sample, except a sodium sulfonate product, is dissolved in ethyl ether and converted to sulfonic acid, using dilute hydrochloric acid. The sulfonic acid after extraction is converted to sodium sulfonate and the isolated sodium sulfonate and mineral oil are dissolved in chloroform. An aliquot of the chloroform solution, or a sample of a sodium sulfonate product, dissolved in chloroform, is placed on a silica gel column. The oil is eluted with chloroform, the sulfonate with ethyl alcohol, and both are determined gravimetrically. Average molecular weight is calculated from the average equivalent