



Designation: ~~D6470 – 99 (Reapproved 2010)~~ D6470 – 99 (Reapproved 2015)

Standard Test Method for Salt in Crude Oils (Potentiometric Method)¹

This standard is issued under the fixed designation D6470; 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 determination of salt in crude oils. For the purpose of this test method, salt is expressed as % (m/m) NaCl (sodium chloride) and covers the range from ~~0.0005~~ 0.0005 to 0.15 % (m/m).

1.2 The limit of detection is 0.0002 % (m/m) for salt (as NaCl).

1.3 The test method is applicable to nearly all of the heavier petroleum products, such as crude oils, residues, and fuel oils. It may also be applied to used turbine oil and marine diesel fuel to estimate seawater contamination. Water extractable salts, originating from additives present in oils, are codetermined.

1.4 The values stated in SI units are to be regarded as the standard.

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

[D329 Specification for Acetone](#)

[D770 Specification for Isopropyl Alcohol](#)

[D843 Specification for Nitration Grade Xylene](#)

[D1193 Specification for Reagent Water](#)

[D4006 Test Method for Water in Crude Oil by Distillation](#)

[D4057 Practice for Manual Sampling of Petroleum and Petroleum Products](#)

[D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products](#)

[D4377 Test Method for Water in Crude Oils by Potentiometric Karl Fischer Titration](#)

[D4928 Test Method for Water in Crude Oils by Coulometric Karl Fischer Titration](#)

[E200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis](#)

3. Summary of Test Method

3.1 After homogenizing the crude oil with a mixer, a weighed aliquot is dissolved in xylene at ~~65°C~~ 65 °C and extracted with specified volumes of alcohol, acetone, and water in an electrically heated extraction apparatus. A portion of the aqueous extract is analyzed for total halides by potentiometric titration.

4. Significance and Use

4.1 A knowledge of water extractable inorganic halides in oil is important when deciding whether or not the oils need desalting. Excessive halide, especially in crude oil, frequently results in higher corrosion rates in refining units.

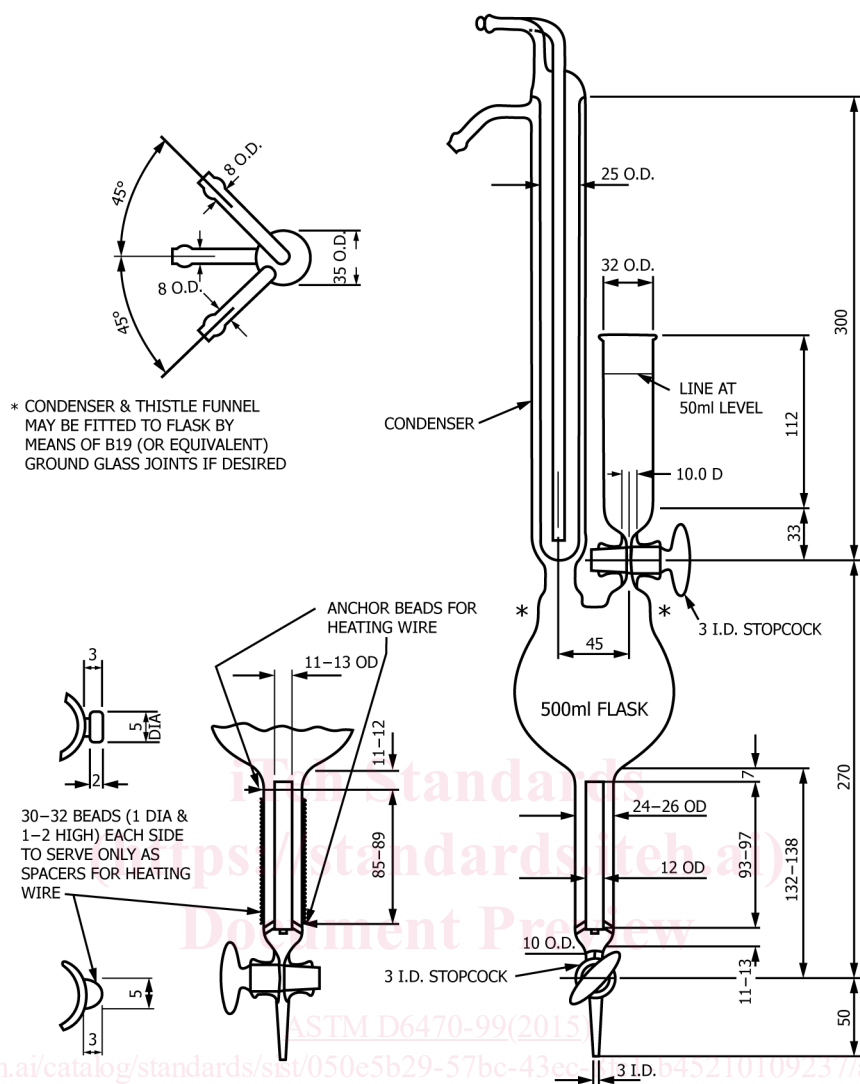
5. Apparatus

5.1 *Extraction Apparatus*, made of borosilicate glass, conforming to the dimensions given in [Fig. 1](#), and consisting of the following component parts:

¹ This test method is under the jurisdiction of ASTM Committee [D02](#) on Petroleum Products—Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee [D02.03](#) on Elemental Analysis.

Current edition approved ~~May 1, 2010~~ April 1, 2015. Published May ~~2010~~ 2015. Originally approved in 1999. Last previous edition approved in ~~2004~~ 2010 as ~~D6470~~ D6470 – 99 (2010). DOI: ~~10.1520/D6470-99R10~~ 10.1520/D6470-99R15.

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.



All dimensions are in millimetres.

NOTE 1—Hopkins-type condenser is used.

FIG. 1 Extraction Apparatus

5.1.1 *Boiling Flask*, 500 mL–500 mL capacity.

5.1.2 *Hopkins Reflux Condenser*, having a vapor outlet connected by a rubber tube to an outside vent or to a suction hood.

5.1.3 *Thistle Tube*, approximately 70 mL–70 mL capacity, with a line to indicate approximately the 50 mL–50 mL level.

5.1.4 *Heating Tube*, containing a chimney for increasing convection in the liquid.

5.1.5 *Heating Coil*, 250 W–250 W, consisting of a suitable gage of Nichrome wire.

5.1.6 *Rheostat*, of suitable resistance and capacity, for regulating the heater.

5.2 *Safety Shield*, colorless safety glass, or equivalent, to be mounted in front of the extraction apparatus (see 5.1).

5.3 *Sampling Tube*, glass, length approximately 600 mm–600 mm, I.D. approximately 5 mm–5 mm, with a bulb having a volume of 100 mL–100 mL, or more, and drawn out at one end to an opening of inside diameter (I.D.) 22 mm to 3 mm–3 mm. A pipette with cut-off tip makes a suitable sample tube.

5.4 *Potentiometric Titration Equipment*, with a measuring accuracy of ± 2 mV, ± 2 mV, or better, provided with a silver indicating and a glass reference electrode and 10 mL–10 mL burette, preferably piston type. If an automatic titrator is used, this shall be capable of adding fixed increments of titrant (see 9.3.3.2).

5.5 *Magnetic Stirrer*, with polytetrafluoroethylene (PTFE)-coated stirring bar.

5.6 *Homogenizer*. A mixer with counter-rotating blades operating at approximately 3000–3000 r/min–min (50/s) is usually suitable for homogenization of samples up to 500 mL–500 mL. Other designs can also be used provided the performance conforms to the requirements described in Annex A1.

5.7 *Oven*, explosion-proof, temperature $65 \pm 5^\circ\text{C}$; $5 \pm 5^\circ\text{C}$.

5.8 *Filter Paper*, Whatman No. 41, or equivalent.

5.9 *Stopwatch*.

6. Reagents and Materials

6.1 *Purity of Reagents*—Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.³

6.2 *Purity of Water*—For all purposes where water is mentioned, reagent water of a suitable purity shall be used. Various types of reagent water are described in Specification **D1193**.

6.3 *Acetone* (2-propanone), conforming to Specification **D329**. (**Warning**—Extremely flammable. Vapors may cause flashfire.)

6.4 *Alcohol*, for example, 95 % (V/V) ethanol, or propan-2-ol (isopropyl alcohol), conforming to Specification **D770**. (**Warning**—Flammable.)

6.5 *Barium Nitrate*, A.R., crystals. (**Warning**—Barium compounds and their solutions present a health risk if incorrectly handled. Prevent all contact.)

6.6 *Hydrochloric Acid*, 10.1 mol/L , aqueous. Add 9 mL of A.R. concentrated hydrochloric acid (density 1.19 g/mL) to 1 L with water. (**Warning**—Corrosive. Causes skin burns.)

6.7 *Nitric Acid*, 55 mol/L , aqueous. Cautiously add 325 mL of A.R. concentrated nitric acid (density 1.42 g/mL) to 1 L water, while stirring. (**Warning**—Corrosive. Causes skin burns.)

6.8 *Silver Nitrate Solution*, standard, $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$, aqueous. Prepare, standardize and store as described in Practice **E200** for 0.1 N aqueous solution, reading concentrations in mol/L in place of normality. Restandardize regularly, but in any case before preparation of the standard 0.01 mol/L solution (see **6.9**)

NOTE 1—Alternatively, ampoules containing concentrated solutions for preparation of standard volumetric solutions are available from various suppliers. (**Warning**—Silver compounds and their solutions present a health risk if incorrectly handled. Prevent all contact.)

6.9 *Silver Nitrate Solution*, standard, $c(\text{AgNO}_3) = 0.01 \text{ mol/L}$, aqueous. Prepare shortly before use by accurately diluting one volume of the recently restandardized 0.1 mol/L silver nitrate solution (**Warning**—see **6.8**) to a tenfold volume with water.

6.10 *Sodium Chloride Solution*, approximately 11 mmol/L , aqueous. Dissolve $5959 \text{ mg} \pm 1 \text{ mg}$ sodium chloride in 1 L water.

6.11 *Xylene*, conforming to Specification **D843**. (**Warning**—Xylene presents a health risk if incorrectly handled. Avoid inhalation. Extract vapor by working in a fume cupboard.)

6.12 *Lead Acetate Paper*.

6.13 *Polishing Paper*, 800 grit, or finer, to polish the silver electrode.

7. Sampling and Sample Preparation

7.1 Sampling is defined as all the steps required to obtain an aliquot representative of the contents of any pipe, tank, or other system, and to place the sample into the laboratory sample container. The laboratory sample container and sample volume shall be of sufficient dimensions and volume to allow mixing, as described in **7.4**. (**Warning**—The results of the round robin have shown that for reliable results, strict adherence to the sampling and mixing procedure is of the utmost importance.)

7.2 *Laboratory Sample*—The sample of crude oil presented to the laboratory or test facility for analysis by this test method. Only representative samples obtained as specified in Practices **D4057** and **D4177** shall be used for this test method.

7.3 *Test Sample*—The sample aliquot obtained from the laboratory sample for analysis by this test method. Once drawn, the entire portion of the test sample will be used in the analysis. Mix the laboratory sample properly, as described in **7.4**, prior to drawing the test sample.

7.4 Homogenize the laboratory sample of crude oil immediately (within 15 min) before drawing the test sample to ensure complete homogeneity. Mix the sample at room temperature (15°C to 25°C), or less, in the laboratory sample container, and record the temperature of the sample in degrees Celsius immediately before mixing. Heat waxy samples, solid at room temperature, to 3°C above their pour point in order to facilitate test sample withdrawal. Select the type of mixer related to the quantity of crude oil in the laboratory sample container. Before any unknown mixer is used, the specifications for the

³ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.