



Standard Test Methods for Determination of Organic Chloride Content in Crude Oil¹

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1. Scope*

1.1 These test methods cover the determination of organic chloride (above 1 $\mu\text{g/g}$ organically-bound chlorine) in crude oils, using either distillation and sodium biphenyl reduction or distillation and microcoulometry.

1.2 These test methods involve the distillation of crude oil test specimens to obtain a naphtha fraction prior to chloride determination. The chloride content of the naphtha fraction of the whole crude oil can thereby be obtained. See Section 5 regarding potential interferences.

1.3 Test Method A covers the determination of organic chloride in the washed naphtha fraction of crude oil by sodium biphenyl reduction followed by potentiometric titration.

1.4 Test Method B covers the determination of organic chloride in the washed naphtha fraction of crude oil by oxidative combustion followed by microcoulometric titration.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard. The preferred concentration units are micrograms of chloride per gram of sample.

1.6 *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

[D86 Test Method for Distillation of Petroleum Products at Atmospheric Pressure](#)

[D1193 Specification for Reagent Water](#)

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

¹ These test methods are under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and are the direct responsibility of Subcommittee D02.03 on Elemental Analysis.

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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.

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

[D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance](#)

3. Summary of Test Method

3.1 A crude oil distillation is performed to obtain the naphtha cut at 204°C (400°F). The distillation method was adapted from Test Method [D86](#) for the distillation of petroleum products. The naphtha cut is washed with caustic, repeatedly when necessary, until all hydrogen sulfide is removed. The naphtha cut, free of hydrogen sulfide, is then washed with water, repeatedly when necessary, to remove inorganic halides (chlorides).

3.2 There are two alternative test methods for determination of the organic chloride in the washed naphtha fraction, as follows.

3.2.1 *Test Method A, Sodium Biphenyl Reduction and Potentiometry*—The washed naphtha fraction of a crude oil specimen is weighed and transferred to a separatory funnel containing sodium biphenyl reagent in toluene. The reagent is an addition compound of sodium and biphenyl in ethylene glycol dimethyl ether. The free radical nature of this reagent promotes very rapid conversion of the organic halogen to inorganic halide. In effect this reagent solubilizes metallic sodium in organic compounds. The excess reagent is decomposed, the mixture acidified, and the phases separated. The aqueous phase is evaporated to 25 to 30 mL, acetone is added, and the solution titrated potentiometrically.

3.2.2 *Test Method B, Combustion and Microcoulometry*—The washed naphtha fraction of a crude oil specimen is injected into a flowing stream of gas containing about 80 % oxygen and 20 % inert gas, such as argon, helium, or nitrogen. The gas and sample flow through a combustion tube maintained at about 800°C. The chlorine is converted to chloride and oxychlorides, which then flow into a titration cell where they react with the silver ions in the titration cell. The silver ions thus consumed are coulometrically replaced. The total current required to replace the silver ions is a measure of the chlorine present in the injected samples.

3.2.3 The reaction occurring in the titration cell as chloride enters is as follows:

*A Summary of Changes section appears at the end of this standard.