



Designation: D8304 – 19

Standard Test Method for Determination of Compounded Forms of Iron in In-service Fluids¹

This standard is issued under the fixed designation D8304; 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 method covers a procedure for determining the concentration of compounded forms of iron in in-service fluids. It is applicable over an iron concentration range of 20 mg/kg to 800 mg/kg (ppm by mass hereafter). The test is not sensitive to metallic iron and thus provides a means of discrimination between the two forms of iron. It is suitable for monitoring the condition of equipment where iron contamination in the lubricating oil or functional fluid may not originate solely from physical wear, for example from corrosion or other forms of undesirable chemical processes within the equipment.

1.2 *Units*—The values stated in SI units are to be regarded as standard. Concentrations are reported as parts per million by mass (ppm) equivalent to iron contents expressed in mg per kg of oil sample (mg/kg).

1.3 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*²

[D1193 Specification for Reagent Water](#)

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

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

[D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants](#)

[E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods](#)

3. Terminology

3.1 *Definitions:*

3.1.1 *condition monitoring, n*—the recording and analyzing of data relating to the condition of equipment or machinery for the purpose of predictive maintenance or optimization of performance.

3.1.2 *machinery health, n*—qualitative indication of the overall condition of equipment or machinery. May depend on data and trend analysis from several sources.

3.1.3 *trend analysis, n*—the interpretation of regular or continuous (in time) condition monitoring data in order to determine any changes indicative of deterioration or incipient failure in equipment or machinery.

3.1.4 *wear, n*—the loss of material from a surface, generally occurring between two surfaces in relative motion, and resulting from mechanical or chemical action, or a combination of both.

4. Summary of Test Method

4.1 A sample of oil is extracted from the equipment and collected in a clean sample bottle. Two small, but equal volumes of the sample are subsequently transferred to two clean optical cuvettes. In one, a combined reagent is added. This combined reagent provides:

4.1.1 a means for reducing any iron present in an oxidation state of +3, ferric iron, to an oxidation state of +2, ferrous iron,

4.1.2 a means of transferring the ferrous iron ions from the oil phase to the aqueous phase of the combined reagent,

4.1.3 a complexing reagent which can react with the ferrous iron ions to form a product having a purple color of peak wavelength 538 nm.

4.2 In the second cuvette, a blank reagent is added. This provides all of the above apart from the complexing reagent in step iii. The intensity of the color developed in the sample

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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.

cuvette is related to the concentration of the ferric and ferrous iron ions in the original oil sample. By comparing this intensity against a calibrated color wheel in an optical comparator, a determination of the compounded iron concentration can be made. The blank cuvette provides a reference for the comparator.

4.3 Two test ranges are covered by this method:

4.3.1 20 ppm to 400 ppm by using a 0.2 mL sample of oil,

4.3.2 40 ppm to 800 ppm by using a 0.1 mL sample of oil.

5. Significance and Use

5.1 This test method is applicable for determining the level of iron contamination in lubricating oils from sources other than physical wear of the machinery or equipment, such as corrosion. It is particularly suited for oils from engines running on sulphur containing fuels where acidic combustion by-products can enter the lubrication system and cause corrosive damage. The test can be performed at the point of use as it does not require extensive laboratory equipment allowing timely intervention if necessary.

6. Interferences

6.1 Some anions and various cations are known to have interfering effects with the thioglycolic acid reagent³, either by enhancing or reducing the intensity of the developed color by the formation of similar complexes as can be formed with ferrous ions, or by causing precipitation. For those additives or contaminants likely to be found in in-service fluids, only the cations noted in the following table have an appreciable effect. The limits are stated as the mass of the cation compared to the mass of the compounded iron present in the sample beyond which a change in the color intensity would be noticeable and lead to an error in the iron determination exceeding 3 %.

Cation	Effect on color intensity	Limits (3 % error)
Cobalt	Enhances	2 % (factor of 0.02)
Nickel	Enhances	10 % (factor of 0.1)
Lead	Reduces	100 % (factor of 1)
Copper	Reduces	1000 % (factor of 10)
Bivalent Tin (Sn ²⁺)	Reduces	20 000 % (factor of 200)

7. Apparatus

7.1 Optical comparator equipped with a calibrated color wheel.⁴

7.2 Cuvettes or vials that fit in the comparator and are transparent over the visible wavelengths.

8. Reagents and Materials

8.1 Unless otherwise indicated, references to water shall be understood to mean Type I or better in Specification **D1193**.

8.2 Three reagents are required for the test comprising as follows:⁵

8.3 *Thioglycolic Acid Reagent*—Contains thioglycolic acid (C₂H₄O₂S) 1 % to 3 % (**Warning**—Toxic and skin corrosive), sodium chloride (NaCl) 10 % to 28 %, pH buffer of 2-aminoethanol (C₂H₇NO) (**Warning**—Toxic and skin corrosive) and ammonia (NH₃) solution (**Warning**—Skin corrosive) 1 % to 8 %, in water.

8.4 *Phase Transfer and Diluent Reagent*—2-propanol (C₃H₇O) (**Warning**—Flammable and eye irritant) 40 % to 75 %, non-polar solvent, for example, light distillate (for example, CAS Number: 64742-47-8) (**Warning**—Combustible, Toxic and irritant) 20 % to 50 %, water 0 % to 3 %.

8.5 *Blank Reagent*—As the thioglycolic reagent but omitting the thioglycolic acid.

9. Hazards

9.1 The specific hazards for the individual constituents of the test reagents are noted in Section 8, however the commercially available formulations contain the hazardous components at low concentrations and can be regarded as non-hazardous. Normal practice regarding the use of hand and eye protection should still be adopted when handling the reagents as a precautionary measure.

9.2 Suitable protective equipment should be worn when preparing the test specimen described in 10.2.

9.3 Disposal of the spent reagents and oil samples should be carried out in accordance with local regulations regarding waste oil and solvent disposal to prevent environmental damage.

10. Sampling, Test Specimens, and Test Units

10.1 In-service fluid samples should be collected in accordance with the practices described in Practices **D4057** or **D4177**, or both, ensuring that a representative sample is collected. The sample should be transferred to clean bottles and sealed until required for use. The minimum quantity of fluid required is 0.4 mL which will be divided into two aliquots for the sample and blank determination. However, it is recommended that a much greater volume than this is collected, approximately 100 mL. Before dividing the collected sample, ensure that it is thoroughly homogenized by shaking for approximately 30 s.

10.2 Test specimens can be prepared by dissolving a suitable soluble iron compound in water at specific concentrations to give the required iron ppm level expected after extraction from an oil sample. One suitable iron compound is Ferrous Sulphate Heptahydrate (FeSO₄·7H₂O, CAS 7782-63-0, molecular weight 278.01 g/mol) (**Warning**—Toxic, skin and eye irritant). This material is readily available and very soluble in

³ Swank, H.W. and Mellon, M.G., *Ing. Eng. Chem. Anal.*, Ed., 10, 7-9, 1938.

⁴ The sole source of supply of the apparatus known to the committee at this time is Parker Hannifin Ltd., Littlehampton, UK, BN17 7LU. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

⁵ The sole source of supply of the reagents known to the committee at this time is Parker Hannifin Ltd., Littlehampton, UK, BN17 7LU. The compositions of the reagents are covered by the following patents, US 9488633, EP 2881735, and others cited therein