



Test Method for Sampling and Testing for Ash and Total Iron in Steel Mill Dispersions of Rolling Oils¹

This standard is issued under the fixed designation D 4042; 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 describes a procedure for sampling and testing dispersions of rolling oils in water from operating steel rolling mills for determination of ash and total iron content. Its purpose is to provide a test method such that a representative sample may be taken and phenomenon such as iron separation, fat-emulsion separation, and so forth, do not contribute to analytical error in determination of ash and total iron.

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

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 and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Note 7, Note 10, Note 12, Note 13, and Note 14.

2. Referenced Documents

2.1 ASTM Standards:

- D 482 Test Method for Ash from Petroleum Products²
- D 1068 Test Methods for Iron in Water³
- D 1193 Specification for Reagent Water³
- D 2795 Test Method for Analysis of Coal and Coke Ash⁴
- E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals⁵
- E 832 Specification for Laboratory Filter Papers⁶

3. Summary of Test Method

3.1 Polyethylene disposable sample bags in a suitable holding device are used to collect representative samples of nonhomogeneous dispersions of rolling mill oils in water from an operating mill coolant system.

3.2 The collected sample and disposable bag are analyzed as one for ash and total iron.

3.2.1 Ash content is determined gravimetrically by evaporating all water from the sample bag before heating the residue and bag in a muffle furnace to a constant weight (Test Method D 482).

3.2.2 Total iron content is determined by digesting the iron in the ashed sample and bag in an acidified solution. The iron in the solution is reduced with stannous chloride and titrated with standardized potassium dichromate.

3.2.3 The ash and total iron contributed by a sample bag alone are determined separately and are subtracted from the values of bag and sample to obtain the values for the sample alone.

NOTE 1—There are a number of alternative methods available for determining total iron in an ashed solution such as Test Methods D 2795 and D 1068.

4. Significance and Use

4.1 The life cycle and cleanliness of a recirculating steel mill rolling oil dispersion is affected by the amount of iron present. This iron consists mainly of iron from acid pickling residues and iron from attrition of the steel sheet or rolls during cold rolling. In sampling rolling oils for total iron it is difficult to prevent adherence of iron containing sludge to the sample container. Thus, the accuracy of a total iron determination from an aliquot sample is suspect. This practice provides a means for ensuring that all iron contained in a sample is included in the analysis.

4.2 Although less significant, the ash content is still an essential part of the procedure for obtaining a total iron analysis. Generally, the ash will be mostly iron, and in many cases, could be used as a substitute for total iron in determining when to change the dispersion.

5. Sampling

5.1 Apparatus:

5.1.1 *Disposable Plastic (Polyethylene) Sampling Bags*⁷ 177 and 473 cm³ (6 and 16 oz) complete with closure wire.

5.1.2 *Holding Fixture.*

⁷ Sampling bags (with wires) can be found in most common laboratory supply catalogues.

¹ This practice is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.L on Industrial Lubricants (Joint ASTM-ASLE).

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² *Annual Book of ASTM Standards*, Vol 05.01.

³ *Annual Book of ASTM Standards*, Vol 11.01.

⁴ *Annual Book of ASTM Standards*, Vol 05.05.

⁵ *Annual Book of ASTM Standards*, Vol 03.05.

⁶ *Annual Book of ASTM Standards*, Vol 14.02.

NOTE 2—A plastic baby bottle holder with an open 177-cm³ (6-oz) sampling bag inserted makes a suitable holder.

5.1.3 Sampling Cup and Handle.

NOTE 3—A hockey stick or similar formed apparatus of steel, plastic, or wood with or without a separate cup can be used to hold the holding fixture or the fixture itself can be attached to the handle (see Fig. 1).

5.2 Sampling Procedure:

5.2.1 Weigh a number of identified sample bags on an analytical balance to the nearest 0.1 mg. Record the weight of each as W_{B1} .

5.2.2 Using a different, preweighed, identified plastic disposable sampling bag for each sample, remove samples from the mill only while the coolant is circulating and the mill running. Take samples from the same location between the second and third stand whenever possible. Fill the bag approximately two thirds full to allow for tying of the bag.

NOTE 4—Occasionally, due to mill configuration or other reasons, it may be advisable to obtain the sample from other points in the mill system. If so, record complete details on location and time of sampling.

5.2.3 Tie and seal each sample with the wire provided in another disposable plastic sampling bag 473 cm³ (16 oz) to prevent loss of material in transit. Individual rigid plastic bottles of suitable size are also satisfactory containers for shipment and can be substituted for the outer 473-cm³ (16-oz) plastic bag.

5.2.4 Take extreme care not to soil the exterior of the sampling bag with the dispersion or any foreign material.

6. Ash Content

6.1 Apparatus:

6.1.1 Borosilicate-Glass Beaker, 400-mL, or Evaporating Dish, 350-mL, designed for ashing in an electric furnace. Do not use platinum crucibles because of the tendency to alloy with metallic iron.

6.1.2 Electric Muffle Furnace, capable of maintaining a temperature of $775 \pm 25^\circ\text{C}$ ($1427 \pm 45^\circ\text{F}$), preferably having suitable apertures at the front and rear that allow a slow natural draft of air to pass through.

6.1.3 Oven, capable of maintaining a temperature of $105 \pm 2^\circ\text{C}$ ($221 \pm 4^\circ\text{F}$).

6.1.4 Analytical Balance, 200-g capacity, accurate to 0.1 mg.

6.1.5 Top-Loading Balance, 600-g minimum capacity, accurate to 0.01 g.

6.2 Procedure:

6.2.1 Heat the empty beaker or evaporating dish at 700 to 800°C (1240 to 1470°F) for 10 min or more. Cool at room temperature in a suitable container (Note 5) and weigh on an analytical balance to the nearest 0.1 mg.

NOTE 5—The container in which the beaker or evaporating dish is cooled should not contain a desiccating agent.

6.2.2 Remove the bagged sample from its carrier container and place it, still tied, in the dish. Remove the wire and weigh the bag with sample on a top loading balance to the nearest 0.01 g. Subtract the weight of the beaker or evaporating dish and record the weight in grams as W_{S1} .

6.2.3 Place the beaker or dish, sample, and bag in an oven at 105°C (221°F) overnight or until the moisture has been evaporated from the sample.

6.2.4 Cover the beaker or evaporating dish with ashless filter paper (Note 6) to prevent loss due to spatter. Add this filter paper to the sample prior to ashing.

NOTE 6—Filter paper should conform to Specification E 832, Type II.

6.2.5 Place the beaker or evaporating dish containing the dried sample, bag, and filter paper in the muffle furnace. Carefully heat the beaker or evaporating dish and sample by progressively advancing it into the muffle furnace where it will eventually ignite.

6.2.5.1 Maintain at such a temperature that the sample continues to burn at a uniform and moderate rate, leaving only ash and carbon when burning ceases.

NOTE 7—Warning: Fumes from burning polyethylene are hazardous.

6.2.6 Heat the residue in the muffle furnace at $775 \pm 25^\circ\text{C}$ ($1427 \pm 45^\circ\text{F}$) until all carbonaceous material has disappeared.

6.2.7 Remove the dish and ashed sample from the furnace and cool in a suitable container to room temperature. Weigh on a 200-g analytical balance to the nearest 0.1 mg.

6.2.8 Reheat the dish to $775 \pm 25^\circ\text{C}$ ($1427 \pm 45^\circ\text{F}$) in the muffle furnace for 30 min, and cool to room temperature in a suitable container and reweigh, as in 6.2.6. Repeat the heating, cooling, and weighing until consecutive weighings differ by not more than 0.5 mg.

6.2.9 Subtract the weight of the evaporating dish (6.2.1) and record the final ash weight as W_{S2} .

6.2.10 Place a new, empty polyethylene bag trimmed to the weight of the original sample bag ± 0.01 g (5.2.2) in a preweighed evaporating dish (6.2.1) and reduce to ash following the procedure described by 6.2.4, 6.2.5, 6.2.6, 6.2.7, and 6.2.8.

6.2.11 Record the final ash weight in grams to the nearest 0.1 mg as W_{B2} .

NOTE 8—In repeat tests for duplicate samples or separate samples, if all sample bags used are from the same source and are of the same size, an average weight from three or more bags can be used for W_{B1} . This avoids having to preweigh all bags as in 5.2.2. Also, the weight of the ashed

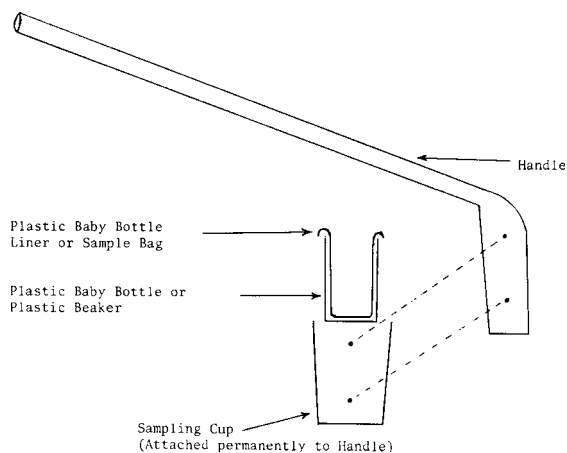


FIG. 1 Possible Holding Fixture and Assembly System