



Designation: D482 – 13



Designation: 4/96

Standard Test Method for Ash from Petroleum Products¹

This standard is issued under the fixed designation D482; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope*

1.1 This test method covers the determination of ash in the range 0.001–0.180 mass %, from distillate and residual fuels, gas turbine fuels, crude oils, lubricating oils, waxes, and other petroleum products, in which any ash-forming materials present are normally considered to be undesirable impurities or contaminants (Note 1). The test method is limited to petroleum products which are free from added ash-forming additives, including certain phosphorus compounds (Note 2).

NOTE 1—In certain types of samples, all of the ash-forming metals are not retained quantitatively in the ash. This is particularly true of distillate oils, which require a special ash procedure in order to retain metals quantitatively.

NOTE 2—This test method is not intended for the analysis of unused lubricating oils containing additives; for such samples use Test Method D874. Neither is it intended for the analysis of lubricating oils containing lead nor for used engine crankcase oils.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only. The preferred expression of the property is mass %.

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.*

2. Referenced Documents

2.1 ASTM Standards:²

D874 Test Method for Sulfated Ash from Lubricating Oils and Additives

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 D02.03 on Elemental Analysis.

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In the IP, this test method is under the jurisdiction of the Standardization Committee. This test method was adopted as a joint ASTM-IP standard in 1965.

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.

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D4928 Test Method for Water in Crude Oils by Coulometric Karl Fischer Titration

D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

D6792 Practice for Quality System in Petroleum Products and Lubricants Testing Laboratories

3. Summary of Test Method

3.1 The sample contained in a suitable vessel is ignited and allowed to burn until only ash and carbon remain. The carbonaceous residue is reduced to an ash by heating in a muffle furnace at 775°C, cooled and weighed.

4. Significance and Use

4.1 Knowledge of the amount of ash-forming material present in a product can provide information as to whether or not the product is suitable for use in a given application. Ash can result from oil or water-soluble metallic compounds or from extraneous solids such as dirt and rust.

5. Apparatus

5.1 *Evaporating Dish or Crucible*, made of platinum, silica, or porcelain, of a capacity of 50 to 150 mL.

NOTE 3—Alumina vessels may be used where appropriate. The use of vessels that are not made of platinum may contaminate the ash.

5.2 *Electric Muffle Furnace*, capable of maintaining a temperature of 775 ± 25°C and preferably having suitable apertures at the front and rear so as to allow a slow natural draught of air to pass through or a programmable furnace controlling both temperature and air flow is suitable.

5.3 *Meeker Gas Burner*, or equivalent.

5.4 *Mechanical Shaker*.

5.5 *Filter Paper*.

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

6. Reagents

6.1 *Propan-2-ol*— (**Warning**—Flammable, can be explosive when evaporated to or near dryness.)

6.2 *Toluene*—(**Warning**—Flammable, toxic.)

6.3 *Quality Control (QC) Samples*, preferably are portions of one or more liquid petroleum materials that are stable and representative of the samples of interest. These QC samples can be used to check the validity of the testing process as described in Section 11.

7. Sampling

7.1 Obtain samples in accordance with the instructions in Practice D4057 or D4177. Before transferring the portion of the sample to be ashed to the evaporating dish or crucible, take particular care to ensure that the portion taken is truly representative of the larger portion. Vigorous shaking can be necessary.

8. Procedure

8.1 Heat the evaporating dish or crucible that is to be used for the test at 700 to 800°C for a minimum of 10 min. Cool to room temperature in a suitable container, and weigh to the nearest 0.1 mg.

NOTE 4—The container in which the dish or crucible is cooled can be a desiccator not containing a desiccating agent. In addition, all weighings of the crucibles should be performed as soon as the crucibles have cooled. If it should be necessary that the crucibles remain in the desiccator for a longer period, then all subsequent weighings should be made after allowing the crucibles and contents to remain in the desiccator for the same length of time.

8.2 When the sample is sufficiently mobile, mix thoroughly before weighing. The mixing is necessary to distribute catalyst fines and other particulate material throughout the sample. Satisfactory mixing can usually be achieved by 10 min of manual shaking or 10 min using a mechanical shaker. Examine the sample for homogeneity before proceeding with 8.3. Continue mixing the sample if it is not homogeneous.

8.2.1 When it is evident that the sample is not homogenized after repeated mixings, or there is a reasonable doubt, a non-aerating, high-speed shear mixer can be used. Such a device is described in Annex A1 of Test Methods D4928.

8.2.2 When the sample cannot be satisfactorily homogenized, reject the sample and acquire a new sample.

8.2.3 When the sample is viscous or solid at room temperature, heat the container carefully until the sample is entirely liquid and mix carefully. An oven at an appropriate temperature can be used.

8.2.4 The sample can contain water. After heating in an oven, the water can boil causing splattering or foaming. The operator shall proceed cautiously with the heating step, wearing appropriate personnel protective equipment, such as safety goggles and gloves. Mixing this type of sample shall be done carefully. Stirring, rather than shaking, is an option.

8.3 The quantity of test specimen taken for testing will depend upon the ash content expected in the sample. Refer to Table 1. The weighing procedure will also depend upon whether the sample requires heating or not, and whether more than one portion has to be weighed.

TABLE 1 Test Specimen Mass versus Ash

| Expected Ash, mass % | Test Specimen, mass, g | Ash Mass, mg |
|----------------------|------------------------|--------------|
| 0.18 | 11 | 20 |
| 0.10 | 20 | 20 |
| 0.05 | 40 | 20 |
| 0.04 | 50 | 20 |
| 0.02 | 100 | 20 |
| 0.01 | 100 | 10 |
| 0.001 | 100 | 1 |

8.4 Using a top-loading balance, weigh into the dish or crucible sufficient test specimen to the nearest 0.1 g to yield no more than 20 mg of ash. Determine the mass of the test specimen used in the analysis at ambient temperature. One way to do this is to take the difference between the initial and final masses of the sample container weighed at ambient temperatures. If one weighing is sufficient, as determined from Table 1, or experience, proceed with steps 8.6 – 8.11.

8.5 If more than one addition of test specimen is required, proceed only through 8.6 (noting 8.6.1 and 8.7) and allow the dish or crucible to cool to ambient temperature before adding more sample as outlined in 8.4. Proceed with steps 8.6 – 8.11.

8.6 Carefully heat the dish or crucible with a Meeker burner or equivalent until the contents can be ignited by the flame. Maintain the dish or crucible at such a temperature that the sample continues to burn at a uniform and moderate rate leaving only a carbonaceous residue when the burning ceases. A hot plate can be used at this stage.

8.6.1 The test sample may contain water that can cause splattering. The operator shall heat the test portion cautiously while wearing appropriate personnel protective equipment, such as safety goggles and gloves. If splattering is very severe, so that material escapes the confines of the dish or the crucible, discard the test portion. If the first test portion is discarded, add 2 ± 1 mL of propan-2-ol (**Warning**—Flammable) to a second test portion while stirring with a glass rod and warming the test portion gently to liquefy if it is solid or near solid. Proceed as described in 8.6. If this is unsuccessful, repeat on a third test portion using a 10 ± 1 mL mixture of 50 ± 5 % by volume toluene (**Warning**— Flammable. Vapor harmful) and 50 ± 5 % by volume of propan-2-ol. In either case, any test specimen that adheres to the glass rod can be returned to the dish using a strip of ashless filter paper. Continue burning as outlined in 8.6.

8.7 Vigilance by the operator is mandatory; burning samples shall never be left unattended.

8.8 Some test specimens will require extra heating after the burning has ceased, particularly heavy samples such as marine fuels which form crusts over the unburned material. The crust can be broken with a glass rod. Any crust that adheres to the glass rod can be returned to the dish using a strip of ashless filter paper. Burn the remaining test specimen.

8.9 The heavier material tends to foam, therefore the operator shall exercise considerable care. Overheating shall be avoided so that neither the test specimen nor the dish are heated to a red hot appearance, as this can result in loss of ash.

Likewise, the flame shall never be higher than the rim of the dish to avoid superheating the crust, thereby producing sparks that can result in considerable loss of ash.

8.10 Heat the residue in the muffle furnace at $775 \pm 25^\circ\text{C}$ until all carbonaceous material has disappeared. Cool the dish to room temperature in a suitable container (Note 4), and weigh to the nearest 0.1 mg.

8.11 Reheat the dish at $775 \pm 25^\circ\text{C}$ for at least 20 min, cool in a suitable container (Note 4), and reweigh. Repeat the heating, cooling, and weighing process until consecutive weighings differ by not more than 0.5 mg.

9. Calculation

9.1 Calculate the mass of the ash as a percentage of the original samples as follows:

$$\text{Ash, mass \%} = (w/W) \times 100 \quad (1)$$

where:

w = mass of ash, g, and
 W = mass of sample, g.

10. Report

10.1 Report the results as follows:

| Test Specimen Mass | Report |
|--------------------|-----------------------|
| 9.00 to 39.99 g | 3 decimal places |
| 40.00 or more g | 3 to 4 decimal places |

10.2 Record the value reported as ash in accordance with Test Method D482, stating the mass of the sample taken.

11. Quality Control:

11.1 Confirm the performance of the instrument or the test procedure by analyzing a QC sample (see 6.3).

11.1.1 When QC/Quality Assurance (QA) protocols are already established in the testing facility, these may be used to confirm the reliability of the test result.

11.1.2 When there is no QC/QA protocol established in the testing facility, Appendix X1 can be used as the QC/QA system.

12. Precision and Bias³

12.1 The precision of this test method as obtained by statistical examination of interlaboratory test results is as follows:

12.1.1 *Repeatability*—The difference between successive tests results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

| Ash, mass % | Repeatability |
|----------------|---------------|
| 0.001 to 0.079 | 0.003 |
| 0.080 to 0.180 | 0.007 |

12.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

| Ash, mass % | Reproducibility |
|----------------|-----------------|
| 0.001 to 0.079 | 0.005 |
| 0.080 to 0.180 | 0.024 |

12.2 *Bias*—The bias of this test method cannot be determined since an appropriate standard reference material containing a known level of ash in liquid petroleum hydrocarbon is not available.

13. Keywords

13.1 ash; crude oils; distillate oils; fuel oils; lubricating oils

³ No ASTM Research Report is available for this standard.

APPENDIX

(Nonmandatory Information)

X1. QUALITY CONTROL

X1.1 Confirm the performance of the instrument or the test procedure by analyzing a QC sample.

X1.2 Prior to monitoring the measurement process, the user of the test method needs to determine the average value and control limits of the QC sample (see Practices D6299 and D6792 and MNL 7).⁴

X1.3 Record the QC results and analyze by control charts or other statistically equivalent techniques, to ascertain the statistical control status of the total testing process (see Practices D6299 and D6792 and MNL 7). Any out-of-control data should

trigger investigation for root cause(s).

X1.4 In the absence of explicit requirements given in the test method, the frequency of QC testing is dependent on the criticality of the quality being measured, the demonstrated stability of the testing process, and customer requirements. Generally, a QC sample is analyzed each testing day with routine samples. The QC frequency should be increased if a large number of samples are routinely analyzed. However, when it is demonstrated that the testing is under statistical control, the QC testing frequency may be reduced. The QC sample precision should be checked against the ASTM method precision to ensure data quality.

X1.5 It is recommended that, if possible, the type of QC sample that is regularly tested be representative of the material

⁴ ASTM MNL 7, *Manual on Presentation of Data Control Chart Analysis*, 6th ed., ASTM International, W. Conshohocken, PA.

routinely analyzed. An ample supply of QC sample material should be available for the intended period of use, and must be homogenous and stable under the anticipated storage condi-

tions. See Practice **D6299** and **D6792** and MNL 7 for further guidance on QC and Control Charting techniques.

SUMMARY OF CHANGES

Subcommittee D02.03 has identified the location of selected changes to this standard since the last issue (D482 – 12) that may impact the use of this standard.

(1) Added information in subsections **5.1**, **5.2**, and **5.5** in Apparatus section to keep consistent with equivalent test method IP 4.

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