



# Standard Test Method for Ash from Petroleum Products<sup>1</sup>

This standard is issued under the fixed designation D 482; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the 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 **D 874**. Neither is it intended for the analysis of lubricating oils containing lead nor for used engine crankcase oils.

1.2 SI units are regarded as the standard. 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:

- D 874** Test Method for Sulfated Ash from Lubricating Oils and Additives<sup>2</sup>
- D 4057** Practice for Manual Sampling of Petroleum and Petroleum Products<sup>3</sup>
- D 4177** Practice for Automatic Sampling of Petroleum and

### Petroleum Products<sup>3</sup>

- D 4928** Test Methods for Water in Crude Oils by Coulometric Karl Fischer Titration<sup>3</sup>
- D 6299** Practice for Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance<sup>4</sup>

## 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 90 mL minimum capacity to 120-mL maximum capacity.

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.

5.3 *Meeker Gas Burner*, or equivalent.

5.4 *Mechanical Shaker*.<sup>5</sup>

## 6. Reagents

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

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products 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.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 05.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 05.02.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 05.03.

<sup>5</sup> The sole source of supply of the mechanical shaker known to the committee at this time is the Eberbach Corp., 505 S. Maple Rd., Ann Arbor, MI 48106-1024. 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, <sup>1</sup> which you may attend.

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

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 D 4057 or D 4177. 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 3—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 D 4928.

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.

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

**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

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  $2 \pm 1$  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  $10 \pm 1$  mL mixture of  $50 \pm 5$  % by volume toluene (**Warning**—Flammable. Vapor harmful) and  $50 \pm 5$  % 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 3), and weigh to the nearest 0.1 mg.

8.11 Reheat the dish at  $775 \pm 25$  °C for at least 20 min, cool in a suitable container (**Note 3**), 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 D 482, 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 Bias <sup>6</sup>

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

<sup>6</sup> 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 Practice **D 6299** and MNL 7).<sup>7</sup>

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 Practice **D 6299** 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 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 conditions. See Practice **D 6299** and MNL 7 for further guidance on QC and Control Charting techniques.

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

## SUMMARY OF CHANGES

Subcommittee D02.03 has identified the location of selected changes to this standard since the last issue (D 482–02) that may impact the use of this standard.

- (1) Updated Table 1 to correct the “Test Specimen, mass, g” of 0.18 to align with the calculated value determined by Eq 1. value from “9” to “11” for an “Expected Ash, mass %” value

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