Designation: D7626 – 19ε1

Standard Test Methods for Determining the Organic Treat Loading of Organophilic Clay[1]

This standard is issued under the fixed designation D7626; 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.

ε[1] NOTE—Corrected data sheet editorially in Appendix X1 in February 2020.

1. Scope

1.1 This standard covers two index test methods that can be used in the evaluation of the amount of organic compound chemically bonded to the base clay portion of a representative sample of organophilic clay.

1.2 The values stated in SI units are to be regarded as the standard. No other units of measurement are included in this standard. Reporting of test results in units other than SI shall not be regarded as nonconformance with this standard.

NOTE 1—This standard is presented using SI units. Use of units other than SI is allowed. However, if other units are used, the performance of a units conversion check of the calculations should be included as a part of the calculations.

1.3 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D6026.

1.3.1 Two test methods are provided in this standard. The methods differ in equipment, the size of the specimen (mass) required and the significant digits reported.

1.3.2 The procedures used to specify how data are collected/recorded or calculated in this standard are regarded as the industry standard. In addition, they are representative of the significant digits that generally should be retained. The procedures used do not consider material variation, purpose for obtaining the data, special purpose studies, or any considerations for the user's objectives; and it is common practice to increase or reduce significant digits of the reported data to be commensurate with these considerations. It is beyond the scope of this standard to consider significant digits used in analysis methods for engineering design.

1.4 *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.*

1.5 *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]

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction

D4753 Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing

D6026 Practice for Using Significant Digits in Geotechnical Data

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

E145 Specification for Gravity-Convection and Forced-Ventilation Ovens

3. Terminology

3.1 *Definitions*—For definitions of common technical terms in this standard, refer to Terminology D653.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *base clay, n*—an untreated naturally occurring clay (for example, bentonite, kaolinite).

3.2.2 *initial mass loss, n*—the amount of water and residual organic compound not bound to the base clay.

[1] These test methods are under the jurisdiction of ASTM Committee D18 on Soil and Rock and are the direct responsibility of Subcommittee D18.21 on Groundwater and Vadose Zone Investigations.

Current edition approved Feb. 1, 2019. Published February 2019. Originally approved in 2010. Last previous edition approved in 2018 as D7626–10(2018). DOI: 10.1520/D7626-19E01.

[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.

3.2.3 *organic treat loading, n*—the amount of organic compound chemically bonded to the base clay portion of an organophilic clay.

3.2.4 *organophilic, adj*—having a strong affinity for oil and other insoluble organic compounds.

3.2.5 *organophilic clay, n*—a manufactured material consisting of base clay to which an organic compound has been chemically bonded to the clay surface.

4. Summary of Test Methods

4.1 *Test Method A*—Initial mass loss is determined by drying an organophilic clay specimen at 160 °C in an oven or muffle furnace. The initial mass loss is expressed as a percentage of the as-received mass. The ash content of the organophilic clay specimen is determined by igniting the dried specimen from the initial mass loss determination in a muffle furnace at 750 °C.

4.2 *Test Method B*—An organophilic clay specimen is heated at a controlled rate in a thermal gravimetric analyzer. Percent initial mass loss and ash are taken from thermal gravimetric analyzer report.

4.3 Some volatile structural matter within the base clay will be lost during the last stage of heating. Organic treat loading is determined on a dry weight basis by a calculation that accounts for the initial mass loss, ash, and a nominal percent of volatile structural matter within the base clay.

NOTE 2—The supplier of the organophilic clay must be able to verify with historical data the nominal percent mass of the base clay (on a dry weight basis) remaining after similar heating. This value is typically in the range of 94 to 96 %.

5. Significance and Use

5.1 This standard test method is intended as an index test to determine the organic treat loading of organophilic clay. This standard test method can be used for manufacturing quality control and construction quality assurance material evaluation.

5.2 The percent organic treat loading of organophilic clay is a relative indicator of its adsorptive capacity. Organophilic clay is used for remediation of contaminated sediment, soil, and groundwater.

5.3 The two test methods denote different devices, a muffle furnace and a thermal gravimetric analyzer. The thermal gravimetric analyzer may be programmed to reach a higher temperature than the muffle furnace, but the organic matter will be burnt off at 750 °C.

NOTE 3—The quality of the result produced by this standard is dependent on the competence of the personnel performing it, and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D3740 are generally considered capable of competent and objective testing/sampling/inspection/etc. Users of this standard are cautioned that compliance with Practice D3740 does not in itself assure reliable results. Reliable results depend on many factors; Practice D3740 provides a means of evaluating some of those factors.

6. Apparatus

6.1 *Oven,* meeting the requirements of Specification E145 and capable of being regulated to a constant temperature of 160 ± 5 °C.

6.2 *Muffle Furnace,* capable of producing constant temperature of 750 °C.

6.3 *TGA,* thermal gravimetric analyzer capable of reaching a minimum 750 °C with a balance having 0.001 mg readability and any ancillary equipment, such as instrumental air, as recommended by the TGA manufacturer.

6.4 *Balance or Scale,* a balance or scale for determining the mass of organophilic clay having a minimum capacity of 100 g and meeting the requirements of Guide D4753 for a balance or scale of 0.01 g readability.

6.5 *Porcelain Container and Cover.*

6.6 *Desiccator.*

6.7 *Mortar and Pestle.*

6.8 *Sieve,* 0.075 mm sieve opening, meeting the requirements of Specification E11.

6.9 *Pan,* slightly larger than sieve.

6.10 *Gloves, Tongs or Other Suitable Equipment or Combinations Thereof,* to safely handle porcelain containers to and from furnace.

7. Sample and Specimen

7.1 Collect a representative sample of organophilic clay. This test method does not address, in any detail, procurement of the sample. It is assumed the sample is obtained using appropriate methods and is representative of the material under evaluation. From the sample obtained from manufacturer, obtain a 50 g sub-sample.

7.2 *Specimen*—Obtain a representative portion from the sub-sample to provide sufficient material passing the 0.075 mm sieve.

7.2.1 Crush organophilic clay sample with mortar and pestle and then screen through 0.075 mm sieve. Retain finer fraction of sub-sample from which the test specimen is to be obtained. Unless otherwise requested, only one specimen is used in the test procedure.

8. Procedures

8.1 *Test Method A—Oven or Muffle Furnace or Combinations Thereof:*

8.1.1 *Initial Mass Loss Determination:*

8.1.1.1 Record to the nearest 0.01 g the mass of a clean, dry porcelain container and cover.

8.1.1.2 Place a test specimen of at least 10 g and no more than 50 g in the porcelain container and record mass to the nearest 0.01 g.

8.1.1.3 Dry uncovered in an oven or muffle furnace for at least 16 h at 160 °C or until there is no change in mass of the specimen after further drying in periods in excess of 1 h.

8.1.1.4 Remove from the oven, cover tightly, cool in a desiccator and record the mass (m_{dry}) to the nearest 0.01 g.

8.1.1.5 Calculate the initial mass loss as percent of as-received mass as follows:

$$M = [(m_i - m_{dry}) \times 100]/m_i \quad (1)$$

where:

M = initial mass loss, as percent of as-received test specimen,
m_i = mass of the as-received test specimen, g, and
m_{dry} = mass of the dried specimen, g.

8.1.2 *Organic Treat Loading Determination:*

8.1.2.1 Place the entire oven-dried specimen and porcelain container from the initial mass loss determination in a muffle furnace. Raise the temperature in the furnace to 750 °C.

8.1.2.2 Heat at 750 °C for a minimum of 1 h. Remove the container, cool in a desiccator, and weigh and record the mass.

8.1.2.3 Replace into furnace and repeat step 8.1.2.2 until weight loss is less than 0.05 % over at least 1 h of heating.

8.1.2.4 Remove the container, cool in a desiccator, and weigh and record the mass of dried ash (m_{ash}) to the nearest 0.01 g.

8.1.2.5 Determination of the Organic Treat Loading requires several computation steps:

Calculate the percent dry weight ash content as follows:

$$\%\ ash_{dry} = (m_{ash} \times 100)/m_{dry} \quad (2)$$

where:

$\%\ ash_{dry}$ = ash content as percent on a dry weight basis,
m_{ash} = mass of ash, g, and
m_{dry} = mass of the dried test specimen, g.

Calculate percent dry base clay as follows:

$$\%\ base\ clay_{dry} = \frac{\%\ ash_{dry}}{\%\ ash_{dry}\ of\ base\ clay} \times 100\,\% \quad (3)$$

where:

$\%\ ash_{dry}\ of\ base\ clay$ = nominal percent of mass of base clay remaining as ash (on a dry weight basis) after similar heating as above, as reported by manufacturer (see 4.3, Note 2), and
$\%\ base\ clay_{dry}$ = percent of mass attributable to base clay on a dry weight basis.

Calculate the Organic Treat loading as follows:

$$\%\ organic\ treat\ loading = 100\,\% - \%\ base\ clay_{dry} \quad (4)$$

where:

$\%\ organic\ treat\ loading$ = percent of organic treat loading on a dry weight basis, and
$\%\ base\ clay_{dry}$ = percent of mass attributable to base clay on a dry weight basis.

8.2 *Test Method B—Thermal Gravimetric Analyzer (TGA):*

8.2.1 Use compressed air as the gas source. Check that air pump is connected and on. Check the gas flow rate ensuring flow is in accordance with manufacturer's standard operating procedure.

8.2.2 Tare a clean, dry porcelain container in the TGA device.

8.2.3 Remove the container from the TGA device and insert a test specimen of at least 50 mg in the container.

8.2.4 Place the container and specimen into the TGA device and allow the balance to stabilize.

8.2.5 Heat at a rate of 20 °C per minute to 160 °C and hold until no further loss of mass and record percent mass loss data as M.

8.2.6 Heat at rate of 20 °C per minute to between 750 °C and 1000 °C.

8.2.7 Hold at final temperature until mass loss is less than 0.05 % over at least 3 min and record percent initial mass remaining data as $\%\ ash_{wet}$ (three significant figures).

8.2.8 *Organic Treat Loading Determination:*

8.2.8.1 Determination of the Organic Treat Loading requires several computation steps:

Determine the percent dry weight ash content:

$$\%\ ash_{dry} = \frac{\%\ ash_{wet}}{100\,\% - M} \times 100\,\% \quad (5)$$

where:

M = percent of initial mass loss determined at 160 °C as reported by TGA,
$\%\ ash_{wet}$ = percent of initial mass remaining as ash after heating to between 750 °C and 1000 °C as reported by TGA, and
$\%\ ash_{dry}$ = percent of mass on a dry weight basis remaining as ash after heating to between 750 °C and 1000 °C as reported by TGA.

NOTE 4—M is the initial mass loss as a percent of as-received mass. The initial mass loss analysis method with oven as described in 8.2.1 could be used to determine this value if it is difficult to obtain it from the TGA report.

Determine the percent dry base clay:

$$\%\ base\ clay_{dry} = \frac{\%\ ash_{dry}}{\%\ ash_{dry}\ of\ base\ clay} \times 100\,\% \quad (6)$$

where:

$\%\ ash_{dry}\ of\ base\ clay$ = nominal percent of mass of base clay remaining as ash (on a dry weight basis) after similar heating as above, as reported by manufacturer (see 4.3, Note 2), and
$\%\ base\ clay_{dry}$ = percent of mass attributable to base clay on a dry weight basis.

Calculate the Organic Treat loading as follows:

$$\%\ organic\ treat\ loading = 100\,\% - \%\ base\ clay_{dry} \quad (7)$$

where:

$\%\ organic\ treat$ = percent of organic treat loading on a dry weight basis, and
$\%\ base\ clay_{dry}$ = percent of mass attributable to base clay on a dry weight basis.

NOTE 5—This standard is presented using SI units. Use of units other than SI is allowed. However, if other units are used, the performance of a units conversion check of the calculations should be included as a part of the calculations.

9. Report: Test Data Sheet(s)/Form(s)

9.1 The methodology used to specify how data are recorded as given below, is covered in 1.3.

9.2 Record as a minimum the following general information (data):