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An American National Standard

Standard Test Method for Quantity of Water-Extractable Matter in Membrane Filters [1]

This standard is issued under the fixed designation D 3861; 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 covers the gravimetric determination of the water-extractable material present in membrane filters and is applicable over the complete concentration range of extractables.

1.2 The analyst should be aware that adequate collaborative data for precision and bias statements as required by Practice D 2777 is not provided. See Section 11 for details.

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]

D 1129 Terminology Relating to Water

D 1193 Specification for Reagent Water

D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D19 on Water

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D 1129.

4. Summary of Test Method

4.1 The quantity of water-soluble extractables present in membrane filters is determined by immersing the preweighed membrane in boiling reagent grade water for an extended time, then drying and reweighing the membrane. A control membrane is employed to eliminate weighing errors caused by balance changes or changing moisture content of the membrane in the weighing procedures. Weight changes of the control membrane are applied as a correction factor to the weight change of the test membrane filters.

5. Significance and Use

5.1 The presence of water-soluble extractables in membrane filters can create errors in test procedures employing membrane filters. However, these errors can be eliminated or significantly reduced if the quantity of water-soluble extractables of the specific membrane is previously determined. Certain membrane filter uses require specifications of maximum water-soluble extractable levels. This test method is intended to be a rapid test to determine the loss of water-soluble compounds such as plasticizers or wetting agents from filtration membranes. This test method is not designed to predetermine the performance of a filter, but is significant in determining the percent extractables of membranes from different sources and lot variations from a single source.

6. Apparatus

6.1 *Beaker*, borosilicate glass, 300-mL capacity.

6.2 *Forceps*, stainless steel, unserrated tips.

6.3 *Desiccator*.

6.4 *Drying Oven*, with thermostatic control.

6.5 *Analytical Balance*, sensitivity 0.01 mg.

6.6 *α-Emitting Polonium Source* [3] (to discharge static charge in balance).

6.7 *Humidity-Controlled Room or Hood*, for balance.

6.8 *Evaporating Dishes*, glass, having sides or other means to keep test filters on the dish while drying.

7. Reagents and Materials

7.1 *Purity of Water*— Unless otherwise indicated, references to water shall be understood to mean Type III reagent water conforming to Specification D 1193.

7.2 *Membrane Filters*, 47-mm diameter (same type as those under test).

8. Preparation of Apparatus and Materials

8.1 *Preparation of Samples and Apparatus*:

8.1.1 Select 47-mm diameter sample (Note 1) membrane filters for the test, and label with water-resistant ink.

NOTE 1—Square or rectangular membrane of equivalent area (17.4 cm^2) may be used.

[1] This test method is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.08 on Membranes and Ion Exchange Materials.

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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.

[3] The "Staticmaster," available from Nuclear Products Inc., El Monte, CA, has been found suitable for this purpose.

8.1.2 To provide a "control filter," select and label one additional 47-mm membrane filter of the same type as those being tested. Take the control filter through all the drying and weighing steps, but do not extract.

8.1.3 Set drying oven at 70°C.

9. Procedure

9.1 Heat the test and control filters in the oven at 70°C for 15 min.

9.1.1 Remove filters from the oven and place them in the desiccator for at least 30 min at room temperature.

9.1.2 Remove filters from desiccator and place them in balance room atmosphere for 10 to 15 min.

9.1.3 In a humidity-controlled balance room or hood, weigh test filters and control filter to 0.1 mg. Employ a static eliminator in the balance.

9.1.3.1 Place the "control" filter in the desiccator.

9.2 Heat 200 mL of the reagent grade water to boiling, insert test membrane filters, and continue heating with mild boiling for 30 min. No more than four membrane filters should be extracted in a single beaker of water. If water level is reduced beyond 50 % of original level, add water to bring volume to original level.

9.3 Remove the filters using unserrated flat-bladed forceps, place them on clean glass evaporating dishes, and dry them in the oven at 70°C for 60 ± 10 min.

9.3.1 Remove the test filters from the oven and examine them to determine that none of the filter material has broken off in the handling and extraction. If any filter has been damaged, discard it and select a replacement for test.

9.4 Place the test filters in the desiccator with the control filter for 30-min minimum time.

9.5 Place the filters in balance room or hood atmosphere for 10 to 15 min. Then weigh test and control filters to 0.1 mg to determine final weight.

10. Calculation

10.1 Determine weight change of the test filter by subtracting the final weight W_2 from the initial weight W_1 of the test filter.

10.2 Determine weight change of the control filter by subtracting the initial weight W_3 from the final weight W_4 of the control filter.

10.2.1 If the weight change of the control filter exceeds 2 mg, the test should be rerun.

10.3 Determine corrected weight change of the test filter by adding weight change of the control filter ($W_4 - W_3$) to the weight change of the test filter.

10.4 Determine percent extractables as follows:

$$E = \frac{(W_1 - W_2) + (W_4 - W_3)}{W_1} \times 100$$

where:

E = percent extractables,
W_1 = initial weight of test filter,
W_2 = final weight of test filter,
W_3 = initial weight of control filter, and
W_4 = final weight of control filter.

11. Precision and Bias

NOTE 2—The range of percent extractables for the samples tested for the round-robin test to determine precision was 1.06 to 3.09 % extractables.

11.1 The precision and bias of this test method was based upon five laboratories with five operators using membrane filters of three different micron ratings.

11.1.1 *Precision*:

11.1.1.1 The overall precision of this test method may be expressed as follows:

$$S_t = 0.54X - 0.37$$

where:

S_t = overall precision, %, and
X = concentration of water extractables.

11.1.1.2 The single-operator precision may be expressed as follows:

$$S_o = 0.64X - 0.61$$

where:

S_o = single-operator precision, %, and
X = concentration of water extractables.

11.1.2 *Bias*—Microporous membranes are inherently variable and therefore no true or absolute value can be established. A bias statement is not applicable to this procedure.

12. Keywords

12.1 extractable; filter; membrane