Designation: D5627 – 94 (Reapproved 2009)$^{\varepsilon 1}$

Standard Test Method for Water Extractable Residue from Particulate Ion-Exchange Resins[1]

This standard is issued under the fixed designation D5627; 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—Added research report information to Section 13 editorially in November 2010.

1. Scope

1.1 This test method covers the measurement of water soluble extractable residue from particulate ion-exchange resins based on elevated temperature extraction and gravimetric determination of residue.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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]

D1129 Terminology Relating to Water

D1193 Specification for Reagent Water

D2187 Test Methods for Physical and Chemical Properties of Particulate Ion-Exchange Resins

D2687 Practices for Sampling Particulate Ion-Exchange Materials

D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D1129.

4. Summary of Test Method

4.1 A sample of particulate ion exchange material is contacted with water at an elevated temperature. After a specified contact time, the concentration of soluble material in the aqueous phase is measured gravimetrically after filtration.

5. Significance and Use

5.1 The presence of water extractables in ion-exchange resins can cause fouling of other materials downstream and contamination of process water. The quantity of water extractables is sometimes used as a specification to indicate resin quality, and typical values are 0.01 to 0.1 %.

5.2 It is recognized that this test method may not remove all potential sloughage products and does not measure volatile compounds. More extensive extraction and identification of compounds may be needed in specific cases.

6. Interferences

6.1 The hygroscopic nature of some extracted compounds can make it difficult to obtain a constant weight. Other extraction times and temperatures may give results that are not comparable to these.

7. Apparatus

7.1 *Funnel,* 60 mL with coarse frit.

7.2 *Filtration assembly.*

7.3 *Glass fiber filters,* without binder, pre-rinsed with water.[3]

7.4 *Weighing vessel*—Platinum, nickel, or aluminum may be used so long as there is no attack by the sample and constant weight can be achieved.

7.5 *Water bath,* for use at 60 ± 1°C.

8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society,

---

[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.

Current edition approved May 1, 2009. Published June 2009. Originally approved in 1994. Last previous edition approved in 2004 as D5627 – 94 (2004). DOI: 10.1520/D5627-94R09E01.

[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] Gelman Type A/E, Millipore Type AP40, Whatman GF/C, or equivalent, have been found suitable for this purpose.

where such specifications are available.[4] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D1193, Type I or II.

9. Sampling

9.1 Collect the sample in accordance with Practices D2687. In-service resins are taken after regeneration and rinse.

10. Procedure

10.1 Drain the sample, if necessary, with the draining apparatus described in the draining apparatus section of Test Methods D2187 to remove free water. Do not wash.

10.2 Weigh (to the nearest 0.1 g) two 50-g portions of the drained sample into clean, dry, 250-mL Erlenmeyer flasks (with stoppers) or BOD bottles. Also weigh (to the nearest 5 mg) a 5-g portion of the drained sample for percent water retention capacity according to Test Methods D2187, Test Method B.

10.3 Transfer 200 mL of water by graduate to each of the two flasks containing sample. (Alternatively, measure the 200 mL of water by weight into each flask.) Prepare another flask and add 200 mL of water to serve as a blank. Stopper the flasks and maintain them at 60 ± 1°C for 18 ± 2 h using a water bath.

10.4 Dry three weighing vessels to constant weight (±1 mg) at 104 ± 2°C, cool in a desiccator and weigh to the nearest 0.1 mg. (Platinum is preferred, but nickel or aluminum may be used.)

10.5 Filter the entire volume of each extract using either a coarse fritted funnel or filter assembly with glass fiber filter. Withdraw a 100-mL aliquot and transfer to a tared weighing vessel. (The remainder of the filtered extracts can be tested for total organic carbon or other parameters as desired.)

10.6 Evaporate the water and then dry the weighing vessels to constant weight (±1 mg) at 104 ± 2°C (which can be done overnight). A hot plate can be used to evaporate nearly all the water so long as a layer of water covers the bottom of the vessel. Final drying is to be done in an oven. Weigh to the nearest 0.1 mg.

11. Calculation

11.1 Calculate the water extractable residue, in percent, for each of the two sample portions as follows:

$$\text{Water Extractable Residue, \% wet basis} = \frac{(A - B) \times 200}{C}$$

$$\text{Water Extractable Residue, \% dry basis} = \frac{(A - B) \times 200}{\left(C \times \frac{D}{100}\right)}$$

A = residue from sample, in g,
B = residue from blank, in g,
C = drained sample used, in g, and
D = percent solids, calculated as 100 % water retention capacity (from Test Methods D2187, Method B).

(These calculations assume a 100-mL aliquot out of 200 mL extract.)

12. Report

12.1 Report the percent water extractable residue as the average of the two values obtained, using the wet or dry, reporting basis or both, as required.

13. Precision and Bias[5]

13.1 Single-operator precision was determined using a composite sample of aged but unused strong acid, gel-type cation resin with an average solids content of 48.6 % by weight. The sample was analyzed in triplicate by six operators in one laboratory. The operators who participated represented a wide range of experience levels. Since other sources of variability should be relatively small (such as from the analytical balances), the multiple laboratory variability is expected to mimic the single-operator precision and is not separately determined. All determinations were made using disposable aluminum weighing dishes. The following results were obtained:

x = 0.393 % by weight, wet basis,
S_o = 0.019 % by weight, wet basis,
x = 0.812 % by weight, dry basis, and
S_o = 0.039 % by weight, dry basis

where x is the arithmetic mean of the 18 determinations and S_o is the single-operator precision calculated in accordance with D2777.

13.2 Since known standards are not available, bias cannot be determined.

14. Keywords

14.1 extractables; fouling; ion exchange; residue; sloughage

---

[4] *Reagent Chemicals, American Chemical Society Specifications* , American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D19-1159.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org). Permission rights to photocopy the standard may also be secured from the ASTM website (www.astm.org/COPYRIGHT/).