



Designation: D 6302 – 98 (Reapproved 2004)

Standard Practice for Evaluating the Kinetic Behavior of Ion Exchange Resins¹

This standard is issued under the fixed designation D 6302; 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 practice is intended to evaluate changes in kinetic performance of ion exchange resins used in mixed beds to produce high purity water. Within strict limitations, it also may be used for comparing resin of different types. This standard does not seek to mimic actual operating conditions. Specific challenge solutions and conditions are specified. At the option of the user, other conditions may be tested.

1.2 *This standard does not purport to address 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 2187 Test Methods for Physical and Chemical Properties of Particulate Ion-Exchange Resins

D 2687 Practices for Sampling Particulate Ion-Exchange Materials

D 5391 Test Method for Electrical Conductivity and Resistivity of a Flowing High Purity Water Sample

3. Terminology

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

4. Summary of Practice

4.1 An apparatus is described in which a specified volume of regenerated resin sample is mixed with a corresponding new resin. The mixed bed then is operated at a controlled high flow rate on an influent of known composition, and the quality of the

effluent is measured by conductivity, and if agreed upon, other appropriate analytical procedures.

5. Significance and Use

5.1 This practice is intended to evaluate changes in the performance of ion exchange resins used in mixed beds operating as polishing systems for solutions of low ionic strength, typically, <10 mg/L dissolved solids, that are intended to produce very high purity effluents. It is recommended that when new resins are installed in a plant it be used to provide a base line against which the future performance of that resin can be judged.

5.2 The conditions of this test must be limiting kinetically, such that kinetic leakage, and not equilibrium leakage, is tested. This leakage is influenced by a combination of influent flow velocity and concentration, as well as bed depth.

5.3 It is recommended that the practice be followed with the resin ratio, flow rate, and influent quality as indicated. The design of the apparatus permits other variations to be used that may be more appropriate to the chemicals used in a specific plant and the nature of its cooling water, but the cautions and limitations noted in the practice must be accommodated.

5.4 It is possible that the cation resin could experience kinetics problems. In many cases, however, the anion resins are more likely to experience the types of degradation or fouling that could lead to impaired kinetics. Testing of field anion and cation resins together is an option, especially when historic data on the mixed bed will be compiled. Recognize, however, that many variables can be introduced, making it difficult to interpret results or to compare to historical or new resin data on separate components.

5.5 Provision is made for calculation of the mass transfer coefficient in the Appendix X1. When such calculation is to be made, a full wet sieve analysis, as described in Test Methods D 2187, also is required. Electronic particle sizing may be substituted if it is referenced back to the wet sieve method.

5.6 This practice is intended to supplement, not displace, other indicators of resin performance, such as exchange capacity, % regeneration, and service experience records.

6. Interferences

6.1 Interferences in the conventional sense are minimal, but variations in test conditions, such as flow rate, temperature, resin ratio, particle size, column configuration, regeneration

¹ This practice is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.08 on Membrane and Ion Exchange Materials.

Current edition approved June 1, 2004. Published June 2004. Originally approved in 1998. Last previous edition approved in 1998 as D 6302 – 98.

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.

efficiency, and influent concentrations can cause major differences in performance. This practice fixes or measures these variables so that true changes in resin kinetics can be demonstrated accurately. Other means will be needed to investigate other resin or equipment problems.

6.2 Contaminant ions in the resins themselves, if present when they are loaded into the test apparatus, may impact performance significantly and must be considered in the interpretation of the results. If the contaminant ions are different from those in the challenge solution, they may be determined by ion chromatography.

6.3 A constant velocity in the range of 50–60 gpm/ft² is used to insure that flow is turbulent and there is little or no resistance to mass transfer from the bulk solution to the resin surfaces. This constant velocity insures the desired testing of surface kinetics at the boundary layer.

7. Equipment

7.1 *Backwash/Separation and Regeneration Apparatus*, see Test Methods **D 2187**. The column should be 50-mm ID × 600 or 900-mm length.

7.2 *Kinetics Test Apparatus* (see **Fig. 1**):

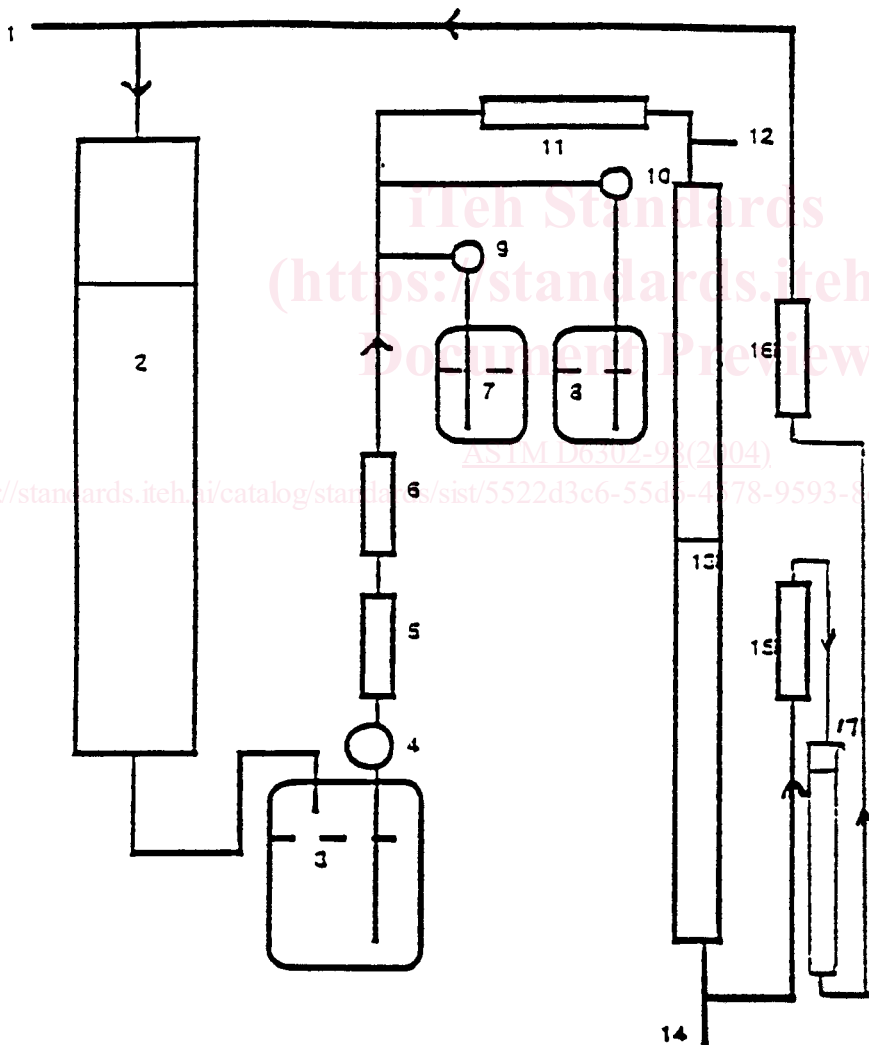
7.2.1 *Feed Pumps*, capable of controlled delivery of 0.5 to 3 mL/min. One is required, the second is optional for use where another reagent, such as ammonia, is to be added.

7.2.2 *Circulating Pump*, capable of delivery of 1 to 1.5 L/min.

7.2.3 *Glass Column*, nominal 25-mm ID × 600 mm. The column shown in **Fig. 1** of Test Methods **D 2187** may be modified for this purpose.

7.2.4 *Mixing Chamber*.

7.2.5 *Conductivity Meter With Recorder and Temperature Compensation*—See Test Method **D 5391**.



1. Water supply, ASTM Type I
2. Mixed bed polishing column (Required for recirc mode)
3. Polished water reservoir (Required for recirc mode)
4. Pump (Required for recirc mode)
5. Conductivity meter (Required for recirc mode)
6. Flow meter
7. and 8. Feed solution reservoir
9. and 10. Proportional metering pump
11. Mixing chamber or static mixer
12. Influent sample tap
13. Test column
14. Effluent sample tap
15. Conductivity meter
16. Conductivity meter
17. Cation column

NOTE—Recirculation of water is optional; final effluent also can be directed to drain.

FIG. 1 Test Apparatus for Kinetics

7.2.6 *Flow Meter*—Capable of measuring flows in the range of about 1 L/min.

7.2.7 *Cation Column*, nominal 25-mm ID × 600-mm column, typically with a 15–45-cm depth of resin. This column should be prepared the day before testing to allow to rinse to >17.5 MΩ (see 8.3).

NOTE 1—Pressure relief should be provided for this system to allow no more pressure than the materials can tolerate, typically 50 psig or less.

8. Reagents

8.1 *Purity of Reagents*—Reagents meeting the specifications of the Committee on Analytical Reagents of the American Chemical Society may not be suitable for use in this practice. All reagents used should be of the highest grade commercially available and should be tested for both anionic and cationic impurities by ion chromatography after the feed solutions have been prepared.^{3,4}

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193, Type I. It shall be checked by ion chromatography at the ppb level prior to use, if ion chromatography will be used for analysis.

8.3 *Standard Cation Resin*—New hydrogen-form, strong acid, cation resin is to be used; nuclear grade is preferred. Do not regenerate this resin. This resin should be stored in impermeable containers at temperatures that do not exceed 25°C. Backwash the resin with water at 100 % expansion for at least 15 min. The resin should be rinsed thoroughly with water to ≥ 17.5 MΩ resistivity before being used in a kinetics test. The same cation resin may be used in the test column, as well as the cation column. It is recommended that a specific type and brand of resin be used consistently where results are to be compared.

8.4 *Standard Anion Resin*—Use new, hydroxide-form, strong base anion resin; nuclear grade preferred. Follow other requirements as given in 8.3.

8.5 *Test Solutions*—Test solutions can be modified for specific systems, however, the following are recommended for routine testing. Although a target feed injection rate of 0.5 mL/min is used here, the feed concentrations and metering pump flows can be altered, so long as the test column influent concentrations and flow rate are nominal as specified.

8.5.1 *Ammonia Feed Solution (3.0 g/L as NH₃) Optional for Use with Ammoniated Systems*—Tare a beaker with about 50 mL of water on an analytical balance with 0.01-g sensitivity. Add 20.9 g of concentrated ammonium hydroxide (sp. gr. 0.90) from a dropping bottle. Transfer to a 2-L volumetric flask, and dilute to volume. Mix well. When delivered at the rate of 0.5

mL/min into 1 L/min flow, the concentration in the influent should be 1.5 mg NH₃/L.

NOTE 2—Ammonium hydroxide generates irritating ammonia vapors.

8.5.2 *Sodium Sulfate Feed Solution (0.9 g Na₂SO₄/L)*—Dry the Na₂SO₄ for 1 h at 100–105°C, then store in a desiccator. Weigh 0.900 g of the anhydrous sodium sulfate, and dissolve it in 1 L of water. Mix well. When delivered at the rate of 0.5 mL/min into 1 L/min flow, the concentration of the influent should be 0.145 mg/L Na and 0.300 mg/L SO₄.

8.6 *Regenerant, Sodium Hydroxide Solution (87 g/L)*—Add 345 g NaOH to 3.5 L of water with stirring. Cool and dilute to 4.0 L. This solution is caustic and liberates heat during dissolution. This is equivalent to 8 % NaOH by weight.

NOTE 3—This solution is intentionally stronger than typical field processes so that maximum % regeneration is achieved.

Reagent grade 50 % NaOH (763 g NaOH/L) also can be used and would require 456 mL to make 4.0 L.

8.7 *Regenerant, Hydrochloric Acid Solution (1 + 9)*—Carefully pour 200 mL of hydrochloric acid (HCl, sp. gr. 1.19) into 1800 mL of water, stirring constantly. Cool to 25 ± 5°C.

NOTE 4—For field cation samples, sulfuric acid typically would be substituted for HCl, since H₂SO₄ is the usual regenerant in the field.

9. Sampling

9.1 Collect the sample in accordance with Practices D 2687. It is extremely important that the resin sample properly represent the entire bed being evaluated. Core sampling is required. A sample containing at least 300 mL of anion, or cation resin, or both, must be provided. The sample may be taken before or after separation of a mixed bed, so long as it is representative. Use a plastic or glass container with a water-tight cap and label in accordance with Practices D 2687.

9.2 Subsamples taken in the laboratory also must be taken by careful coring to preserve the representativeness of the sample.

10. Backwash and Separation Procedure

10.1 Place about 800 mL of mixed bed resin sample or about 500 mL of individual resin sample in the backwash/separation apparatus. Backwash with water at a flow sufficient to give about 50 % bed expansion. This should allow crud to rinse away while separating any cation from the anion in the sample.

10.2 Using a siphon or aspiration assembly, remove and collect the resin of interest, anion resin (above the interface) or cation resin. Try to minimize cross-contamination by leaving behind or wasting resin as needed. This, however, must be minimized in order to avoid sample bias. Inspection of the interface with a hand lens may show a bead size variation at the interface. If less than 300 mL of the resin of interest is recovered, repeat 10.1 with another portion of sample.

10.3 Remove a small amount of the separated resin to a plastic petri dish and examine under low power (12–15X) magnification to estimate the percentage of whole beads. If the resin is less than about 90 % whole beads, this practice should not be continued.

NOTE 5—Ion exchange kinetics are affected by particle size and shape.

³ *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.

⁴ McNulty J. T., Bevan C. A., et al., “Anion Exchange Resin Kinetic Testing: An Indispensable Tool for Condensate Polisher Troubleshooting,” Proceedings of the 47th International Water Conference, Engineers’ Society of Western Pennsylvania, October 1986.