INTERNATIONAL

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Standard Guide for Detection of Fouling and Degradation of Particulate Ion Exchange Materials[1]

This standard is issued under the fixed designation D5217; 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 guide presents a series of tests and evaluations intended to detect fouling and degradation of particulate ion exchange materials. Suggestions on reducing fouling and on cleaning resins are given.

1.2 This guide is to be used only as an aid in the evaluation of particulate ion exchange material performance and does not purport to address all possible causes of unsatisfactory performance. The evaluations of mechanical and operational problems are not addressed.

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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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]

D1129 Terminology Relating to Water
D1782 Test Methods for Operating Performance of Particulate Cation-Exchange Materials
D2187 Test Methods and Practices for Evaluating Physical and Chemical Properties of Particulate Ion-Exchange Resins
D2332 Practice for Analysis of Water-Formed Deposits by Wavelength-Dispersive X-Ray Fluorescence
D2687 Practices for Sampling Particulate Ion-Exchange Materials
D3087 Test Method for Operating Performance of Anion-Exchange Materials for Strong Acid Removal
D3375 Test Method for Column Capacity of Particulate Mixed Bed IonExchange Materials
D3682 Test Method for Major and Minor Elements in Combustion Residues from Coal Utilization Processes
D3683 Test Method for Trace Elements in Coal and Coke Ash by Atomic Absorption
D5042 Test Method for Estimating the Organic Fouling of Particulate Anion Exchange Resins
D6302 Practice for Evaluating the Kinetic Behavior of Ion Exchange Resins
E830 Test Method for Ash in the Analysis Sample of Refuse-Derived Fuel (Withdrawn 2011)[3]

3. Terminology

3.1 ~~*Definitions*—~~*Definitions:* ~~For definitions of terms used in this guide, refer to Terminology D1129.~~

3.1.1 For definitions of terms used in this standard, refer to Terminology D1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *air lance, v*—to subject to a stream of air under pressure.

3.2.2 *organic ~~fouling—~~fouling, n—*the buildup of organic material in or on anion exchange resins by sorption during the service cycle and incomplete removal during normal regeneration.

[1] This guide 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 last approved version of this historical standard is referenced on www.astm.org.

4. Significance and Use

4.1 Resins used in demineralization systems may deteriorate due to many factors including chemical attack, fouling by organic and inorganic materials, mishandling, or the effects of aging. Detection of degradation or fouling may be important in determining the cause of poor demineralizer performance.

5. Sampling

5.1 Follow the recommendations of Practices D2687 for obtaining samples of particulate ion exchange materials. Core samples are important for obtaining representative samples; however, special problems may dictate other sampling requirements, such as surface, interface, or other samples.

6. Preliminary Examination

6.1 Examine the sample visually or with the aid of a magnifier for any abnormalities. Note any unusual color, precipitates, biological material (slime), particulate matter, or small pieces or fragments of resin. Note that the color of resin may vary from lot to lot or with normal use and would not be considered unusual.

TABLE 1 Detection of Fouling and Degradation of Particulate Ion Exchange Materials

Section No.	Property Tested	Test Results	Possible Indications
6	Visual appearance	Unusual color or precipitates Pieces/fragments present	Coating on beads from foulants or improper regeneration Physical degradation
6	Odor	Unusual odor	Fouling of resin by oil, solvents, etc. or biological activity
7	Moisture	Higher than expected (>10 % above) Lower than expected	Degradation of resin causing decrosslinking Fouling of resin by heavy materials, such as metal oxides
7	Particle size distribution	Smaller sizes than expected Larger sizes than expected	Physical degradation or non-representative sample Loss of smaller beads by backwash or through strainers
8	Mixed bed resin separation	Poor separation	Ionic form of resin may not be correct Resin may be fouled Particle size distribution of beads may be incorrect
9	Ash content and metals	Higher than expected	Fouling of resin by expected metal oxides or silica (from corrosion products, influent water, or regenerants)
9	Ash content and metals	Higher than expected	Fouling of resin by expected metal oxides or silica (from corrosion products, influent water, or regenerants)
10	Organic fouling of anion resins	Moderate to severe	Presence of sufficient organic fouling to affect performance
11	Column performance	Poorer than expected	Degradation or fouling sufficient to affect performance
12	Kinetics	Poorer than expected	Degradation or fouling sufficient to affect performance

6.2 Note any peculiar odor associated with the sample, such as from oil, solvents, or biological activity.

7. Moisture and Particle Size Distribution

7.1 Follow procedures given in Test Methods D2187, Test Methods A, B, and D for determining moisture (water retention capacity) and particle size distribution.

7.2 Compare the values obtained in 7.1 to those expected for the resin when in good condition. It is preferred that new resin, treated in the same way, be used for this comparison, but manufacturer's specifications can also be used.

8. Mixed-Bed Resin Separation

8.1 Observe resin during separation according to Test Methods D2187, Test Method A. Adjust backwash rate to give optimiumoptimum separation, then let resin settle and observe interface and note degree of cross-mixing.

9. Ash Content and Metals Analysis

9.1 Follow the procedure given in Test Method E830 for determining the ash content of the pretreated and dried sample. A larger sample portion may be used for low-ash resins.

9.2 Analyze the ash for silica or metals such as iron, copper, manganese, barium, aluminum, calcium, magnesium, or others which might be suspected as contaminants. Use X-ray fluorescence analysis to determine major elements (see Practice D2332). Employ digestion, fusion, and analysis techniques as would be used for other types of ash. (Seeash (see Test Methods D3682 and D3683.)). Note that some elements may be lost during the 575°C ashing, and spike recoveries must be checked.

10. Detection of Organic Fouling of Anion Resins

10.1 Follow procedures given in Test MethodsMethod D5042 for estimation of the degree of organic fouling of anion resins.

10.2 For a more rapid, but less reliable evaluation of the resin, the caustic-brine extract from Test MethodsMethod D5042 may be judged by color rather than by total organic carbon measurement: the darker the color, the heavier the organic fouling. Note that colorless foulants such as detergents or synthetic polyelectrolytes will not be detected.

11. Column Performance Testing

11.1 Follow procedures given in Test Methods D3375, D3087, or D1782 as needed to evaluate the performance of mixed bed, anion, or cation exchange materials, respectively.

12. Kinetics Testing

12.1 The evaluation of the kinetics properties of ion-exchange resins is especially important for anion resins used in high flow rate applications such as condensate polishing.

12.2 Test the resin's kinetics properties according to Practice D6302 or published procedures such as those by the Central Electricity Generating Board[4] and Rohm & Haas CompanyCompany.[5].

13. Interpretation of Results

13.1 Table 1 gives general guidelines for the interpretation of results from these tests. Note that in most cases, test results must be compared to those obtained for resins of the same type which are in good operating condition.

13.2 Caution must be exercised in applying these test results to the evaluation of operating demineralizer systems. However, Appendix X1 and Appendix X2X3 give some suggestions for pretreatment and resin cleaning procedures. The user should also consult with the resin supplier before using any new treatment process.

14. Precision and Bias

14.1 No statement is made about either the precision or the bias of this guide since the result merely states whether there is conformance to the criteria for success specified in the procedure.

15. Keywords

15.1 degradation; fouling; ion exchange; kinetics; resin

APPENDIXES

(Nonmandatory Information)

X1. METHODS FOR PREVENTING OR REDUCING FOULANTS TO CONTAMINANTS SUSPECTED OF FOULING OR DEGRADING ION EXCHANGE RESINS[6]

X1.1 Organics

Humic or fulvic solubles in water
Humic or fulvic leakage from pretreatment-coagulation or organic traps
Colloidal color from influent water
Cation exchange resin degradation products
Oil, soluble, or grease
Organic or vegetable fibers

[4] Harris, R. R., "Anion Exchange Kinetics in Condensate Purification Mixed Beds-Assessment and Performance Prediction," *Proceedings of EPRI Condensate Polishing Workshop*, October 1985, pp. 31–40.

[5] McNulty, J. T., et al., "Anion Exchange Resin Kinetic Testing: An Indispensable Diagnostic Tool for Condensate Polisher Troubleshooting," *Proceedings of International Water Conference*, October, 1986.

[6] Crits, G. J., "The Prevention of Organics and Other Foulants in Ion Exchange Resins," *24th Annual Liberty Bell Corrosion Course*, April 1986.

Filter media, siliceous (Celite,[7] perlite)
Filter media, cellulose (Solka-Floc[8])
Micro-organisms, algae, bacteria, slime, etc.
Detergents, ABS/LAS, anionic
Detergents, cationic
Air-borne dusts, micro-organisms
Solvents/detergents from new resins
Amines from anion resins
Sloughage from aged exhausted activated carbon
Organic leakage from weak base or Type II strong base anion exchange resins
Resin leakage, fines or beads
Polyelectrolytes/coagulation aids

X1.2 Metals or Non-MetalsX1.2 These are only suggested treatments; the resin supplier should be consulted before any new treatment process is used.

Suspected Contaminant or Foulant Organics
Humic or fulvic solubles in water
Humic or fulvic leakage from pretreatment coagulation or organic traps
Colloidal color from influent water
Cation degradation products
Oil, soluble or grease
Organic or vegetable fibers
Filter media, celite/siliceous
Filter media, cellulose (solka floc)
Micro-organisms, algae, bacteria, slime, etc.
Detergents, ABS/LAS, anionic
Detergents, cationic
Air-borne dusts, micro-organisms
Solvents/detergents from new resins
Amines from anion resins
Sloughage from aged exhausted activated carbon
Organic leakage from weak/Type II
Resin leakage, fines or beads
Polyelectrolytes/coagulation aids
Metals or Non-Metals:
Silt, clay, turbidity (colloidal)
Colloidal silica (insoluble)
Silica gelation (due to high soluble silica and strong caustic)
Manganese on cation resin with HCl regeneration causing oxidative attack by chlorine
Manganese on cation resin with hydrochloric acid (HCl) regeneration causing oxidative attack by chlorine
Iron, soluble or insoluble, influent (greater than 0.5 mg/L per 24-h run)
Iron, soluble, to 30 mg/L (no air)
Corrosion products, iron, copper, etc., in cation water or regeneration dilution water
Iron in caustic, above 10 mg/L (50 % sodium hydroxide basis)
Sulfur precipitate, above 0.5 mg/L per 24-h run
Aluminum floc/aluminum precipitation (above 0.3 mg/L per 24 h)
Barium, strontium, calcium forming sulfate precipitate
Chlorine, ozone, oxidation
Chlorine, ozone, hydrogen peroxide, other oxidants
Physical/Radiological:
High operating water temperature
Radiation (less than 1 r/day)
Osmotic regeneration shocks
Air mixing in mixed beds/oxidation of cation resins

X1.3 Physical/RadiologicalX1.3 **Warning—**Treatments used on potable water production systems must meet all applicable safety and health regulations.

Possible Pretreatment or Method to Reduce Fouling
Coagulation, lime softening, organic traps
Activated carbon, caustic/salt treatment
Coagulation, lime softening, ultra filter
Rinse new resins, sulfite/SO_2 feeds
Pretreatment, coagulation, filter
Coagulation and filter

[7] Celite is a trademark of Imerys Minerals California, Inc. in San Jose, CA.
[8] Solka-Floc is a trademark of Solvaira Specialties, Inc. in North Tonawanda, NY.