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

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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 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
D1782 Test Methods for Operating Performance of Particulate Cation-Exchange Materials
D2187 Test Methods for 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
E830 Test Method for Ash in the Analysis Sample of Refuse-Derived Fuel (Withdrawn 2011)[3]

3. Terminology

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

3.2 *Definitions of Terms Specific to This Standard:*

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

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.

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[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.

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	Coating on beads from foulants or improper regeneration
		Pieces/fragments present	Physical degradation
6	Odor	Unusual odor	Fouling of resin by oil, solvents, etc. or biological activity
7	Moisture	Higher than expected (>10 % above)	Degradation of resin causing decrosslinking
		Lower than expected	Fouling of resin by heavy materials, such as metal oxides
7	Particle size distribution	Smaller sizes than expected	Physical degradation or non-representative sample
		Larger sizes than expected	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)
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 Particule Size Distribution

7.1 Follow procedures given in Test Methods D2187, 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, Method A. Adjust backwash rate to give optimium 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. (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 Methods 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 Methods 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 published procedures such as those by the Central Electricity Generating Board[4] and Rohm & Haas Company[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 X2 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

[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.

APPENDIXES

(Nonmandatory Information)

X1. METHODS FOR PREVENTING OR REDUCING FOULANTS TO ION EXCHANGE RESINS[6]

X1.1 These are only suggested treatments; the resin supplier should be consulted before any new treatment process is used.

X1.2 **Warning—**Treatments used on potable water production systems must meet all applicable safety and health regulations.

Suspected Contaminant or Foulant Organics	*Possible Pretreatment or Method to Reduce Fouling*
Humic or fulvic solubles in water	Coagulation, lime softening, organic traps
Humic or fulvic leakage from pretreatment-coagulation or organic traps	Activated carbon, caustic/salt treatment
Colloidal color from influent water	Coagulation, lime softening, ultra filter
Cation degradation products	Rinse new resins, sulfite/SO_2 feeds
Oil, soluble or grease	Pretreatment, coagulation, filter
Organic or vegetable fibers	Coagulation and filter
Filter media, celite/siliceous	Better control of filter operation
Filter media, cellulose (solka floc)	Better control of filter operation (add another filter)
Micro-organisms, algae, bacteria, slime, etc.	Chlorine/coagulation/filtration
Detergents, ABS/LAS, anionic	Activated carbon, foam fractionation
Detergents, cationic	Activated carbon
Air-borne dusts, micro-organisms	Add filter to air blowers/compressors
Solvents/detergents from new resins	Warm rinse or brine acid treatment, or both
Amines from anion resins	Warm rinse
Sloughage from aged exhausted activated carbon	Renew carbon, caustic-salt treatments
Organic leakage from weak/Type II	Pretreat/coagulation/caustic-salt treatments
Resin leakage, fines or beads	Improve underdrain collector
Polyelectrolytes/coagulation aids	Reduce dosage of aids
Metals or Non-Metals:	
Silt, clay, turbidity (colloidal)	Coagulation/filtration pretreatment
Colloidal silica (insoluble)	Coagulation/filtration
Silica gelation (due to high soluble silica and strong caustic)	Reduce temperature, sodium hydroxide strength, step regeneration
Manganese on cation resin with HCl regeneration causing oxidative attack by chlorine	Aeration/filtration, with or without coagulation
Iron, soluble or insoluble, influent (greater than 0.5 mg/L per 24-h run)	Aeration/coagulation/filtration
Iron, soluble, to 30 mg/L (no air)	Brine regeneration with reducing agents
Corrosion products, iron, copper, etc., in cation water or regeneration dilution water	Use plastic materials, stainless steel
Iron in caustic, above 10 mg/L (50 % sodium hydroxide basis)	Evaluate resin replacement versus sodium hydroxide
Sulfur precipitate, above 0.5 mg/L per 24-h run	Aeration and filter, with or without coagulation
Aluminum floc/aluminum precipitation (above 0.3 mg/L per 24 h)	High rate polishing filter
Barium, strontium, calcium forming sulfate precipitate	Lime softening pretreatment
Chlorine, ozone, oxidation	Activated carbon, sulfite/SO_2 feed
Physical/Radiological:	
High operating water temperature	Evaluate temperature reduction versus resin cost
Radiation (less than 1 r/day)	Evaluate radiation versus resin replacement
Osmotic regeneration shocks	Reduce regenerant strengths, limit exchange capacity
Air mixing in mixed beds/oxidation of cation resins	Restrict air mixing time to minimum

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

X2. CLEANING AGENTS AND PROCEDURES FOR ION EXCHANGE RESINS[7]

X2.1 These are only suggested treatments; the resin supplier should be consulted before any new treatment process is used.

X2.2 **Warning—**Treatments used on potable water production systems must meet all applicable safety and health regulations.

X2.3 *Procedures* :

X2.3.1 Brine at least 8 lb/ft^3 (at 12 to 15 %).

X2.3.2 Air scour—at minimum 4 ft^3/min/ft^2 with minimum 24 in. water above bed

X2.3.3 Air lance (at 4 ft^3/min/ft^2) along with soak is recommended.

X2.3.4 Always backwash thoroughly after treatment, for at least 30 min.

X2.3.5 For softener after acid treatments, always brine twice.

X2.4 These cyclic caustic/salt applications should be performed at regular intervals such as each week, month, and so forth, to prevent the fixing or polymerization of the organics or color.

[7] Crits, G. J., "Resin Cleaning Methods and Are They Effective," *24th Annual Liberty Bell Corrosion Course*, April 1986.

Equipment System	Type Resin	Suspected Contaminants	Possible Cleaning Agents and Procedures
Softener	Strong acid cation	Iron, manganese	Brine, 8 lb/ft^3 Air scour Brine with 1 lb, sodium hydrosulfite, soak 2 h
Softener	Strong acid cation	Iron, manganese	Brine Air scour 2 lb sulfamic, or citric, or oxalic acid per ft^3 at 2.5 % Air lance, soak for 2 to 4 h
Softener	Strong acid cation	Iron, manganese, silica gels, aluminum floc	Brine Air scour Caustic, 2 lb/ft^3 at 2 to 4 % Air lance, soak 2 h
Softener	Strong acid cation	Slime, iron chrenothrix algae, etc.	H + Beds—must brine first Air scour Brine with 0.8 to 1.2 oz. sodium hypochlorite solution (5 %)/ft^3 Soak 2 h, air lance
Softener/Demineralizer/ Mixed Bed	Strong acid cation	Slime, bacteria, algae, etc.	Brine, 8 lb/ft^3 Formaldehyde, peracetic, or H_2O_2 at ¼ to ½ %, soak 3 h or more Air lance
Softener/Demineralizer	Strong acid cation	Slime, bacteria, algae, etc.	Apply 0.5 to 1 mg/L chlorine (on line feed) sodium hypochloritesolution for 2 to 5 h (also, I_2, O_3, H_2O_2, hot 70°C water may be used)
Softener/Demineralizer/ De-alkalizer	Strong acid or cation anion resin	Oil, fibers, sulfur, algae organics	Brine (to remove acid and hardness) Caustic, 2 lb/ft^3 (1 to 3 %) with non-ionic surfactant Air lance Air scour
Softener/Demineralizer/ De-alkalizer	Strong acid or weak acid cation	Calcium sulfate, iron	HCl, 2 to 3 gal. of 30 %/ft^3 (add extra acid for conversion of resin to H + form); equipment must be HCl corrosion proof
Softener/Demineralizer/ De-alkalizer (Problems without oil)	Strong acid or weak acid cation	Organic fibers, cellulose filter media	Air scour Brine with or without 0.05 to 0.1 % detergent (non-ionic surfactant) Air lance
Demineralizer/Organic trap/ De-alkalizer	Strong or weak base anion	Iron, calcium, magnesium	Exhaust and brine ^{A} 2 gal of 30 % HCl per ft^3 For calcium and magnesium, apply 3 % HCl For iron, apply 15 % HCl Flow slowly for 2 h
Demineralizer/Organic trap	Strong base anion	Silica precipitates or gels	Regenerate with normal sodium hydroxide, air scour or lance Apply extra 2 lb sodium hydroxide/ft^3 at 2 to 4 % and air lance for 1 h Rinse, backwash, and regenerate again
Demineralizer/Organic trap	Strong base anion Type 1	Organics, color, foulants	Cyclic caustic/salt Sodium hydroxide (warm 150°F) 2 lb/Cu. at 3 %. Displace with water, 10min. salt (warm), 6 to 8 lb/ft^3 at 12 %. Displace with water, 10 min. Repeat 3 times or until color in effluent dropsbelow 20 % of the maximum observed. (Alternatively 2 to 3 bed volumes of a combined 10 % NaCl and 1 to 2 % NaOH solution at 120 to 140°F can be used, soaking on the last volume.)
Demineralizer/Organic trap/ De-alkalizer	Strong Base Type I Type II	Organics with iron/heavy metals	Cyclic caustic/salt treatments followed by HCl^B ; 2 gal. of 30 % HCl per ft^3 applied to drained bed (water level below bed 3 in.) Soak and displace slowly over 1 or 2 h; note effluent color
Demineralizer/Organic trap/ De-alkalizer	Strong base anion	Organics and/or bacteria/ slimes	Air scour Regenerate with sodium hydroxide—no acidity Brine with 0.5 to 1.0 oz. of sodium hypochlorite solution (5 %) per ft^3 Soak 2 h or more Air lance if slimes are high
		Slimes, bacteria	Hot water soak/flow at 70°C for 3 h or more^C

^{A} For strong base resin with high silica exchanged (over 7 %), regenerate with normal caustic dosage first to remove silica, then brine to exhaust or remove the alkalinity.
^{B} Equipment must be HCl-corrosion proof when using HCl acid.
^{C} Internal surfaces of the unit, piping, regen. tank must be contacted by the sterilizing agent to prevent re-infection of the resin beds.