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ABSTRACT

This document is an instructional module package prepared in objective form for use by an instructor familiar with the operation of an ion exchange softening system. It includes objectives, an instructor guide, student handouts and transparency masters. This is the third level of a three module series. This module considers the theory of ion exchange, interpretation and application of laboratory data for optimal operation of an ion exchange unit and laboratory evaluation of resins. (Author/RH)

ADVANCED ION EXCHANGE SOFTENING

Training Module 2.212.4.77

Prepared for the

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September, 1977

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INSTRUCTOR GUIDE

for

Training Module II4AFWS

Module No: II4AFWS	Module Title: Advanced Ion Exchange Softening
	Submodule Title:
Approx. Time: 11 hours	Topic: Summary

Objectives: Upon completion of this module, the participant will be able to:

1. Describe the theory of ion exchange.
2. Determine ~~the~~ optimal operation of an ion exchange.
3. Describe the laboratory evaluation of resins.

Instructional Aids:

1. Handout
2. Transparencies #1 - #27

Instructional Approach:

Discussion

References:

1. Elements of Ion Exchange, Kunin
2. Ion Exchange Resins, Kunin

Class Assignments:

1. The participant will read Handout

Module No: II4AFWS	Topic: Summary
Instructor Notes:	**Instructor Outline:**
1. Distribute handout 2. Present transparencies	1. Discuss the theory, optimal operation and laboratory evaluation resins for exchange softeners. 2. Give evaluation of 30 questions.

Module No: I14AFWS	Module Title: Advanced Ion Exchange Softening
	Submodule Title:
Approx. Time: 1 hour	Topic: Introduction

Objectives: Upon completion of this topic, the participant will be able to:
1. Describe ion exchange principles.
2. Describe ion exchange preventive maintenance.
3. Design an ion exchange softener.
4. Describe ion exchange analytical control.

Instructional Aids:
1. Handout - Introduction
2. Transparency #1-Softening Reactions
3. Transparency #2-Regeneration Reactions
4. Transparency #3-Preventive Maintenance
5. Transparency #4-#6-Ten States Standards for Softeners
6. Transparency #7-Ten States Standards for Brine Tank
7. Transparency #8-Laboratory Control

Instructional Approach:

Discussion

References:
1. Elements of Ion Exchange, Kunin
2. Ion Exchange Resins, Kunin

Class Assignments:

1. The participant will read Handout-Introduction

Module No:	Topic:
II4AFWS	Introduction

Instructor Notes:	Instructor Outline:
1. Present Transparency #1	1. Review the principles of ion exchange softening.
2. Present Transparency #2	2. Review the principles of regeneration of ion exchange softening.
3. Present Transparen y #3	3. Review the preventive maintenance program for an ion exchange softener.
4. Present Transparency #4-#6	4. Review the design standards for softeners.
5. Present Transparency #7	5. Review the design standards for brine tanks.
6. Present Transparency #8	6. Review the laboratory control for an ion exchange softener.

Module No: II4AFWS	Module Title: Advanced Ion Exchange Softening
	Submodule Title:
Approx. Time: 2 hours	Topic: Theory of Ion Exchange

Objectives: Upon completion of this topic, the participant will be able to:
1. Describe the theory of ion exchange.
2. Select the appropriate ion exchange resin for a partical problem.
3. Determine the appropriate regeneration for the resin.

Instructional Aids:
1. Handout - Theory of Ion Exchange
2. Transparency #9-Selectivity Coefficient
3. Transparency #10-Typical Selectivity Coefficients
4. Transparency #11-"Rules of Thumb" for Selectivity Coefficients
5. Transparency #12-Types of Resins
6. Transparency #13-Types of Regenerates

Instructional Approach:

Discussion

References:
1. Elements of Ion Exchange, Kunin
2. Ion Exchange Resins, Kunin

Class Assignments:
1. The participant will read Handout-Introduction

Module No:	Topic:
II4AFWS	Theory of Ion Exchange

Instructor Notes:	Instructor Outline:
1. Present Transparency #9	1. Discuss the selectivity coefficient and how it relates to the operation of an ion exchange softener.
2. Present Transparency #10	2. Discuss the relative locations of the various ions in the chart and its impact on operation.
3. Present Transparency #11	3. Discuss "rules of thumb" and demonstrate the rules to the table values.
4. Present Transparency #12	4. Discuss the different classes of resins and their applications.
5. Present Transparency #13	5. Discuss the different regenerations and their role in operation.

Module No:	Module Title:
II4AFWS	Advanced Ion Exchange Softening
	Submodule Title:
Approx. Time:	Topic:
3 hours	Laboratory Evaluation of Resins

Objectives: Upon completion of this topic, the participant will be able to:

1. Select the appropriate test to evaluate a resin.
2. Describe the tests to evaluate a resin.

Instructional Aids:

1. Handout - Laboratory Evaluation of Resins
2. Transparency #14-Laboratory tests to evaluate a resin
3. Transparency #15-#20 - Laboratory tests

Instructional Approach:
Discussion

References:

1. Elements of Ion Exchange, Kunin
2. Ion Exchange Resins, Kunin

Class Assignments:

1. The participant will read Handout-Laboratory Evaluation of Resins

Module No:	Topic:
II4AFWS	Laboratory Evaluation of Resins

Instructor Notes:	Instructor Outline:
1. Present Transparency #14	1. Discuss in general the various tests conducted on ion exchange resins. Indicate the importance of each to operation.
2. Present Transparency #15	2. Discuss the screen test for ion exchange resins and its application to ion exchange softener control.
3. Present Transparency #16	3. Discuss in general the total capacity measurements for ion exchange resins and its application to operation.
4. Present Transparency #17	4. Discuss pretesting resin conditioning and its importance to the rest of the testing procedure.
5. Present Transparency #18	5. Discuss solids content determination.
6. Present Transparency #19	6. Discuss cation exchange capacity.
7. Present Transparency #20	7. Discuss anion exchange capacity.
NOTE: If at all possible do a demonstration of the tests along with the transparencies.	

Module No: II4AFWS	Module Title: Advanced Ion Exchange Softening
	Submodule Title:
Approx. Time: 2 hours	Topic: Interpretation and application of laboratory data

Objectives: Upon completion of this topic, the participant will be able to:

1. Interpret laboratory data to check compliance with design standards.
2. Interpret laboratory data to evaluate resin problems.
3. Interpret laboratory data to optimally control an ion exchange softener
4. Interpret laboratory data to determine optimal salt regeneration.

Instructional Aids:
1. Handout - Interpretation and Application of Laboratory Data
2. Transparency #21-Interpretation and Application of Laboratory Data
3. Transparency #22-Typical Resin Specifications
4. Transparency #23-Evaluate Resin Problems
5. Transparency #24-Optimal Control of Ion Exchange Units

Instructional Approach:

Discussion

References:

1. <u>Elements of Ion Exchange</u>, Kunin
2. <u>Ion Exchange Resins</u>, Kunin

Class Assignments:

1. The participant will read Handout-Interpretation and application of laboratory data

Module No:	Topic:
II4AFWS	Interpretation and Application of Laboratory Data

Instructor Notes:	Instructor Outline:
1. Present Transparency #21	1. Discuss generally the areas of application of laboratory data.
2. Present Transparency #22	2. Discuss typical specifications and how to take laboratory data and determine compliance with specifications.
3. Present Transparency #23	3. Discuss the use of laboratory data in evaluating operational problems that are related to the resin.
4. Present Transparency #24	4. Discuss the use of laboratory data in determining the optimal control of an ion exchange unit.

Module No:	Module Title:
II4AFWS	Advanced Ion Exchange Softening
	Submodule Title:
Approx. Time: 2 hours	Topic: Special Applications of Ion Exchange

Objectives: Upon completion of this topic, the participant will be able to:

1. Describe principles of deionization.
2. Describe operation of deionization.

Instructional Aids:

1. Handout - Special Applications of Ion Exchange
2. Transparency #25-Principles of deionization
3. Transparency #26-Types of deionization
4. Transparency #27-Operation of mixed bed deionizer

Instructional Approach:

Discussion

References:

1. Elements of Ion Exchange, Kunin
2. Ion Exchange Resins, Kunin

Class Assignments:

1. The participant will read Handout-Special Applications of Ion Exchange

Module No: II4AFWS	Topic: Special Applications of Ion Exchange

Instructor Notes:	Instructor Outline:
1. Present Transparency #25	1. Discuss the principles of deionization.
2. Present Transparency #26	2. Discuss the different types of deionization. Include advantages and disadvantages to each and the quality of water you expect for each.
3. Present Transparency #27	3. Discuss the operation of a deionizer. Include safety and regeneration of separate units when discussing the mixed bed deionizer.

Module No: II4AFWS	Module Title: Advanced Ion Exchange Softening
	Submodule Title:
Approx. Time: 1 Hour	Topic: Evaluation

Objectives:

The participant should be able to answer correctly 25 of the 30 questions asked.

Instructional Aids:

None

Instructional Approach:

Examination

References:

None

Class Assignments:

None

Module No: II4AFWS	Topic: Evaluation

Instructor Notes:	Instructor Outline:
1. Distribute exam. Each participant is to complete the exam independently and with no books or notes. Collect after 1 hour.	

TRANSPARENCIES

for

Training Module II4AFWS

SOFTENING REACTIONS

HARD WATER		SODIUM EXCHANGE RESIN	SOFT WATER	EXHAUSTED EXCHANGE BED

$$\left\{\begin{matrix} Ca \\ Mg \end{matrix}\right\} \left\{\begin{matrix} (HCO_3)_2 \\ SO_4 \\ Cl_2 \end{matrix}\right\} + Na_2R \longrightarrow Na_2 \left\{\begin{matrix} (HCO_3)_2 \\ SO_4 \\ Cl_2 \end{matrix}\right\} + \left\{\begin{matrix} Ca \\ Mg \end{matrix}\right\} R$$

REGENERATION REACTIONS

Exhausted Exchange Bed		Sodium Chloride (Salt) Solution		Sodium Exchange Bed		Waste Brine
$\left\{ \begin{matrix} Ca \\ Mg \end{matrix} \right\} R$	+	$2\ NaCl$	$\longrightarrow$	Na_2R	+	$\left\{ \begin{matrix} Ca \\ Mg \end{matrix} \right\} Cl_2$

$$\left\{ \begin{matrix} Ca \\ Mg \end{matrix} \right\} R + 2\ NaCl \longrightarrow Na_2R + \left\{ \begin{matrix} Ca \\ Mg \end{matrix} \right\} Cl_2$$

PREVENTATIVE MAINTENANCE

A. ACCURATE RECORD OF PERFORMANCE
   1. PERIODIC CAPACITY CHECKS
   2. PERIODIC BRINE FLOW CHECKS

B. KEEP ALL PARTS WELL PAINTED TO PREVENT CORROSION

C. PROPERLY TREAT UNITS WHEN THEY ARE LAID UP
   1. FOR ABOVE FREEZING TEMPERATURES
      A) BACKWASH AND REGENERATE
      B) LEAVE A VALVE OPEN TO RELEASE ANY PRESSURE BUILDUP
   2. FOR BELOW FREEZING TEMPERATURES
      A) BACKWASH
      B) FILL TANK WITH STRONG BRINE
      C) LEAVE A VALVE OPEN TO RELEASE ANY PRESSURE BUILDUP

TEN STATES STANDARDS FOR SOFTENERS

4.4.2 Cation exchange process

Alternative methods of hardness reduction should be investigated when the sodium content and dissolved solids concentration is of concern. Iron, manganese, or a combination of the two, should not exceed 0.3 milligrams per liter in the water as applied to the ion exchange resin. Pre-treatment is required when the content of iron, manganese, or a combination of the two, is one milligram per liter or more.

4.4.2.1 Design

The units may be of pressure or gravity type, of either an upflow or downflow design. Automatic regeneration based on volume of water softened should be used unless manual regeneration is justified and is approved by the reviewing authority. A manual override shall be provided on all automatic controls.

4.4.2.2 Exchange capacity

The design capacity for hardness removal should not exceed 20,000 grains per cubic foot when resin is regenerated with 0.3 pounds of salt per kilograin of hardness removed.

4.4.2.3 Depth of resin

The depth of the exchange resin should not be less than three feet.

4.4.2.4 Flow rates

The rate of softening should not exceed seven gallons per minute per square foot of bed area and the backwash rate should be six to eight gallons per minute per square foot of bed area.

4.4.2.5 Freeboard

The freeboard will depend upon the specific gravity of the resin and the direction of water flow.

4.4.2.6 Underdrains and supporting gravel

The bottoms, strainer systems and support for the exchange resin shall conform to criteria provided for rapid rate gravity filters. (see Sections 4.2.1.6 and 4.2.1.7).

TEN STATES STANDARDS FOR SOFTENERS

4.4.2.7 Brine distribution

Facilities should be included for even distribution of the brine over the entire surface of both upflow and downflow units.

4.4.2.8 Cross connection control

Backwash, rinse and air relief discharge pipes should be installed in such a manner as to prevent any possibility of back-siphonage.

4.4.2.9 Bypass

A bypass must be provided around softening units to produce a blended water of desirable hardness. Totalizing meters must be installed on the bypass line and on each softener unit. An automatic proportioning or regulating device and shut-off valve should be provided on the bypass line. In some installations it may be necessary to treat the bypassed water to obtain acceptable levels of iron and/or manganese in the finished water.

4.4.2.10 Additional limitations

Waters having five units or more turbidity should not be applied directly to the cation exchange softener. Silica gel resins should not be used for waters having a pH above 8.4 or containing less than six milligrams per liter silica and should not be used when iron is present. When the applied water contains a chlorine residual the cation exchange resin shall be a type that is not damaged by residual chlorine. Phenolic resin should not be used.

4.4.2.11 Sampling taps

Smooth-nose sampling taps must be provided for the collection of representative samples. The taps shall be located to provide for sampling of the softener influent, effluent and blended water. The sampling taps for the blended water shall be at least 20 feet downstream from the point of blending. Petcocks are not acceptable as sampling taps.

4.4.2.14 Stabilization

Stabilization for corrosion control shall be provided. (see Section 4.8.5).

4.4.2.15 Waste disposal

Suitable disposal must be provided for brine waste. (see Section 4.11).

4.4.2.16 Construction material

Pipes and contact materials must be resistant to the aggressiveness of salt.

4.4.2.17 Housing

Salt-storage shall be enclosed and separated from other operating areas in order to prevent damage to equipment.

4.11.2 Brine waste

The waste from ion exchange plants, demineralization plants, etc. may be disposed of by controlled discharge to a stream if adequate dilution flow is available. Stream requirements of the regulatory agency will control the rate of discharge. Except when discharging to large waterways, a holding tank of suffucient size should be provided to allow the brine to be discharged over a twenty-four hour period. Where discharging to a sanitary sewer, a holding tank may be required to prevent the overloading of the sewer and/or interference with the waste treatment processes. The effect of brine discharge to sewage lagoons may depend on the rate of evaporation from the lagoons.

TEN STATES STANDARDS FOR BRINE TANK

4.4.2.12 Brine and salt storage tanks

Brine measuring or salt dissolving tanks and wet salt storage facilities must be covered and must be constructed of corrosion-resistant material. The make-up water inlet must have a free fall discharge of two pipe diameters above the maximum liquid level of the unit or be otherwise protected from back-siphonage. Water for filling the tank should be distributed over the entire surface by pipes above the maximum brine level in the tank. The salt shall be supported on graduated layers of gravel under which is a suitable means of collecting the brine. Wet salt storage basins must be equipped with manhole or hatchway openings having raised curbs and watertight covers having overhanging edges similar to those required for finished water reservoirs. Overflow, where provided, must be turned down, have a proper free fall discharge and be protected with corrosion-resistent screens or self-closing flap valves.

4.4.2.13 Salt storage capacity

Salt storage should have sufficient capacity to store in excess of 1½ carloads or truckloads of salt, and provide for at least 30 days of operation.

LABORATORY CONTROL

A. PHYSICAL
   1. TEMPERATURE - FINAL
   2. PRESSURE LOSS THROUGH SOFTENER

B. CHEMICAL
   1. ALKALINITY - FINAL
   2. TOTAL AND CALCIUM HARDNESS - RAW AND FINAL
   3. TOTAL DISSOLVED SOLIDS - FINAL
   4. pH - FINAL
   5. SOAP TEST - FINAL

SELECTIVITY COEFFICIENT

A. CATION RESIN

$$RA + B^+ \rightleftarrows RB + A^+$$

$$K_D = \frac{[A]_S}{[B]_S} \frac{[B]_R}{[A]_R}$$

K_D DEPENDS ON:

1. NATURE OF THE IONS
2. NATURE OF THE RESIN
3. EXTERNAL FACTORS SUCH AS TEMPERATURE
4. DEGREE OF SATURATION OF THE RESIN

B. ANION RESIN

$$RC + D^- \rightleftarrows RD + C^-$$

$$K_D = \frac{[C]_S}{[D]_S} \frac{[D]_R}{[C]_R}$$

TRANSPARENCY II4AFWS-#9

ION SELECTIVITIES

Cation	Selectivity	Anion	Selectivity
H^+	1.0	OH^-	1.0
Li^+	0.9	Benzene Sulfonate	500
Na^+	1.3	Salicylate	450
NH_4^+	1.6	Citrate	220
K^+	1.75	I^-	175
Rb^+	1.9	Phenolate	110
Cs^+	2.0	HSO_4^-	85
Ag^+	6.0	NO_3^-	65
Mn^{+2}	2/2	Br^-	50
Mg^{+2}, Fe^{+2}	2.4	CN^-	28
Zn, Co, Cu, Cd, Ni^{+2}	2.6-2.9	Cl^-	22
Ca^{+2}	3.4	HCO_3	6.0
Sr^{+2}	3.85	IO_3^-	5.5
Hg^{+2}, Pb^{+2}	5.L-5.4	Formate	4.6
Ba^{+2}	6.15	F^-	1.6

Transparency II4AFWS-#10

SELECTIVITY "RULES OF THUMB"

A. AT LOW CONCENTRATIONS SELECTIVITY INCREASES WITH INCREASED VALENCE.

B. AT LOW CONCENTRATION SELECTIVITY INCREASES WITH INCREASED ATOMIC NUMBER.

TYPES OF RESINS

A. CATION RESINS

1. STRONG ACID (SULFONIC)
   A) OPERATED IN H MODE OR NA MODE
2. WEAK ACID (CARBOXYLIC)
   B) OPERATED IN H MODE

B. ANION RESINS

1. STRONG BASE (QUATERNARY)
   A) OPERATED IN OH MODE OR OTHER (HCO_5)
2. WEAK BASE (TERTIARY AMINE)
   A) OPERATED IN OH MODE

C. MACRORETICULAR RESINS

1. VARIOUS ACTIVE GROUPS AND OPERATIONS

REGENERATES

A. Cation resins

1. Hydrochloric acid
2. Sulfuric acid
3. Nitric acid
4. Sodium chloride

B. Anion resins

1. Sodium hydroxide
2. Sodium bicarbonate
3. Sodium chloride
4. Ammonia

C. Macroreticular resins

1. Hydrochloric acid
2. Sodium chloride
3. Methonal

LABORATORY EVALUATION OF RESINS

A. PHYSICAL PROPERTIES
   *1. SCREEN ANALYSIS
   2. ATTRITION RESISTANCE
   3. CHEMICAL ATTRITION
   4. DENISITY
   5. VOID VOLUME
   6. HYDRAULIC PROPERTIES
   7. SWELLING
   8. SOLUBILITY
   9. AESTHETIC PROPERTIES

B. CHEMICAL PROPERTIES
   1. MOISTURE-HOLDING CAPACITY
   2. CHARACTERIZATION OF FUNCTIONAL GROUPS
   *3. TOTAL CAPACITY MEASUREMENTS
   4. SELECTIVITY AND EQUILIBRIUM MEASUREMENTS
   5. KINETIC MEASUREMENTS

TRANSPARENCY II4AFWS - #14

TRANSPARENCIES 15-20 REMOVED PRIOR TO BEING SHIPPED TO EDRS FOR FILMING DUE TO COPYRIGHT RESTRICTIONS.

INTERPRETATION AND APPLICATION OF LABORATORY DATA

A. CHECK COMPLIANCE WITH DESIGN STANDARDS

B. EVALUATE RESIN PROBLEMS

C. OPTIMAL CONTROL OF ION EXCHANGE UNITS

TYPICAL RESIN SPECIFICATIONS

PROPERTY	SPECIFICATION
MOISTURE	44-48%
VOLUME CAPACITY	1.90 MEQ/ML (MINIMUM)
WEIGHT CAPACITY	4.5 MEQ/GRAM (MINIMUM)
COLOR THROW	25 APHA (MAXIMUM)
ODOR AND TASTE	NONE
pH	6.0 - 7.0
HYDRAULIC EXPANSION	37 - 75% (700 ML/MIN FLOW RATE, 25°C, 2-IN. DIAMETER
DENSITY	43-54 LB/CU FT.
WHOLE BEADS	70% (MINIMUM)
COMPRESSIBILITY	5.0 GAL/SQ FT/MIN -0.6 LB/SQ FT/FT MAXIMUM 2.5 GAL/SQ FT/MIN -0.29 LB/SQ FT/FT
EFFECTIVE SIZE	0.4-0.6 MM
UNIFORMITY COEFFICIENT	1.7 (MAXIMUM)

EVALUATE RESIN PROBLEMS

A. CHECK PHYSICAL CHARACTERISTICS FOR EFFECTIVE SIZE TRENDS

B. CHECK CAPACITY TRENDS

C. CHECK DIFFERENT CHEMICAL METHODS FOR EFFECT ON RESIN CLEAN-UP

OPTIMAL CONTROL OF ION EXCHANGE UNIT

A. DETERMINE THE TRACE CAPACITY AND THE TRUE 2/3 POINT THROUGH THE LIFE OF THE UNIT.

B. DETERMINE WHICH REGENERATE WORKS BEST.

C. DETERMINE THE PROPER BACKWASH RATE.

D. DETERMINE WHEN ECONOMICALLY THE RESIN SHOULD BE CHEMICALLY CLEANED UP.

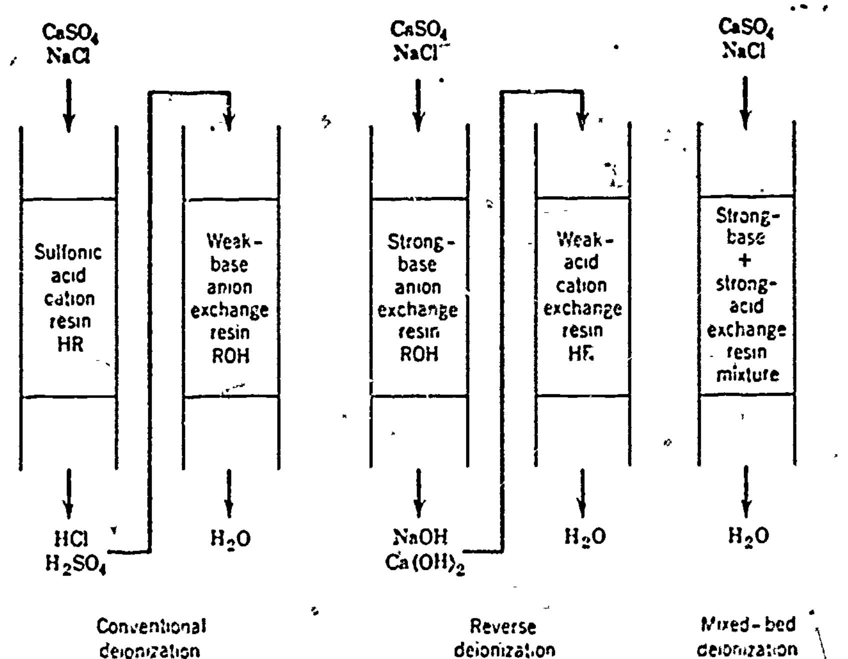
E. DETERMINE THE OPTIMAL REGENERATE DOSAGE AND TIME OF CONTACT.

PRINCIPLES OF DEIONIZATION

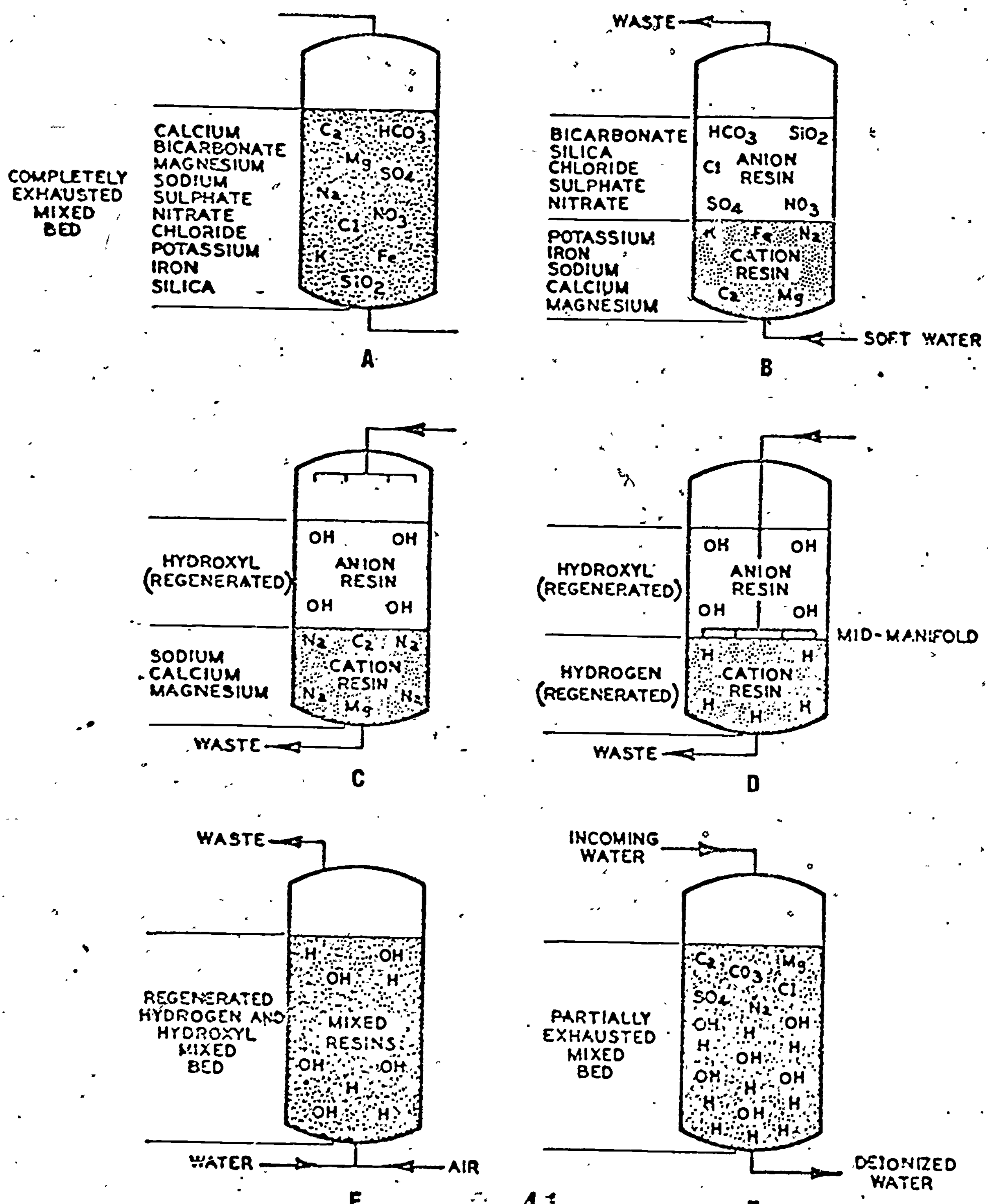
HARD WATER		CATION RESIN		EXHAUSTED CATION RESIN		ACIDS
$\left\{\begin{array}{l} CA\,(HCO_3)_2 \\ MG\,SO_4 \\ NA\,CL \end{array}\right\}$	+	$H\,R$	$\longrightarrow$	$\left\{\begin{array}{l} CA \\ MG \\ NA \end{array}\right\} R$	+	$\left\{\begin{array}{l} H_2CO_3 \\ H_2SO_4 \\ HCL \end{array}\right\}$

ACIDS		ANION RESIN		EXHAUSTED ANION RESIN		WATER
$\left\{\begin{array}{l} H_2CO_3 \\ H_2SO_4 \\ HCL \end{array}\right\}$	+	$R\,OH$	$\longrightarrow$	$R\left\{\begin{array}{l} CO_3 \\ SO_4 \\ CL \end{array}\right\}$	+	H_2O

TYPES OF DEIONIZATION



OPERATION OF MIXED BED DEIONIZER



TRANSPARENCY II4AFWS - #27

CLASS HANDOUT

for

Training Module II4AFWS

Handout for II4AFWS - ADVANCED ION EXCHANGE SOFTENING

I. Introduction

A. Principles of ion exchange softening.
B. Preventative maintenance for ion exchange softener.
C. Design evaluation for ion exchange softener.
D. Laboratory control forion exchange softener.

II. Theory of Ion Exchange

A. Selectivity coefficient

$$K_D = \frac{[A]_S}{[B]_S} \frac{[B]_R}{[A]_R}$$

Cation	Selectivity[b]	Anion	Selectivity[c]
H^+	1.0	OH^-	1.0
Li^+	0.9	Benzene sulfonate	500
Na^+	1.3	Salicylate	450
NH_4^+	1.6	Citrate	220
K^+	1.75	I^-	175
Rb^+	1.9	Phenolate	110
Cs^+	2.0	HSO_4^-	85
Ag^+	6.0	NO_3^-	65
Mn^{2+}	2.2	Br^-	50
Mg^{2+}, Fe^{2+}	2.4	CN^-	28
Zn, Co, Cu, Cd, Ni	2.6-2.9	Cl^-	22
Ca^{2+}	3.4	HCO_3^-	6.0
Sr^{2+}	3.85	IO_3^-	5.5
Hg^{2+}, Pb^{2+}	5.1-5.4	Formate	4.6
Ba^{2+}	6.15	F^-	1.6

RULES OF THUMB

1. At low concentrations selectivity increases with increased valence.
2. At low concentrations selectivity increases with increased atomic number.

B. Types of resins

1. Cations Resins

a) strong acid
b) weak acid

2. Anion Resins

a) strong base
b) weak base

3. Macroreticular Resins

C. Types of regenerates

1. Cation resins

a) hydrochloric acid
b) sulfuric acid
c) nitric acid
d) sodium chloride

2. Anion resins

a) sodium hydroxide
b) sodium bicarbonate
c) sodium chloride
d) ammonia

3. Macroreticular resins

a) hydrochloric acid
b) sodium chloride
c) methonal

III. Laboratory Evaluation Resins

A. General tests

1. Physical Properties

a) screen analysis
b) attrition resistance
c) chemical attrition
d) density
e) void volume
f) hydraulic properties
g) swelling
h) solubility
i) aesthetic properties

2. Chemical properties

a) moisture-holding capacity
b) characterization of functional groups
c) total capacity measurements
d) selectivity and equilibrium measurements
e) kinetic measurements.

B. Screen analysis

See Figure 1.

C. Total capacity measurements

1. General

See Figure 2.

2. Pretesting resin conditioning

See Figure 3.

3. Solids (or moisture) content determination

See Figure 4.

4. Cation exchange capacity

See Figure 5.

5. Anion exchange capacity

See Figure 6.

IV. Interpretation and Application of Laboratory Data

A. Check compliance with design standards.
B. Evaluate resin problems.
C. Optimal control of Ion exchange units.

V. Special Applications of Ion Exchange

A. Principles of deionization

$$\begin{matrix} Ca(HCO_3)_2 \\ MgSO_4 \\ NaCl \end{matrix} + HR \rightarrow \begin{matrix} Ca \\ Mg \\ Na \end{matrix} R + \begin{matrix} H_2CO_3 \\ H_2SO_4 \\ HCL \end{matrix}$$

$$\begin{matrix} H_2CO_3 \\ H_2SO_4 \\ HCL \end{matrix} + ROH \rightarrow R \begin{matrix} CO_3 \\ SO_4 \\ CL \end{matrix} + H_2O$$

B. Types of deionization

1. conventional deionization
2. reverse deionization
3. mixed-bed deionization

C. Operation of mixed-bed deionization

The method selected for screen analysis of ion-exchange resins is a function of the particle size of the resin to be screened and the use for which the resin is intended. Where the majority of the resin sample is retained on 100-mesh screens and the resin is to be used in aqueous systems a wet screening of the desired ionic form is to be preferred. Samples finer than 100 mesh, on the other hand, are more conveniently screened air- or oven-dried. In cases where dry-screening techniques are used, the fact that the majority of resins swell on hydration must be recognized. The methods employed are identical to those employed for sands, gravel, coal, etc. Normally one chooses 8-in. diameter screens of No. 16, 20, 30, 40, 50, 60, 70 and 100 U.S. screen sizes as a nest through which the sample is screened, and the amount retained by each screen is measured.

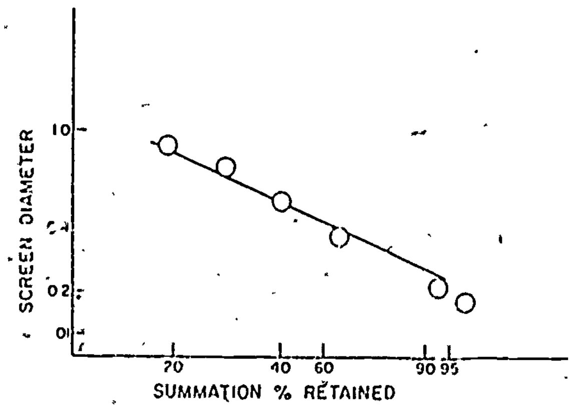


Figure 4.1. Screen analysis plot.

The screen analysis data are used to calculate the effective size and the uniformity of the sample. From the volume (or weight) of resin per screen cut and the total volume of all the cuts, the per cent of the sample in each screen cut and the cumulative per cent are calculated. Typical results are shown in Table 4.1. Using these results, a plot of cumulative per cent retained as a function of the screen opening in millimeters is made on normal probability paper. (Figure 4.1.)

TABLE 4.1. TYPICAL SCREEN ANALYSIS DATA

Screen Size (U.S.)		Screen Opening (mm)	Volume Resin (ml)	% Resin	Cumulative %
Retained on	16	1.19	0.5	0.24	0.24
	20	0.84	4.6	2.2	2.4
	30	0.59	37.6	18.0	20.4
	40	0.42	89.2	42.7	63.1
	50	0.30	61.7	29.5	92.6
	60	0.25	8.6	4.1	96.7
	70	0.21	5.0	2.4	99.1
	100	0.15	1.5	0.72	99.8
Passing	100		0.2	0.09	99.9

The best straight line is drawn through the points, giving greater weight to the points representing the largest resin fractions. From this line the effective size, which is the screen opening that will retain 90 per cent of the sample, is determined. From the data in Fig. 4.1 the effective size would be 0.34 mm. The mesh size retaining 40 per cent of the sample is also noted. From these two values the uniformity, which is equal to:

$$\frac{\text{Mesh size (mm) retaining 40\% of the sample}}{\text{Mesh size (mm) retaining 90\% of the sample}}$$

is calculated. In the illustrative example this would be

$$\frac{0.51}{0.34} \text{ or } 1.50$$

TOTAL CAPACITY MEASUREMENTS

The following section describes methods of testing for one of the most important characteristics of ion exchange resins—total capacity. Such total capacity is a constant for any given resin and must be distinguished from column capacities which define only the potential of a resin to remove specified ions under actual conditions of use to a target leakage value.

The techniques described here express total capacities in terms of milliequivalents per gram of dry resin. In order to obtain these values, it is necessary to know the percent solids of each resin being tested. This procedure is described below. In order to achieve some degree of standardization, capacity determinations are usually run on specific ionic forms for each type of exchanger. Thus, the capacity procedures speak of "conditioned" resin, and also listed below are suggested conditioning steps for each exchanger.

Although these determinations will yield only weight capacities, volume capacities can be determined where desirable by determining the density of the conditioned resin. This can be done by measuring a known weight of the conditioned resin in a graduated cylinder, but most workers find it more convenient and accurate to employ calibrated ion exchange columns in which the actual capacity will be determined. A buret-type column, therefore, is extremely useful. The volume capacity of any resin can be found by the following equation:

$$\text{Capacity } \frac{\text{meq.}}{\text{ml.}} = \text{Capacity } \frac{\text{meq.}}{\text{g.}} \times \text{Density } \frac{\text{g.}}{\text{ml.}} \times \frac{\%\ \text{Solids}}{100}$$

PRETESTING RESIN CONDITIONING

Before capacities can be determined, each ion exchange resin should be conditioned with an appropriate regenerant as listed below. Enough resin should be placed into a conditioning column to allow both a capacity determination and a separate amount for a solids determination (e.g., about five grams hydrated resin for capacity plus at least one gram and preferably three to five grams for solids). In each case about 10 bed volumes of regenerant should be passed through the column at a rate of 0.5 ml. per ml. of resin per minute, followed by a rinse with deionized water to the prescribed endpoint.

Type of Exchanger	Regenerant Solution	Rinse Requirement
strong acid cation	10% NaCl (SSCC)	Free of Cl—
	4% HCl (CEC)	Neutral to methyl orange
weak acid cation	4% HCl	Neutral to methyl orange
strong base anion	10% NaCl	Free of Cl—
weak base anion	4% NaOH	Until 1 drop of 0.1 N H_2SO_4 neutralizes 12 ml of effluent

As with all column operations, this regeneration technique should be preceded by a thorough backwashing. If the resin sample is dirty, backwashing should be continued until the overflow runs clear.

After rinsing, the sample should be transferred from the column to a Buchner funnel where excess moisture should be removed by drawing air through the funnel for five minutes using a water suction pump. The drained sample is now in the proper form for determining solids and capacity.

SOLIDS CONTENT DETERMINATION

All samples for this determination should be weighed at the same time as the sample for capacity determination. It is suggested that no less than one gram of sample be used for this determination (three to five grams are preferable). Accurately weigh the sample on an analytical balance, in a dry, tared weighing pan. Oven dry at 110° C. for at least eight house. Cool in a desiccator and weigh. The calculation is:

$$\frac{\text{Wt. oven dried resin} \times 100}{\text{Wt. resin before drying}} = \%\ \text{Solids}$$

$$100 - \%\ \text{Solids} = \%\ \text{Moisture}$$

FIGURE IIIAP.S - 14

CATION EXCHANGE CAPACITY

Principle: To neutralize the acid form of a cation exchange resin by contacting it with a known excess of a standard alkali. The residual alkali is tritrated with standard acid.

Reagents: 0.1 *N*. NaOH prepared in 5 percent NaCl
0.1 *N* HCl
Phenolphthalein indicator
Deionized water

Procedure:

1. Weigh accurately a one-gram sample of *conditioned* cation exchange resin together with another sample for solids determination. NOTE: If sample is a strong-acid cation exchange resin it should be conditioned with HCl rather than NaCl.
2. Place the capacity sample into a dry 250-ml. Erlenmeyer flask and pipette in exactly 200 ml. of standardized 0.1 *N* NaOH which has been prepared in 5 percent sodium chloride.
3. Stopper the flask and allow to stand overnight.
4. Back-titrate 50-ml. aliquots of the supernatent liquid with standardized 0.1 *N* HCl using a phenolphthalein endpoint. Be sure not to draw off any ion exchange resin beads.
5. Calculate capacity:

$$\frac{(200 \times N_{NaOH}) - 4(ml._{acid} \times N_{acid})}{\text{Sample wt.} \times \frac{\%\ \text{Solids}}{100}} = \frac{\text{Meq. cation exchange capacity}}{\text{Gram of dry H-form resin}}$$

FIGURE III-AP-S - 5

ANION EXCHANGE CAPACITY

Principle: Anion exchange capacity is determined by first converting the anion exchange resin to the chloride form and then eluting it with sodium nitrate.

Reagents: 4 percent HCl
Alcohol (acidified with 0.5 cc conc. HCl per liter alcohol)
1 *N* $NaNO_3$
Methyl orange indicator
0.1 *N* NaOH
5 percent K_2CrO_4
0.05 *N* $AgNO_3$

Procedure:

1. Weigh out accurately 5 grams of the conditioned sample along with another sample for solids determination.
2. Quantitatively transfer the capacity sample to an ion exchange column.
3. Pass through the column 1-liter of 4 percent HCl. Rinse with about 1 liter of alcohol. Flow rate should be 5 bed volumes per minute.
4. Pass through the column exactly 1 liter of 1 *N* $NaNO_3$, collecting the effluent in a 1-liter volumetric flask. Use the same flow rate.
5. Shake the flask well and transfer 100-ml. aliquots to an Erlenmeyer flask.
6. Add 2 drops of methyl orange and, if pink, just enough 0.1 *N* NaOH to change the color back to yellow.
7. Add 1 ml. of 5 percent K_2CrO_4 and titrate with 0.05 *N* $AgNO_3$ until color changes from yellow to yellow-orange.
8. Calculate the anion exchange capacity using the following equation:

$$\frac{\text{ml.}_{AgNO_3} \times N_{AgNO_3} \times 10}{\text{Sample wt.} \times \frac{\%\ \text{Solids}}{100}} = \frac{\text{Meq. anion exchange capacity}}{\text{Gram of dry free-base form resin}}$$

FIGURE III/APS - #5

EXAMINATION

for

Training Module II4AFWS

Examination for II4AFWS - Advanced Ion Exchange Softening

1. For each mg/l of magnesium removed __________ mg/l of sodium are released into the water in ion exchange softening.

2. List four periodic checks necessary for a good preventative maintenance program on an ion exchange softener.

   a.
   b.
   c.
   d.

3. List two physical and five chemical laboratory tests used to control an ion exchange softener.

   Physical:

   a.
   b.

   Chemical:

   a.
   b.
   c.
   d.
   e.

4. The two "Rules of Thumb" used to determine ion selectivity are:

   a.
   b.

5. List three general types of resins

   a.
   b.
   c.

6. List one physical and one chemical property of ion exchange resins that should be periodically checked in the laboratory.

   Physical:

   a.

   Chemical:

   a.

TRUE OR FALSE - CIRCLE THE CORRECT ANSWER

T or F 7. Softeners in Iowa are designed under the "Ten States Standards"

T or F 8. Softeners should be operated at 2/3 capacity and regenerated at .3 lb salt/1000 grains removed.

T or F 9. Deionization removes all ion except potassium.

T or F 10. The selectivity coefficient is used to determine the amount of salt needed for regeneration.

T or F 11. Generally the lower the valence of an ion the higher the selectivity.

T or F 12. Weak acid cation ion resins are very easy to regenerate, however, they are very expensive which limits their use.

T or F 13. Macoreticular resins have been developed only recently for use in deionization.

T or F 14. Cation resins can be regenerated with any strong acid.

T or F 15. Total exchange capacity for a softener generally increase with age due to the beads swelling and giving more exchange are.

T or F 16. Deionization is misleading because the process also removes non-ionizable material from the water.

T or F 17. Ion exchange softening replaces all hardness ion with sodium and anions with chloride.