

American Water Works Association

AWWA STANDARD

for

CATION EXCHANGER TEST PROCEDURES

(Ion-Exchange Materials—Cation Exchangers Operating on the Sodium Cycle—Commonly Termed "Zeolites")

This "Standard for Cation Exchanger Test Procedures" is intended to be a general guide for persons having occasion to purchase cation exchangers to be used in water purification, but it is not intended to be a substitute for, nor to be superior to, competent engineering judgment based upon a complete understanding of special conditions.

Approved as Standard by AWWA on May 4, 1951

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Foreword

The Water Purification Division of the American Water Works Association, acting in its field of routine activities, in 1932 organized a committee to develop specifications and methods of testing zeolites used in water treatment. A "Tentative Manual of Zeolite Test Procedures" was approved and published in 1943. The committee has been continued under the chairmanship of D. E. Davis and in 1948 submitted a series of revisions to the text of the Manual. The title of the earlier document has been changed to: "Standard for Cation Exchanger Test Procedures."

The committee feels that, since the main body of the Standard has been used quite extensively in the trade and has been sufficiently discussed, its acceptance as a permanent standard is warranted. Items about which opinion is still in a state of flux, however, have been included as an Appendix to the document.

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AWWA Standard for

Cation Exchanger Test Procedures**(Ion-Exchange Materials—Cation Exchangers Operating on the Sodium Cycle—Commonly Termed "Zeolites")****Section 1—Scope***Sec. 1.1—Sodium Cycle Only*

The term "cation exchanger," as hereinafter used in this Standard shall be taken to mean all granular cation-exchange materials operating on the sodium cycle, for removing hardness (Ca, Mg, etc., or iron (Fe) or manganese (Mn) in solution) from a water supply, employing common salt (sodium chloride (NaCl)) as a regenerant. This manual deals with cation exchangers operating on the sodium cycle only.

Sec. 1.2—Anion Exchangers Excluded

Anion exchangers employing alkaline compounds such as hydroxides and carbonates as regenerants require separate test procedures which are not included in this manual.

Sec. 1.3—Cation-Exchange Materials

Cation-exchange materials may be classed as:

1.3.1. Inorganic Materials (Siliceous):

- (1) Glauconite, or greensand (low capacity and high capacity)
- (2) Precipitated synthetic gel (sometimes called wet process)
- (3) Fused synthetic
- (4) Clay group or "Bentonites"
- (5) Miscellaneous

1.3.2. Organic Materials:

- (1) Coal derivatives (made from

coal, lignite, peat (humus-containing), etc.)

- (2) Resins
- (3) Miscellaneous

Sec. 1.4—Object of Tests

The object of the tests herein described is to determine, as nearly as possible, the physical and chemical properties of exchangers by the use of laboratory methods, keeping in mind the necessity of correlating laboratory tests with actual performance of exchangers in service. It is recognized that laboratory tests do not indicate the quality of an exchanger over a period of time and under definite conditions of service as reliably as does experience.

Sec. 1.5—Limitations of Tests

The tests are only an indication of apparent results (comparative in nature), with actual operating results to be determined (as far as exchange capacity, in grains or kilograins, is concerned) by properly regulated acceptance tests to be determined in each case, preferably after the material has been properly conditioned or normalized—(1) at the beginning of operations and (2) at the end of a week or a month or any other suitable period.

Section 2—Suggested Definitions*Sec. 2.1—Zeolite*

A zeolite is defined as any member of the family of hydrous silicates

(aluminosilicates), such as the phillipsite, the montmorillonite or the natrolite groups (which are so called because many species intumesce before the blowpipe) and some clay minerals, ultramine, glauconite and chabazites. A smaller degree of ion exchange is found in feldspars and kaolinite. All of these minerals contain alkali or alkaline earth metals, and it is these metals which will ionize and exchange with ions in solutions. They are considered as analogous to the feldspars in chemical constitution. Since the original designation of zeolites, it has been found that organic materials as well as inorganic materials can be used to produce cation-exchange materials. There is no scientific justification for using the term "zeolite" to designate any of the cation-exchange materials in use today. Some thirty or forty years' use of the word "zeolite," however, has identified it so closely with base exchange and cation exchange in general, that the term is now generally accepted as covering both base-exchange and cation-exchange substances.

Although there has been no serious objection to extending the use of the word "zeolite" from the mineralogical field to the field of water treatment because of the similarity between the cation-exchange reactions of these minerals in nature to the reactions of the cation exchangers in water treatment processes, the increasing deviation of the composition of cation exchangers from the zeolitic minerals makes it desirable to use the more precise term "cation exchanger."

Sec. 2.2—Cation

The cation is the positively charged particle, or ion, in an electrolytic solution which travels to the cathode (a metallic ion and H^+) and, after being

discharged, is evolved or deposited; hence it is any positive ion.

Sec. 2.3—Cation Exchange

Cation exchange is the property of exchanging a cation existing in a solid (cation exchanger) for another cation in solution. Exchange reactions are reversible and are used to a great extent in the field of water treatment.

Sec. 2.4—Anion

In an electrolytic solution, the anion is the negatively charged particle, or ion, which travels to the anode; hence it is any negative ion.

Sec. 2.5—Ion Exchange

Ion exchange is a reversible interchange of ions between a liquid phase and a solid (cation exchanger) involving no radical change in the structure of the solid.

Section 3—Suggested Terminology

Sec. 3.1—Samples

The procedures outlined in *Standard Method for the Examination of Water and Sewage* * [hereinafter called *Standard Methods*; all page numbers refer to the ninth edition] re: "Collection of Samples" (page 1) are suggested for sampling, preservation, etc., of any water to be tested according to the directions contained in this manual.

Sec. 3.2—Reports

The procedure outlined in *Standard Methods* re: "Expression of Chemical Results" (page 5) is suggested for reporting quantity determinations on any water tested according to directions contained in this manual. (See also footnote, page 468 of this manual.)

* *Standard Methods for the Examination of Water and Sewage*. Am. Public Health Assn. & Am. Water Works Assn., New York (9th ed., 1946).

Sec. 3.3—Supplemental Reports

This form of report may be supplemented by customarily accepted methods of stating mineral analyses of water.

Section 4—Taking of Samples

Sec. 4.1—Representative Samples

The importance of obtaining a truly representative sample cannot be over-emphasized. A sampling tube (Fig. 1) or a grain sampler* shall be employed in obtaining a representative sample, care being exercised to secure a full cross section, so that the effects of automatic sizing or segregation are taken into account.

Sec. 4.2—Samples for Shipping

If the material is shipped in containers, samples shall be taken from at



FIG. 1. Sampling Tube

least 5 per cent of them, scattered throughout the entire shipment. If the material is shipped in bulk, samples shall be taken from top, half way down and bottom from at least sixteen locations in the car. Samples from operating tanks or units shall total at least four samples per 100 sq.ft. of surface area, and in layers of lesser area a minimum of four samples shall be obtained.

These portions shall be mixed thoroughly into one composite sample of

* Grain Sampler No. 12, $\frac{3}{8}$ in. in diameter, as made by Eimer & Amend Co., is suggested. Either dry or wet samples can be obtained. This grain sampler consists of two revolving concentric tubes with suitable annular openings, the inner tube being provided with knobs for rotating purposes.

approximately 50 lb., then quartered or riffled to approximately 15 lb. of greensand or 9 lb. of synthetic or organic exchanger and immediately placed in an airtight container.

Sec. 4.3—Air-Drying Not Recommended

Air-drying is not recommended, since exchangers, particularly the organic types, vary greatly in water content, even when in equilibrium with "air," and the density (apparent) is markedly influenced by the water content. When the tests for moisture, density and porosity have been determined by the procedures later described, it is believed that the assembled data will make it possible to compare exchangers on a convenient and practical basis.

Sec. 4.4—Draining for Sampling

To obtain, with the sampling tube (Fig. 1), a sample that will contain material from the entire depth of the exchanger layer, the water shall be drained from the entire exchanger layer down to the level of the top part of the supporting gravel layer. The sampling tube shall be driven into the drained layer.

Great care shall be exercised in re-wetting the exchanger to remove air pockets by opening valves slowly, to fill the unit from below.

Section 5—Amount of Cation Exchanger for Tests

The approximate amounts of glauconite or greensand (at approximately 90 lb. per cubic foot) required for the various tests to be made according to these procedures are given in Table 1. Amounts of other exchangers shall be in the ratio of their dry weights to that of greensands.

TABLE 1
Amount of Exchanger for Tests

Test	Weight g.
Total moisture	3
Weight after backwashing	1,550
Sieve analysis	200
Exchange value	*
Chemical composition	10
Total	1,763

* Exchange value tests may be run on same sample weight-after-backwashing tests.

or thickened at the ends it will prove more sturdy.

Sec. 6.2—Calibration Procedure

A rubber stopper of proper size, containing a piece of glass tubing approximately $\frac{1}{4}$ in. in diameter, with the end flush to the small end of the stopper, shall be prepared. To the long end, a short piece of rubber tubing shall be attached. A "Hoffman"

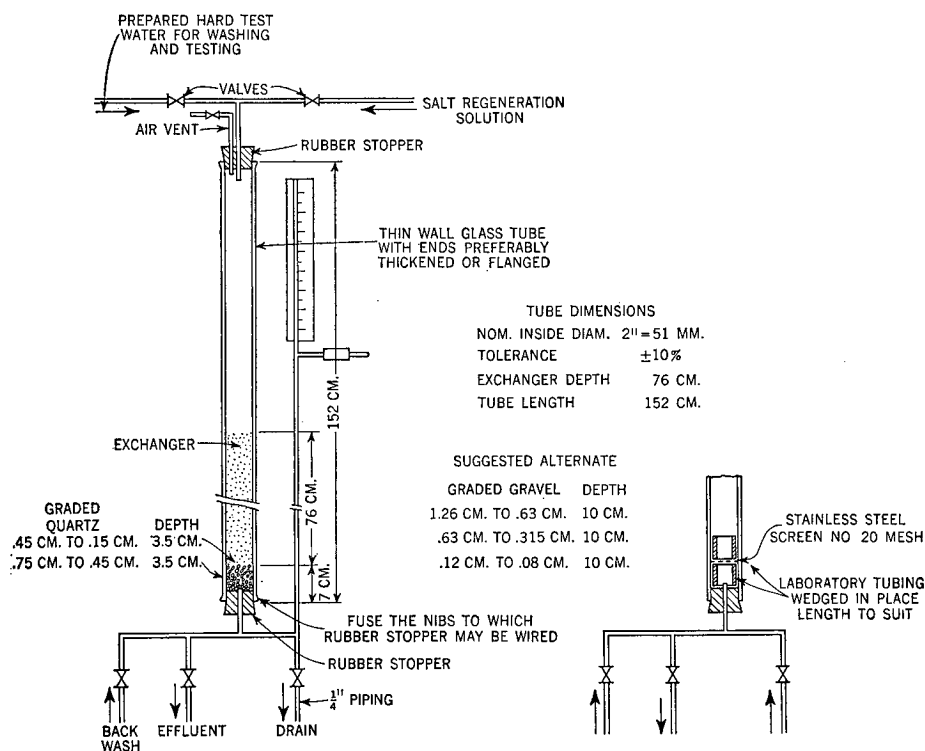


FIG. 2A. Standard Tube Arrangement, Showing Direction of Flow

Section 6—Tube Preparation and Calibration

Sec. 6.1—Type of Tube

The standard test tube unit shall be of light-walled glass (or other suitable material), with dimensions as shown in Fig. 2A. If the glass tube is flanged

screw clamp shall be used to regulate or to shut off flow. The rubber stopper shall be taped in place, for the weight of the layer of cation-exchange material is sufficient to loosen it. In mounting the tube, the bottom shall rest on a suitable support, and the tube shall be truly vertical, to prevent un-

desirable turning of the layer of exchanger.

On the bottom shall be placed 0.63 cm. graded quartz or suitable silica gravel (dirt- and oil-free), round and clean, to a depth of about 3.5 cm. and, on top, 0.315 cm. graded material to a depth of 3.5 cm. All of this material in place shall be washed by upflow until it is clean.

The tube shall be filled with water at 70°F. $\pm$ 10°F. (21°C. $\pm$ 6°C.) A height of 76 cm. above the quartz layer shall be measured with exactness, marking both top and bottom of the 76-cm. length; then the water shall be drained down to the top mark. Following this, the water between the top and bottom marks shall be drained into a graduate, measuring carefully the volume of water between the two marks.

Assuming that the measured volume equals 1,550 ml. and that 1 ml. = 0.0000354 cu. ft., then 1,550 ml. = 0.0548 cu. ft. occupied by 76 cm. in height. On this basis $\frac{0.0548}{2.5} = 0.02192$ sq. ft. equals the average area in the 76-cm. layer. The tube shall be permanently marked to show its area when the determination has been made.

Details of Fig. 2A tube arrangements are shown in Fig. 2B.

Sec. 6.3—Calibration of Entire Tube for Organic Cation Exchangers

For those exchangers in which the volume changes during the operation cycle, it may prove desirable to calibrate the entire tube by attaching a strip of graph paper,* $\frac{1}{2}$ in. wide, along the axis of the tube. Calibration may be made by successive additions of water (10 ml.) from the burette, with volumes marked on the graph in convenient

* 10 $\times$ 10 per $\frac{1}{2}$ in., tenth lines heavy—equivalent to K & E No. 359-11.

units. In such case, the "volume" factor shall be marked on the tube.

Sec. 6.4—Various Test Tube Diameters

Test tubes having a diameter other than that herein designated are being used, but it is felt that the diameter chosen will permit reasonably accurate and reproducible results with a minimum of sample (material) and test water requirements.

Section 7—Air-Drying

Sec. 7.1—Limitation

It is to be noted that under Sec. 4.3 air-drying is not recommended. Certain tests covered in this manual, however, state that air-drying of samples is a permissible procedure if reproducible results can be obtained. Therefore, a suitable air-drying procedure is given.

Sec. 7.2—Air-Drying Procedure

To insure reproducible results when air-drying materials, the following procedure shall be used:

The sample shall be spread over the bottom of a suitable container to a depth of $\frac{1}{2}$ in. Drying shall continue for 24 hr. and shall be facilitated by thorough mixing and spreading of the sample three times, at intervals of eight hours, for the organic materials. Twice the time and number of stirrings shall be employed for inorganic materials. On completion of the sieve analysis, the moisture content of the sample shall be determined. (See Supplementary Notes, p. 24.)

Sec. 7.3—Moisture and Density

The total moisture of this sample shall be determined as in Sec. 10 (see p. 7) and the density of the dried material determined as in Sec. A1 of the Appendix (see p. 17). The density of