

JIS

JAPANESE INDUSTRIAL STANDARD

Testing methods for determination of anions in highly purified water

JIS K 0556—1995

Translated and Published

by

Japanese Standards Association

In the event of any doubt arising,
the original Standard in Japanese is to be final authority.

UDC 628.1.038 : 543.34 : 546.12 : 546.226-325 :
546.175-323

JAPANESE INDUSTRIAL STANDARD

J I S

Testing methods for determination of
anions in highly purified water

K 0556-1995

1. **Scope** This Japanese Industrial Standard specifies the testing methods for determination of anions (fluoride ion, chloride ion, nitrite ion, bromide ion, phosphate ion, nitrate ion, and sulfate ion) in highly purified water.

Remarks 1. These methods can be applied to the determination of anions contained in very pure water, too, other than highly purified water.

2. The standards cited in this Standard are listed in Attached Table 1.

2. **Matters in common** The matters in common shall be as follows.

(1) **General rules** The general matters commonly applicable to chemical analysis shall follow JIS K 0050.

(2) **Ion chromatography** The general matters commonly applicable to ion chromatography shall follow JIS K 0127.

(3) **Definitions** The definitions of main terms used in this Standard shall follow those in JIS K 0101, JIS K 0102, JIS K 0127, JIS K 0211, and JIS K 0215.

(4) **Precision of repeatability** The precision of repeatability shall be shown by the variation coefficient (%) which has been obtained in repeated tests carried out in the determination range of each testing method⁽¹⁾.

Note (1) Variation coefficient (%) = $\frac{\sigma}{\bar{x}} \times 100$

where, σ : standard deviation

$\bar{x}$: averaged value

(5) **Water** Employ the water A1 to A3 specified in JIS K 0557, but when there is a specification in each procedure, follow it.

(6) **Reagents** Matters in common to reagents shall be as follows.

(a) When reagent is specified in Japanese Industrial Standards, use the reagent which is the best one or is suitable one of the kind, and when there is no one specified in Japanese Industrial Standards, use the reagent giving no hindrance to test.

(b) The concentration of solution of reagent is generally shown by g/l (when reagent is compound, use the mass of anhydrous) or mol/l (or mmol/l). The concentration of reference solution, however, is shown by mg/ml, µg/ml, or ng/ml.

- (c) The name of reagent is, principally coordinated with the nomenclature of compounds specified by the Chemical Society of Japan, based on the nomenclature system for inorganic compounds and for organic compounds prescribed by the International Union of Pure and Applied Chemistry, and with nomenclature system for JIS reagents.
 - (d) When handling reagents, waste, or others, be sufficiently careful according to laws and regulations concerned.
- (7) Glassware Glassware shall be principally the ones specified in JIS R 3503 and JIS R 3505.

The desiccant used in a desiccator shall be principally silicagel⁽²⁾.

Note ⁽²⁾ Use silicagel desiccant for packing, type A class 1 specified in JIS Z 0701.

- (8) Working curve (ion chromatography) When a working curve is to be plotted, divide from 4 steps to 6 steps the determination range shown in testing method, and take reference solution to suit these steps. Prepare the working curve to fall in the range of determination.

In case of ion chromatography, use a newly plotted working curve when test is carried out, and when a lot of samples are tested successively, confirm indicated values, employing reference solution, timely in the middle of the test.

- (9) Environment for test Carry out preferably these tests in the environment (for instance, in a clean room), which has cleanliness class 3 or higher specified in JIS B 9920.
- (10) Notes and Remarks Notes and Remarks are serially numbered in each clause.

Remarks: At sampling and any procedure in testing, it is important to pay the utmost attention not to get any contamination on the sample. Give consideration to the shape and material of tools used and their washing method.

Be so careful that contamination may not come from an analyzer. It is preferable to let him put on the gloves used in a clean room.

3. Sampling Sampling shall be as follows.

- (1) Container for sample The container for sample shall be the airtight made of hard glass, polyethylene, polypropylene, or polystyrene.

(2) Cleaning of sample container

- (a) Wash sufficiently sample containers with A1 water.
- (b) Put A1 water into a sample container by a quarter of its volume, stopper it, and shake violently for about 30 seconds to make washing. Repeat this procedure five times.
- (c) Fill it with A3 water (or the same water as the water to be tested), stopper closely it, allow it to stand for 16 hours or longer, and discard the water.
- (d) Fill it with the same water as that at (c), stopper closely it, and allow it to stand until sampling is tried.

(3) Sampling procedures

- (a) Wash sufficiently the outlet of water purifying apparatus or the end of sampling valve⁽¹⁾ of main piping, with the water for test, and fix a sample introducing pipe (made of soft synthetic resin) which has been sufficiently washed with A1 water⁽²⁾ previously.
- (b) Discard the water in a sample container, and carry out the procedure in (2)(b) with the water to be tested.
- (c) Make the end of the sample introducing pipe touch bottom of the sample container, let flow out sample by about 5 times volume of sample container, take out the sample introducing pipe, and stopper closely it after fully washing the stopper with sample.

Notes (1) Sampling valve shall be made of anticorrosive material such as stainless steel or the like.

- (2) When a sample introducing pipe has long distance, let flow out sample to completely displace the water staying from the sampling valve to the end of sample introducing pipe, and then further let flow out freely the sample for about 5 minutes successively. In case of sampling from main piping, it is preferable to flow out always sample at the rate of about 1 l/min.

4. Fluoride ion (F⁻) Fluoride ion (F⁻) in sample shall be determined with applying ion chromatography.

Determination range: 0.5 µgF⁻/l to 10 µgF⁻/l⁽¹⁾, precision of repeatability: 5% to 20% by variation coefficient⁽¹⁾.

Note (1) It depends upon apparatus and measuring condition.

(1) Reagents Reagents shall be as follows.

- (a) Water A2 or A3 water specified in JIS K 0557, or the water purified to the same level as this. Confirm its usability by practicing a blank test. Use this water when preparing reagent solution for this test, or when carrying out operations.
- (b) Eluent The type of eluent⁽²⁾ differs according to the kind of apparatus and of anion exchanger to be packed in a separation column, therefore it shall be needed to confirm the separation condition owing to the operation shown in Note⁽⁵⁾.
- (c) Regenerant Regenerant⁽³⁾ is needed when a suppressor is used, but its type differs according to the kind of apparatus and of suppressor. In advance, combine the suppressor and separation column, and carry out the procedures in Note⁽⁵⁾ to confirm the performance of regenerant.
- (d) Fluoride ion reference solution (0.1 mgF⁻/ml) Heat sodium fluoride, reference material for volumetric analysis, specified in JIS K 8005, at 500°C for about 1 hour, and allow it to cool in a desiccator. Weigh its 0.221 g, dissolve it in a little of water, transfer it into a 1000 ml measuring flask, and add water up to the marked line. Store it in a polyethylene bottle.
- (e) Fluoride ion reference solution (0.5 µgF⁻/ml) Take 5 ml of fluoride ion reference solution (0.1 mgF⁻/ml) into a 1000 ml measuring flask, and add water up to the marked line. Prepare it when it is needed.
- (f) Fluoride ion reference solution (50 ngF⁻/ml) Take 10 ml of fluoride ion reference solution (0.5 µgF⁻/ml) into a 100 ml measuring flask, and add water up to the marked line. Prepare it when it is needed.

Notes ⁽²⁾ For example, the eluent shall be prepared as follows:

Case of using a suppressor Choose one method of the following three.

Sodium hydrogencarbonate solution (1.2 mmol/l + sodium carbonate solution (4.4 mmol/l) Weigh 0.101 g of sodium hydrogencarbonate specified in JIS K 8622 and 0.466 g of sodium carbonate specified in JIS K 8625, dissolve both in a little of water, and add water to make total 1 l.

Sodium hydrogencarbonate solution (1 mmol/l) Weigh 0.084 g of sodium hydrogencarbonate specified in JIS K 8622, dissolve it in a little of water, and add water to make total 1 l.

Sodium borate solution (5 mmol/l) Weigh 1.907 g of sodium tetraborate decahydrate (borax) specified in JIS K 8866, dissolve it in a little of water, and add water to make total 1 l.

Case of no suppressor Choose either of the following.

Sodium 1-octanesulfonate solution (1 mmol/l) + boric acid solution (20 mmol/l) Weigh 0.216 g of sodium 1-octanesulfonate and 1.237 g of boric acid specified in JIS K 8863, dissolve both in a little of water, and add water to make total 1 l.

4-Hydroxybenzoic acid solution (8 mmol/l) + 2-[bis(hydroxyethyl)imino]-2-(hydroxymethyl)-1,3-propanediol solution (3.2 mmol/l) (pH 4.0) Weigh 1.105 g of 4-hydroxybenzoic acid and 0.670 g of 2-[bis(hydroxyethyl)imino]-2-(hydroxymethyl)-1,3-propanediol[bis(2-hydroxyethyl)iminotris(hydroxymethyl)methane], dissolve both in a little of water, and add water to make total 1 l.

(³) For example, the regenerant shall be prepared as follows.
Choose either of the following.

Sulfuric acid (15 mmol/l) Take sulfuric acid (0.5 mol/l) (add little by little 30 ml of sulfuric acid specified in JIS K 8951 in about 500 ml of water, and after cooling add water to make 1 l.) 30 ml, and dilute it with water to make total 1 l.

Sulfuric acid (25 mmol/l) Take 50 ml of sulfuric acid (0.5 mol/l), and dilute it with water to make total 1 l.

(2) Implements and apparatus Implements and apparatus shall be as follows.

(2.1) Microsyringe 0.1 ml to 2 ml

(2.2) Ion chromatograph Though there are two types as the type combined both separation column and suppressor, and the type of separation column alone, either will do, but satisfying the following conditions and capable of separating fluoride ion and chloride ion.

(a) Separation column Pipe for column made of stainless steel or synthetic resin(⁴), and packed with strong basic anion-exchanging resin (type I) (surface-layer coated type or porous silica type)(⁵).

(b) Suppressor Serviceable for exchanging cation in eluent with hydrogen ion, and composed of cation-exchange membrane or cation-exchanger with equal performance to the membrane. It is alternatively called a removing column.

(c) Detector Electric conductivity detector

(2.3) Recorder Follow 4.2(6) of JIS K 0127.