

JIS

JAPANESE INDUSTRIAL STANDARD

**Testing methods for
determination of silica in
highly purified water**

JIS K 0555—1995

Translated and Published

by

Japanese Standards Association

Printed in Japan

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

JAPANESE INDUSTRIAL STANDARD

J I S

Testing methods for determination of silica
in highly purified water

K 0555-1995

1. Scope This Japanese Industrial Standard specifies the testing methods for determination of silica in highly purified water.

Remarks 1. These methods can be applied to the determination of silica 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) Absorptiometry The general matters commonly applicable to absorptiometry shall follow JIS K 0115.

(3) Electrothermal-type atomic absorptiometry The general matters commonly applicable to electrothermal-type atomic absorptiometry (hereafter referred to as "electrothermal atomic absorptiometry") shall follow JIS K 0121.

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

(5) Range of determination The range of determination by absorptiometry shall be shown by the mass (μg) in the final solution, and that by electrothermal atomic absorptiometry by the concentration ($\mu\text{g/l}$) in the final solution.

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

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

where, σ : standard deviation

$\bar{x}$: average

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

(8) 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. The concentration of reference solution, however, is shown by mg/ml or µg/ml.
 - (c) The concentration, given in parentheses behind the name of the solution of reagent, means the approximate value except the case of reference solution. For instance, ascorbic acid solution (100 g/l) means L(+)-ascorbic acid solution whose concentration is approximately 100 g/l.
 - (d) The concentration of liquid reagent is expressed by the mixing ratio with water [reagent (a+b)]. This expression means mixing of a ml of reagent and b ml of water, and can be used in case of nitric acid or sulfuric acid according to JIS K 0050. When these reagents are used not diluted, however, the name of reagent alone will do.
 - (e) When handling reagents, waste, or others, be sufficiently careful according to laws and regulations concerned.
- (9) Glassware Glassware shall be principally the ones specified in JIS R 3503 and JIS R 3505.
- The desiccant used in a dessicator shall be principally silica gel ⁽²⁾.
- Note ⁽²⁾ Use silica gel desiccant for packing, type A class 1 specified in JIS Z 0701.
- (10) Working curve (absorptiometry, electrothermal atomic absorptiometry)
When a working curve is to be plotted, divide from 4 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 electrothermal atomic absorptiometry, 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.
- In case of absorptiometry, the working curve, which has been plotted in advance, can be used.
- (11) 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.
- (12) Note, Remarks, and Figure Note, Remarks, and Figure shall be serially numbered in each clause.
- (13) Marking of result When there are 2 or more testing methods for making test, testing method shall be attached.

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

Be so careful that contamination may not come from an analyst. 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 container made of polyethylene, polypropylene, or polystyrene.
- (2) Cleaning of sample container
 - (a) Wash sufficiently sample container 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 with water the outlet of water purifying apparatus or the end of sampling valve ⁽¹⁾ of main piping, 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 a sample introducing pipe touch bottom of a 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 let sample flow out always at the rate of about 1 l/min from the valve.

4. Testing method For the determination of silica, either molybdenum-blue extraction absorptiometry or electrothermal atomic absorptiometry shall be adopted.

4.1 Molybdenum-blue extraction absorptiometry Make ionic silica react with hexaammonium heptamolybdate to produce heteropoly compound, reduce it with L(+)-ascorbic acid to get molybdenum blue, extract it with 1-butanol, and measure the absorbance found in organic phase to determine silica.

Determination range: SiO₂ 0.5μg to 10 μg

Precision of repeatability: 5 % to 20 % by variation coefficient