



JAPANESE
INDUSTRIAL
STANDARD

Translated and Published by
Japanese Standards Association

JIS K 0068 : 2001

(JCIA/JSA)

**Test methods for water content
of chemical products**

ICS 71.040.40

Descriptors : water content determination, karl fischer method, karl fischer reagent,
moisture measurement, distillation methods of analysis, evaporation
residue determination

Reference number : JIS K 0068 : 2001 (E)

Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee, as the result of proposal for revision of Japanese Industrial Standard submitted by the Japan Chemical Industry Association (JCIA)/the Japanese Standards Association (JSA) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law. Consequently **JIS K 0068 : 1992** is replaced with this Standard. Attention is drawn to the possibility that some parts of this Standard may conflict with a patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have technical properties. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying the patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have the said technical properties.

Date of Establishment: 1966-08-01

Date of Revision: 2001-04-20

Date of Public Notice in Official Gazette: 2001-04-20

Investigated by: Japanese Industrial Standards Committee
Divisional Council on Chemical

JIS K 0068:2001, First English edition published in 2001-11

Translated and published by: Japanese Standards Association
4-1-24, Akasaka, Minato-ku, Tokyo, 107-8440 JAPAN

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Printed in Japan

Test methods for water content of chemical products

Introduction This Japanese Industrial Standard has been prepared based on the first edition of **ISO 760 : 1978** *Determination of water—Karl Fischer method (General method)*, however, this International Standard has specified only Karl Fischer method that is traditional volumetric titration method, which means this method is behind the times, therefore the required methods (such as Karl Fischer method by coulometric titration, drying loss method, distillation method and so on) are supplemented in this Japanese Industrial Standard, and additionally the reagents and others concerning the testing methods have been specified after their technical contents in International Standards are modified.

1 Scope This Japanese Industrial Standard specifies the general methods to measure water content in solid or liquid chemical products.

Remarks 1 The chemical products shall be the overall substance manufactured by chemical reactions, and when the test methods other than those in this Standard are specified in the standard for respective product or for the group of products, the methods specified in the standard shall be conformed.

2 Because some chemical products have volatility, explosiveness, radioactivity, and other properties, this Standard may not be able to secure sufficient safety condition when being applied. The methods adopted in this Standard are general methods, therefore they shall be applied only when the safety measures are sufficiently confirmed.

3 The standard corresponding to this Standard is as follows.

In addition, abbreviations which denote the degree of correspondence in the contents between the relevant International Standard and **JIS** are IDT (identical), MOD (modified) and NEQ (not equivalent) according to **ISO/IEC Guide 21**.

ISO 760 : 1978 *Determination of water—Karl Fischer method (General method)* (NEQ)

2 Normative references The standards indicated in Attached Table 1 contain provisions which, through reference in this Standard, constitute provisions of this Standard. The most recent editions of the standards (including amendments) shall be applied.

3 Definition For the purpose of this Standard, the definitions given in **JIS K 0211** and **JIS K 0213** and the following definition apply.

a) **titer** When water is titrated with Karl Fischer reagent, the titer means the mass of water equivalent to unit volume of Karl Fischer reagent. It is expressed with mg/ml.

4 Matters in common Matters in common to measurements shall follow the description in **JIS K 0050**, and the method for rounding-off of numerical values follow **JIS Z 8401**. Matters in common to Karl Fischer titration shall follow **JIS K 0113**.

5 Kinds of test methods The kinds of test methods shall be as follows.

a) **Karl Fischer titration method**

- 1) Volumetric titration method
- 2) Coulometric titration method
- 3) Water vaporizing—volumetric titration method, or water vaporizing—coulometric titration method

b) **Drying loss method**

c) **Distillation method**

6 Karl Fischer titration method

6.1 Summary Karl Fischer titration method shall be the method to determine water content by means of titrating water using Karl Fischer reagent for finding titrant required, and is classified into volumetric titration method, coulometric titration method, and the methods which combine these methods with water vaporizing method, depending on the property of sample or its water content.

Remarks 1 This cannot be applicable to the sample containing the substance, other than water, which reacts with Karl Fischer reagent (hereafter referred to as “interfering substance”). If the sample solvent or electrolyte previously prepared to eliminate interference is used or if water vaporizing method is used, however, this method can be applicable. The typical interfering substances are as follows:

- a) Those reacting with iodine: strong basic substance, oxidizing substance, reducing substance.
- b) Those reacting other components than iodine: ketones or the like which react with methanol to produce water.

2 Coulometric titration method is usually applicable to the case of low water content, and for the sample containing 2 % or more of water, volumetric titration method is recommended.

6.2 Sampling Sampling shall be as follows.

- a) A sample container shall be tightly closed, and be dried up at about 105 °C before being used, then cooled in a desiccator. Immediately after taking it out from the desiccator, it shall be stoppered.
- b) Carry out sampling quickly to make the amount of sample become 80 % or more capacity of the container, and stopper closely it immediately.
- c) Sufficiently stir the sample taken in the container to make it uniform.
- d) When solid sample makes lump or granule, crush it to suitable grain size as powder. In this case, be careful about dispersion of water or being moistened.
- e) When the temperature of the sample is other than room temperature, carry out sampling after sample temperature becomes room temperature owing to letting it stand in room for a while.

- f) Immediately after opening the container, take the sample quickly, and stopper soon it closely.
- g) The amount of sample shall be decided as shown in Table 1 and Table 2 depending on the amount of water contained.

Table 1 Amount of sample for volumetric titration method

Expected water content %	Amount of sample ⁽¹⁾ ml or g
0.1 or under	10 to 20
0.1 to 0.5	5
0.5 to 1	2
1 to 5	0.5
5 to 10	0.3
10 to 50	0.1
50 or over	0.03

Note (1) The sample amount in Table 1 was calculated on the base that the titer of Karl Fischer reagent is 3 mgH₂O/ml. Therefore, in case of the titer having seriously different value from this, the amount of sample must be controlled.

Table 2 Amount of sample for coulometric titration method

Expected water content %	Amount of sample ml or g
0.05 or under	5 to 10
0.05 to 0.1	2
0.1 to 0.2	1
0.2 to 0.5	0.5
0.5 to 2.0	0.1

6.3 Volumetric titration method

6.3.1 Summary Put sample solvent in a titration vessel, drop Karl Fischer reagent to make it water-free condition, and add a sample into this solution to dissolve it or extract water from it. Then, titrate using Karl Fischer reagent and find water content from the amount of titrant.

6.3.2 Equipment and apparatus Equipment and apparatus shall be as follows.

- a) **Apparatus for volumetric titration** This apparatus⁽²⁾ is composed of a titration part, controlling part, and indicating and recording part. The example of this construction is shown in Fig. 1. The volumetric titration apparatus shall conform to the following except those specified in **JIS K 0113**.

Note (2) Every connection of glass parts shall be ground, and they shall be applied with the grease, which does not react with or is not dissolved in Karl Fischer reagent, to prevent air moisture.

Remarks : In a volumetric titration apparatus, a Magic Eye or a voltage-controlled current detector with ammeter can be used for finding an end point. In this case, titration shall be manually carried out.

- 1) **Titration vessel** This vessel shall be a 100 ml to 250 ml capacity glass-made flat-bottomed flask equipped with a sample charging aperture, detector (a pair of platinum electrodes), titration nozzle, and a drying tube keeping desiccant such as silica gel, and after putting a rotor it is placed on a magnetic stirrer whose stirring rate was suitably adjusted. The aperture for sample charging should be closely stoppered using a glass plug, but in case of using a microsyringe or injector for sample charging, the stopper made of either stainless steel or tetrafluoroethylene resin with a packing shall be attached instead. The example of the stopper is shown in Fig. 2.
- 2) **Buret** The buret shall be a 10 ml or 20 ml capacity piston-type buret driven by a pulse motor, and its smallest discharged amount shall be 0.01 ml to 0.02 ml.

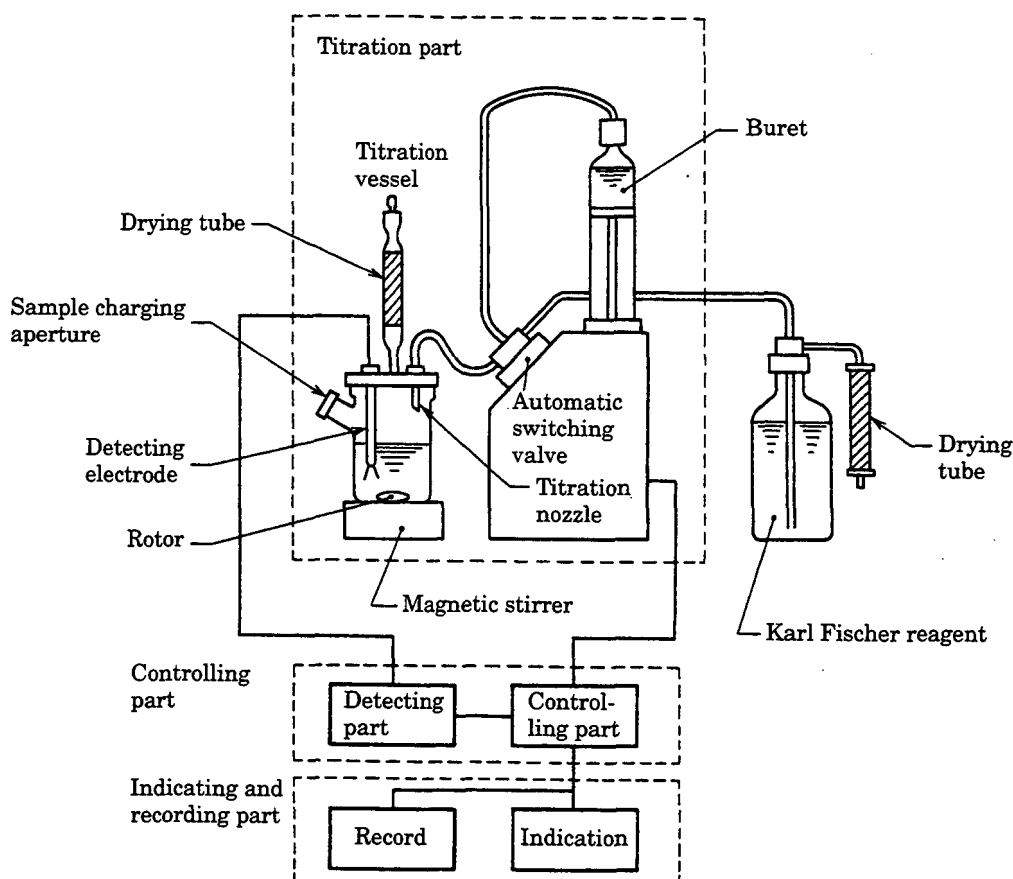


Fig. 1 Example of automatic titration apparatus for volumetric titration

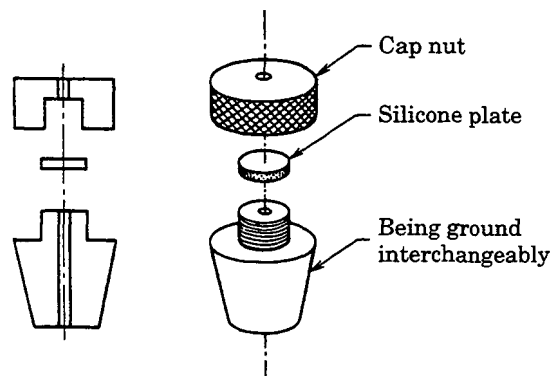


Fig. 2 Example of stopper

b) **Sampler** Sampler shall be either of the following two.

1) **Sampler for liquid sample**

- 1.1) **Microsyringe** The capacity of a microsyringe shall be 10 μ l, 25 μ l, 50 μ l, or 100 μ l, and the length of a needle be 50 mm or longer.
- 1.2) **Injector** The capacity of an injector shall be 1 ml to 20 ml, and the needle measures 1 mm to 2 mm in outside diameter and 50 mm to 100 mm in length. A rubber plug shall be fixed at the end of the needle for sealing, and a round-type silicone rubber, measuring 13 mm to 15 mm in diameter and 5 mm to 7 mm in thickness, can be used for this purpose.
- 1.3) **Transfer pipet** 5 ml to 20 ml
- 1.4) **Dropping bottle** This dropping bottle should be equipped with a dropping pipet whose one drop has about 0.03 ml volume. Its example is shown in Fig. 3.

2) **Sampler for solid sample**

- 2.1) **Straight pipe-type sampler for solid sample** Its example is shown in Fig. 4. This sampler can be used for the sample shaped as fluidized powder, granule, or small lump.
- 2.2) **Sampler for small amount of solid sample** Its example is shown in Fig. 5. This sampler can be used for the sample whose amount is about 0.4 g or less.
- 2.3) **Sampler for high viscous semisolid sample** Its example is shown in Fig. 6. This sampler can be used for high viscous semisolid.
- 2.4) **Syringe-type sampler for solid sample** Its example is shown in Fig. 7. This sampler can be used for solidified powder or viscous solid.

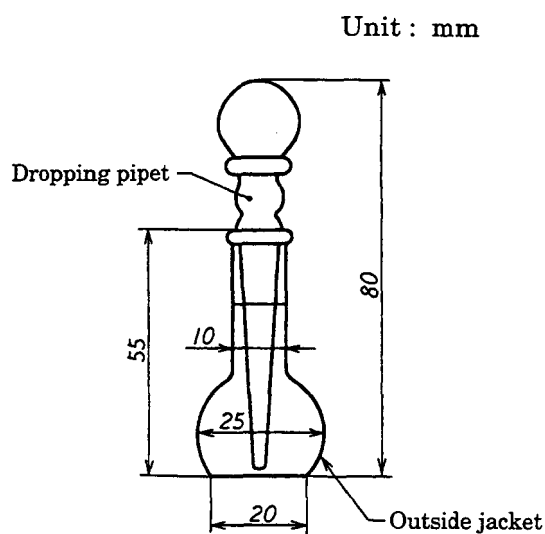


Fig. 3 Example of dropping bottle

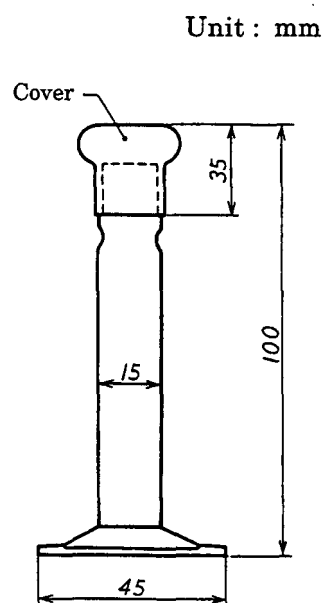


Fig. 4 Example of straight pipe-type sampler for solid sample

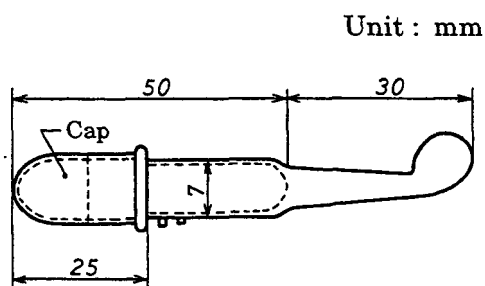


Fig. 5 Example of sampler for small amount of solid sample

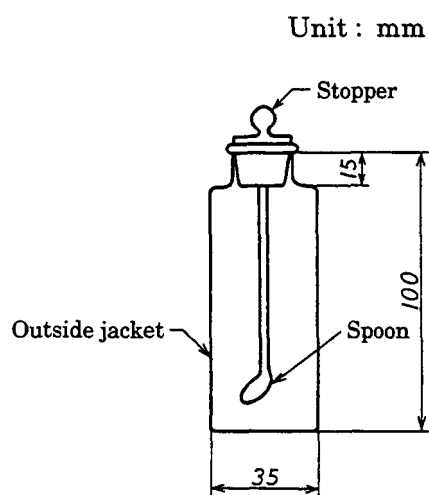


Fig. 6 Example of sampler for high viscous semisolid sample

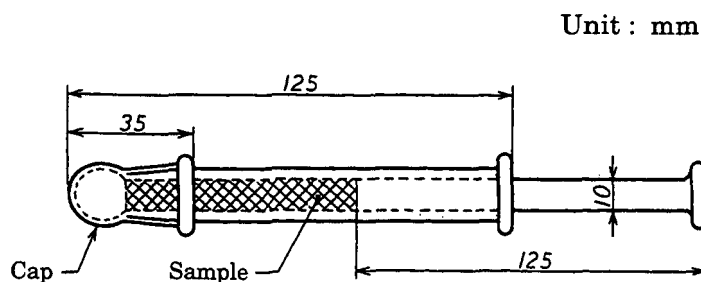


Fig. 7 Example of syringe-type sampler for solid sample

- c) **Balance (chemical balance)** Chemical balance or electrobalance.
Remarks : An electrobalance can be conveniently used if it is connected to an automatic titration device through a suitable interface.
- d) **Desiccator** The desiccator shall be specified in **JIS R 3503**, and contain silica gel⁽³⁾ as desiccant.
Note ⁽³⁾ Use the silica gel previously heated at about 150 °C, and treat it similarly when regenerating.
- e) **Flask** 2 L Erlenmeyer flask which has been ground interchangeably.

6.3.3 Reagents Reagents shall be as follows.

- a) **Methanol** Place 1 000 ml methanol specified in **JIS K 8891** in a 2 L interchangeably ground Erlenmeyer flask, add 5 g of magnesium powder specified in **JIS K 8876**, and if necessary add 0.5 g of iodine specified in **JIS K 8920** for accelerator of reaction. After evolution of gas finished, distill methanol while shielding moisture, and store it with close stoppering. Make water content 0.05 % or less.
- b) **Chloroform** Place 1 000 ml of chloroform specified in **JIS K 8322** in a 2 L interchangeably ground Erlenmeyer flask, add 30 g of synthesized zeolite for drying, and let it stand for about 8 h with close stoppering. While being let stand, swirl it slightly from time to time. Then, let it stand for another 16 h, take an aliquot of chloroform, and store it with close stoppering. Make water content 0.01 % or less.
- c) **2-methoxyethanol** Distill 2-methoxyethanol specified in **JIS K 8895** while shielding moisture, discard initial distillate, and store the intermediately distilled distillate with close stoppering. Make water content 0.05 % or less.
- d) **Diethylene glycol monoethyl ether** Employ diethylene glycol monoethyl ether whose purity is 98 % or more and whose water content is 0.05 % or less. When its water content is over this value, add 30 g of synthesized zeolite for drying in 1 000 ml diethylene glycol monoethyl ether, stopper it closely, let it stand for about 8 h while gentle swirling from time to time, let it stand for another 16 h, and use transparent supernatant diethylene glycol monoethyl ether. Store it while shielding moisture.
- e) **Propylene carbonate** Employ propylene carbonate whose purity is 98 % or more and whose water content is 0.3 % or less.
- f) **Pyridine** Place 1 000 ml of pyridine specified in **JIS K 8777** in a 2 L interchangeably ground Erlenmeyer flask, add either about 10 g of potassium hydroxide specified in **JIS K 8547** or about 10 g of barium oxide, store it for several days with close stoppering, distill it while shielding moisture, and store with close stoppering. Make water content 0.05 % or less.
- g) **Imidazole** Employ imidazole whose purity is 99 % or more and whose water content is 0.3 % or less.
- h) **2-(methylamino) pyridine** Employ 2-(methylamino) pyridine whose purity is 98 % or more and whose water content is 0.3 % or less.

- i) **Sodium (+)-tartrate dihydrate** Recrystallize sodium (+)-tartrate dihydrate specified in **JIS K 8540** from water solution, remove carefully attached water, make its water content constant by letting it stand under room temperature and about 50 % of relative humidity, and then use it⁽⁴⁾.

Note ⁽⁴⁾ Put it in the desiccator for moisture control for 48 h or more at 10 °C to 30 °C.

- j) **Sample solvent for titration** As a sample solvent for titration, any of the following solvents can be used according to the property of sample or the purpose of test. Water content in any solvent shall be 0.05 % or less.

Remarks : The sample solvent for titration found on the market can be used.

- 1) **Methanol** Follow a).

- 2) **Methanol—chloroform mixture solvent** Mix methanol and chloroform with volumetric ratio of 1 : 3 or 1 : 4. This solvent can be used for such as petroleum or oil-and-fat.

Remarks : Which of mixture ratios should be chosen is depending on the solubility of sample. When large amount of sample is necessary because of trace of moisture, the mixture ratio with large chloroform is chosen.

- 3) **Methanol—formamide mixture solvent** Mix methanol and formamide specified in **JIS K 8873** with volumetric ratio of 1 : 2. This solvent can be used for such as saccharides or proteins.

- 4) **Chloroform—propylene carbonate mixture solvent** Mix chloroform and propylene carbonate with volumetric ratio of 7 : 1. This solvent can be used for such as ketones, organic acids, aminobenzenes.

- 5) **Pyridine—ethylene glycol mixture solvent** Mix pyridine and ethylene glycol specified in **JIS K 8105** with volumetric ratio of 5 : 1. This solvent can be used for such as carbonyl compounds.

- 6) **Pyridine—propylene glycol mixture solvent** Mix pyridine and propylene glycol specified in **JIS K 8837** with volumetric ratio of 3 : 1. This solvent can be used for such as aldehydes.

- 7) **Salicylic acid—methanol solution** Dissolve 150 g of salicylic acid specified in **JIS K 8392** in 1000 ml of methanol. This solvent can be used for the amine whose pKa value is 9 or more.

- k) **Karl Fischer reagent**

- 1) **Preparation** Dissolve 85 g of iodine specified in **JIS K 8920** in 670 ml of methanol, add 270 ml of pyridine, and after sufficiently mixing cool with icy water. Pass dried sulfur dioxide through this solution with care not to make it over 20 °C temperature, and continue on this operation until its weight increase reaches 65 g⁽⁵⁾.

In this case, instead of methanol, 2-methoxyethanol, chloroform, diethylene glycol monoethyl ether, propylene carbonate, or their mixture is effective. Instead of pyridine, imidazole or 2-(methylamino) pyridine is effective. When the titer of Karl Fischer reagent is controlled by dilution, the solvent, which