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Test methods for water content of chemical products

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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.

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In the event of any doubts arising as to the contents,
the original JIS is to be the final authority.

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 mgH_2O/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.