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JAPANESE INDUSTRIAL STANDARD

Test methods for water content of chemical products

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Test methods for water content of chemical products

K 0068-1992

1. Scope This Japanese Industrial Standard specifies general methods to test water content of chemical products which are liquid or solid.

Remarks 1. The chemical products mentioned here generally mean various products prepared through chemical reaction, however, when measuring methods other than these methods are prescribed in the standard of other individual product or group of products, the test should conform to the method in the standard.

2. In some chemical products, the safety for the tests can not always be secured when the tests are carried out because of its volatility, explosiveness, or radioactivity. The methods prescribed in this standard should be applicable to the products of which safety is satisfactorily confirmed since they are only general methods.

3. The standards cited in this standard are shown in Attached Table 1.

4. The International Standard corresponding to this standard is shown in Attached Table 2.

2. General matters

2.1 Definition of terms The definition of main terms used in this standard shall be as follows except those defined in JIS K 0211 and JIS K 0213.

(1) Titer When water is determined with Karl Fischer reagent, the titer means the mass of water equivalent to unit volume of Karl Fischer reagent and is expressed with mg/ml.

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

3. Kind of test methods

The kind of test methods shall be as follows.

(1) Karl Fischer titration method

(a) Volumetric titration method

(b) Coulometric titration method

(c) Water vaporizing methods

(2) Drying loss method

(3) Distillation method

4. Karl Fischer titration method

4.1 Summary Karl Fischer titration method shall be the method to determine water content by means of titrating water using Karl Fischer reagent for finding titant required, and is classified into volumetric titration method, coulometric titration method and 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 can react 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:

(1) Those reacting with iodine: strong basic substance, acidic substance, reducing substance.

(2) Those reacting other 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 water volumetric titration method is recommended.

4.2 Sampling Sampling shall be as follows.

(1) 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 a desiccator, it shall be stoppered.

(2) Carry out sampling quickly to make the amount of sample become 80 % or more capacity of the container, and stopper it immediately.

(3) Sufficiently stir the sample taken in the container to make it uniform.

(4) 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.

(5) 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.

(6) Immediately after opening the container, take the sample, and quickly stopper it closely.

(7) 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 less	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 more	0.03

Note ([1]) The sample amount in Table 1 was calculated on the base that the titer of Karl Fischer reagent is 3 mg H_2O/ml. Therefore, in case of the titer having seriously different value to 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 to 0.05	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

4.3 Volumetric titration method

4.3.1 Summary Put sample solvent in a titration vessel, drip 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.

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

(1) Apparatus for volumetric titration This apparatus([2]) is composed of a titration part, controlling part, 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, to prevent air moisture, which does not react with or dissolve Karl Fischer reagent.