



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 6283-2008

**Chemical products -- Determination of water Karl-Fischer
method (general method)
(English Translation)**

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Foreword

This Standard is not equivalent for the degree of consistency with ISO 760:1978 Determination of Water Karl - Fischer Method (General Method).

This Standard replaced GB/T 6283-1986 Chemical Products - Determination of Water.

Compared with GB/T 6283-1986, this Standard mainly has the following changes.

--- Add the content of "this Standard is not applicable to the sample that can react with the main components of Karl - Fischer Reagent and produce water; neither applicable to the determination of water in sample that can reduce the iodine or oxidized iodide";

--- Change the "water equivalent" of Karl - Fischer Reagent into "titer";

--- Add the content of "Select other formulation of Karl- Fischer Reagent on the market according to the nature of the sample";

--- Rearrange the order of the standard appendixes.

This Standard's Appendix A, B, C, D are the normative ones.

This Standard was proposed by China Petroleum and Chemical Industry Federation.

This Standard shall be under the jurisdiction of National Technical Committee for Standardization of Chemical (SAC/TC 63).

Drafting organizations of this Standard. China Petroleum & Chemical Corporation Beijing Yanshan Branch, and Zhonghua Chemical Industry Institute of Standardization.

Chief drafting staffs of this Standard. Cui Guanghong, Yang Jianhai, Weijing, Bi Xiaoxia, and Su Xiaoyan.

The historical edition replaced by this Standard is as follows.

--- GB/T 6283-1986.

Chemical Products - Determination of Water Karl -Fischer Method (General Method)

1 Scope

This Standard specifies a general method for the determination of the free or crystal water content of a sample by the Karl - Fischer visual method and the coulometric method.

This Standard is applicable to the determination of free or crystal water content in the most organic and o inorganic solid, liquid chemical products.

This Standard is not applicable to the sample that can react with the main components of Karl - Fischer Reagent and produce water; neither applicable to the determination of water in sample that can reduce the iodine or oxidized iodide.

In some cases, the sample requires pretreatment measures, which shall be specified in the corresponding national standards.

When there is no instrument for the coulometric method, the visual method can be used; it is a direct titration, but only can used for the colorless solution. The coulometric method includes direct titration and back titration methods. No matter which titration is adopted, the result is more accurate; therefore, the coulometric method is recommended to be used.

2 Normative References

The provisions in following documents become the provisions of this Standard through reference in this Standard. For dated references, the subsequent amendments (excluding corrigendum) or revisions do not apply to this Standard, however, parties who reach an agreement based on this Standard are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

GB/T 6682 Water for Analytical Laboratory Use - Specification and Test Methods (GB/T 6682-2008, ISO 3696:1987, MOD)

3 Principle

Any water (free or crystal water) present in the sample is quantitatively reacted with Karl - Fischer Reagent (a solution consists of iodine, sulfur dioxide, pyridine and methanol) of known titer.

5.1 Water

Tested water shall meet the specification of Grade-III water stipulated in GB/T 6682.

5.2 Methanol

Analytically pure. If the mass fraction of water in reagent is greater than 0.05%, add 50g of 5A molecular sieve into 500mL of methanol; plug the bottle stopper; place overnight; absorb the supernatant to use.

5.4 Iodine

Analytically pure.

5.5 Pyridine

Analytically pure. If the mass fraction of water in reagent is greater than 0.05%, add 50g of 5A molecular sieve into 500mL of pyridine; plug the bottle stopper; place overnight, absorb the supernatant to use.

5.6 Glacial acetic acid

Analytical pure.

5.9 Anhydrous sodium sulfite

Chemically pure.

5.10 Sulfur dioxide

The sulfur dioxide both loaded with steel bottle and generated through sulfuric acid decomposing the sodium sulfite shall go through the dehydrating and drying treatment.

The sulfur dioxide generation unit can refer to Appendix A.

5.11 Sample solvent

The mixture containing 4 volumes of methanol and 1 volume of pyridine; or the mixture containing 4 volumes of ethylene glycol monomethylether and 1 volume of pyridine (especially applicable to the carbonyl-containing compound). In special cases, other solvent

can also be used, such as glacial acetic acid, pyridine, or mixture containing 1 volume of methanol and 3 volumes of chloroform.

5.14 Sodium tartrate ($\text{Na}_2\text{C}_4\text{H}_4\text{O}_6 \cdot 2\text{H}_2\text{O}$)

Analytically pure.

5.15 Water-methanol standard solution (10g water/L)

Use micro burette or pipette to inject 1mL of pure water into fully dried 100mL volumetric flask containing about 50mL of methanol;

5.16 Water-methanol standard solution (2g water/L)

Use micro burette or pipette to inject 1mL of pure water into fully dried 500mL volumetric flask containing about 100mL of methanol;

5.17 Silicone grease

Used for lubricating frost glass joints.

6 Apparatus

All used glassware shall be pre-dried at 130°C oven for 30min; then stored and cooled off in the desiccator containing the desiccant.

6.1 Direct titration (visual method or coulometric method)

6.1.1

The apparatus in this method can refer to Appendix C, the apparatus of this Standard consists of the following parts.

6.1.1.1

Automatic burette. 25mL, fine tip, scale division of 0.05mL, connect the protection tube filled with desiccant, and prevent the atmospheric moisture into the tube.

6.1.1.5 Reagent bottle with Karl - Fischer reagent, capacity 3L, brown bottle, filling

tube inserting into automatic burette through the ground plug.

6.1.1.6 Bi-join rubber ball, connect with the drying bottle filled with desiccant; so that transfer the dry air to the reagent bottle, and fill the burette with the reagent.