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GB/T 6283-2008

Replacing GB/T 6283-1986

Chemical Products – Determination of Water Karl • Fischer Method (General Method)

(ISO 760: 1978, Determination of Water Karl • Fischer Method
(General Method), NEQ)

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Table of Contents

Foreword.....	3
1 Scope	4
2 Normative References.....	4
3 Principle	5
4 Reaction Formula	5
5 Reagents and Materials	5
6 Apparatus.....	8
7 Visual Method.....	10
8 Direct Coulometric Titration Method	12
9 Coulometric Back-Titration Method	14
Appendix A (Normative) Sulfur Dioxide Generating Equipment	18
Appendix B (Normative) Water-Methanol Standard Solution for Calibration of Karl • Fischer Reagent.....	19
Appendix C (Normative) Visual ¹⁾ or Direct Coulometric Titrator	21
Appendix D (Normative) Coulometric Back-Titration Apparatus.....	24

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)

bottle with stopper; add about 85g of iodine, shake till the iodine is totally dissolved; then add 270mL of pyridine, tighten the stopper, shake till the fully mixing. Take the following methods to dissolve 65g of sulfur dioxide into the solution.

When injecting the sulfur dioxide, replace the stopper with a rubber plug where the thermometer, inlet glass tube (about 10mm from the bottle bottom, and tube diameter about 6mm), and air-ventilating capillary are equipped with.

Place the whole device and the ice bath on the balance, weight, accurate to 1g; use the hose to connect the sulfur dioxide steel bottle (or sulfur dioxide generator outlet) with drying tower filled with desiccant and air inlet glass tube; then slowly open the air inlet switch.

Adjust the flow rate of sulfur dioxide, so that it is completely absorbed; the liquid level in the inlet tube has no rising phenomenon.

As the mass slowly increases, keep balance through adjusting the balance weights, the solution temperature shall not exceed 20°C; when mass reaching 65g, immediately close the air inlet switch.

Quickly remove the connecting hose, then weigh the glass bottle and air inlet device; dissolve the mass of sulfur dioxide to be 60g~70g. Slightly excessive is not a problem.

Tighten the stopper, mix the solution, leave it in the dark for at least 24h before use.

The titer of such reagent is about 3.5mg/mL~4.5mg/mL. If it is prepared by methanol, it need day-to-day calibration; if it is prepared by ethylene glycol monomethylether, then it needn't calibration.

Use sample solvent to dilute the prepared solution, then the Karl • Fischer reagent with lower titer can be obtained.

The reagent shall be stored in the brown reagent bottle, placed in the dark, and prevent the influence from the atmospheric moisture.

NOTE: since the reaction is heat removing, from the beginning of the reaction, cool off the brown bottle, and maintain at about 0°C. E.g.: immerse into the ice batch or shredding solid carbon dioxide (dry ice).

In the newly prepared reagent, due to some unknown reactions, the titer of the reagent may decrease quickly at the beginning; after that it decreases very slowly.

Other formulation of Karl • Fischer reagent on the market can also be selected according to the sample nature. The determination result after such selection shall be consistent with that of preparing Karl • Fischer reagent stipulated in this Standard.

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

150r/min~300r/min.

6.2.1.6 End-point electricity measuring device, see Figure D.3 in Appendix D.

6.2.2 Medical syringe, with appropriate volume, which has been corrected.

6.2.3 Small glass tube (called sample tube), one end closed, the other end sealed by rubber plug; used for weighing specimen and adding materials into the titration vessel; e.g.: weigh sodium tartrate (about 0.250g) for calibration of Karl • Fischer reagent, or weigh solid specimen.

7 Visual Method

7.1 End-point determination principle

The color of iodine in the Karl • Fischer reagent vanished as encountered water in the to-be-tested specimen; the first excessive drop of reagent shall show color.

7.2 Operating procedures

7.2.1 Calibration of Karl • Fischer reagent

7.2.1.1 Assemble the apparatus as per the Appendix C. Lubricate the joints with silicone grease; use syringe, through rubber plug, to inject 25mL of methanol into the titration vessel; start the electromagnetic stirrer; in order to react with slight amount of water in the methanol, add Karl • Fischer reagent with automatic burette, till the solution turns to brown.

7.2.1.2 In small glass tube, weigh 0.250g of sodium tartrate, accurate to 0.0001g; remove the rubber plug, add it swiftly into titration vessel within several seconds, then weigh the small tube, the mass of used sodium tartrate (m_1) is determined through subtraction.

Also can use burette to add about 0.040g of water for calibration. Weigh the burette mass before and after the titration; the mass of used water (m_2) is determined through subtraction.

Use water-methanol standard solution for calibration; refer to B.1 in Appendix B.

Use Karl • Fischer reagent for calibration to titrate the known amount of water till the solution turns to brown like the 7.2.1.1; then record the volume of consumed Karl • Fischer reagent (V_1).

7.2.2 Determination

Discharge the residual liquid in the titration vessel thoroughly through the discharge

Where:

m_0 – specimen mass (solid specimen), in g;

V_0 – specimen volume (liquid specimen), in mL;

V_2 – when testing, the volume of consumed Karl • Fischer reagent, in mL;

ρ – specimen density at 20°C (liquid specimen), in g/mL;

T – titer of Karl • Fischer reagent calculated by 7.3.1, in mg/mL.

8 Direct Coulometric Titration Method

8.1 End-point determination principle

Ensure there is a potential difference between two platinum electrodes; when the solution contains water, the cathodic polarization reverses the current flow; then titrate the end-point through cathodic depolarization accompanied by abruptly increasing current (shown by suitable electrical device).

8.2 Operating procedures

8.2.1 Calibration of Karl • Fischer reagent

8.2.1.1 Assemble the apparatus as per the Figure C.1 in Appendix C. Lubricate the joints with silicone grease; use syringe, through rubber plug, to inject 25mL of methanol into the titration vessel; start the electromagnetic stirrer; and connect with the end-point electricity measuring device.

Adjust the apparatus, so that the electrodes have a 1V~2V potential difference; at the same time, the ammeter indicates a low current, generally about a few microamps. In order to react with the slight amount of water in the methanol, add Karl • Fischer reagent till the current indicated by the ammeter is increased to 10 μ A~20 μ A, and maintain stable for 1min.

8.2.1.2 In small glass tube, weigh 0.250g of sodium tartrate, accurate to 0.0001g; remove the rubber plug, add it swiftly into titration vessel within several seconds, then weigh the small tube, the mass of used sodium tartrate (m_3) is determined through subtraction.

Also can use burette to add about 0.040g of water for calibration. Weigh the burette mass before and after the titration; the mass of used water (m_4) is determined through subtraction.

Use water-methanol standard solution for calibration; refer to B.2 in Appendix B.

(m_5) here] is determined through subtraction.

Also can use burette to add about 0.040g of water for calibration. Weigh the burette mass before and after the titration; the mass of used water (m_6) is determined through subtraction.

Then add the known excessive volume (V_5) of Karl • Fischer reagent till the solution turns to brown, wait for 30s; use standard water-methanol solution to back-titrate the excessive reagent till the ammeter pointer is suddenly back to zero; record the volume of the consumed standard solution.

9.2.2 Corresponding values between Karl • Fischer reagent and standard water-methanol solution

Partially release the titration vessel, ensure the electrodes are still immersed in the solution stipulated in 9.2.1.

Use the first burette to add 20mL to-be-tested Karl • Fischer reagent; use the water-methanol standard solution in the second burette to titrate till the ammeter pointer is suddenly back to zero; record the volume of the consumed standard solution (V_7).

9.2.3 Determination

Discharge the residual liquid in the titration vessel thoroughly through the discharge nozzle; use syringe, through the rubber plug, to inject 25mL (the stipulated volume of the to-be-tested specimen) of methanol or other solvent; start the electromagnetic stirrer; in order to react with slight amount of water in the methanol, add excessive (about 2mL) Karl • Fischer reagent; then titrate water-methanol standard solution till the ammeter pointer is suddenly back to zero.

The specimen injection, if it is liquid, use syringe to inject; if it is solid powder, add with small tube, accurate to 0.0001g.

Add the known excessive volume (V_8) of Karl • Fischer reagent till the solution turns to brown, wait for 30s; use water-methanol standard solution to back-titrate the excessive reagent till the ammeter pointer is suddenly back to zero. Record the volume of the consumed standard solution (V_9).

NOTE: in order to accurately determine the moisture of the specimen, take proper amount of specimen as per its water content; so that the volume of Karl • Fischer reagent used for titration can be read; if necessary, increase the specimen amount and solvent in proportion, and use suitable volume of titration vessel.

9.3 Result expression

9.3.1 The titer of Karl • Fischer reagent T is expressed in mg/mL; and calculated by Formula (9) or (10):

Appendix B

(Normative)

Water-Methanol Standard Solution for Calibration of Karl • Fischer Reagent

B.1 Visual method

B.1.1 If use water-methanol standard solution to replace sodium tartrate for calibration of Karl • Fischer reagent; the relevant visual titration in 7.2.1.2 is modified as follows:

Use syringe to inject 10.0mL of methanol into the titration vessel; use Karl • Fischer reagent to titrate till the solution turns to brown the same as 7.2.1.1; record the volume of consumed solution (V_{10}).

In the same method, inject 10.0mL of water-methanol standard solution; use the Karl • Fischer reagent waiting for calibration to titrate and add known amount of water, till the solution turns to the same brown; record the volume of consumed solution (V_{11}).

B.1.2 In addition, 7.3.1 is modified as follows:

The titer of Karl • Fischer reagent T is expressed in mg/mL; and calculated by Formula (B.1):

$$T = \frac{100}{V_{11} - V_{10}} \dots\dots\dots (B.1)$$

Where:

100 – mass of water contained in 10mL of water-methanol standard solution, in mg;

V_{10} – volume of consumed Karl • Fischer reagent for titrating 10mL of methanol; in mL;

V_{11} – volume of consumed Karl • Fischer reagent for titrating 10mL of water-methanol standard solution; in mL.

B.2 Direct Coulometric Titration Method

B.2.1 If use water-methanol standard solution to replace sodium tartrate for calibration of Karl • Fischer reagent; the relevant direct coulometric titration in 8.2.1.2 is modified as follows:

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