GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 65.080
G 20

GB/T 8577-2010

Replacing GB/T 8577-2002

Determination of free water for compound fertilizers - Karl Fischer method

复混肥料中游离水含量的测定 卡尔•费休法

Issued on: September 26, 2010 **Implemented on: March 1, 2011**

Issued by: General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China;

Standardization Administration of the People's Republic of China.

Table of Contents

Determination of free water for compound fertilizers - Karl Fischer method

1 Scope

This Standard specifies the determination of free water content for compound fertilizers by the method of using dioxane to extract the free water from the fertilizer and then using the Karl Fischer reagent to titrate.

2 Normative references

The following standards contain provisions which, through reference in this Standard, constitute provisions of this Standard. For dated reference, subsequent amendments to (excluding corrections to), or revisions of, any of these publications do not apply. However, the parties to agreements based on this Standard are encouraged to investigate the possibility of applying the most recent editions of the standards. For undated references, the latest edition of the normative document referred to applies.

GB/T 6283 Chemical products - Determination of water - Karl Fischer method (general method) (GB/T 6283-2008, ISO 760:1978, NEQ)

GB/T 8571 Preparation of laboratory samples for compound fertilizers

HG/T 2843 Chemical fertilizer products - Standard volumetric standard reagent and indicator solutions for chemical analysis

3 Principle

The free water in the sample is quantitatively reacted with the Karl Fischer reagent with the known titer of water. The reactions are as follows:

$H_2O + I_2 + SO_2 + 3C_5H_5N = 2C_5H_5N \cdot HI + C_5H_5N \cdot SO_3$

$C_5H_5N \cdot SO_3 + CH_3OH = C_5H_5NH \cdot OSO_2OCH_3$

4 Reagents and materials

The reagents, solutions and water used in this Standard shall comply with the specifications of HG/T 2843 when their specification and formulation are not specified.

(except that it must be used for arbitration. Under normal cases, absolute ethanol or methanol can be used); SHAKE or oscillate for several minutes; STAND for 15 min, and then SHAKE or oscillate for several minutes. After the sample is slightly settled, TAKE part of the solution to centrifuge in the centrifuge tube with a brine bottle rubber stopper.

DRAIN the residual solution in the titration vessel through the outlet; ADD 50 mL of methanol to the titration vessel; the amount of methanol shall be enough to submerge the electrode; TURN on the power; TURN on the electromagnetic stirrer; as the same with titration of Karl Fischer reagent; USE Karl Fischer reagent to titrate until the galvanometer has the same skew as the calibration; MAINTAIN stable for 1 min.

TAKE 5.0 mL of dioxane extract from the centrifuge tube by syringe; INJECT into the titration vessel via the feed port; TITRATE to the end with Karl Fischer reagent; RECORD the volume (V_1) of the consumed Karl Fischer reagent.

When dioxane is used as the extractant, the residue solution in the titration vessel shall be drained after three titrations; ADD methanol; TITRATE to the end with Karl Fischer reagent, followed by the next determination.

In the same manner, determine the volume (V_2) of the Karl Fischer reagent consumed by 5.0 mL of dioxane.

7 Expression of analysis results

7.1 Calculation of analysis results

Free water content w, expressed as mass fraction (%), is calculated according to formula (1):

$$w = \frac{T(V_1 - V_2)}{m \times \frac{5}{50} \times 1\,000} \times 100 = \frac{(V_1 - V_2)T}{m} \quad \cdots\cdots (1)$$

where:

V_1 - the value of the volume of the Karl Fischer reagent consumed by titrating 5.0 mL of dioxane extracts, in milliliters (mL);

V_2 - the value of the volume of the Karl Fischer reagent consumed by titrating 5.0 mL of dioxane, in milliliters (mL);

T - the value of the titer of water of Karl Fischer reagent on, in milligrams per milliliter (mg/mL);

m - the value of the sample mass, in grams (g).

This is an excerpt of the PDF (Some pages are marked off intentionally)

------ The End ------