ICS 65.100
CCS G23

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 1600-2021

Replacing GB/T 1600-2001

Testing method of water for pesticides

农药水分测定方法

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Table of Contents

1 Scope

GB/T 1600-2021 is the Chinese national standard on testing method of water for pesticides, in the field of agriculture. The /T suffix marks it as a recommended standard: it is not compulsory by itself, but it becomes binding as soon as a contract, a tender or a customer specification calls it up - which in practice is how most foreign buyers meet it. It was issued on 31 December 2021 by the State Administration for Market Regulation; Standardization Administration of China, and has been in force since 1 July 2022. Classification: ICS 65.100, CCS G23. This page is published from the official record of the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

This document describes testing methods of water for pesticides by the Karl Fischer method and the azeotropic distillation method. This document applies to the determination of water for pesticides and their processed preparations.

1.5 V or

2.0 V battery pack: connected in parallel with a variable resistor of about 2 000 Omega. A microammeter is connected in series with the platinum electrode. When

0.2 mL excess Karl Fischer reagent flows through the platinum electrode, adjust the variable resistor so that the initial current does not exceed 20 mA. Each time Karl Fischer reagent is added, the ammeter pointer deflects once, but quickly returns to its original position. When the titration reaches the end point, the deflection time lasts longer. Ammeter:

full-scale deflection is not greater than 100 μA.

4.2.1.4 Reference materials calibrated by Karl Fischer reagent 4.2.1.4.1 Sodium tartrate dihydrate as reference material Add 20 mL of methanol to the titration container, use Karl Fischer reagent to titrate to the end point; quickly add

0.15 g ~

0.20 g (accurate to

0.1 mg) of sodium tartrate; stir until it is completely dissolved (about 3 min); then, continue to add Karl Fischer reagent dropwise at a speed of 1 mL/min to the end point. The water equivalent of Karl Fischer reagent is calculated according to Formula (1): Where: w1 - mass fraction of water, expressed as percentage (%); Vt - volume of Karl Fischer reagent consumed by the titration reagent, in milliliters (mL); cE - water equivalent of Karl Fischer reagent, in milligrams per milliliter (mg/mL); ms - mass of the sample, in grams (g).

4.2.1.6 Tolerance When the mass fraction of water is greater than 1%, the relative difference between two parallel measurements shall not be greater than 5%; when the mass fraction of water is 0.1% ~ 1%, the relative difference between two parallel measurements shall not be greater than 10%; when the mass of water is less than 0.1%, the relative difference between two parallel measurements shall not be greater than 20%. Respectively take the arithmetic mean value as the measurement result.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the version corresponding to that date is applicable to this document; for undated references, the latest version (including all amendments) is applicable to this document.

GB/T 8170-2008, Rules of rounding off for numerical values & expression and judgment of limiting values

3 Terms and definitions

No terms and definitions need to be defined in this document.

4 Test method

Warning - Personnel using this document shall have practical experience in laboratory work. This document does not address all safety concerns. It is the user's responsibility to take appropriate safety and health measures.

4.1 General provisions The reagents and water used in this document refer to analytical reagents and distilled water unless other requirements are noted.

4.2.1 Karl Fischer chemical titration method

4.2.1.1 Method summary Disperse the sample in methanol; use a standard Karl Fischer reagent of known water equivalent for titration.

4.2.1.2 Reagents and solutions 4.2.1.2.1 Anhydrous methanol: The mass fraction of water shall not be greater than 0.03%. Take 5 g ~ 6 g of magnesium (or magnesium strips) with a smooth surface and

0.5 g of iodine; place them in a round-bottomed flask; add 70 mL ~ 80 mL of methanol; heat and reflux on a water bath until all the magnesium forms flocculent magnesium methoxide; then, add 900 mL of methanol; continue to reflux for 30 minutes; then, carry out fractionation, and collect anhydrous methanol at 64.5 °C ~ 65 °C. The instrument used shall be dried in advance, and the part connected to the atmosphere shall be connected to a drying tube filled with calcium chloride or silica gel. 4.2.1.2.2 Anhydrous pyridine: The mass fraction of water shall not be greater than 0.1%. Pyridine passes through a glass tube filled with granular potassium hydroxide. The length of the tube is 40 cm ~ 50 cm, the diameter is

1.5 cm ~ 2 cm, and the height of the potassium hydroxide is about 30 cm. After treatment, carry out fractionation and collect the fractions at 114 °C ~ 116 °C. 4.2.1.2.3 Iodine: sublimate again and place it in a sulfuric acid desiccator for 48 hours before use. 4.2.1.2.4 Silica gel: containing color change indicator. 4.2.1.2.5 Sulfur dioxide: Add concentrated sulfuric acid dropwise into a branch flask containing a paste aqueous solution of sodium sulfite (or sodium bisulfite); cool the generated sulfur dioxide (as shown in Figure 1) to a liquid state by cold hydrazine (add dry ice and ethanol or ice and salt mixed outside of the cold hydrazine). Before use, place the cold hydrazine containing liquid sulfur dioxide in the air for vaporization; dry in a concentrated sulfuric acid and calcium chloride drying tower. Key: A - 10 mL automatic burette; B - reagent bottle; C - drying tube; D - titration bottle; E - galvanometer or galvo; F - variable resistor; G - switch; H -

4.2.2 Karl Fischer volumetric method

4.2.2.1 Method summary The Karl Fischer volumetric method is often used to determine sample with a water content greater than 0.1%. Place the sample in the titration cell; use titrant for titration; let the water in the sample reacts with the titrant; detect the titration end point by a potentiometer. The optimal environment for titration shall be pH 5 to pH 7. If the pH value of the sample solution exceeds this range, buffer solution shall be added or acid or alkali shall be used for neutralization.

4.2.2.2 Reagents 4.2.2.2.1 Single-component system reagents:

a) Solvent: suitable alcohols, such as methanol;

b) Titrant: an alcoholic solution of known water equivalents of sulfur dioxide, iodine and a base (such as imidazole). 4.2.2.2.2 Two-component system reagents:

a) Solvent: alcohol solution containing sulfur dioxide, alkali (such as imidazole);

b) Titrant: an alcoholic solution containing iodine of a known water equivalent.

4.2.2.3 Instruments Karl Fischer automatic titration system: a drying tube filled with fresh molecular sieves to ensure that the titration vessel is completely unaffected by moisture in the air. Built-in burette: 10 mL. Analytical balance: accuracy

0.1 mg;

4.2.2.4 Calibration of Karl Fischer reagent 4.2.2.4.1 Pre-titration Titrate to remove residual moisture in the titration chamber; select the appropriate titrant based on the expected moisture content of the sample. See Table 1 for details. 4.2.2.4.2 Calibration of titrant Calibration substances can be selected from water, aqueous standard alcohol solution and crystallized sodium tartrate dihydrate. The recommended sample weight (accurate to

0.1 mg) is as follows:

a) Distilled or deionized water: 30 mg ~ 40 mg (m1);

b) Water standard solution (mass fraction w about 1%): 3 g ~ 4 g (m2);

c) Sodium tartrate dihydrate (moisture content: 15.66% ± 0.05%):

0.20 g ~

4.3 Azeotropic distillation method

4.3.1 Method summary The water and toluene in the sample are distilled to form an azeotropic binary mixture and are distilled out. Water and toluene have different densities and are stratified in the receiver. According to the volume of distilled water, calculate the moisture content. The measured sample cannot contain substances that form an azeotrope with toluene. If the sample contains substances that form an azeotrope with toluene, other testing methods shall be selected.

4.3.2 Reagents Toluene.