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

GB/T 20378-2006

**Native starch -- Determination of starch content -- Ewers
polarimetric method**

原淀粉 淀粉含量的测定 旋光法

(English Translation)

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1 Scope

This Standard specifies a polarimetric method for the determination of the starch content of native starch.

This Standard is not applicable to the determination of starch content in native starch, modified starch and pregelatinized (cold water soluble) starch with high amylose content.

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.

ISO 1666 Starch - Determination of Moisture Content - Oven-Drying Method

GB/T 6682 Water for Analytical Laboratory Use - Specification and Test Methods

3 Principle

The method includes two intermediate determination steps.

3.1

A portion of the sample is hydrolysed with dilute hydrochloric acid and the optical rotation measured polarimetrically after clarification and filtration.

3.2

A second portion of the sample is treated with 40 % (V/V) ethanol to extract soluble sugars and polysaccharides of lower molecular mass. The filtrate is then subjected to the procedure given in 3.1. The difference between the measurements 3.1 and 3.2, multiplied by a factor, gives the starch content of the sample.

NOTE: Key parameters of the method are the time and temperature of the hydrolysis, and the correct use and calibration of the polarimeter. Consequently, the method includes constant agitation in the water bath, which should be of a size appropriate to ensure rapid temperature rise and steady temperature conditions.

4 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified, and water complying with grade 2 in accordance with GB/T 6682.

4.1 Dilute hydrochloric acid (HCl) solution

$c(\text{HCl}) = 7.7 \text{ mol/L}$, dilute 63.7 mL of hydrochloric acid ($\rho_{20} = 1.19 \text{ g/mL}$) with water up to 100mL.

4.2 Dilute hydrochloric acid (HCl) solution

$c(\text{HCl}) = 0.309 \text{ mol/L}$, dilute 25.6 mL of hydrochloric acid ($\rho_{20} = 1.19 \text{ g/mL}$) with water up to 1000mL.

NOTE: The concentration should be verified using sodium hydroxide solution [$c(\text{NaOH}) = 0.1 \text{ mol/L}$] and Methyl red as indicator: 10 mL HCl should consume 30.94 mL of 0.1 mol/L NaOH.

4.3 Dilute ethanol solution

40 % (V/V) ($\rho_{20} = 0.948 \text{ g/mL}$).

4.4 Carrez solution I

Dissolve 10.6 g of potassium hexacyanoferrate (II) trihydrate $[K_4[Fe(CN)_6] \cdot 3H_2O]$ in water.

Dilute to 100 mL with water.

4.5 Carrez solution II

Dissolve 21.9 g of zinc acetate dihydrate $[Zn(CH_3COO)_2 \cdot 2H_2O]$ and 3 g of glacial acetic acid in water. Dilute to 100 mL with water.

5 Apparatus

5.1

Volumetric flasks, of capacity 100mL.

5.2

Boiling water bath equipped with a vibrator or a magnetic stirrer.

5.3

Polarimeter, adjusted to a wavelength of 589.3 nm, with 200 mm tubes.

5.4

Analytical balance, capable of weighing to the nearest 0.001 g.

6 Processing of Sample

If the particle size of the laboratory sample exceeds 0.5 mm, grind the sample to pass a sieve with 0.5 mm apertures. Homogenize the sample thus prepared.

7 Procedure

Carry out weighing to the nearest 0.001 g (see 5.4).

7.1 Determination of optical rotation of a total portion

7.1.1

Weigh $2.5 \text{ g} \pm 0.05 \text{ g}$ (m1) of the test sample and transfer it to a volumetric flask (5.1).

Add 25 mL of the dilute hydrochloric acid (4.2) and agitate to distribute the test sample evenly.

Add a further 25 mL of the dilute hydrochloric acid (4.2).

7.1.2

Immerse the flask in the boiling water bath and shake continuously or immerse the flask in the boiling water bath equipped with a magnetic stirrer and stir at minimum speed.

7.1.3

Leave the flask for 15 min $\pm$ 5 s in the boiling water bath and stop shaking or stirring shortly before removing it. Immediately add 30 mL of cold water and cool rapidly under flowing water to 20 °C $\pm$ 2 °C.

7.1.4

Add 5 mL of the Carrez solution I (4.4) and shake for 1 min.

7.1.5

Add 5 mL of the Carrez solution II (4.5) and shake for 1 min.

7.1.6

Dilute to the mark with water. Homogenize and filter the solution through a suitable filter funnel and paper. If the filtrate is not perfectly clear, repeat the operations with 10 mL of each of the Carrez solutions.

7.1.7

Measure the optical rotation (α_D^{20}) of the solution in a 200mm tube with the polarimeter (5.3).

7.2 Determination of optical rotation of substances soluble in 40 % (V/V) ethanol

7.2.1

Weigh 5 g $\pm$ 0.1 g (m_2) of the sample and transfer to a 100 mL volumetric flask (5.1). Add about 80 mL of the ethanol solution (4.3). Leave the flask to stand for 1 h at room temperature;

shake vigorously six times during the hour to ensure thorough mixing of the test sample with the ethanol. Dilute to 100 mL with ethanol (4.3), homogenize and filter.

7.2.2

Pipette 50 mL of the filtrate (equivalent to 2.5 g of the test portion) into a volumetric flask (5.1). Add 2.1 mL of the dilute hydrochloric acid (4.1) and shake vigorously. Fit a reflux condenser to the flask and immerse the flask in a boiling water bath. Remove the flask from the boiling water bath after 15 min $\pm$ 5 s. Cool to 20 °C $\pm$ 2 °C.

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