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

GB/T 12008.6-2010

**Plastics -- Polyether polyols -- Part 6: Determination of degree
of unsaturation**

塑料 聚醚多元醇 第6部分：不饱和度的测定

(English Translation)

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1 Scope

This Part of GB/T 12008 specifies the method for the determination of degree of unsaturation of polyether polyols.

This Part is applicable to the determination of degree of unsaturation of polyether polyols. It can be used for quality control and scientific research, but it does not apply to compounds, in which, the unsaturated part of the molecule is connected to the carbonyl group, carboxyl group or nitrile group.

This Part does not address any safety issues associated with its use. Prior to use, it is the user's responsibility to establish appropriate safety and health measures and determine the scope of application of regulatory limitations.

2 Normative References

The clauses in the following documents become clauses of this Part through reference in this

Part of GB/T 12008. In terms of references with a specified date, all subsequent amendments (excluding corrigenda) or revisions do not apply to this Part. However, parties to an agreement based on this Part are encouraged to explore the possibility of adopting the latest versions of these documents. In terms of references without a specified date, the latest version applies to this Standard.

GB/T 2035-2008 Terms and Definitions for Plastics (ISO 472:1999, IDT)

GB/T 12008.5-2010 Plastics - Polyether Polyols - Part 5: Determination of Acidity as Acid Number

3 Terms and Definitions

The terms and definitions established in GB/T 2035-2008 and the following apply to this Part.

3.1

unsaturation

The content of carbon-carbon double bonds in a compound or polymer.

4 Principle

The carbon-carbon unsaturated compounds in the specimen react with mercuric acetate-methanol in methanol solution and generate mercuric acetate methoxy compounds and acetic acid. Use a standard titration solution of potassium hydroxide in alcohol to titrate the generated acetic acid, thereby, calculating the degree of unsaturation. Due to the formation of insoluble mercuric oxide, in the presence of excess mercuric acetate, the acid cannot be titrated; add sodium bromide to convert the excess mercuric acetate into mercuric bromide, which does not affect the determination. This method is based on acid form titration, with phenolphthalein as the indicator, and if the sample is not neutral, it shall be appropriately adjusted. During the test, attention shall be paid to eliminate carbon dioxide, as it will be titrated as an acid and lead to erroneous results.

There are two methods for the determination of the degree of unsaturation of polyether polyols:

Method A: high-volume reagent method---use 25 mL of 0.1 mol/L mercuric acetate-methanol solution and 15 g or more sample, and the indicator to determine the end point of color development. It is recommended for the determination of polyols with a low degree of unsaturation (lower than 0.01 mol/kg).

Method B: low-volume reagent method---use 2 mL of 0.05 mol/L mercuric acetate-methanol solution and 1 g or less sample, and potentiometrically titrate to indicate the end point.

5 Application

Due to the side reaction of unsaturated compounds in propylene oxide, a small amount of monohydroxy polymers will be generated. These unsaturated polymers reduce the degree of functionality and molecular weight, and the overall molecular weight distribution of the polymers becomes broadened.

6 Interference

Since the reaction products of compounds, in which, the unsaturated part of the molecule is connected to the carbonyl group, carboxyl group or nitrile group, are easily hydrolyzed to generate alkaline mercury salts, the system shall be anhydrous. Although the presence of low- concentration acetone makes the end point less obvious, the effect is not significant. The sample shall be free of inorganic salts, especially halides, because even small amounts of salts can render the reaction of mercuric acetate with unsaturated compounds ineffective.

7.1 Instruments

7.1.1

Pipette

25 mL.

7.1.2

Conical flask

250 mL, with a glass stopper.

D---the volume of 0.05 mol/L potassium hydroxide-ethanol standard titration solution consumed by titration of the test portion, expressed in (mL);

E---the volume of 0.05 mol/L potassium hydroxide-ethanol standard titration solution consumed by titration of the blank, expressed in (mL);

N---the concentration of potassium hydroxide-ethanol standard titration solution, expressed in (mol/L);

C---the acidity value of the specimen (calculated as the amount of KOH consumed), expressed in (mg/g);

W---the mass of the test portion, expressed in (g);

56.1---the numerical value of the molar mass of potassium hydroxide, expressed in (g/mol).

7.5 Precision

The difference between parallel results obtained by the same person shall not exceed 0.002.

8.1 Instruments

8.1.1

Pipette

2 mL.

8.1.2

Automatic titrator

It can determine the acid form end point and is equipped with a 5 mL burette.

8.1.3

Complex, glass and pH electrode for use with the automatic titrator.

8.1.4

Analytical balance

Accurate to 0.1 mg.

8.1.5

Titration container

With a capacity of 100 mL.

8.2 Reagents

Unless otherwise stated, only analytically pure reagents are used.

8.2.1

Methanol.

8.2.2

Mercuric acetate-methanol solution

C = 0.05 mol/L.

Dissolve 16 g of mercuric acetate $[\text{Hg}(\text{C}_2\text{H}_3\text{O}_2)_2]$ in 1 L of methanol solution, add a sufficient amount of glacial acetic acid, so that 2 mL of the reagent blank consumes 0.5 mL ~ 1 mL of

0.05 mol/L potassium hydroxide-methanol standard titration solution. Usually, it is necessary to add a few drops of glacial acetic acid. The solution needs to be weekly prepared and filtered before use.

8.2.3

Potassium hydroxide-methanol standard titration solution

C (KOH) = 0.05 mol/L.

Dissolve potassium hydroxide in methanol to prepare a 0.05 mol/L solution. Use standard potassium hydrogen phthalate for calibration.

8.2.4

Hydrochloric acid-methanol standard titration solution

C (HCl) = 0.05 mol/L.

Add concentrated hydrochloric acid to methanol solution to prepare a 0.05 mol/L solution.