



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

GB 31604.45-2016

**Food contact materials for export -- Polymer materials --
Determination of isocyanates by high performance liquid
chromatography**

食品安全国家标准 食品接触材料及制品 异氰酸酯的测定

(English Translation)

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Foreword

This Standard replaces GB/T 23296.22-2009 "Food contact materials - Determination of isocyanates in plastics - High performance liquid chromatography" AND SN/T 2741-

2010 "Food contact materials for export - Polymer materials - Determination of

isocyanates by high performance liquid chromatography".

Compared with GB/T 23296.22-2009 AND SN/T 2741-2010, main changes of this Standard are as follows.

- Modify the Standard name to "National food safety standard - Food contact materials and articles - Determination of isocyanate";
- Increase the number of analyte to 7;
- Cancel the preparation of blank samples;
- Modify the matrix standard curve to the standard-solution's standard curve;
- Modify the expression of the analysis results.

National Food Safety Standard - Food Contact

Materials and Articles - Determination of Isocyanate

1 Scope

This Standard specifies the determination methods for toluene-2,6-diisocyanate, diphenylmethane-4,4'-diisocyanate, toluene-2,4-diisocyanate, naphthalene-1,5-diisocyanate, phenyl isocyanate, hexamethylene diisocyanate and cyclohexyl isocyanate in plastics for food contact material and their products; this Standard specifies the determination method for the total amount of the above 7 isocyanates. This Standard applies to the detection of toluene-2,6-diisocyanate, diphenylmethane-4,4'-diisocyanate, toluene-2,4-diisocyanate, naphthalene-1,5-diisocyanate, phenyl isocyanate, hexamethylene diisocyanate and cyclohexyl isocyanate in polyurethane plastics and their products; this Standard applies to the detection of the total amount of the above 7 isocyanates.

2 Principle

In the sample, after isocyanate is vibrated-extracted by dichloromethane, isocyanate starts derivative reaction with 9-N-methylaminomethyl hydrazine. The derivatized products are separated by C18 liquid chromatography column, determined by fluorescence detector AND quantified by external standard method.

3 Reagents and materials

Unless otherwise specified, all reagents shall be the analytically pure. The water shall be the Grade-1 water specified in GB/T 6682.

3.1.1 Dichloromethane (CH_2Cl_2). it shall be dried by 5A molecular sieves, stay for overnight for standby application.

3.4.1 Isocyanate stock solution (1000 mg/L). weigh and take 0.01 g (accurate to 0.1

mg) of various isocyanate standard materials; dissolve the dichloromethane; then transfer it to a volumetric flask of 10 mL; dilute the dichloromethane to the constant volume. Stock solution shall be stored at -20 °C under dry preservation; it shall be kept

from the light. The term of validity is 1 month.

3.4.2 Standard intermediate-solution of isocyanate (100 mg/L). respectively weigh and

take 5mL of dichloromethane in six 10 mL volumetric flasks; respectively transfer them into various 1.0 mL of isocyanate stock solution; dilute them to the constant volume with dichloromethane. The standard intermediate-solution shall be stored at -20 °C under dry preservation; it shall be kept from the light. The term of validity is 1 month.

3.4.3 Standard working solution of Isocyanate (1 mg/L). respectively weigh and take

5mL of dichloromethane into six 10mL volumetric flasks; respectively add 100 µL of various standard intermediate-solutions of isocyanate; dilute them to the constant volume with dichloromethane; mix them evenly. Standard working solution shall be stored at -20 °C under dry preservation; it shall be kept from the light. The term of validity is 2 weeks.

3.4.4 Standard working solution of mixed isocyanate. respectively weigh and take 5

mL of dichloromethane into six 10 mL volumetric flasks; successively add 0.00 mL,

0.01 mL, 0.05 mL, 0.10 mL, 0.25 mL and 0.50 mL of various standard intermediate-solutions of isocyanate; dilute them to the constant volume with dichloromethane; mix them evenly; the mixed standard working solutions whose isocyanate's concentration are respectively 0 mg/L, 0.10 mg/L, 0.50 mg/L, 1.0 mg/L, 2.5 mg/L and 5.0 mg/L can be acquired. These mixed standard working solutions shall be stored at -20 °C under dry preservation; they shall be kept from the light. The term of validity is 2 weeks.

4.5 Analytical balance. the sensitivity shall be 0.0001 g and 0.01 g.

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