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Plastics — Thermoplastic polyurethanes for moulding and extrusion

Part 3: Distinction between ether and ester polyurethanes by determination of the ester group content

Plastiques — Polyuréthannes thermoplastiques pour moulage et extrusion —

Partie 3: Distinction entre polyuréthannes éther et ester par dosage des groupements esters

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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ISO 16365-3 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

This second/third/... edition cancels and replaces the first/second/... edition (), [clause(s) / subclause(s) / table(s) / figure(s) / annex(es)] of which [has / have] been technically revised.

ISO 16365 consists of the following parts, under the general title *Plastics — Thermoplastic Polyurethanes for moulding and extrusion*:

— *Part 1: Designation system and basis for specifications*

— *Part 2: Preparation of test specimen and determination of properties*

— *Part 3: Distinction of Ether TPU and Ester TPU and mixtures by analysis*

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Introduction

The saponification value is a fast and easy determination method, to distinguish between polyesterol and polyetherol based thermoplastic polyurethanes. However, the saponification value strongly depends on the reaction conditions. Higher saponification values are resulted in longer hydroxylation times and / or higher temperatures. With this procedure the theoretically expected values have been found.

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Plastics — Thermoplastic Polyurethanes for moulding and extrusion — Part 3: Distinction of Ether TPU and Ester TPU and mixtures by analysis

1 Scope

This part of the standard provide a method for detection of esters including ester TPU in ether TPU for quality control without the necessity of sophisticated instrumentation and avoids or minimize long term microbial testing for TPU materials. The method described is used to distinguish major TPU types and characterize TPU mixtures.

The method is applicable on all linear polyurethanes based on 4,4'-diphenylmethane diisocyanate (MDI) and is intended e.g. to minimize long term tests related to microbe resistance and hydrolysis.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 385; *Laboratory glassware — Burettes*

ISO 1042; *Laboratory glassware — One-mark volumetric flasks*

ISO 3696; *Water for analytical laboratory use — Specification and test methods*

ISO 3681;1998 - *Binders for paints and varnishes - Determination of saponification value - Titrimetric method (ISO 3681:1996)*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply:

3.1
Total hydrolysable groups (S_{Z1})
number of milligrams of potassium hydroxide required to neutralize the free acids of ester and urethane groups present in 1 g of thermoplastic polyurethanes, when determined in accordance with the procedure specified in this International Standard.

NOTE The acid value is expressed in milligrams per gram.

3.2
Hydrolysable Urethanes (S_{Z2})
number of milligrams of potassium hydroxide required to neutralize the free acids of only urethane groups present in 1 g of thermoplastic polyurethanes, when determined in accordance with the procedure specified in this International Standard.

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NOTE The acid value is expressed in milligrams per gram.

3.3
Ester content (E)
number of milligrams of potassium hydroxide required to neutralize the free acids of only ester groups present in 1 g of thermoplastic polyurethanes, when determined in accordance with the procedure specified in this International Standard.

NOTE The acid value is expressed in milligrams per gram.

4 Principle

The content of ester groups in TPU is determined by alkaline hydrolysis. The amount of urethane groups is determined and subtracted from the determined total amount of hydrolysable compounds, i.e. esters and urethanes. The difference is a measure of the content of ester groups in mg KOH/g. Pure polyether-TPU yields results close to 0 mg KOH/g.

5 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

5.1 Dimethylsulfoxide

5.2 Potassium hydroxide solution, c(KOH) = 0.5 mol/l accurate to 0,001 mol/l, methanol solution

5.3 Sulfuric acid, $c(H_2SO_4)$ = 0.5 mol/l accurate to 0,001 mol/l, aqueous solution

5.4 Phenolphtaleine, 1% in methanol or ethanol

5.5 Methanol, for analysis

5.6 Water, distilled or of equivalent purity according ISO 3696

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6 Apparatus

6.1 Centrifugal mill with sieve of 2 mm

6.2 Analytical balance, accuracy 0,1 mg or better

6.3 Erlenmeyer flask, 250 ml which can be closed

6.4 Automatic burette, 20 ml or similar

6.5 Magnetic stirrer, with heater

6.6 Reflux cooler with Teflon manchette

6.7 Magnetic stirrer, 40 mm

6.8 Volumetric measuring cylinder 50 ml

6.9 Soxhlet apparatus

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7 Sample preparation

Cut a representative fraction of the sample to be analysed into pieces of approximately 2 mm in diameter.

For Thermoplastic urethane materials containing phosphorous plasticizers, extract 3 g of the powdered sample for 4 hours in methanol using a Soxhlet extractor (6.9). Remove the sample from the extractor and dry for 30 minutes at 60 °C in an oven.

NOTE Phosphorous esters used as plasticizers for TPU have no influence on the resistance of the material against microbes. However, the presence of Phosphorous ester will cause a higher ester content result.

8 Procedure

At least two determinations shall be carried out.

Weigh a test portion of 0,5 g to the nearest 1 mg (E gram) into an Erlenmeyer flask of 250 ml (6.3). Add 40 ml dimethylsulfoxide (5.1) and a magnetic stirrer (6.7), close the flask and the stir for 15 minutes.

NOTE The sample may only be partially dissolved after 15 minutes.

Add 20 ml (5.2) of a solution of potassium hydroxide (5.2) under continuous stirring to avoid coagulation. The sample is hydrolysed for 5 hours under reflux conditions and continuous stirring (6.6) on the magnetic stirrer with heater (.5).

Two blank determinations are prepared following the same procedure.

Cool to room temperature and rinse the reflux cooler and Erlenmeyer wall with 50 ml of water. The surplus amount of KOH is titrated by sulphuric acid (5.3) from red to colourless using 10 droplets of phenolphthalein solution as indicator (sample: V1 ml, average blanks: V1b ml) but avoiding an excess of sulphuric acid.

After titration add 10 ml of H_2SO_4 (5.3) by a burette and boil for 10 minutes on the magnetic stirred with heater while continuously stirring. It is essential that all carbon dioxide is removed from the solution.

After cooling to room temperature the surplus amount of sulphuric acid is titrated back by KOH (5.2) using phenolphthalein as indicator. The titration is carried out from colourless to slightly pink (sample: V2 ml, average blanks: V2b ml).

NOTE Fillers like $CaCO_3$ and other additives like some flame retardants that may form salts might have an affect on the result.

NOTE Pigments may change the visibility of the color change of the indicator which will make it difficult or impossible to carry out a visual titration.

NOTE Potentiometric titration is also possible.

9 Calculation

Calculate the Ester content in mg KOH/g as follows:

$$E = S_{Z1} - S_{Z2}$$

Where

E = Ester content in mg KOH/g

$$S_{Z1} = (V_{1b} - V_1) * 56{,}1 * Titre_{H_2SO_4}$$

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