Application Note AN-T-167

Isocyanate content of polyurethane raw materials

Titration according to EN ISO 14896 for unsaturated polyester resin and polyurethane resin

Polyurethane (PU) is a class of very important polymers due to its flexibility and insulating properties. It is used in various industries such as the automobile industry, in building construction, as well as in the production of synthetic fibers. PU is mostly produced via a chemical reaction between polyisocyanates and polyols. This results in linked networks forming «duroplasts», while the use of diisocyanates and dioles will lead to linear polymers, so-called «elastomers».

The isocyanate (NCO) content in the raw material is crucial to control its properties. If the isocyanate content of the raw material is unknown, a polyurethane with undesirable properties might be obtained. It is therefore quite important to determine the isocyanate content in these compounds. This Application Note shows an easy and straightforward way to determine the NCO content in polyurethane raw materials using a fully automated titration system from Metrohm.

SAMPLE AND SAMPLE PREPARATION

The method is demonstrated on a polyurethane resin (PUR) and an unsaturated polyester resin (UPR). For both samples, no sample preparation is required.

EXPERIMENTAL

The analyses are performed fully automatically using an 814 USB Sample Changer in combination with a 907 Titrando and a Solvotrode easyClean. The sample is weighed into a beaker, and toluene along with the reaction solution (consisting of dibutylamine in toluene) is added. After a reaction time of 15 minutes, acetone is added and the solution is titrated with hydrochloric acid until after the equivalence point is reached.

The blank is determined in the same way, but by omitting the sample.

Figure 1. Titrando system consisting of an 814 USB Sample Changer in combination with a 907 Titrando and tiamo.

RESULTS

Steep and smooth titration curves (see Figure 2) are obtained for all analyses. The automated analysis leads to reproducible results with a RSD < 2% as shown in Table 1.

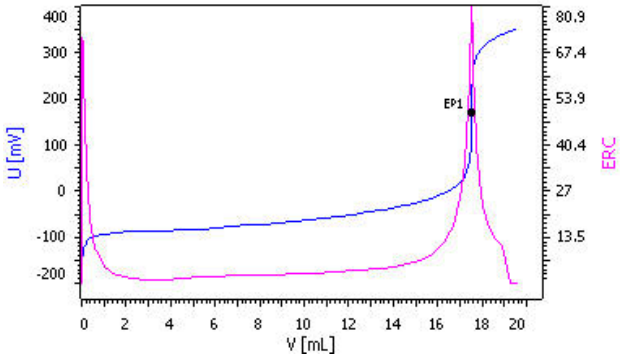

Figure 2. Titration curve of the determination of the NCO value in polyurethane resin.

Table 1. Results of the determination of the NCO content in polyurethane resin (PUR) and unsaturated polyester resin (UPR)

	n	Mean value /%	SD(abs)/ %	SD(rel) /%
Polyurethane resin (PUR)	5	2.335	0.022	0.94
Unsaturated polyester resin (UPR)	5	0.826	0.016	1.94

CONCLUSION

The NCO content determination according to EN ISO 14896 is carried out without difficulties and can easily be automated. The waiting time of 15 minutes must be followed strictly, otherwise results might become falsified as the reaction time is elongated. Therefore, the automated addition of the auxiliary solutions is highly recommended.

Internal reference: AW TI CH1-1228-102016

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CONFIGURATION

907 Titrando

High-end titrator for potentiometric and volumetric Karl Fischer titration with one measuring interface and Dosino dosing units.

- up to four dosing device systems of the 800 Dosino type
- dynamic (DET), monotonic (MET) and endpoint titration (SET), enzymatic and pH-STAT titrations (STAT), Karl Fischer titration (KFT)
- Measurement with ion-selective electrodes (MEAS CONC)
- "iTrode" intelligent electrodes
- Dosing functions with monitoring, liquid handling
- four MSB connectors for additional stirrers or dosing device systems
- USB connector
- For use with OMNIS Software, *tiamo* software, or Touch Control
- Complies with GMP/GLP and FDA regulations such as 21 CFR Part 11, if required

814 USB Sample Processor (1T/1P)

USB Sample Processor with one workstation and one built-in membrane pump for the automatic processing of routine samples in series with small to medium quantities. In addition to the built-in pump, an additional one (membrane or peristaltic) and up to three dosing devices for Liquid Handling tasks can be connected.

Because of the multitude of application variants, rack, stirrer, titration head, Swing Head and sample vessels must be tailored to the application and ordered separately.

The control is "stand alone" using Touch Control. The following software products can be selected for the PC control: tiamoTM titration software, MagIC Net chromatography software, viva voltammetry software, or OMNIS.

Solvotrode easyClean (fixed cable 1.2 m)

Combined pH electrode with flexible easyClean diaphragm and fixed cable (1.2 m) for all non-aqueous acid/base titrations.

The glass membrane is optimized for poorly conducting solutions, and thanks to the easy-to-clean easyClean diaphragm, the electrode is also well suited for heavily contaminated samples, such as used oil.

This electrode can be used with non-aqueous reference electrolytes (lithium chloride or tetraethylammonium bromide). Storage in the respective reference electrolyte.

The Solvotrode easyClean is also available under the article number 6.0229.020 with a fixed cable length of 2.0 m.