



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 diols 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.

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 tiemo.

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**.

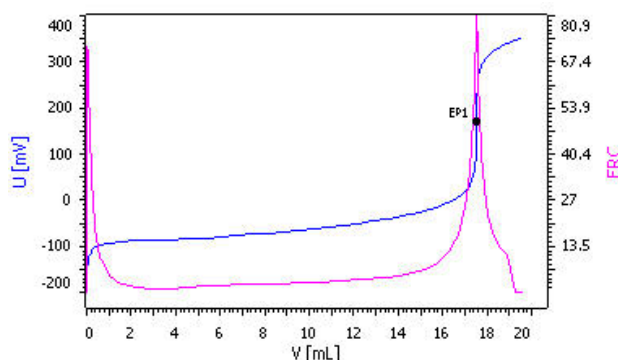


Figure 2. Titration curve of the determination of the NCO value in polyurethane resin.

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

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

CONTACT

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CONFIGURATION



907 Titrando

Dosino

- 800 Dosino 4
- (DET) (MET) (SET) pH STAT (STAT) (KFT)
- (MEAS CONC)
- iTrode
- LQH
- 4 MSB
- USB
- OMNIS Software **tiamo** Touch Control
- GMP/GLP FDA 21 CFR Part 11 ()



814 USB Sample Processor (1T/1P)

USBLQH()1()3

Swing Head

Touch ControlIPC: tiamoTMMagIC NetvivaOMNIS



Solvotrode easyClean (1.2 m)

/easyClean (1.2 m) pH

easyClean ()

()

Solvotrode easyClean6.0229.0202.0m