Application Note AN-T-167

聚氨酯原材料的异氰酸酯含量

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.

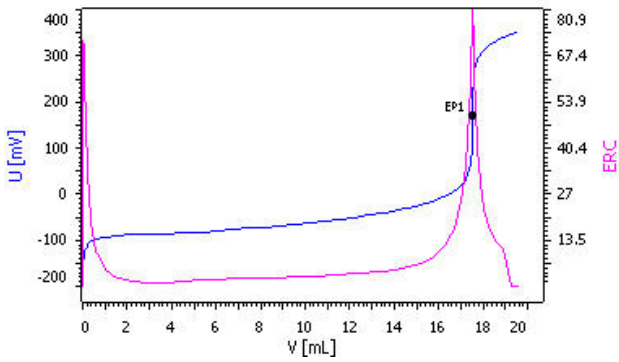

Figure 2. Titration curve of the determination of the NCO value in polyurethane resin.

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.

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CONFIGURATION

907 Titrando
用于使用一个测量接口和 Dosino 加液单元电位分析和容量卡尔·费休滴定法滴定的电位滴定仪。

- 多达四套 800 Dosino 加液系统
- 动态(DET)、等量(MET)和终点滴定(SET),酶和 pH-STAT 滴定(STAT)、卡尔·费休容量滴定(KFT)
- 使用离子选择性电极测量(MEAS CONC)
- 智能电极“iTrode”
- 带监控的加液功能,LQH
- 用于其他搅拌器或加液器系统的四个 MSB 接口
- USB 接口
- 使用 OMNIS-Software、*tiamo*-软件或 Touch Control
- 如果需要,满足 GMP/GLP 和 FDA 要求,比如 21 CFR 第 11 部分

814 USB Sample Processor (1T/1P)

USB Sample Processor 包括一个工作站和一个内置的隔膜泵,用于自动处理少量至中等量的常规样品系列。除了内置的泵之外,还可再连接一台泵(隔膜泵或蠕动泵)和多达三台加液单元用来进行 LQH 加液处理。

由于其应用范围很广,因此必须根据具体应用来选择合适的样品盘、搅拌器、滴定头和 Swing Head 以及样品容器并单独订购。
通过 Touch Control 实现单机控制。 有以下 PC 控制用软件产品可供选择: 滴定软件 tiamoTM、色谱分析软件 MagIC Net、伏安法软件 viva 或 OMNIS。

Solvotrode easyClean1.2 m

带灵活的 easyClean 隔膜的组合 pH 电极和固定电缆 (1.2 m) 用于所有非水性酸/碱滴定。
玻璃膜针对导电性差的溶液进行了优化,并且由于易于清洁的 easyClean 隔膜,该电极也适用于非常脏的样品(例如:旧油)。
电极可与非水参比电解质(氯化锂或四乙基溴化铵)一起使用。储存在相应的参比电解质中。
物品编号为 6.0229.020 的 Solvotrode easyClean 同样有 2.0 m 固定电缆长度的版本可供选购。