

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.

## 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.

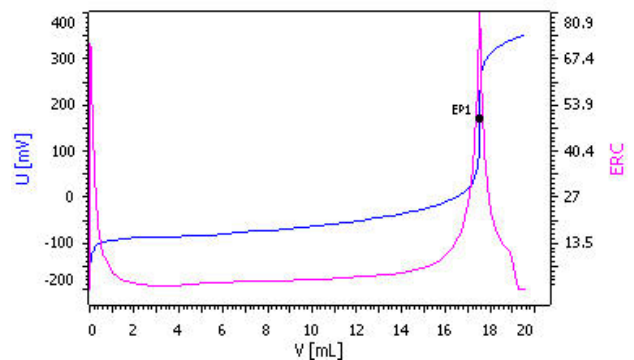
The blank is determined in the same way, but by omitting the sample.



**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

用于使用一个量接口和 Dosino 加液元位分析和容量-休滴定法滴定的位滴定。

- 多四套 800 Dosino 加液系
- (DET)、等量(MET)和点滴定(SET),和 pH-STAT 滴定(STAT)、·休容量滴定(KFT)
- 使用子性量(MEAS CONC)
- 智能“iTrode”
- 控的加液功能,LQH
- 用于其他拌器或加液器系的四个 MSB 接口
- USB 接口
- 使用 OMNIS-Software、*tiamo*-件或 Touch Control
- 如果需要,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,用于自理少量至中等量的常品系列。除了内置的之外,可再接一台(隔膜或蠕)和多三台加液元用来行 LQH 加液理。

由于其用范很广,因此必根据具体用来合的品、拌器、滴定和 Swing Head 以及品容器并独。

通 Touch Control 通 "stand alone" 控制。有以下 PC 控制用件品可供: 滴定件 *tiamo*TM、色分析件 MagIC Net、伏安法件 *viva* 或 OMNIS。



### Solvotrode easyClean 1.2 m

活的 easyClean 隔膜的合 pH 和固定 (1.2 m) 用于所有非水性酸/滴定。

玻璃膜性差的溶液行了化,并且由于易于清的 easyClean 隔膜,也用于非常的品(例如:旧油)。

可与非水参比解(化或四乙基化)一起使用。存在相的参比解中。

物品号 6.0229.020 的 Solvotrode easyClean 同有 2.0 m 固定度的版本可供。