Titration of NCO value in resins according to DIN EN ISO 14896

Description

Titration of Isocyanates (NCO value) in polyester and polyurethane resins according to DIN EN ISO 14896. This method is used to determine of reactive Isocyanate groups in resins and similar materials. The Isocyanate reacts in toluene with an excess of Di-n-butylamine to form a urea. Unreacted (excess) amine is determined by back titration with Hydrochloric acid.
The result is calculated as % NCO.

Instruments

Titrator	TL 7000 or higher
Electrode	N 6480 Eth (electrolyte L 5034)
Cable	L1A
Stirrer	Magnetic stirrer TM 235 or similar
Titration tip	Long version TZ 1643 required
Lab accessoires	Glas beaker 250 ml tall form without spout
	Watch glass or Parafilm
	Magnetic stirrer bar 30 mm

Reagents

1	Hydrochloric acid 1 mol/l in Isopropylalcohol (aqueous hydrochloric acid is also suitable for most applications)
2	Di-n-butylamine, 0.9 mol/l solution in Toluene
3	Toluene, anhydrous
4	Methanol
6	Molecular sieve
All reagents should be in analytical grade or better.	

Titration procedure

Reagents

The Toluene is dried over molecular sieve.

Dibutylamine solution 0.9 mol/l in toluene:
118 g Di-n-butylamine are weighed out in a volumetric flask and made up to 1000 ml with dried toluene.
A 1 mol/l solution can also be used, but it can happen that the burette must fill during the Blank titration when the consumption of HCl is greater than 20 ml, if a 20ml burette is used.

Titer determination of the HCl is described in the application “Titer strong acids”.

Cleaning of the electrode

The electrode is first rinsed with Toluene, ethanol or 2-propanol, then with dist. water and then with ethanol or 2-propanol. The electrode is placed always after the reaction time in the titration beaker because the alcohol can react with the isocyanate groups. The electrode is stored in a solution of 1.5 mol/l LiCl in Ethanole (or, if another electrolyte is used, in this electrolyte solution).

Blank value

This titration is performed as back titration, therefore the blank value must first be determined. The blank value determination is carried out under the same conditions as the sample titration: 30 ml of dried Toluene are placed in a 250 ml glass beaker (tall form, without spout), 20.0 ml of the 0.9mol/l Di-n-butylamine solution are added, the beaker is sealed with a watch glass (or Parafilm) and the mixture is stirred for 10 minutes. After 10 minutes, 30ml Methanol are added. This mixture is titrated with 1mol/l Hydrochloric acid to an equivalence point. Titration parameters for the Blank value are described below.
The blank value should be in the range of 18 - 20 ml.

Sample preparation

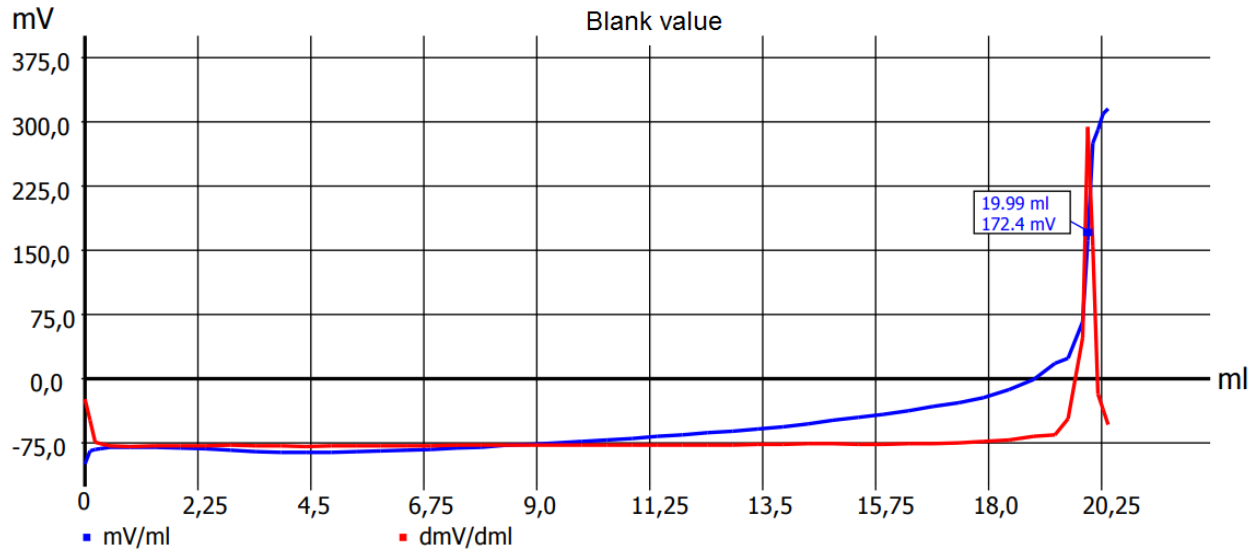
The sample is placed in a 250 ml glass beaker (tall form, without spout), 30 ml of dried Toluene are added, the beaker sealed with a watch glass and the mixture is stirred until the sample is dissolved. This mixture can be slightly heated in case of a poor solubility of the sample. After the complete dissolution of the sample, the mixure is cooled down to room temperature. 20.0 ml of the 0.9 mol/l Di-n-butylamine solution are added and the beaker is sealed with a watch glass. This mixture is stirred for 10 minutes. After this, 30ml Methanol are added. This mixture is titrated with 1 mol/l Hydrochloric acid until an equivalence point.
When aqueous HCl is used, it is necessary to stir vigorously because 2 phases are formed during the titration.

The required amount of sample can be estimated according to this rule of thumb:

$$W(g) = \frac{40}{expected\ NCO\ value}$$

Titration parameter

Blank value



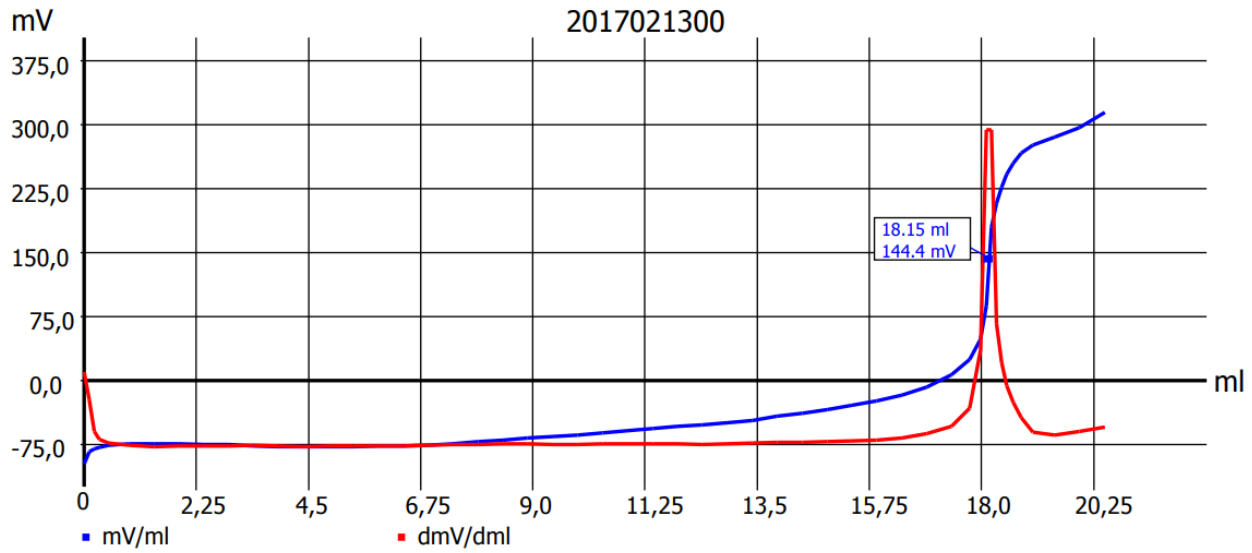
Default method	-		
Method type	Automatic titration		
Modus	Dynamic		
Measured value	mV		
Measuring speed / drift	User defined	Minimum holding time	4 s
		Maximum holding time	20 s
		Measuring time	3 s
		drift	10 mV/min
Initial waiting time	0 s		
Dynamic	User-defined	Max step size	0.5 ml
		Slope max ml	20
		Min. step size	0.05 ml
		Slope min. ml	120
Damping	Average	Titration direction	increase
Pretitration	off	Delay time	0 s
End value	off		
EQ	on	Slope value	700
Max. titration volume	25 ml		
Dosing speed	100%	Filling speed	30 s

Calculation:

$$ml = EQ1$$

The result is saved in a global memory, e.g. M01. We recommend to use statistics = 3.

Sample titration



For the sample titration, the same parameters are used as for the blank determination.

Calculation:

$$NCO\ [\%] = \frac{(B - EQ1) * T * M * F1}{W * F2}$$

B	M01	Blank value, saved in global Memory M01
EQ1		Consumption of titrant at first Equivalence point
T	WA	concentration of the titrant
M	42.02	Molecular mass of NCO
W	man	Weight of the sample in g
F1	0.1	Conversion factor
F2	1	Conversion factor

Any questions? Please contact the application team:

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