Determination of isocyanate (NCO-) content

Application

Use

This method is applicable for material containing reactive isocyanate groups.The isocyanate is reacted with di-n-butyl amine to form a urea. Unreacted (excess) amine is determined by backtitration with hydrochloric acid.

Appliances

- Titrator: TL 7000/TL 7750 M1/20 or 50
- Basic device
- Magnetic stirrer TM 235
- 20/50 mL exchange unit WA 20/50, with amber glass bottle for the titrant, complete
- Erlenmeyer flask 250 or 300 ml, Syringe 2 or 5 ml for high viscosity samples

Electrodes

- Electrode: N 6480 eth + TZ 1643 titration tip
- Electrolyte: L 5034 (LiCl/ethanol)
- Calibration: n.a.

Reagents

- Titrant: HCl 1 mol/l aqueous or HCl in methanol. Sometimes HCl 0.5 mol/l
- Titer determination: TRIS (Tris hydroxyl aminomethan)
- Reagent mixture: Di-n-butylamine solution 1 mol/l
- Solvent: Toluene dried with molecular sieve 4A
- Additional solvent: acetone or other suitable solvents

Description

Determination of the exact concentration of the HCL titrant

We recommend ready to use HCl titrant. The exact concentration of the HCL 1.000 mol/l can be determined using the titrimetric standard Tris hydroxy amino methane (TRIS). The standard can be dried for 24 h in an exsiccator at room temperature.

1-2 g of the standard are weighed accurately in a 150 mL beaker and dissolved in 80 mL of dist. water with stirring. It is titrated with the 1 mol/l HCl solution.

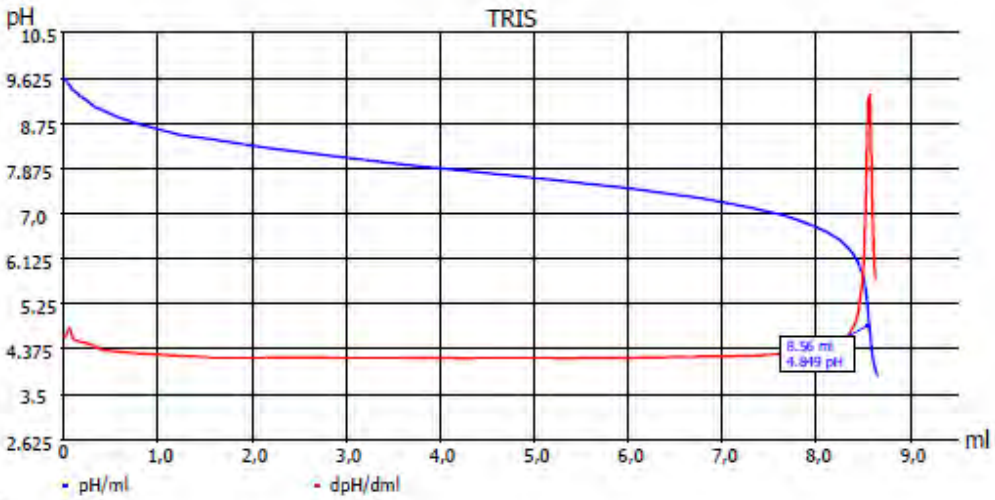
Repeat the standardization two times. The average value is stored automatically in the exchangeable unit.

Page 1: Curve and result: Titer determination

The curve shows the use of an HCl 0.1 mol/l. The sample amount of TRIS should be 10 – 15 times higher for the 1 mol/l titrant.

GLP documentation

Titration graph



Method data

Method name:	Titre HCl	Titration duration:	3 m 8 s
End date:	13.09.12	End time:	14:39:30

Titration data

Sample ID:	TRIS	Weight:	0.1038 g
Start pH:	pH 9.590	End pH:	pH 3.864
Start temperature:	25.0 °C (m)	End temperature:	25.0 °C (m)
Zero point:	pH 6.83 / -10.0 mV	Slope:	100.6 % / -59.5 mV/pH
EQ:	8.560 ml / pH 4.849	Titre:	0.1001 mol/l

Calculation formula

Titre:	(W*F2)/((EQ1-B)*M*F1) -> M103
Mol (M):	121.14000

Weight (W):	man	Factor 2 (F2):	1000.0000
Blank value (B):	0.0000 ml	Factor 1 (F1):	1.0000
Statistics:	Off		

Page 2: Method parameters Titer determination:

Method data overall view

Method name:	Titre HCl	Created at:	09/13/12 14:23:02
Method type:	Automatic titration	Last modification:	09/13/12 14:27:56
Measured value:	pH	Damping settings:	None
Titration mode:	Dynamic	Documentation:	GLP
Dynamic:	Steep		
Measuring speed / drift:	Normal:	minimum holding time:	02 s
		maximum holding time:	15 s
		Measuring time:	02 s
		Drift:	20 mV/min
Initial waiting time:	0 s		
Titration direction:	Decrease		
Pretitration:	Off		
End value:	2.500 pH		
EQ:	On (1)		
Slope value:	Steep	Value:	700

Dosing parameter

Dosing speed:	100 %	Filling speed:	30 s
Maximum dosing volume:	50.00 ml		

Unit values

Unit size:	20ml
Unit ID:	10039005
Reagent:	HCl 0.1 mol/L
Batch ID:	no Charge
Concentration [mol/l]:	0.10070
Determined at:	12/05/11 19:18:45
Expire date:	08/18/12
Opened/compounded:	09/10/11
Test according ISO 8655:	05/10/11
Last modification:	09/13/12 14:35:18

Di n-butylamine solution

129 g di-n-butyl amine are weight in a 1 L volumetric flask and filled up to the mark with dried toluene.

Titration of the sample

There are different procedures. Sometimes the di n-butylamine solution is pipetted first and then the sample is weight in. Iin some procedures the sample is weight in first and the reagent mixture is added afterwards.

The ISO 14896 (part A) describes the pipetting of the di n-butylamine solution into an Erlenmeyer flask (250-300 ml) and then the addition of the sample. We are following this procedure.

Pipette 50.00 ml using a volumetric pipette into an Erlenmeyer flask with stopper. The sample is weight in using a 2 or 5 ml syringe by different weight. Sample weight should be between 2- 5 g.

Close the flask with a stopper and gently stir the mixture on the magnetic stirrer until the sample is completely dissolved. Wait then additional 15 min for the reaction + 5 – 10 minutes until the mixture cooled down to room temperature. Add 100 – 150 ml acetone (or any other suitable solvent) carefully.

Place the Erlenmeyer flask on the magnetic stirrer (if not already) and start the titration method. After the titration rinse the electrode and burette tip with ethanol. For each set of samples perform a blank titration.

Result calculation

% NCO

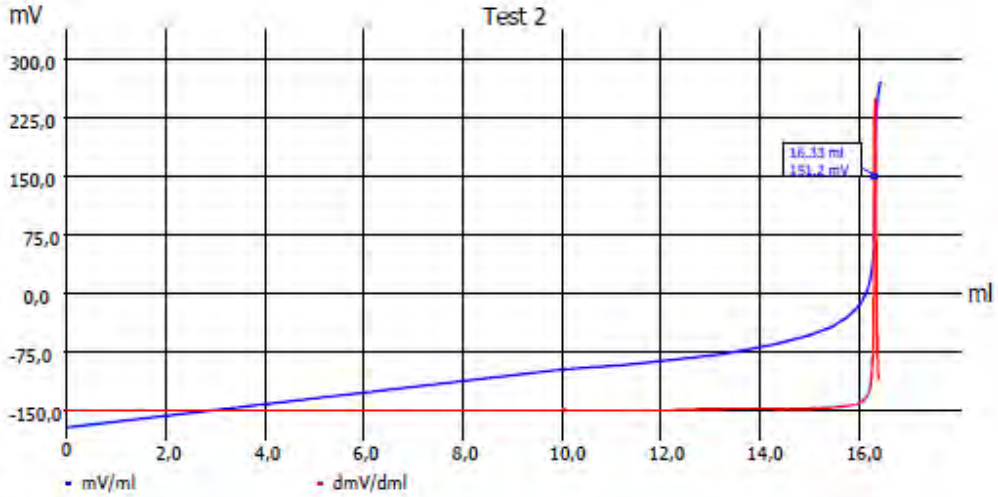
% NCO = (EQ1-B) * M * T * F1/(W*F2)

EQ1: ml consumption at the equivalence point
B: ml consumption for the blank titration
M: 4.202 equivalent weight including conversation factor to % NCO
T: concentration of the HCl titrant (e.g.1.002 mol/l)
W: sample weight in g
F1,F2: 1

Blank titration page 1: Curve and result

GLP documentation

Titration graph



Method data

Method name:	NCO blank titration	Titration duration:	2 m 13 s
End date:	27.06.12	End time:	10:53:46

Titration data

Sample ID:	Test 2	Weight:	1.0000 g
Start mV:	-173.3 mV	End mV:	272.0 mV
EQ:	16.330 ml / 151.2 mV	Blank:	16.330 ml

Calculation formula

Blank:	EQ1 -> M01
Mol (M):	1.00000
Statistics:	Off

Blank titration page 2: method

The pre-titration volume can also be changed to 25-40 ml. The end volume can also be changed to 55 or 60 ml.

Method data overall view

Method name:	NCO blank titration	Created at:	11/17/11 11:29:35
Method type:	Automatic titration	Last modification:	06/27/12 10:48:12
Measured value:	mV		
Titration mode:	Dynamic	Documentation:	GLP
Dynamic:	Steep		
Measuring speed / drift:	Normal:	minimum holding time:	02 s
		maximum holding time:	15 s
		Measuring time:	02 s
		Drift:	20 mV/min
Initial waiting time:	0 s		
Titration direction:	Increase		
Pretitration:	10.000 ml	Delay time:	10 s
End value:	Off		
EQ:	On		
Slope value:	Steep	Value:	700

Dosing parameter

Dosing speed:	100 %	Filling speed:	30 s
Maximum dosing volume:	50.00 ml		

Unit values

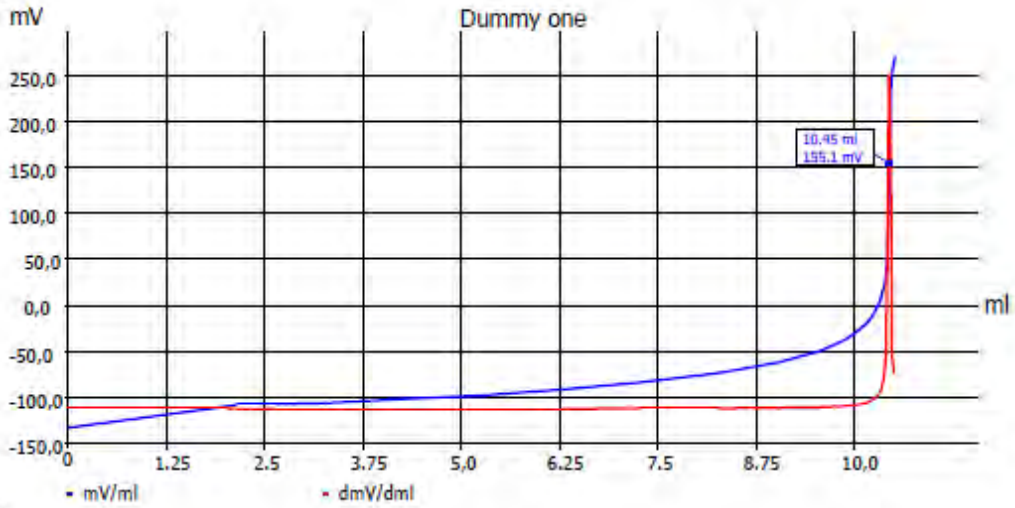
Unit size:	20ml
Unit ID:	10039060
Reagent:	HCl 1 mol/l
Batch ID:	no entry
Concentration [mol/l]:	1.00000
Determined at:	06/27/12 16:03:58
Expire date:	--
Opened/compounded:	--
Test according ISO 8655:	--
Last modification:	06/27/12 9:04:36

Sample titration page 1: Curve and result

The result calculation was wrong here for this titration because of using a wrong factor.

GLP documentation

Titration graph



Method data

Method name:	NCO sample	Titration duration:	2 m 11 s
End date:	27.06.12	End time:	11:03:06

Titration data

Sample ID:	Dummy one	Weight:	5.5463 g
Start mV:	-133.7 mV	End mV:	271.1 mV
EQ:	10.448 ml / 155.1 mV	free NCO:	942.928 %

Sample titration page 2: method

The pre-titration volume can also be changed to 5, 10 or more ml.

Method data overall view

Method name:	NCO sample	Created at:	12/06/11 12:43:30
Method type:	Automatic titration	Last modification:	06/27/12 10:24:02
Measured value:	mV		
Titration mode:	Dynamic	Documentation:	GLP
Dynamic:	Steep		
Measuring speed / drift:	Normal:	minimum holding time:	02 s
		maximum holding time:	15 s
		Measuring time:	02 s
		Drift:	20 mV/min
Initial waiting time:	0 s		
Titration direction:	Increase		
Pretitration:	2.000 ml	Delay time:	10 s
End value:	Off		
EQ:	On		
Slope value:	Steep	Value:	700

Dosing parameter

Dosing speed:	100 %	Filling speed:	30 s
Maximum dosing volume:	50.00 ml		

Unit values

Unit size:	20ml
Unit ID:	10039060
Reagent:	HCl 1 mol/l
Batch ID:	no entry
Concentration [mol/l]:	1.00000
Determined at:	06/27/12 16:03:58
Expire date:	–
Opened/compounded:	–
Test according ISO 8655:	–
Last modification:	06/27/12 9:04:36

Notes

If you have any questions on the application, you can feel free to contact us..

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