SCHOTT Instruments

Determination of Hydroxy number according to ASTM E 1899-02

Application

Content

Use

This test method covers the determination of hydroxyl groups attached to primary and secondary carbon atoms in aliphatic and cyclic compounds and phenols. It is not suitable for determination of hydroxyl groups attached to tertiary carbon atoms. This test method is applicable to polyacetals, temperature sensitive materials, high solids polymer polyols, and rigid polyols.

Appliances

Titrator:	TitroLine Alpha plus (TL 20 plus or TL 50 plus)
Titration tip:	TZ 1643
Magnetic stirrer:	TM 135
other appliances:	250 ml ”erlenmeyer” with sleeve or COD-reaction vessel

Electrodes

Electrode:	N 6480 SA LiCl/Ethanol
Electrolyte:	LiCL/Ethanol L 503 4

Reagents

Solvent:	Acetonitrile
Standardisation:	Potassium Hydrogen Phthhale
Titrant agent:	Tetrabutyl ammonium hydroxide 0,1 mol/L in Methanol/2-Propanol
Acylise mixture	TSI (p-Toluolsulfonyl Isocyanate) 20 ml filled up to 500 ml with Acetonitrile

Description

Preparation of the Tetrabutylammonium Hydroxide 0,1 mol/L in Methanol

Take Tetrabutyl ammonium hydroxide 1 mol/L in Methanol 100 ml and fill up to 1000 ml with 2-Propanol.

Standardisation of the NaOH

With dried (about 1 hour at 120 °C) potassium phthalic acid.

Preparation of the TSI Reagent

TSI (p-Toluolsulfonyl Isocyanate) 20 ml filled up to 500 ml with Acetonitrile

Titration

The sample is weighed in a 100 ml beaker with accuracity of 0,1 mg. The weight is calculated with following formula:

$$\text{weight}[g] = \frac{40}{\text{expected OH - number}}$$

Add to the sample 10 ml of Acetonitrile, add a magnetic stirring bar and stir until the sample is dissolved.

Add 10,0 ml of TSI reagent, cover the beaker and stir slowly for 5 minutes.

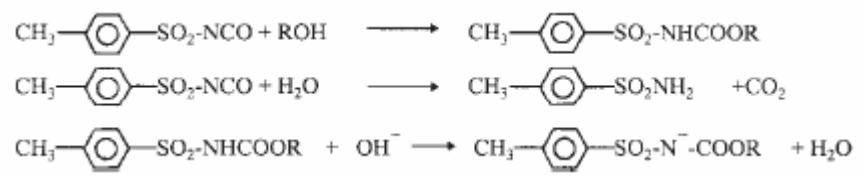
Add 0,5 ml of water and stir slowly for 1 minute.

Add 30 ml Acetonitrile, immerse electrode and start titration.

Electrode handling

As long as the N 61 or N 6480 electrode is not being used it should be stored in the electrolyte solution. For further details, please refer to the electrode's operating instructions.

Theory



Calculation

$$\text{OH - number}\ [mg/g] = \frac{(V_1 - V_2) \cdot c_T \cdot M_{KOH}}{m(\text{sample})}$$

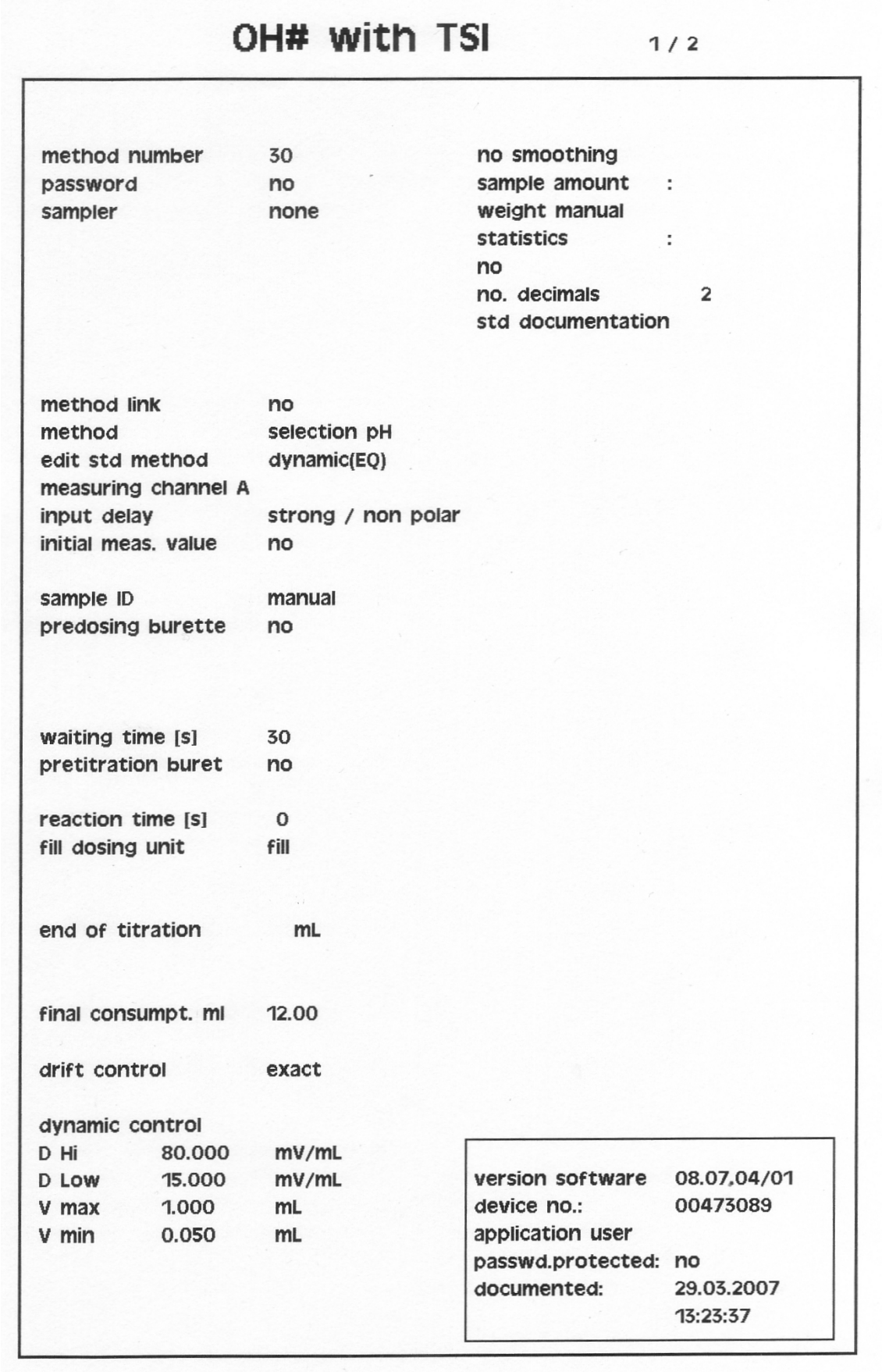
V_1: consumption EQ 1 [mL]
V_2: consumption EQ 2 [mL]
c_T: concentration of Tetrabutyl ammonium hydroxide (0,1 mol/L)
M_{KOH}: molar mass of KOH (56,1 g/mol)
m(sample): amount of the sample [g]

Hazards:

p-Toluenesulfonyl Isocyanate (TSI) is harmful by inhalation, in contact with skin and if swallowed. It may cause sensitization by inhalation and skin contact. It reacts violently with water. It causes severe irritation. It is a Lachrymator. In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. Never add water to this product.
Wear suitable protective clothing, gloves and eye/face protection when handling *p*-Toluenesulfonyl Isocyanate. Use only in a chemical fume hood. Do not breathe vapor. Do not get in eyes, on skin, or on clothing.

Method

sample titration (page 1):



OH# with TSI 1 / 2

method number	30	no smoothing
password	no	sample amount :
sampler	none	weight manual
		statistics :
		no
		no. decimals 2
		std documentation
method link	no	
method	selection pH	
edit std method	dynamic(EQ)	
measuring channel A		
input delay	strong / non polar	
initial meas. value	no	
sample ID	manual	
predosing burette	no	
waiting time [s]	30	
pretitration buret	no	
reaction time [s]	0	
fill dosing unit	fill	
end of titration	mL	
final consumpt. ml	12.00	
drift control	exact	

dynamic control

D Hi	80.000	mV/mL
D Low	15.000	mV/mL
V max	1.000	mL
V min	0.050	mL

version software	08.07.04/01
device no.:	00473089
application user	
passwd.protected:	no
documented:	29.03.2007 13:23:37

sample titration (page 2) :

OH# with TSI

2 / 2

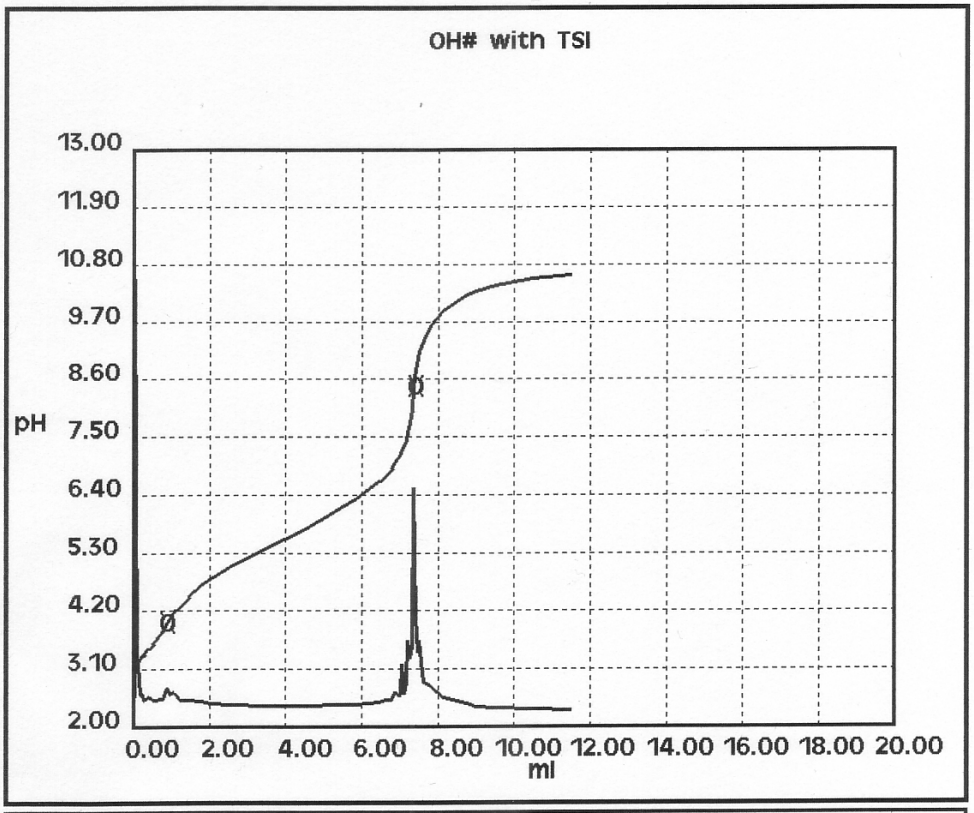
method number	30
calculation :	
formula	mL*F1*F2/Q
formula no.	EQ 1
value F1	1.0000
value F2	1.0000
value Q	1.0000
identifier	
unit	
formula	(mL1-mL2)*F1*F2/Q
formula no.	EQ 2
value F1	0.1050
value F2	56.1100
value Q	1.0000
identifier	OH#
unit	mg/g

version software	08.07,04/01
device no.:	00473089
application user	
passwd.protected:	no
documented:	29.03.2007 13:24:27

Example

sample titration:

titration protocol



method	30		OH# with TSI				
date:	29.03.2007		time:	13:21:34			
sample ID	= 01			duratien	419.96	s	
EQ 1	pH =	3.943	ml =	0.874 (	69	mV/mL)	
EQ 2	pH =	8.442	ml =	7.374 (	432	mV/mL)	
weight		0.6017	g				
initial meas. value		no					
		1.45					
OH#		63.64	mg/g				

Hints

If you have any questions concerning the application, you are welcome to contact us.

Literature

ASTM E 1899-02

An other method to determine the hydroxy number is described in DIN 53 240-02.

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