

Organic
Chemical

Hydroxyl value of Polyether polyol

Acid-Base Titration by Automatic Potentiometric Titrator

Standard	JIS	K 1557-1
	ISO	14900
	JIS	K 0070

1. Abstarct

Hydroxyl value is obtained from a sample of pyridine containing acetic anhydride, which is first acetylated, of which excessive acetylated reagent then hydrolyzes with water, and thus generated acetic acid is titrated with potassium hydroxide up to the endpoint.

The endpoint is determined by the inflexion point on titration curve.

Hydroxyl value of polyether polyol is calculated from titration volume.

2. Reference

- 1) JIS K 1557-1:2007 Part I: Test Method for Determination of Hydroxyl Value of Plastics — Polyols for Polyurethane Products
- 2) ISO 14900:2001 Plastics -- Polyols for use in the production of polyurethane -- Determination of hydroxyl number
- 3) JIS K 0070-1992 Test Method for Acidity, Saponification value, Ester value, Iodine value and Hydroxy value of Chemical products and Unsaponifiables

3. Cautions in measurement

- 1) If the sample contains excessive water, normal acetylation may not be obtained.
- 2) Pyridine and acetic anhydride are harmful and inflammable. Acetic anhydride is corrosive. Caution must be taken when handle these chemicals.

4. Post measurement care

Clean the electrode with pure water each time after measurement is finished.

5. Test equipment

Main unit: Automatic potentiometric titrator (Standard preamplifier: STD—)

Electrode: Glass electrode

Cork type reference electrode

6. Reagent

Titrant : 0.5mol/L Potassium hydroxide Ethanol solution (f=1.00)

Reagent : Pure water, Pyridine, Acetic ether, Perchloric acid (78%), Acetic anhydride

7. Measurement procedure

—Pretreatment—

- 1) Add 4.0g (2.35mL) perchloric acid to 400mL acetic ether.
- 2) Add 8mL acetic anhydride, and leave at room temperature for 30 minutes.
- 3) Cool it to 5°C, and add 42mL chilled acetic anhydride. Leave at 5°C for one hour.
- 4) The solution warmed at room temperature is the acetic anhydride solution.
- 5) Add 100mL water to 300mL pyridine to make pyridine + water (3+1).

—Measurement—

- 1) Sample 3g in a 100mL beaker.
- 2) Add 10.0mL acetic anhydride, and stir for 15 minutes.
- 3) Add 2mL water and 10.0mL pyridine + water (3+1), and stir for 5 minutes.
- 4) Add 10.0mL pyridine.
- 5) Titrate with 0.5mol/L potassium hydroxide ethanol to obtain hydroxyl value.
- 6) Perform blank test likewise.

8. Formula

Hydroxyl value (mg/g) = (BLI - EPI) × TF × Cl × K1 / SIZE

EPI : Titration volume (mL)

BLI : Blank level (56.1542mL)

TF : Factor of titrant (1.00)

Cl : Concentration conversion coefficient (28.05mg/mL)
(Formula mass of potassium hydroxide: 56.11×1/2)

K1 : Unit conversion coefficient (1)

SIZE : Sample size (g)

9. Example of measurement

— Ambient condition —

Room temperature : 23.9 °C	Humidity : 48 %	Weather : Cloudy
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-Titration parameter-

Model : AT-510 Method No. : 12 Titr.mode : Auto Titration Titr.form : EP Stop [Titration parameter] Form : EP Stop APB No. : 1 Unit No. : 1 Detector No. : 1 Unit : mV Max. Volume : 80.0mL Wait Time : 0s Direction : Auto [Control parameter] End Point No. : 1 End sense : Auto End Point Area : Off Separation : Off Over Titr.Vol. : 0mL Gain : 1 Data samp.Pot. : 4.0mV Data samp.Vol. : 0.5mL Control Speed : Medium	[Result parameter] <Calculation> Calc.Type : Sample Conc.1 : Set CO1= (BL1-EP1)*TF*C1*K1/SIZE Unit : mg/g EP No. : 1 Temp.Comp. : Off <Constant> C1(mg/mL) : 28.05 K1 : 1 [Titr. constant] Factor : 1.00 [Blank list] Blank1 : 56.1542
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-Titration curve-

<pre> *** R e s u l t *** Sample No. : 03-08 Date : 1999/10/26 10:42 Sample ID : Method No. : 12 <Auto Titration> Method Name: Method 12 Dose Reagent Name: ----- Titr.Reagent Name: ----- <Blank> Blank 1 : 56.154 Init.Level : 69.2mV Fin.Level :- 284.4mV Fin.Volume : 40.9mL Titr.Time : 00:06:20 Size : 3.0134g MW : 1.000 EGN : 1.000 Conc-1 : 143.37mg/g End Point-1 Volume : 40.7515mL Potential :- 236.1mV - 300 [mV] 100 0.000 64.000 [mL] </pre>	
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(The above printout data were obtained from titration by AT-510 unit)

«Titration parameter»

Form: of titration / APB No. the burette used in titration / Unit No.: APB Unit File number

Detector No.: the detector used in titration/ Unit of potential / Max Volume: of titration

Wait Time: before titration starts/ Direction.: of titration

«Control parameter»

End Point No.: number of EPs / End sense: of EP detection / End Point Area: EP detection area

Separation: of potential / Over Titr.Vol. over-titrated volume / Gain: sensitivity of detection signal

Data samp.Pot.: potential change of sampling signal / Data samp.Vol. : titration volume of sampling signal

Control Speed: control mode of titration speed

«Calculation parameter»

Calc.Type: of formula / Conc.1 formula 1 / Unit: of results

EP No. of calculation / Temp.Comp.: temperature compensation / C1(mg/mL): concentration conversion coefficient

K1: unit conversion coefficient / TF: factor of titration liquid / Blank1: blank level 1

—Measurement results—

n	Sample (g)	Titration (mL)	Hydroxyl value(mg/g)	Hydroxyl value	
				Mean	SD
1	<u>3.0134</u>	<u>40.7515</u>	<u>143.37</u>	141.83 mg/g	1.3588 mg/g
2	3.0427	40.8803	140.81	141.83 mg/g	1.3588 mg/g
3	3.0127	40.9781	141.30	141.83 mg/g	0.958 %

*The above test results were obtained by 3 tests of the same sample.

* Red underline shows the data from page 3/4.

10.Summary

Hydroxyl value is expressed by potassium hydroxide in mg required to neutralize acetic acid coupled with hydroxyl group when 1 gram of sample is acetylated.

Hydroxyl value of polyol is important for quality control in production of polyurethane.

The sample measurement this time shows a good repeatability with 1% of relative standard deviation.

Precise and reliable measurement is assured by the automated potentiometry.

Hydroxyl value of polyether polyol can be perfectly measured by any of the following titration systems manufactured by Kyoto Electronics (KEM).

【AT-610】



Awarded Product of Supreme Technology from Kyoto City

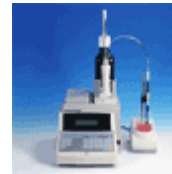
- Easy key entry by touch panel of large color LCD (8-inch wide)
- Simultaneous titration in parallel
- Both potentiometric and Karl Fischer moisture titration (coulometric·volumetric) can be performed at a time.

【AT-510】



- Compact and cost performance model
- PC card expands data memory for convenience and versatility.

【AT-500N-1】



- Low cost and high performance
- Easy view with back light LCD
- GLP/GMP conformed model

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