

<i>Category</i>	<i>Potentiometric Titrator COM series</i>
Resins/Rubber/Adhesives /Paints/Electronics etc...	Total amine number in amine hardener for epoxy resin by Acid/base titration method (non-aqueous)
Referenced methods	JIS K7237

Key words; epoxy resins, amine, hardener, curing agent

Outline

Epoxy resins have excellent properties of chemical and heat resistance, electrical insulating property and adhesion, a wide range of applications, especially utilized as sealant for electronic elements on the IC.

Epoxy is the general term for a group of chemical compounds in which two or more epoxide groups ($\begin{matrix} \text{CH}_2 & - & \text{CH} \\ & \diagdown & / \\ & \text{O} & \end{matrix}$) are bound in each monomer. Epoxy does not become harden in itself, thus it is necessary to add curing agent or catalyst to form hardener compounds. By adding the hardener such as fatty amine, the amine groups react with the epoxide groups to form a covalent bond, resulting a thermosetting resin. The amount of the hardening agent corresponds to the epoxy equivalent of each epoxy resin (mass of the resin containing one equivalent of epoxy group). Thus, the process of determining the epoxy equivalent is an important part of quality control.

Amine number is expressed as milligrams (mg) of potassium hydroxide (KOH) required to neutralize the total basic nitrogen compounds contained in 1g of sample.

Reagents

<i>Titrant</i>	:	0.1mol/L perchloric acid (in acetic acid)
<i>Titration solvent</i>	:	Mixture of o-nitrotoluene 900mL and acetic acid 100mL 100mL per measurement In this analysis, 50mL acetic acid used as alternate

Instruments & Electrodes

Recommended automatic titrator	COM-1700S / COM-1600S
<ul style="list-style-type: none"> • GE-101B Glass electrode • RE-201 Reference electrode* 	} standard accessories
(*replace the electrolyte with saturated sodium perchlorate in acetic acid)	



Summary of Test Method

A weighed sample is dissolved in a mixture of o-nitrotoluene and acetic acid, and titrated potentiometrically with 0.1mol/L perchloric acid in glacial acetic acid.

Recommended size of test portion is equilibrium to 2 ~ 3mmol basic nitrogen.

Condition parameters (example)

Master File 1 Condition 1 (for BLANK)				Master File 2 Condition 2 (for measurement)			
Method	Auto			Method	Auto		
Buret No.	1	Mode No.	19	Buret No.	1	Mode No.	5
Amp. No.	1	Pre Int	0 sec	Amp No.	1	Pre Int	0 sec
Meas Unit	mV	Del K	0	Meas Unit	mV	Del K	5
S-Timer	10 sec	Del Sens	0 mV	S-Timer	10 sec	Del Sens	0 mV
CP mL	0 mL	Int Time	5 sec	CP mL	0 mL	Int Time	5 sec
DP mL	0 mL	Int Sens	3 mV	DP mL	0.1 mL	Int Sens	3 mV
End Sens	500	BrT Speed	2	End Sens	300	BrT Speed	2
Over mL	0.1 mL	Pulse	16	Over mL	0.2 mL	Pulse	40
Max volume	20 mL			Max Volume	20 mL		
Unit	mL			Unit	mg/g		
Size	0 g			Size	--- g		
Blank	0 mL			Blank	BLANK result value		
Factor	0			Factor	Titre of the titrant		
Molarity	0			Molarity	0.1 mol/L		
K	0			K	56.11		
L	0			L	0		
Formula	D			Formula	(D-B)xKxFXM/S		

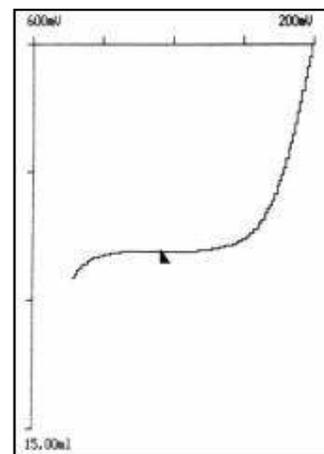
Tips for Measurements

1. Effect of water on the titration curve

If water is mixed into the non-aqueous system, change in slope of the titration curve is very subtle and no clear end point is obtained.

2. Temperature of the titrant

Since the titration solvent used is acetic acid-base, the expansion coefficient is large. Thus, it is necessary to compensate with the factor value which is calculated from the temperature differences between the temperature of the titrant for the standardization and that for the measurement.



- Titration curve example -

For more information, please feel free to contact:

Hiranuma Sangyo Co., Ltd.

1739 Motoyoshida-cho, Mito, Ibaraki 310-0836 JAPAN

Phone: +81-29-247-7343 / Fax: +81-29-247-0381

URL <http://www.hiranuma.com> E-mail info@hiranuma.com



ISO 9001
ISO 13485 Certified



ISO 14001 Certified