

Organic
Chemical

Isocyanic Group in Curing Agent

Acid-base titration by
Automatic Potentiometric Titrator

Standards	JIS	K 7301	ISO	14896
	JIS	K 1603		
	JIS	K 1556		

1. Abstract

Measurement of Isocyanic group content is standardized by the below quoted test methods.

Sample liquid after dissolved in dry toluene reacts with excessive di-n-Butylamine, and back titrate residual di-n-Butylamine with hydrochloric acid up to the endpoint. The content of Isocyanic group in curing agent is calculated from titration volume.

2. Reference

- 1) JIS K 7301-1995 Test Method for Tri-range Isocyanic Prepolymer for Heat-curable Urethane Elastomer
- 2) JIS K 1603-1985 Test Method for Polymethylene Polyphenyl Polyisocyanate
- 3) JIS K 1556-2006 Test Method for Tolunediisocyanate — Polyurethane raw materials (Appendix 1)
- 4) ISO 14896-2006 Plastics -- Polyurethane raw materials -- Determination of isocyanate content

3. Cautions in measurement

- 1) Repeated measurement lowers sensitivity of glass electrode, and regeneration process of electrode is required each time a measurement is finished. To regenerate the electrode, dip it in an alcohol solution once for cleaning, and then dip it in pure water for 10 to 60 min. (depends on how much contaminated)
- 2) Handle chemicals in a well ventilated room or use a draft.

4. Post-measurement care

After a series of measurements for the day is over, be sure to regenerate the electrode according to the preceding "3. Cautions in measurement".

5. Test equipment

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

Electrode: Glass electrode

Cork type reference electrode

Temperature compensation electrode

6. Reagent

Titrant : 1.0 mol/L Hydrochloric acid (f = 1.00)

Solvent : Toluene + 2-Propanol

Reagent : 1.5N di-n-Butylamine · Toluene solution

7. Measurement procedure

—Measurement—

- 1) Prepare 1g sample in a 200mL beaker.
- 2) Add 20mL Toluene.
- 3) Add 20mL of 1.5N di-n-Butylamine · Toluene solution
- 4) Leave it for 15 minutes.
- 5) Add 70mL of 2-propanol.
- 6) Titrate with 1.0 mol/L Hydrochloric acid up to the endpoint.
- 7) Likewise, perform blank test.

8. Formula

Content of Isocyanic group (wt%) = (BL1 - EP1) × FA1 × C1 × K1 / SIZE

EP1 : Titration volume (mL)

BL1 : Blank level (33.255mL)

FA1 : Titant factor (1.00)

C1 : Concentration conversion (42.0)

(Chemical formula weight (g/mol) of Isocyanic group)

K1 : Coefficient (0.1)

SIZE : Sample size (g)

9. Example of measurement

— Ambient condition —

Room temperature : 25 °C	Humidity : 62 %	Weather : Fair
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- Titration parameter -

Model : AT-420	
Method No. : 13	
Titr.mode : Auto Titr	
Titr.form : EP Stop	
[TITR. PARA]	
Form : EP Stop	Sample Measurement
Buret No. : 1	Conc1 CalcuNo. 2
Preamp : STD	Conc1 Dim. [%]
Detector No. : 1	Conc1 EP Position 1
Dimension : mV	Data [f(EP2-EP1)]
Max.Vol : 40.0mL	T.Type [Back]
W.Time : 2s	Data [f(T.Vol)]
Direction : Auto	Local Blank
	Blank 33.255mL
	Common T.Factor
	K1 0.1
	C1 42.0mg/mL
	Temp.Comp. [Off]
[CTRL. PARA]	
End Point No. : 1	
S(dE) : 50	
S(E/mL) : 100	
O.Titr : 0mL	
Gain : 1	
C.Speed : 4	
S.Pot : 4.0mV	
M.Unit : 0.5mL	
Separation : Off	
A.Simulation : Off	

- Titration curve -

No. 09 - 05	
Date 99/10/20 14:17	
Combine-0 (Method 20)	
4th-Combine	
Method	13 [Auto Titr]
I.Lev	99.2mV
F.Vol	31.4350ml
F.Lev	- 271.4mV
D.Vol	20.0000ml
Time	00:03:47
Size	1.0468g
Conc-1	8.0915%
EP-1	31.2383ml - 150.5mV

(The above printout data were obtained from titration by AT-420)

«TITR. PARA: Titration parameter»

Form: of titration / Buret No.: the burette used in titration / Detector No.: the detector used in titration
Dimension: potential unit/ Max Volume: of titration /Wait Time: before titration starts /Direction.: of titration

«CTRL. PARA: Control parameter»

End Point No. number of EPs detected / S(dE): EP potential (difference) / S(E/mL): EP potential (differential)
Over Titr.Vol. over-titration volume /Gain: sensitivity of detection signal/ C.Speed: dose speed/ S.Pot.: potential changes of sampling signal /Separation: of potential / Stab.: stability sense potential/ L.Time: time for stability check
M.Unit.: titration for sampling signal /Separation: of potential/ A.Simulation: redetection of EP

«CALCU. PARA: Result parameter»

Conc.1: formula 1 / Conc1 Dim: unit of concentration/Conc1EP Position: EP at conc. 1
K1: unit conversion coefficient/ Factor: of reagent /Blank : blank level /Data: titration form used in calculation
T.Type: titration type (normal or back) /Common T.Factor: factor of titrant registered
C1(mg/mL): conc. at EP1/Data: calculation of titration / Temp.Comp.: temperature compensation

—Measurement results—

n	Sample (g)	Titration (mL)	Content (wt%)
1	1.0468	31.2383	8.0915
2	1.0654	31.1947	8.1221
3	1.0352	31.2594	8.0965

Content of Isocyanic group	
Mean	8.1034 %
SD	0.0164 %
RSD	0.2026 %

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

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

10. Summary

The chemical compound Isocyanate having chemical structure of $-N=C=O$ is also called Isocyanic ester.

The above sample test shows a good repeatability of 0.2% relative standard deviation as the automated potentiometry is the most reliable measurement technique.

The analysis of Isocyanic group in curing agent can be easily and precisely made by any of the following titration systems manufactured by Kyoto Electronics (KEM).

【AT-610】



Awarded Product of Supreme Technology from Kyoto City

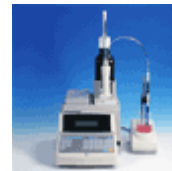
- Easy key entry by touch panel of large color LCD (8-inch wide)
- Simultaneous titration in parallel
- Both potentiometric and Karl Fischer moisture titration (coulometric • volumetric) can be performed at a time.

【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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