

Sheet No.

**GT200-PT036E**

# Determination of isocyanate group content in Methylenediphenyl 4,4'-Diisocyanate

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Method	: Acid-base back titration
Apparatus	: Automatic Titrator model GT-200 (GT0EF) Electrodes: Combined Glass Electrode (GTPC1B) *Inner solution: 3.3mol/l Potassium chloride
Titration mode	: INF, Detection: pH
Related standard	: JIS K7301: Testing methods for tolylene diisocyanate type prepolymers for thermosetting urethane elastomers JIS K1603: Aromatic isocyanates for use in the production of polyurethanes. Part1: Determination of isocyanate content

\*This application sheet is provided as reference, and does not assure the measurement results. Please consider analysis environment, external factors and sample nature for optimal conditions before the measurement.

## Outline

Isocyanate content rate is measured by back titration with the following procedure. After dissolve the sample into dehydrated toluene, add excess volume of Di-n-Butylamine / toluene solution. Isocyanate is converted to urea by the above reaction and back titrate the remained Di-n-Butylamine by Hydrochloric Acid Titrant.

## Reagents

[ Titrant ]

■0.5mol/L-hydrochloric acid solution (Volumetric analysis grade)

[ Reagents ]

■Dehydrated toluene: Moisture content must be less than 50ppm

Di-n-Butylamine solution: Prepare 100ml of the solution by dissolving 1.3g of Di-n-Butylamine into dehydrated toluene.

## Analytical Procedure

- (1) Take 0.6g of sample accurately into a 200ml co-stoppered Erlenmeyer flask.
- (2) Add 25ml of dehydrated toluene and dissolve the sample.
- (3) Add 10ml of Di-n-Butylamine solution and allow to react for 15min.
- (4) Add 100ml of 2-propanol and titrate with 0.5mol/L hydrochloric acid solution.
- (5) Determine blank by running all of the above procedures without sample.

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[ Calculation ]

$$\text{Isocyanate content (\%)} = ((\text{BL}-\text{A1}) \times \text{M} \times f \times \text{FW}) / (\text{S} \times 1000) \times 100$$

BL : Titration volume of 0.5mol/L Hydrochloric acid titrant from blank test

A1 : Titration volume of 0.5mol/L Hydrochloric acid titrant from sample test

M : Molar concentration of 0.5mol/L hydrochloric acid titrant

f : Factor of 0.5mol/L hydrochloric acid titrant

FW : Formula weight of isocyanate group, 42.02 (g/mol)

S : Sample size (g)

1000 : Unit conversion factor (mg g)

100 : Unit conversion factor (to percentage)

**Other Requirements**

- Before the testing, pH calibration is required.
- Set the Buret Initial suction position to 75%.
- Make sure to confirm labels and safety data sheets of reagents and gases used for the measurement and handle them with enough care.
- Wear protective equipment (eye protector, gloves and others) when handling reagents. Measurement results

**Measurement Results**

	Sample size (g)	Titration volume (ml)	Results (%)
1	0.6306	10.0552	33.12
2	0.6039	10.4533	33.20
3	0.6110	10.4259	32.91

Nos. of data (n) 3  
 Average 33.07  
 Standard deviation (SD) 0.15  
 Relative S.D. (RSD%) 0.4600

Blank value 19.9872ml

Isocyanate content of commercially available Methylenediphenyl 4,4'-Diisocyanate was tested.

Result measured by GT-200 was 33.07%.

Repeatability was good with relative standard deviation (RSD%) less than 0.5%.

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ID No. : 1 GT No.1

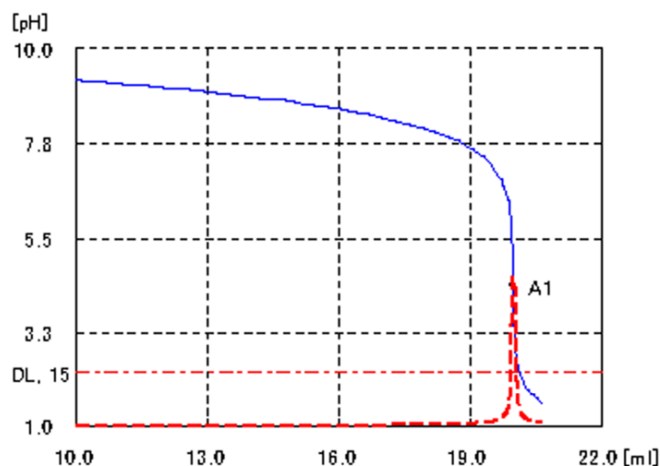
User : GT-200

Measurement : 2016/01/27 11:28

Sample name : Blank test

Type : Sample Titr

Sample size(S) : 1 [ml]



C1 : 19.9872 [ml]

A1 : 19.9872 [ml] 4.371 [pH]

Initial potential(Pi) : 11.393 [pH]

Start (Start) : 10 [ml]

9.228 [pH]

End (End) : 20.638 [ml]

1.575 [pH]

Measurement time(Time) : 9'50"

Run file No. : 16 Isocyanate group

Titration file No. : 21 Blank test for isocyanate group content rate

Mode : INF

End1, End1 Width : 5 [pH]  $\pm$  5.7 [pH]

Detect : pH

BRT No. : 1

Reagent : 39

Preset1 Mode : V Buret(BRT) : 1

WTint : 10 [sec]

Injection volume(Vol) : 10 [ml]

Vup : 300 [ $\mu$ l]

Position(Pos) : Titration

Vlow : 20 [ $\mu$ l]

dE : 0.05 [pH]

dT : 4 [sec]

DL : 15 [pH/ml]

DetCnt : 6

Vmax : 50 [ml]

Vover : 0.5 [ml]

C1 : A1

[ml]

Reagent(Reag) : HCl

Equivalent(E) : 1

Molarity(M) : 0.5 [Mol/l]

Factor(f) : 1.001

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ID No. : 2 GT No.1

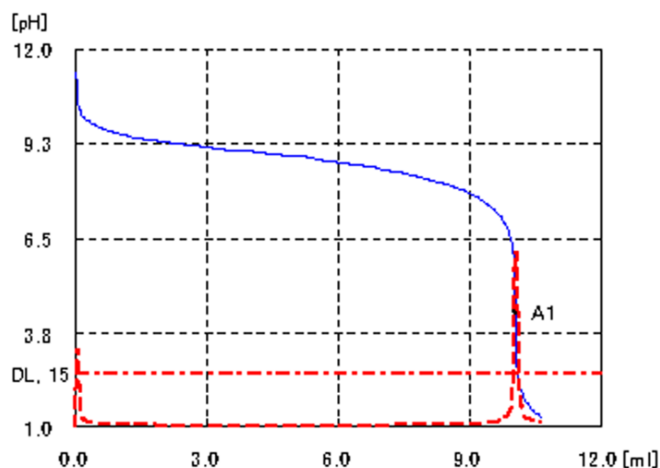
User : GT-200

Measurement : 2016/01/27 14:58

Sample name : TEST SAMPLE

Type : Sample Titr

Sample size(S) : 0.6306 [g]



C1 : 33.124 [%]

A1 : 10.0552 [ml] 4.384 [pH]

Initial potential(Pi) : 11.295 [pH]

Start(Start) : 0 [ml] 11.295 [pH]

End(End) : 10.666 [ml] 1.272 [pH]

Measurement time(Time) : 9'29"

Run file No. : 16 Isocyanate group

Titration file No. : 11 Isocyanate group content rate

Mode : INF End1, End1 Width : 5 [pH] ± 5.7 [pH]

Detect : pH

BRT No. : 1

Reagent : 39

WTint : 10 [sec]

Vup : 300 [μl]

Vlow : 20 [μl]

dE : 0.05 [pH]

dT : 4 [sec]

DL : 15 [pH/ml]

DetCnt : 6 C1 : ((BL-A1)\*M\*\*FW)/(S\*1000)\*100

Vmax : 50 [ml]

Vover : 0.5 [ml] [%]

Reagent(Reag) : HCl

Equivalent(E) : 1

Molarity(M) : 0.5 [Mol/l]

Factor(f) : 1.001

Blank(BL) : 19.9872 [ml]

Formula weight(FW) : 42.02

Buret Injection Speed : 500 [ul/sec]