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HIRANUMA APPLICATION DATA		Automatic Titrator	Data No.	M9	Apr. 19, 2018
Resins, Oils and Fats, Rubber, Adhesives, Paints	Determination of isocyanate (NCO) content in adhesives				

1. Abstract

Synthetic adhesives like hydrophilic macromolecule –Isocyanate type wood adhesives are consisted of base compound and cross-linker; the principal components of base compound are macromolecule aqueous solution or aqueous dispersing element, or those combination. The principal components of cross-linker is isocyanate compounds. The measurement procedure of isocyanate (NCO) content described in this report is standardized by JIS K 6806. NCO content is determined by the neutralization titration which excess di-n-butylamine is titrated with hydrochloric acid standard solution after sample and di-n-butylamine are mixed and reacted. A measurement example of potentiometric titration for NCO determination is introduced in this report.

2. Configuration of instruments and reagents

(1) Configuration

Main unit	: Hiranuma Automatic Titrator COM series
Electrode	: Glass – Reference electrode GR-522BZ
Option	: Buret tip (Tube Type)

(2) Reagents

Titration	: 0.5 mol/L Hydrochloric acid standard solution
Solvent	: 2-propanol (Isopropyl alcohol)
Additive	: Di-n-butylamine – toluene solution Dissolve 130 g of di-n-butylamine in dehydrated toluene.

3. Measurement procedure

- (1) Take about 2 g of the sample into Erlenmeyer flask and accurately weigh it.
- (2) Dispense 25 mL of di-n-butylamine – toluene solution with volumetric pipette.
- (3) Add stirring bar and close it with a stopper. Gently stir the solution for 15 min with stirrer.
- (4) Add 150 mL of 2-propanol.
- (5) Immerse the electrode and titrate with 0.5 mol/L hydrochloric acid standard solution.
- (6) Measure the blank value by operation of (2) ~ (5) without sample.

4. Measurement conditions and results

Examples of titration conditions

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Buret No.	1	Size	0	g	Mode No.	4	Pre Int	0	sec
Amp No.	1	Blank	0	mL	Del K	9	Del Sens	0	mV
D. Unit	pH	Molarity	0.5	mol/L	Int Time	3	Int Sens	3	mV
S-Timer	5 sec	Factor	1.005		BrT Speed	2	Pulse	40	
C.P. mL	45 mL	K	0						
T Timer	0 sec	L	0						
D.P. mL	0 mL	Unit		mL					
End Sens	1000	Formula		D					
Over mL	0.3 mL	Digits		3					
Max.Vol.	60 mL	Auto In Pram.		Non					

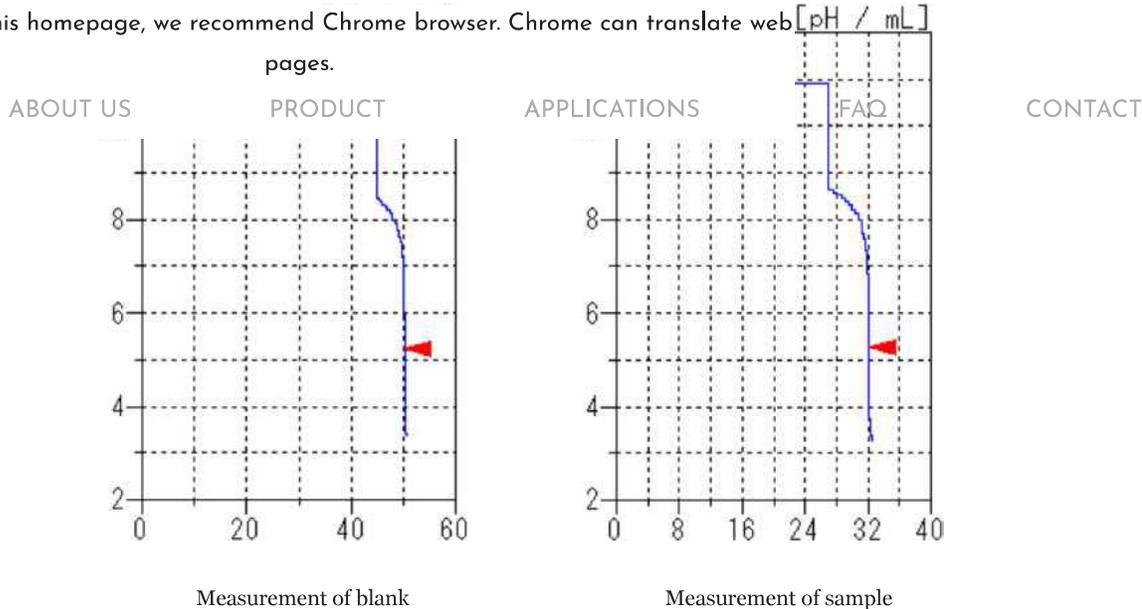
Measurement of sample

Cndt No.	2	ConstantNo.	2	Mode No.	4
Method	Auto	Size	2.0412 g	Pre Int	0 sec
Buret No.	1	Blank	50.196 mL	Del K	9
Amp No.	1	Molarity	0.5 mol/L	Del Sens	0 mV
D. Unit	pH	Factor	1.005	Int Time	3 sec
S-Timer	10 sec	K	42.02	Int Sens	3 mV
C.P. mL	27 mL	L	0	BrT Speed	2
T Timer	10 sec	Unit	%	Pulse	40
D.P. mL	0 mL	Formula	(B-D)*K*F*M/(S*10)		
End Sens	1000	Digits	4		
Over mL	0.3 mL	Auto In Pram.	Non		
Max.Vol.	60 mL				

Measurement results

	Number of Measurements	Size (g)	Titrant volume (mL)	NCO content (%)	Statistical calculation results	
Blank	1	-	50.178	-	Avg. (Blank)	50.196 mL
	2	-	50.214	-		
Sample	1	2.0412	32.105	18.714	Avg. SD CV	18.74 % 0.0287 % 0.15 %
	2	2.0429	32.035	18.771		
	3	2.0473	32.017	18.749		

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Examples of titration curves

5. Note

- (1) Dehydrated toluene (less than 50 ppm water content) is used as a solvent to dissolve sample. Please note that the water in solvent causes measurement error because it reacts with isocyanate group.
- (2) Electrode which can be directly inserted into Erlenmeyer flask is preferred for this titration.

Keywords: Isocyanates, NCO, Neutralization titration, JIS K 6806, JIS K 1603, JIS K 7301

*Some measurement would not be possible depending on optional configuration of system.

