| *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

| | | |
|---|---|---|
| Titrant | : | 0.5 mol/L Hydrochloric acid standard solution |
| Solvent | : | 2-propanol (Isopropyl alcohol) |
| Additive | : | Di-n-butylamine - toluene solution<br>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

Measurement of blank

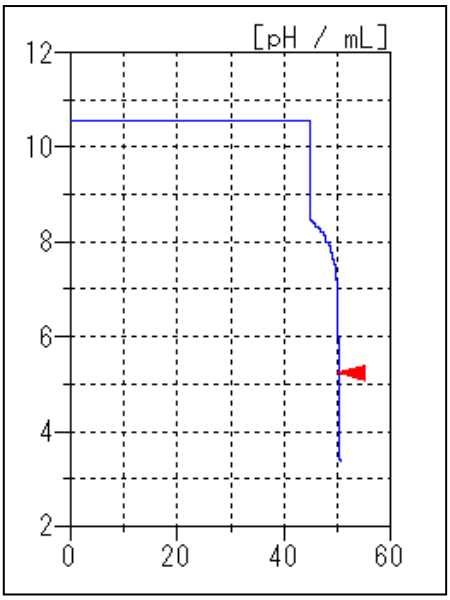
| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Cndt No. | 1 | | | | | | | |
| Method | Auto | | ConstantNo. | 1 | | Mode No. | 4 | |
| Buret No. | 1 | | Size | 0 | g | Pre Int | 0 | sec |
| Amp No. | 1 | | Blank | 0 | mL | Del K | 9 | |
| D. Unit | pH | | Molarity | 0.5 | mol/L | Del Sens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

Measurement of sample

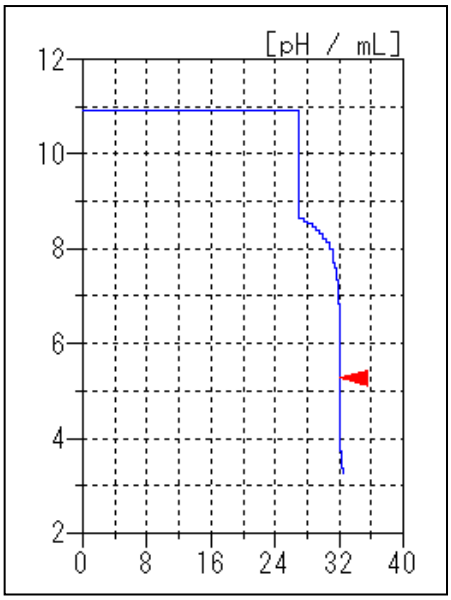
| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Cndt No. | 2 | | | | | | | |
| Method | Auto | | ConstantNo. | 2 | | Mode No. | 4 | |
| Buret No. | 1 | | Size | 2.0412 | g | Pre Int | 0 | sec |
| Amp No. | 1 | | Blank | 50.196 | mL | Del K | 9 | |
| D. Unit | pH | | Molarity | 0.5 | mol/L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

### 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. | 18.74 % |
| | 2 | 2.0429 | 32.035 | 18.771 | SD | 0.0287 % |
| | 3 | 2.0473 | 32.017 | 18.749 | CV | 0.15 % |




Measurement of blank




Measurement of sample

Examples of measurement 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.