

Paint, Adhesive

Chloride Ion of Adhesive

Precipitation titration by
Automatic Potentiometric Titrator

Standard

1. Abstract

The concentration of chloride ion in adhesive is measured as follows: the sample is first dissolved in solvent to extract chloride ion, and then, a constant and excess amount of silver nitrate solution was added. the excess silver ion is back titrated with potassium thiocyanate solution until the endpoint is reached on titration curve.

The concentration of chloride ion is calculated from the difference between titration volume and blank value, which is first obtained by blank test in advance.

2. Reference

- 1) JIS K 0050-2005 General Method for Chemical Analysis
- 2) JIS K 8001-1998 General Rule for Reagent Test Method

3. Cautions in measurement

- 1) In order to avoid measurement error due to flowing out chlorine, use a silver sulfate reference electrode or silver chloride reference electrode of which junction liquid is replaced with 1 mol/L potassium nitrate solution.
- 2) Wipe the tip of silver electrode with polishing paper before use.

4. Post-measurement care

After a series of measurements for the day is over, clean the electrode tip with polishing paper as instructed in the above step 3. "Cautions in measurement".

5. Test equipment

Main unit : Automatic potentiometric titrator (Standard preamplifier: STD-)
Electrode : Option Combination silver electrode
(Junction liquid : 1mol/L-potassium nitrate)

6. Reagent

Reagent : 0.02mol/L potassium thiocyanate solution (f=1.063)
Solvent : 2-propanol, methanol, toluene
Additive : Nitric acid, 0.02mol/L silver nitrate solution

7. Measurement procedure

—Blank test—

- 1) Titrate 25.0mL of 0.02mol/L silver nitrate solution with 0.02mol/L potassium thiocyanate solution, and obtain blank value.

—Measurement—

- 1) Prepare 2.5g sample in a 200mL beaker.
- 2) Add 10mL 2-propanol, 5mL methanol and 25mL toluene, and close the beaker. Stir for 15 minutes to dissolve the sample.
- 3) Transfer the solution to a funnel, and add 25mL pure water. Shake it for one (1) minute to extract chloride ion in water phase.
- 4) Transfer the chloride extracted solution to a 200mL beaker.
- 5) Repeat step 3) and 4) for two times.
- 6) Add 5mL nitric acid and then 25.0mL of 0.02mol/L silver nitrate solution onto the beaker, and precipitate chloride ion in sample liquid as silver chloride.
- 7) Back titrate the excess silver ion with 0.02mol/L potassium thiocyanate, and obtain chloride ion concentration from the difference between titration volume and blank value.

8. Formula

Chloride ion concentration (%) = (BLI - EPI) × TF × Cl × K1 / SIZE

- EPI : Titration volume (mL)
BLI : Blank level (24.774mL)
TF : Reagent factor (1.063)
Cl : Concentration conversion coefficient (0.709mg/mL)
(Chloride ion in mg equivalent to 1mL of 0.02mol/L AgNO₃)
K1 : Unit conversion coefficient (0.1)
SIZE : Sample size (g)

9. Example of measurement

— Ambient condition —

Room temperature : 27 °C	Humidity : 69 %	Weather : Fair
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-Measurement parameter-

<p>Model : AT-510 Method No. : 01 Titr.mode : Auto Titration Titr.form : EP Stop</p> <p>[Titration parameter]</p> <p>Form : EP Stop APB No. : 1 Unit No. : 1 Detector No. : 1 Unit : mV Max. Volume : 40.0mL Wait Time : 7s Direction : Auto</p> <p>[Control parameter]</p> <p>End Point No. : 1 End sense : Auto End Point Area : Off Separation : Off Over Titr.Vol. : 0mL Gain : 1 Data samp.Pot. : 4.0mV Data samp.Vol. : 0.5mL Control Speed : Medium</p>	<p>[Result parameter]</p> <p><Calculation> Calc.Type : Sample Conc.1 : Set CO1= (BL1-EP1)*TF*C1*K1/SIZE</p> <p>Unit : % EP No. : 1 Temp.Comp. : Off</p> <p><Constant> C1(mg/mL) : 0.709 K1 : 0.1</p> <p>[Titr. constant] Factor : 1.063</p> <p>[Blank list] Blank1 : 24.774</p>
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-Titration curve-

<p>*** Result ***</p> <p>Sample No. : 01-01 Date : 2007/09/03 14:14 Sample ID :</p> <p>Method No. : 01 <Auto Titration> Method Name :</p> <p>Titr.Time : 00:04:20</p> <p>Size : <u>2.5067g</u></p> <p>Conc-1 : <u>0.2149%</u></p> <p>End point-1 Volume : <u>17.6251mL</u> Potential : 202.0mV</p>	<p style="text-align: center;">100 [mV] 400 0.000</p> <p style="text-align: center;">32.000 [mL]</p> <table border="1" style="width: 100%; border-collapse: collapse; margin-top: 10px;"> <thead> <tr> <th>No.</th> <th>dE</th> <th>dE/dmL</th> <th>St</th> </tr> </thead> <tbody> <tr> <td>56</td> <td>1.67E 2</td> <td>6.07E 2</td> <td>3</td> </tr> </tbody> </table>	No.	dE	dE/dmL	St	56	1.67E 2	6.07E 2	3
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(The above parameters and titration curve were printed out by AT-510)

<p>«Titration parameter» Form: of titration / APB No. the burette used in titration / Unit No.: APB Unit File number Detector No.: the detector used in titration / Unit: of potential / Max Volume: of titration Wait Time: before titration starts / Direction.: of titration</p> <p>«Control parameter» End Point No. number of EPs detected /End sense: EP detection /End Point Area: detection area Separation: of potential / Over Titr.Vol. over-titration volume / Gain: sensitivity of detection signal Data samp.Pot.: potential changes of sampling signal /Data samp.Vol.: titration volume of sampling signal Control Speed: of dosing</p> <p>«Result parameter» Calc.Type: of formula / Conc.1: formula 1) / Unit of result /EP No. EP number for calculation Temp.Comp.: temperature compensation / EP1: titration volume / BL1: blank level C1(mg/mL): concentration conversion coefficient / K1: unit conversion coefficient</p>

–Measurement results–

n	Sample (g)	Titration (mL)	Concentration (%)
1	<u>2.5067</u>	<u>17.6251</u>	<u>0.2149</u>
2	2.5063	17.6350	0.2147
3	2.5067	17.3587	0.2229

Concentration of chloride ion	
Mean	0.2175 %
SD	0.0047 %
RSD	2.1506 %

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

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

10. Summary

Depending on the kind of adhesive, lower level of chloride ion is preferred in the quality control.

This measurement demonstrated as above shows a good repeatability with 2.1% relative standard deviation.

The use of automatic potentiometric titrator enables precise and stable measurement.