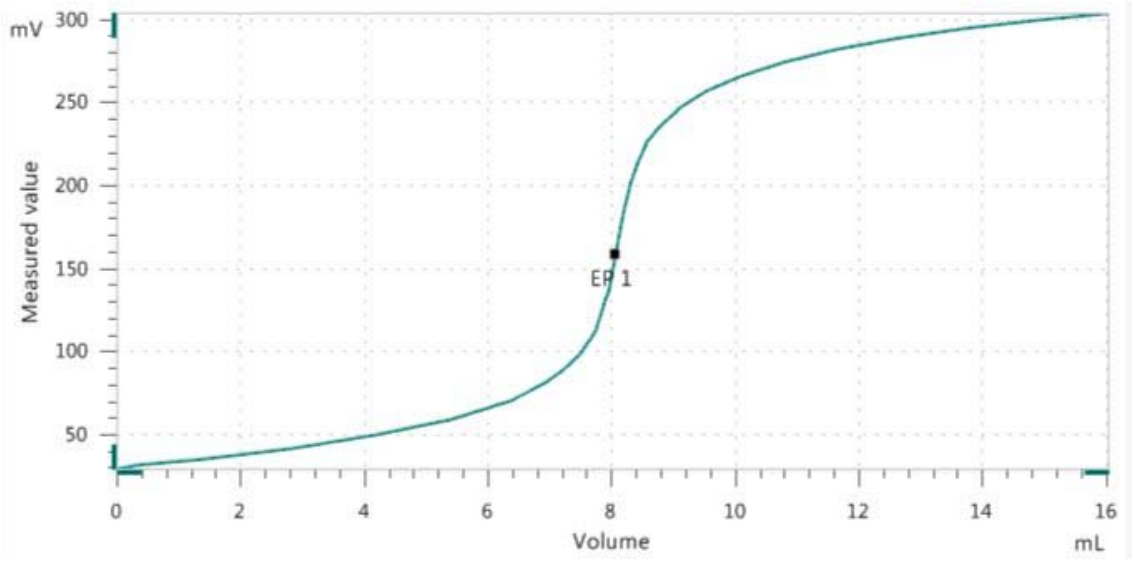
Titration Application Note T-174

# Determination of hydrolyzable chloride content in liquid epoxy resins according to ASTM D1726 (method B)




The hydrolyzable chloride content of epoxy resins is an important variable in determining their reactivity and the properties of coating made from them.

With the OMNIS system a fast and accurate determination by a potentiometric titration using the dAg ring electrode and silver nitrate as titrant is realized.

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# Method description

### Samples

Liquid epoxy resin

### Sample preparation

No sample preparation is required.

### Configuration

| | |
|---|---|
| OMNIS Advanced Titrator with stirrer | 2.1001.0220 |
| OMNIS Dosing Module, 2x | 2.1003.0010 |
| Cable MDL PL/SO 1 m, 2x | 6.02102.020 |
| OMNIS 20 mL cylinder unit, (titrant) | 6.03001.220 |
| OMNIS 50 mL cylinder unit, 2x (KOH, glacial acetic acid) | 6.03001.250 |
| Digital measuring module | 6.02100.010 |
| Stirring bar / 30 mm, 5x | 6.1903.060 |
| Heating plate with stirrer | - |
| Reflux condenser | - |
| Erlenmeyer flask, glass 250 mL | - |
| Electrode cable plug-in head Q / plug P, 0.55 m | 6.02104.300 |
| OMNIS Stand-alone license (including one instrument license) | 6.06003.010 |
| dAg ring electrode | 6.00402.300 |

### Solutions

| | |
|---|---|
| Titrant | $c(AgNO_3)$ = 0.0025 mol/L in methanol |
| Potassium hydroxide | c(KOH) = 0.1 mol/L in methanol |
| Acetone | w(acetone) ≥ 99.5% |
| Toluene | w(toluene) ≥ 99.3% |
| Glacial acetic acid | $w(C_2H_4O_2)$ ≥ 99.0% |

### Analysis

*Blank*

The blank is determined the same way as the sample, just without sample.

*Sample*

2 – 3 g sample is pipetted into an Erlenmeyer flask. 50 mL c(KOH) = 0.1 mol/L in methanol, 20 mL toluene and 20 mL acetone are added to the sample and the flask is connected to a reflux condenser. The solution is refluxed for 15 ± 1 min at 120 °C. Afterwards, the Erlenmeyer flask with condenser was removed from the heating plate and the solution was allowed to cool down to room temperature. The condenser is rinsed with 20 mL acetone and then the flask is removed. Afterwards, the solution is diluted with acetone to about 125 mL. While stirring, 50 mL glacial acetic acid is added and the solution is then titrated with $c(AgNO_3)$ = 0.0025 mol/L until after the equivalence point using the dAg ring electrode.

### Parameters

| | |
|---|---|
| Mode | DET U |
| Pause | 30 s |
| Start volume | 0 mL |
| Stirring rate | 6 |
| Min. increment | 10 µL |
| Max. increment | Off |
| Signal drift | 20 mV/min |
| Max. waiting time | 38 s |
| Min. waiting time | 5 s |
| Dosing rate | Maximum |
| Stop volume | 20 mL |
| Stop EP | 1 |
| Volume after EP | 8.0 mL |
| EP criterion | 30 mV |
| EP recognition | Greatest |

### Results

| Hydrolyzable chloride content / (ppm) (n = 5) | s(rel) / % |
|---|---|
| 225 | 0.81 |

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