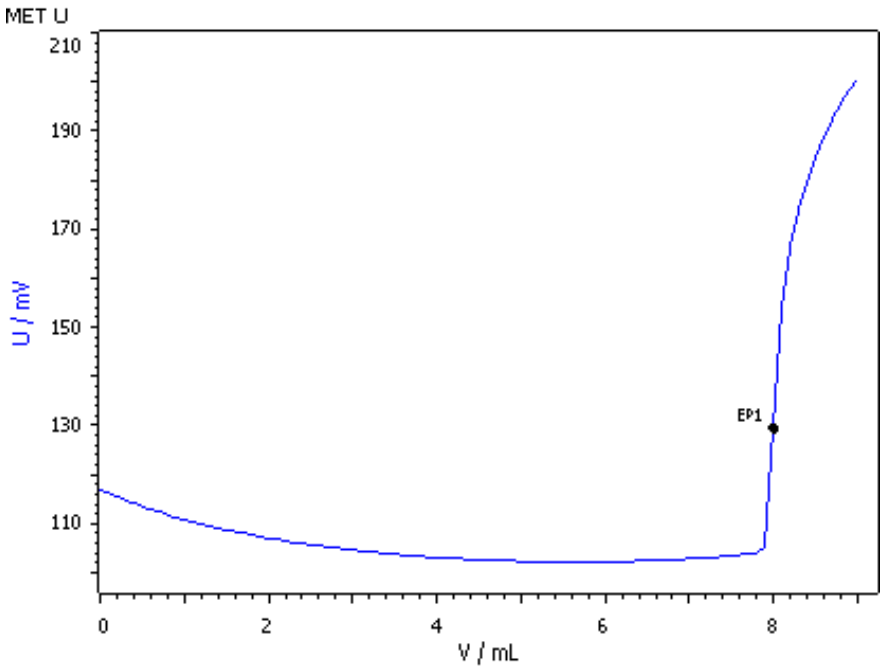
Titration Application Note T–123

# Automated determination of zirconium in aqueous solution using the Cu ISE




Zirconium in aqueous solutions can be analyzed by back-titration in slightly acidic solution. The ion-selective copper electrode is used as indicator electrode.

Metrohm

# Method description

### Sample

Aqueous solution of zirconium

### Sample preparation

No sample preparation is required.

### Configuration

| | |
|---|---|
| 907 Titrando | 2.907.0010 |
| 815 Robotic USB Sample Processor XL | 2.815.0020 |
| 786 Swing head | 2.786.0040 |
| Swing arm | 6.1462.070 |
| Titration head | 6.1458.010 |
| Sample rack 28 × 200 mL | 6.2041.830 |
| 800 Dosino, 5 × | 2.800.0010 |
| 802 Stirrer | 2.802.0020 |
| 10 mL Dosing unit, 3 × | 6.3032.210 |
| 20 mL Dosing unit | 6.3032.220 |
| 50 mL Dosing unit | 6.3032.250 |
| Disposable PP sample beakers, 200 mL, 1000 pieces | 6.1459.310 |
| Cu ISE | 6.0502.140 |
| LL ISE Reference | 6.0750.100 |

### Solutions

| | |
|---|---|
| Titrant | $c(CuSO_4)$ = 0.1 mol/L<br>If possible this solution should be bought from a supplier. |
| EDTA solution | c(EDTA) = 0.1 mol/L<br>If possible this solution should be bought from a supplier. |
| Buffer solution<br>pH = 4.7 | 123 g sodium acetate and 86 mL conc. acetic acid are dissolved in deion. $H_2O$ and made up to 1 L. |

### Analysis

Pipet a sample volume containing no more than 70 mg $Zr^{4+}$ into the titration vessel and dilute with 50 mL deion. $H_2O$. Add 10.00 mL c(EDTA) = 0.1 mol/L and 5 mL buffer solution pH = 4.7. If necessary adjust the pH to 4.7 with ammonia. After stirring for 30 s back-titrate the EDTA excess with $c(CuSO_4)$ = 0.1 mol/L until after the first equivalence point.

### Parameters

| | |
|---|---|
| Mode | MET U |
| Pause | 30 s |
| Stirring rate | 8 |
| Signal drift | 50 mV/min |
| Min. waiting time | 5 s |
| Max. waiting time | 26 s |
| Volume increment | 0.1 mL |
| Stop EP | 1 |
| Volume after EP | 1 mL |
| EP criterion | 30 mV |
| EP recognition | greatest |

### Results

Mean results (n = 5)

| | |
|---|---|
| Zr content / (g/L) | 2.993 |
| s(rel) / % | 0.60 |