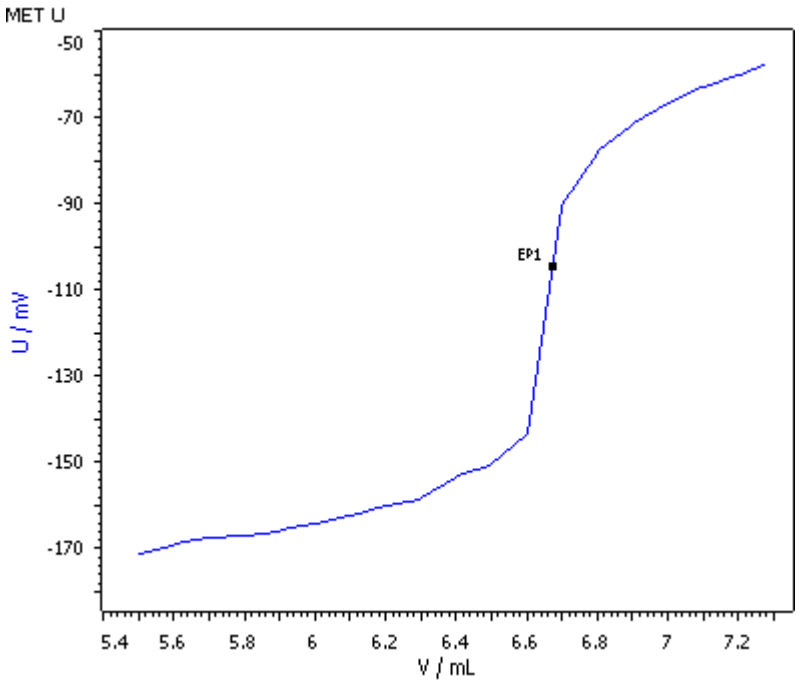
Titration Application Note T–118

Automated determination of zinc and magnesium in mixtures using the Cu ISE



Mixtures of zinc and magnesium ions can be analyzed by back-titration at different pH values. The ion-selective copper electrode is used as indicator electrode. First the zinc is determined in acidic solution, then the magnesium in alkaline solution.

Metrohm

Method description

Sample

Aqueous mixture of zinc and magnesium

Sample preparation

No sample preparation 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, 4 ×	6.3032.210
50 mL Dosing unit	6.3032.250
200 mL PP beakers (1000 ×)	6.1459.310
Cu ISE	6.0502.140
LL ISE Reference	6.0750.100
Solitrode	6.0220.100

Solutions

Titrant	$c(CuSO_4)$ = 0.1 mol/L If possible this solution should be bought from a supplier.
DCTA solution	c(DCTA) = 0.1 mol/L Dissolve 36.463 g DCTA in 400 mL c(NaOH) = 0.5 mol/L and make up to 1 L with deion. H_2O.
Acetate buffer pH = 4.7	Dissolve 123 g sodium acetate and 86 mL conc. acetic acid in deion. H_2O and make up to 1 L.
Ammonium buffer pH = 10	54 g NH_4Cl is weighed into a 1 L volumetric flask and dissolved in deion. H_2O. 350 mL $w(NH_3)$ = 25% is added and the mixture made up to 1 L with deion. H_2O.

Analysis

Pipet a sample volume containing not more than 50 mg Zn^{2+} or 20 mg Mg^{2+} into the titration vessel and dilute with 50 mL deion. H_2O. First the zinc is determined. Add 10.00 mL c(DCTA) = 0.1 mol/L and 10 mL acetate buffer and allow to react for 2 min under stirring. If necessary adjust the pH to 4.7. Afterwards the DCTA excess is back-titrated with $c(CuSO_4)$ = 0.1 mol/L until after the first equivalence point.

Now the magnesium can be determined. Add another 10.00 mL c(DCTA) = 0.1 mol/L and 10 mL ammonium buffer to the titrated sample solution and back-titrate the DCTA excess with $c(CuSO_4)$ = 0.1 mol/L until after the first equivalence point.

Parameters

Mode	MET U
Stirring rate	8
Signal drift	30 mV/min (Zn) 50 mV/min (Mg)
Min. waiting time	12 s (Zn) 5 s (Mg)
Max. waiting time	32 s (Zn) 26 s (Mg)
Volume increment	0.1 mL
Stop EP	1
Volume after EP	1 mL
EP criterion	30 mV (Zn) 15 mV (Mg)
EP recognition	greatest

Results

Mean results (n = 5)

Zn content / (g/L)	1.569
s(rel) / %	0.13
Mg content / (g/L)	0.549
s(rel) / %	1.05