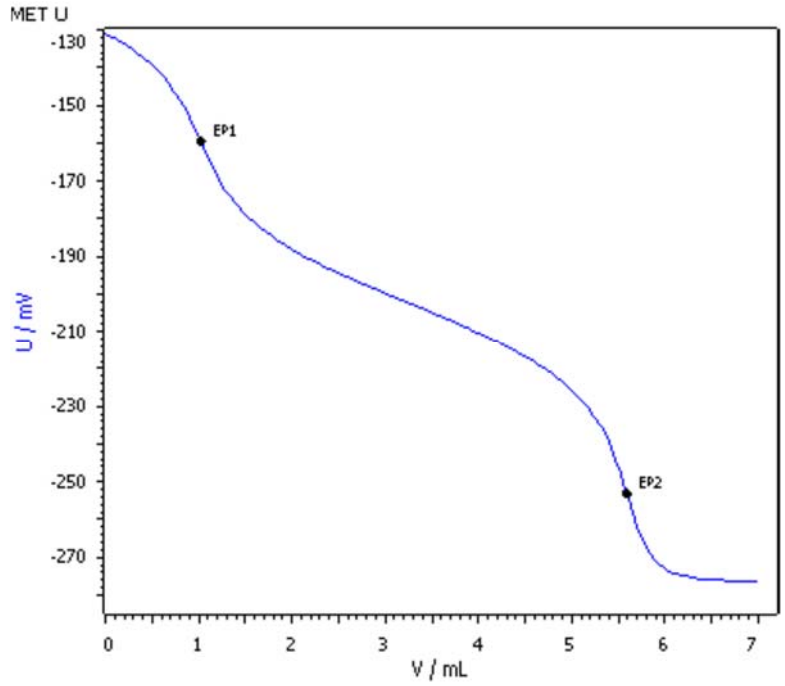
Titration Application Note T–116

Automated determination of sulfate in aqueous solution using the combined Ca ISE



This application note describes the automated determination of sulfate using the combined Ca ISE as sensor. The sulfate is precipitated with an excess of barium, which is then back-titrated with a standard EGTA solution.

Metrohm

Method description

Sample

Aqueous sulfate solution

Sample preparation

No sample preparation is required

Configuration

907 Titrando	2.907.0020
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, 6 ×	2.800.0010
802 Stirrer	2.802.0020
5 mL Dosing unit	6.3032.150
10 mL Dosing unit, 2 ×	6.3032.210
20 mL Dosing unit, 2 ×	6.3032.220
50 mL Dosing unit	6.3032.250
Disposable plastic beaker	6.1459.310
Combined polymer Ca ISE	6.0510.100
Solitrode	6.2020.100
200 mL PP beaker (1000×)	6.1459.310

Solutions

Titrant	c(EGTA) = 0.05 mol/L 19.4 g EGTA sodium salt is suspended in 200 mL deion. H_2O. While stirring c(NaOH) = 10 mol/L is added until all EGTA is dissolved. After cooling down the solution is made up to 1 L with deion. H_2O.
Barium chloride solution	c($BaCl_2$) = 0.05 mol/L 12.34 g $BaCl_2 \cdot 2\ H_2O$ is dissolved in c(HCl) = 0.01 mol/L and made up to 1 L with deion. H_2O.
Buffer solution	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.
Nitric acid solution	c(HNO_3) = 1 mol/L
Calcium standard	6.2303.070

Analysis

First a blank needs to be determined. 50 mL deion. H_2O, 0.5 mL calcium standard and 7.50 mL c($BaCl_2$) = 0.05 mol/L are dosed into a titration vessel. After a reaction time of 3 min, 5 mL buffer solution is added. The solution is then back-titrated with c(EGTA) = 0.05 mol/L until after the second equivalence point. The difference between the two equivalence points is saved as blank value.

A sample solution containing less than 20 mg sulfate is diluted with 50 mL deion. H_2O. The pH is measured and if necessary set to pH 3 … 4 with a SET titration using c(HNO_3) = 1 mol/L. 0.5 mL calcium standard and 7.50 mL c($BaCl_2$) = 0.05 mol/L are added and after a reaction time of 3 min, 5 mL buffer solution is added. The solution is then back-titrated with c(EGTA) = 0.05 mol/L until after the second equivalence point.

Parameters

Mode	MET U
Stirring rate	8
Pause	30 s
Signal drift	50 mV/min
Min. waiting time	5 s
Max. waiting time	26 s
Volume increment	0.1 mL
EP criterion	15 mV
EP recognition	all

Results

Mean result for the aqueous sulfate solution (n = 5)

Sulfate Content / (g/L)	s(rel) / %
4.721	0.58