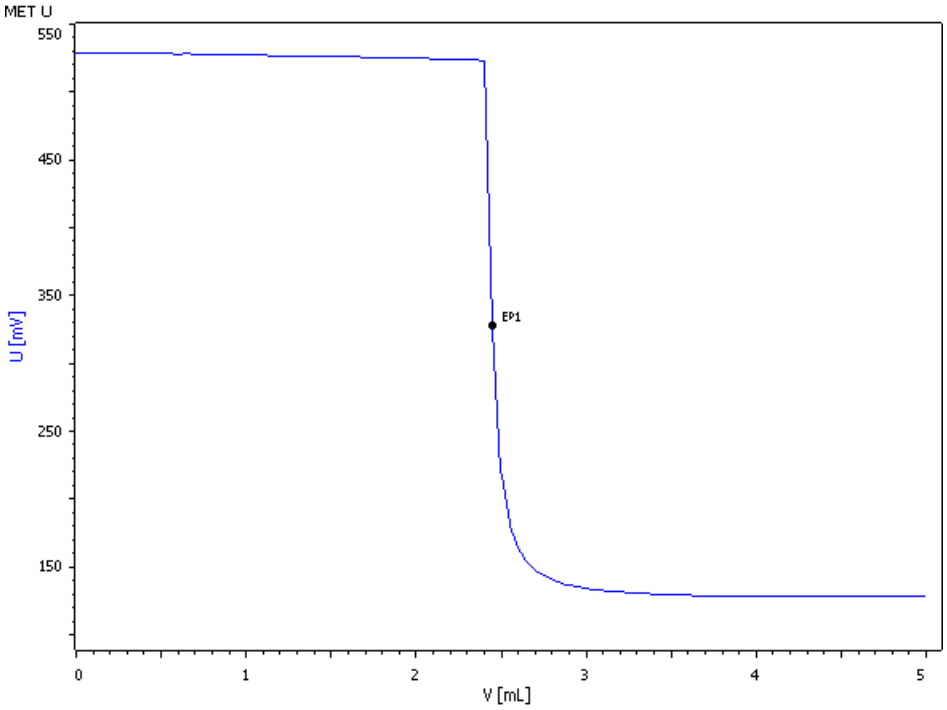
Titration Application Note T–140

Automated photometric determination of lead using the Optrode



Lead is determined at pH 4 to 5 by back-titration with zinc sulfate. To visualize the equivalence point, xylenol orange is used as indicator, and the equivalence point is detected with the Optrode at a wavelength of 574 nm.

Metrohm

Method description

Sample

Aqueous solution of lead (0.05 mol/L)

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 x 200 mL	6.2041.830
800 Dosino, 5x	2.800.0010
802 Stirrer	2.802.0020
Stirring propeller	6.1909.020
5 mL Dosing unit	6.3032.150
10 mL Dosing unit, 2x	6.3032.210
20 mL Dosing unit	6.3032.220
50 mL Dosing unit	6.3032.250
Disposable PP sample beaker, 200 mL, 1000 pieces	6.1459.310

Solutions

Titrant	c($ZnSO_4$) = 0.1 mol/L 28.9 g $ZnSO_4 \cdot 7\ H_2O$ (99.5%) is weighed into a 1000 mL volumetric flask and dissolved in approx. 500 mL deion. water. After addition of 0.5 mL w(H_2SO_4) = 25% the solution is filled up to the mark with deion. water.
Na_2EDTA solution	c(Na_2EDTA) = 0.1 mol/L If possible this solution should be bought from a supplier.
Buffer solution pH = 4.9	123 g sodium acetate and 50 mL glacial acetic acid are given into a 1 L volumetric flask, dissolved in approx. 800 mL deion. water and filled up to the mark with deion. water.
Xylenol orange	100 mg Xylenol orange is dissolved in 100 mL deion. water.
Buffer solution pH 10	54 g NH_4Cl is weighed into a 1 L volumetric flask and dissolved in deionized water. 350 mL w(NH_3) = 25% is added and the mixture made up to 1 L with deionized water.

Analysis

5 – 7 mL lead standard solution is pipetted into a 200 mL plastic beaker and 90 mL deion. water is added. After addition of 10 mL acetate buffer, 5 mL c(Na_2EDTA) = 0.1 mol/L and 0.5 mL xylenol orange indicator solution the solution is titrated with c($ZnSO_4$) = 0.1 mol/L until after the equivalence point.

Parameters

Mode	MET U
Stirring rate	8
Pause	60 s
Signal drift	20 mV/min
Min. waiting time	0 s
Max. waiting time	26 s
Volume increment	0.05 mL
EP criterion	15 mV
EP recognition	greatest

Results

Mean results (n = 6)

Pb content / (g/L)	10.557
s(rel) / %	1.33