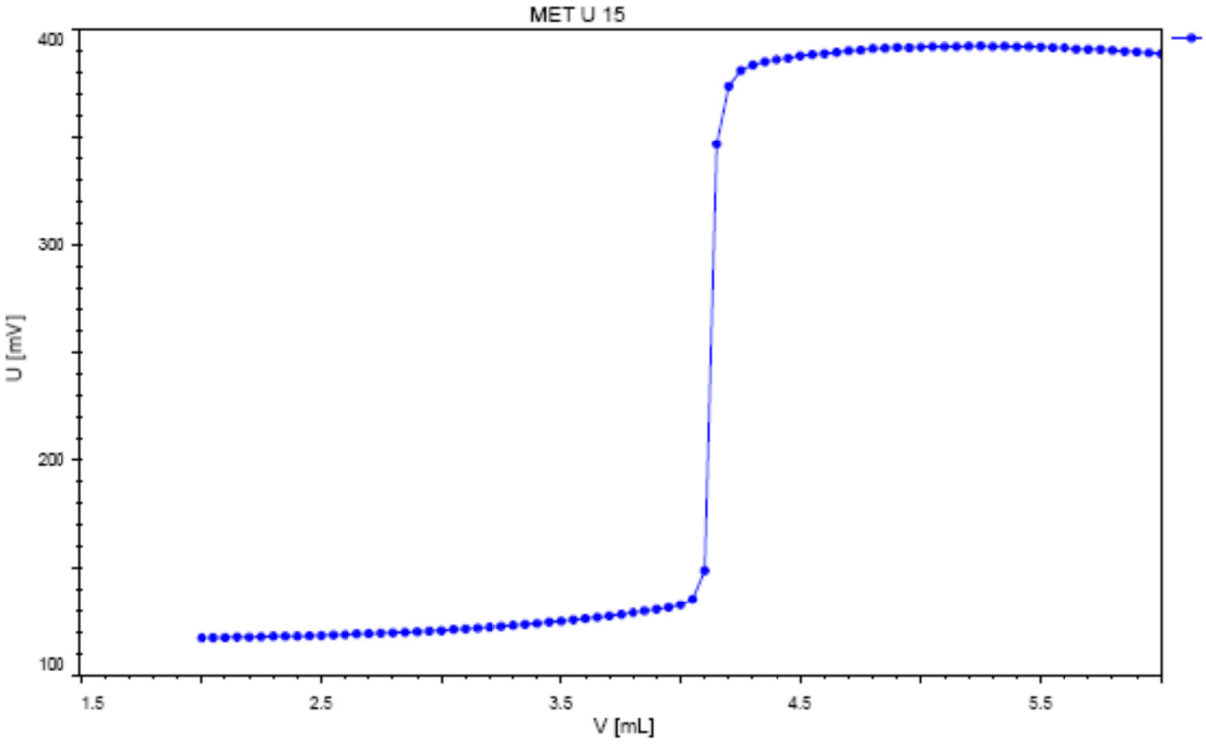
Titration Application Note T–81

Determination of magnesium in cement by photometric titration of the solubilized product



This Application Note describes the photometric determination of magnesium in cement by using the Optrode (610 nm). After digestion of a cement aliquot, magnesium is titrated with EDTA.

Metrohm

Method description

Sample

Portland cement CEM 42.5

Sample preparation

2.5075 g cement is mixed with 1.5 g NH_4Cl and treated with 8 mL conc. HCl and 0.5 mL HNO_3 (1:1). The mixture is boiled over a flame for 40 min with occasional agitation.

After uptake of the residue in 50 mL hot water, the solution is filtered through a black band filter S&S 589 into a 500 mL flask and filled up with dist. water to 500 mL.

Configuration

907 Titrando	1 x 2.907.0010
800 Dosino	5 x 2.800.0010
Dosing unit 2 mL	2 x 6.3032.120
Dosing unit 5 mL	1 x 6.3032.150
Dosing unit 10 mL	2 x 6.3032.210
Dosing unit 50 mL	1 x 6.3032.250
802 Rod Stirrer	1 x 2.802.0020
815 Robotic USB SP	1 x 2.815.0110
Sample beaker 250 mL	1 x 6.1432.320
Sample rack 28 x 250 mL	1 x 6.2041.820
Optrode (at 610 nm)	1 x 6.1115.000
Unitrode with Pt1000	1 × 6.0258.600
Electrode cable	1 × 6.2104.600

Solutions

Titrant $c(Na_2EDTA)$ = 0.1 mol/L	Dissolve 37.224 g $Na_2EDTA \cdot 2H_2O$ in dist. water, add 10 mL c(NaOH) = 1 mol/L and make up to 1 L with dist. water.
Ammonia $c(NH_3)$ = 25%	CAS 1336-21-6
Indicator methylthymol blue sodium salt	w(methylthymol blue sodium salt) = 0.01% CAS 1945-77-3

Analysis

10 mL of sample solution are diluted to 100 mL with distilled water and the pH adjusted to 10.0 with ammonia.

After addition of 1 mL methylthymol blue indicator, the solution is titrated with EDTA at **λ** = 610 nm. The color changes from blue to brownish pink.

Parameters

Titration mode	MET U
Measurement drift	50 mV/min
Min. waiting time	0 s
Max. waiting time	26 s
Volume increment	0.05 mL
Breakpoint evaluation	
EP criterion	0.3 mV
Slope	0.9
Smoothing factor	5
Window	off
Stirring speed	8

Calculations

$$\% MgO = (BP1 - C01) \times C02 \times C03 \times C04/C00 \times 100$$

BP1 = titrant consumption in mL
C00 = sample weight in mg
C01 = titrant consumption of Ca determination in mL
C02 = concentration of the EDTA titrant in mol/L (0.1)
C03 = titer EDTA (dimensionless unit)
C04 = molecular weight of MgO in g/mol (40.32)

Results

MgO in %	Mg in mg
1.08 ± 0.009 (n = 3)	0.33 ± 0.002 (n = 3)

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