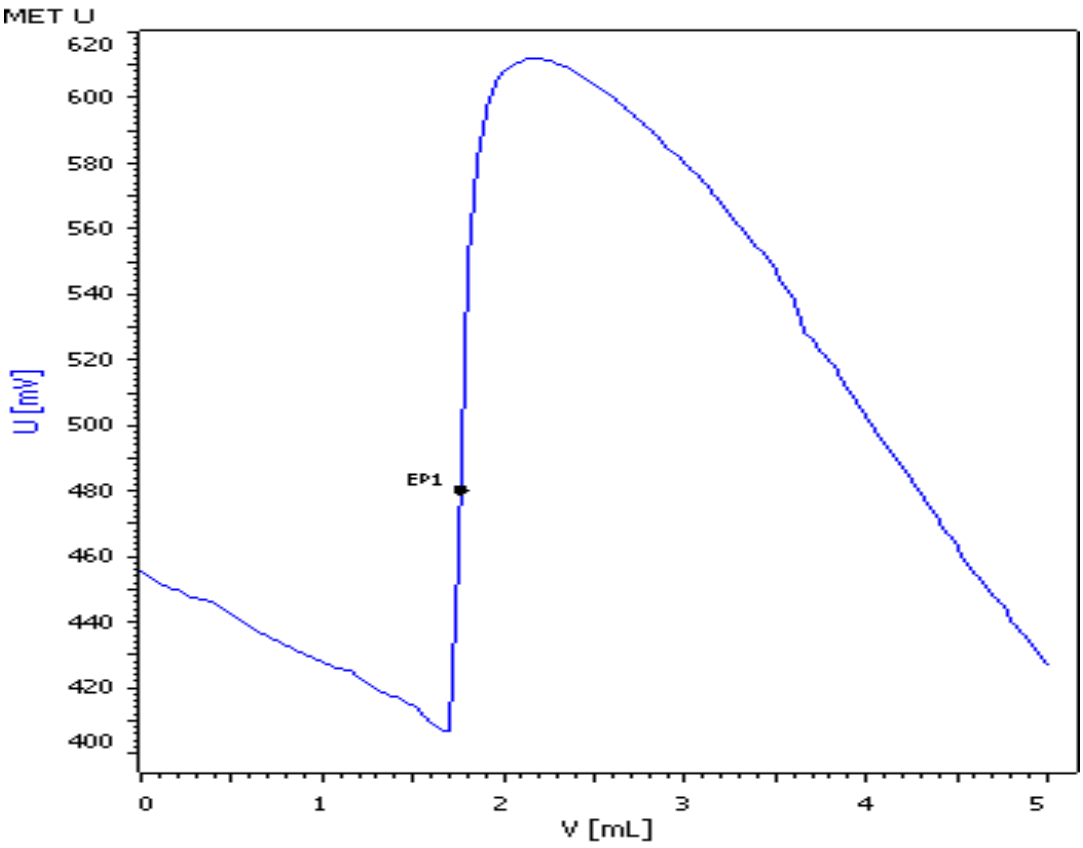
Titration Application Note T–91

Fully automatic determination of the total content of Ba, Ca, Mg, Pb, and Zn in unused lubricating oils



This Application Note focuses on the determination of the total content of barium, calcium, magnesium, lead, and zinc in unused lubricating oils by using the Optrode (610 nm). The metals are complexed with an excess of EDTA. Unreacted EDTA is back-titrated with magnesium chloride solution to the Eriochrome Black T endpoint.

Metrohm

Method description

Sample

New lubricating oil

Sample preparation

3.02455 g sample was dissolved in 100 mL of toluene.

Configuration

907 Titrando	1 x 2.907.0010
800 Dosino	6 x 2.800.0010
Dosing unit 10 mL	2 x 6.3032.210
Dosing unit 50 mL	1 x 6.3032.250
Dosing unit 5 mL	2 x 6.3032.150
Dosing unit 2 mL	1 x 6.3032.120
802 Rod Stirrer	1 x 2.802.0020
815 Robotic USB SP	1 x 2.815.0110
Sample beaker 250 mL	1 × 6.1432.320
Sample Rack 28 x 250 mL	1 x 6.2041.820
Optrode (at 610 nm)	1 x 6.1115.000

Solutions

Titrant $c(MgCl_2)$ = 0.025 mol/L	5.083 g $MgCl_2 \cdot 6H_2O$ is dissolved in dist. H_2O and then made up to 1 liter.
Indicator solution Eriochrome black T	0.1 g Eriochrome Black T and approx. 0.2 g ascorbic acid (vitamin C) are dissolved in dist. H_2O and then made up to 100 mL.
Buffer solution pH = 10	54 g NH_4Cl is dissolved in dist. H_2O, treated with 350 mL $w(NH_3)$ = 25% and made up to 1 liter with dist. H_2O.
Auxiliary solution $c(Na_2EDTA)$ = 0.025 mol/	9.325 g $Na_2EDTA \cdot 2H_2O$ is dissolved in dist. H_2O, treated with 10 mL c(NaOH) = 1 mol/L and made up to 1 liter with dist. H_2O.
Solvent	isopropanol (CAS 67-63-0)
Solvent	toluene (CAS 108-88-3)

Analysis

10 mL sample solution were pipetted into a sample beaker, treated with 10 mL toluene, 90 mL isopropyl alcohol, 5 mL buffer solution (pH 10), and 5 mL Na_2EDTA. If oil droplets are formed then further toluene is added until they have dissolved.
The Optrode and buret tip are immersed in the solution, 0.5 mL Eriochrome Black T is added and the excess EDTA is back-titrated with $c(MgCl_2)$ = 0.025 mol/L.

Parameters

Titration mode	MET U
Measurement drift	20 mV/min
Min. waiting time	0 s
Max. waiting time	38s
Volume increment	0.05 mL
EP criterion	30 mV
EP recognition	greatest
Stirring speed	8

Calculations

$$\text{mmol/kg } Me^{2+} = (C01 - EP1) \times C02/(C00 \times 1000)$$

EP1 = titrant consumption in mL
C01 = volume of the EDTA solution in mL
C02 = concentration of the titrant in mol/L (0.025)
C00 = sample size in g

Results

Metal content in mmol/kg	
1.101 ± 0.014 (n = 6)	