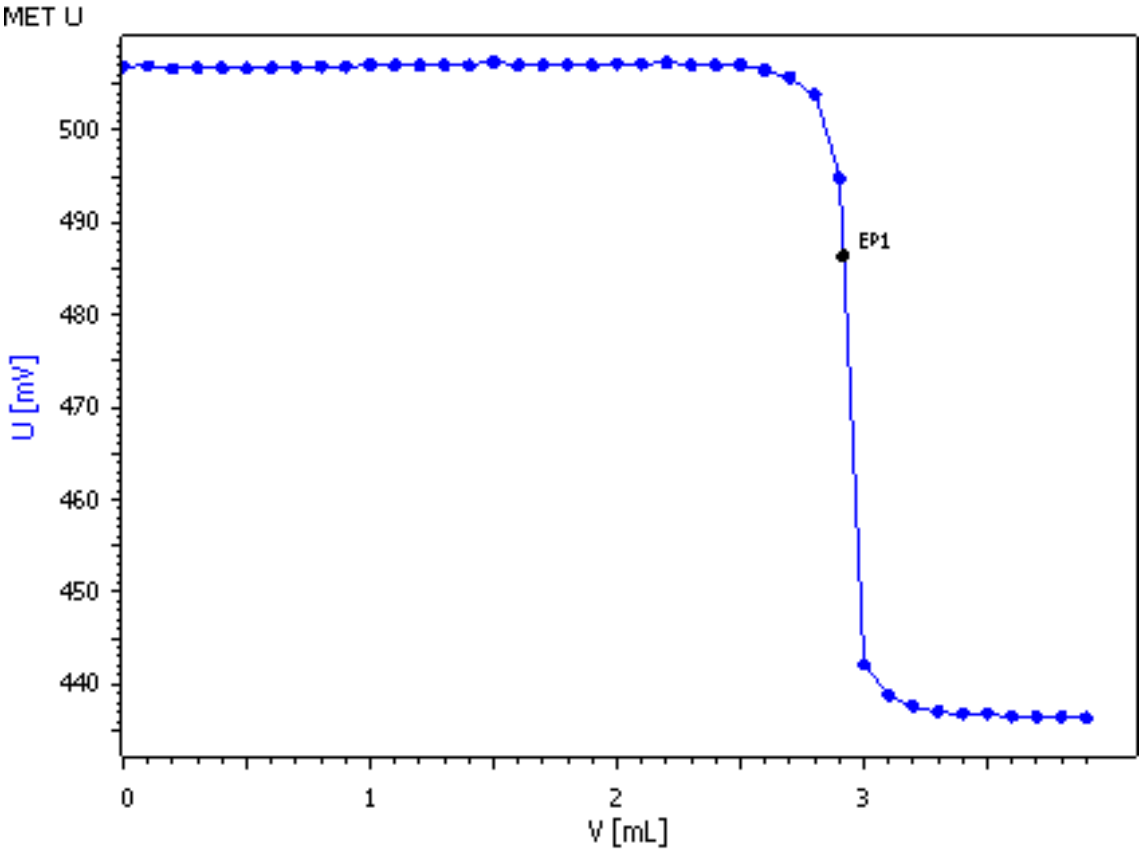
Titration Application Note T–84

Fully automatic determination of the total, calcium, and magnesium hardness of water samples by photometric titration according to DIN 38406-3



This Application Note describes the photometric determination of the total, calcium, and magnesium hardness of water samples by using the Optrode (610 nm). The total hardness is determined using an EDTA titrant and Eriochrome Black T as indicator. For the calcium hardness, EDTA titrant and calconcarboxylic acid indicator are used. Subtraction of calcium hardness from total hardness provides the magnesium hardness.

Metrohm

Method description

Sample

Tap water (Herisau)

Sample preparation

No sample preparation required

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 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	1 x 6.1115.000

Solutions and reagents

Titrant c (EDTA) = 0.1 mol/L	Dissolve 37.224 g $Na_2EDTA \cdot 2H_2O$ in dist. Water in a 1 L volumetric flask, add 10 mL c(NaOH) = 1 mol/L and make up to 1 L with dist. water.
Buffer solution pH 10	Dissolve 54 g NH_4Cl and 300 mL w (NH_3) = 25% in dist. water and make up to 1 L.
Eriochrome Black T CAS 1787-61-7	Dilute 100 mg Eriochrome Black T in 100 mL dist. water. Add 100 mg vitamin C to stabilize.
Calconcarboxylic acid indicator CAS 3737-95-9	Dissolve 50 mg calconcarboxylic acid in 100 mL c(NaOH) = 0.1 mol/L
Sodium hydroxide,	c(NaOH) = 2 mol/L
Magnesium complexonate	MgEDTA CAS 14402-88-1

Analysis

a) Determination of the total hardness (MET U TH).
Add ca. 0.1 g MgEDTA and 10 mL buffer solution pH 10 to 100 mL sample. After addition of 0.25 mL Eriochrome Black T indicator, titrate with c(EDTA) = 0.1 mol/L.

b) Determination of the calcium hardness (MET U CaH)
Add 1.5 mL calconcarboxylic acid indicator and 4 mL c(NaOH) = 2 mol/L to 100 mL sample. Titrate immediately with c(EDTA) = 0.1 mol/L.

c) Determination of the magnesium hardness
Subtraction of the calcium hardness from the total hardness.

Parameters (MET U TH and MET U CaH)

Titration mode	MET U
Measurement drift	50 mV/min
Min. waiting time	0 s
Max. waiting time	26 s
Volume increment	0.1 mL
EP criterion	30 mV
EP recognition	greatest
Stirring speed	8

Calculations

$$\text{Total hardness} = \frac{V_{EP1} \times c(EDTA) \times f \times 1000}{V_S}$$

$$\text{Ca hardness} = \frac{V_{EP1} \times c(EDTA) \times f \times 1000}{V_s}$$

Total hardness: Total hardness in mmol/L

Ca hardness: Calcium hardness in mmol/L

V_{EP1}: Titrant consumption until the first equivalence point in mL

c(EDTA): Concentration of titrant in mol/L; here c(EDTA) = 0.1 mol/L

f: Correction factor («titer») without unit

1000: Conversion factor to obtain result in mmol/L

V_S: Sample size in mL

Results

	EP1 TH / mL	TH in mmol/L	EP1 CaH / mL*	CaH in mmol/L
1	2.9107	2.911	1.3599	2.667
2	2.9161	2.916	1.3674	2.682
3	2.9273	2.927	1.3664	2.680
Aver-age		2.918 ± 0.0082		2.676 ± 0.0081

*This measurements where done with a sample size of 50 mL.

Metrohm