Determination of Sulfate in drinking, surface and waste water

Description

The determination of the Sulfate is possible using the Ca-ISE and EGTA as titrant. An excess of BaCl2 is added to the water sample to precipitate the sulfate. The not reacted Ba-ions are back titrated with EGTA titrant. The titration curve shows two equivalence points. The first one corresponds to the Ca and the difference between the second and first EQ corresponds to the Ba. Mg is not determined with this titrant.

Instruments

Titrator	TL 7000 or higher
Electrode	Ca 1100 PLH
Cable	L 1 A
Reference electrode	B 2920+
cable	L 1 N
Stirrer	Magnetic stirrer TM 235 or similar
Lab accessory	Glass beaker 100 ml
	Magnetic stirrer bar 30 mm

Reagents

1	EGTA 0.05 mol/l
2	$BaCl_2$ 0.05 mol/l
3	Buffer solution pH 10
4	Distilled Water
5	HCl 2 mol/l, HCL 0.01 mol/l, NaOH 10 mol/l
6	Ca standard solution 0.1 mol/l
7	Electrolyte solution KCL 3 mol/l (L 300)
All reagents should be of analytical grade or better.	

Reagents

Titrant EGTA 0.05 mol/l
19.4 g ethylene glycol-bis-(2-aminoethyl)-tetra acetic acid (98%) is weighed into a beaker and about 200 ml dist. H_2O are added to mix the EGTA under stirring. Then NaOH 10 mol/l is added until the EGTA is dissolved completely. After cooling down, the solution is transferred quantitatively to a 1000 mL volumetric flask with dist. H_2O, filled up to the mark and mixed.

The titer determination of the EGTA solution is carried out as described in the application note "Titer determination of EGTA".

$BaCl_2$ = 0.05 mol/l
12.34 g $BaCl_2$ x 2 H2O (99%) is weighed out into a 1000 mL volumetric flask, dissolved in HCl = 0.01 mol/l, filled up to the mark with it and mixed.

Buffer solution pH 10
9 g NH_4Cl is weighed into a 1000 mL volumetric flask and dissolved in about 800 mL dist. H_2O. After the addition of 60 mL NH_4OH = 25% the solution is filled up to the mark with dist. H_2O and mixed.

HCl = approx. 2 mol/l
Concentrated HCl is diluted 1:5 with dist. H_2O.

Cleaning of the electrode

The electrodes are cleaned with distilled water. The Ca 1100PLH is stored clean and dry, for the storage of the reference electrode use KCl 3 mol/l electrolyte solution (L 300).

Titration procedure

Blank titration

A blank titration has to be carried out before. To 40 mL dist. H_2O are pipetted 5.00 ml of the $BaCl_2$ 0.05 mol/l solution into the glass beaker. After the addition of 0.5 mL of the Ca standard solution and 5 ml buffer pH 10 the solution is stirred for 3 min. The solution is then back titrated with the EGTA titrant to two EQ´s. The difference between the EQ2-EQ1 is stored in the global memory M01.

Sample preparation

The water sample is pipetted into the glass beaker. The sample volume depends on the expected SO_4 concentration.

The pH is adjusted to pH 4 by adding carefully the HCl 2 mol/l. Then 5 ml of the $BaCl_2$ 0.05 mol/l solution is added to the sample. After three minutes of reaction time under stirring 5 ml of the buffer solution pH 10 are added. After additional three minutes of reaction time the sample is back titrated with the EGTA titrant to two EQ´s.

Titration parameter

Blank value

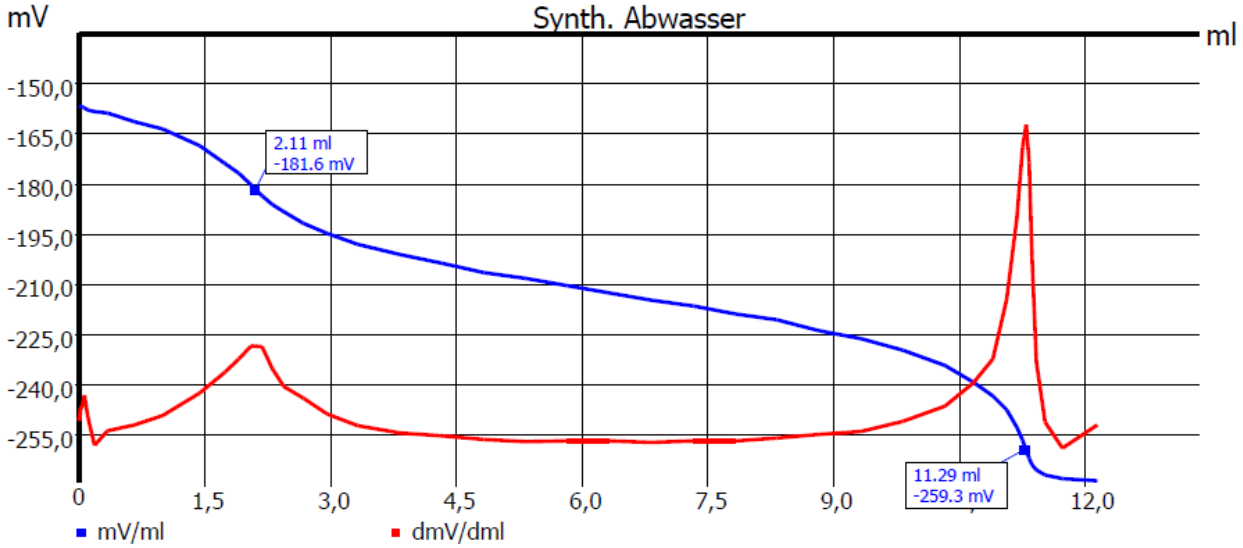
Default method	---		
Method type	Automatic titration		
Modus	Dynamic		
Measured value	mV		
Measuring speed / drift	User defined	Minimum holding time	5 s
		Maximum holding time	12 s
		Measuring time	4 s
		Drift	3 mV/min
Initial waiting time	10 s		
Dynamic	flat	Max step size	0.5 ml
		Slope max ml	10
		Min. step size	0.05 ml
		Slope min. ml	50
Damping	none	Titration direction	decrease
Pretitration	off	Delay time	0 s
End value	off		
EQ	On (2)	Slope value	120
Max. titration volume	20 ml		
Dosing speed	100%	Filling speed	30 s

Calculation:

$$Result\ EQ1 = EQ1$$

$$Result\ \text{blank value}\ [ml] = EQ2 - EQ1$$

Sample titration



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Pretitration	off	Delay time	0 s
End value	off		
EQ	On (2)	Slope value	120
Max. titration volume	20 ml		
Dosing speed	100%	Filling speed	30 s

Calculation:

$$Result\ Ca^{2+}\ [mg/l] = \frac{EQ1 * T * M_{Ca} * 1000}{V}$$

$$Result\ SO4^{2-}\ [mg/l] = \frac{((M01 - (EQ2 - EQ1)) * T * M_{SO4} * 1000}{V}$$

EQ1		Consumption of titrant at first Equivalence point
EQ2		Consumption of titrant at second Equivalence point
T	WA	Actual concentration of the titrant (e.g.0.0511 mol/l)
M_{Ca}	40,08	Molecular mass of Ca
M_{SO4}	96,06	Molecular mass of SO_4
V	man	sample volume [ml]

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