

Determination of Sulfate (with Thorin indicator)

Description

This application describes the determination of sulfate by titration with barium perchlorate and Thorin indicator. The titration is done in an alcoholic solution. The sample solution should contain at least 70% isopropanol before the titration.

If gas bubbles stick at the electrode during the titration, a silicone oil-free defoamer or a non-foaming wetting agent can be added.

The content of Sulfate is calculated in mg/L.

Instruments

Titration	TL 7000, TL 7750 or TL 7800
Exchangeable head	WA 10 or WA 20
Electrode	Optiline 6
Stirrer	TM 235
Lab accessory	Glass beaker 100 ml
	Magnetic stirrer bar 30 mm

Reagents

1	Barium perchlorate $\text{Ba}(\text{ClO}_4)_2$ 0,005 mol/L in Isopropyl alcohol / Water 80/20
2	Isopropyl alcohol
3	Perchloric acid HClO_4 , diluted
4	Thorin
5	Defoamer Surfynol DF 110 (or similar silicone-free defoamer)
6	Distilled Water
All reagents should be of analytical grade or better.	

Reagents

Titrant Bariumperchlorate $\text{Ba}(\text{ClO}_4)_2$ 0,005 mol/L

$\text{Ba}(\text{ClO}_4)_2$ 0,005 mol/L in Isopropyl alcohol / Water 80/20 is available as ready to use solution.
1.7 g $\text{Ba}(\text{ClO}_4)_2$ are dissolved in 200 ml Water and made up to 1L with Isopropyl alcohol.

Perchloric acid, diluted (~1 mol/L)

14,3 g Perchloric acid 70% are dissolved in about 80ml Water and made up 100mL.

Thorin solution 2 g/L

0.2 g of Thorin are dissolved in a little water and made up to 100mL.

Defoamer solution

10 g of Surfynol DF 110 are dissolved in Isopropyl alcohol and made up to 100mL.

Cleaning of the electrode

The electrode is cleaned with distilled water. Optiline 6 is stored clean and dry.

Titration procedure

Sample preparation

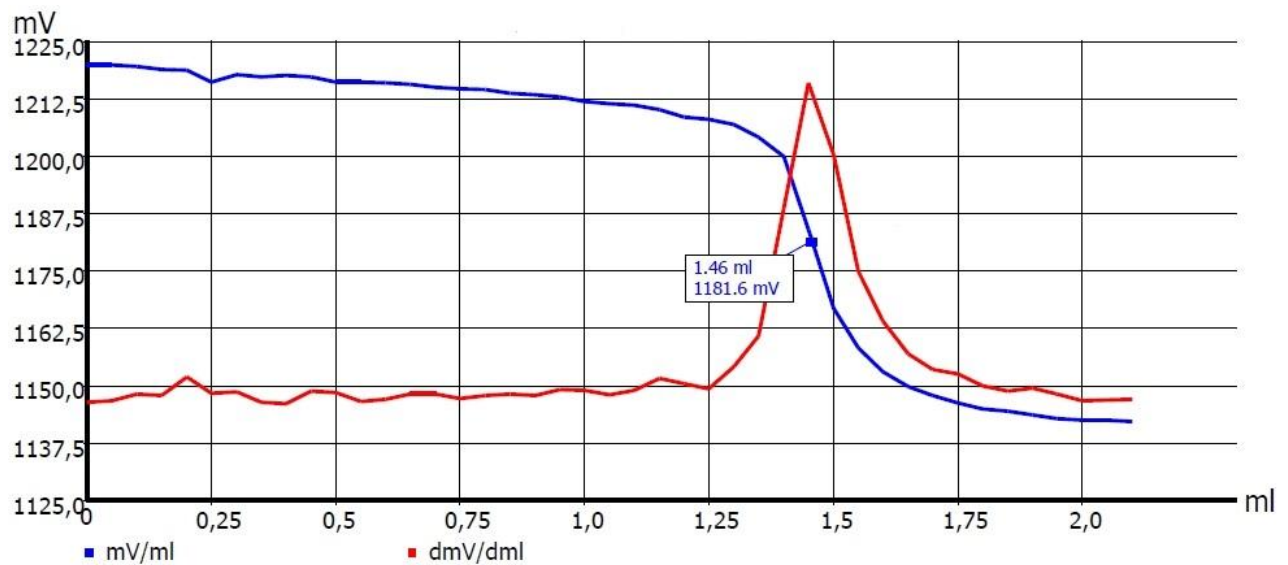
The sample is pipetted into a beaker and made up to approx. 10 - 15 mL with dist. Water. Then it is made up to 60-70mL with Isopropyl alcohol and 0.5 mL diluted HClO_4 are added. For alkaline samples, more HClO_4 is needed. 5 drops of Thorine solution 2 g/L are added and titrated with $\text{Ba}(\text{ClO}_4)_2$ 0.005 mol/L to an equivalence point. If needed, one drop of defoamer solution is added before the titration.

The required sample amount can be estimated using this rule of thumb:

$$V(\text{mL}) = \frac{150000 * \text{Titer} \left[\frac{\text{mol}}{\text{L}} \right]}{\text{expected content} [\text{mg/L}]}$$

Titration parameter

Sample titration



Default method	---		
Method type	Automatic titration		
Modus	Linear		
Measured value	mV(E)		
Measuring speed		Fixed delay time	10 s
Optiline 6 settings		Wavelength	520 nm
		Intensity	average
		Smoothing	strong
Initial waiting time	0 s		
Linear step size	0.05 mL		
Pretitration	off	Titration direction	decrease
End value	off	Delay time	0 s
EQ	On (1)		
Max. titration volume	10 ml	Slope value	250
Dosing speed	100%	Filling speed	30 s

The mV(E) value should be <2000 mV at the start of the titration; a value in the range of around 1000 mV is best. This can be adjusted via the intensity in the Optiline settings.

Calculation:

$$\text{Sulfate [mg/L]} = \frac{(EQ1 - B) * T * M * F1}{V * F2}$$

EQ 1		Consumption of the titrant on the first EQ [mL]
B	0	Blank
T	WA	Actual concentration of the titrant [mol/L]
M	96,06	Molecular weight of Sulfate [g/mol]
V		Sample amount [mL]
F1	1000	Conversion factor 1
F2	1	Conversion factor 2