

Determination of Silver in alloy

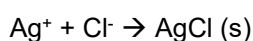
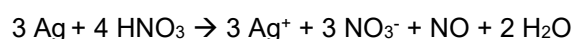
Description

The determination of silver in alloys according to ISO 13756 is done by titration with a NaCl or KCl solution. To do this, the sample is dissolved in nitric acid.

Palladium interferes with the determination and must be removed by precipitation with Dimethylglyoxime.

The silver content of ores can be determined in the same way.

The silver content is expressed in ‰.



Instruments

Titration	TL 7000 or higher
Exchangeable head	WA 50
Electrode	AgCl 62 RG
Cable	L 1 A
Stirrer	Magnetic stirrer TM 235 or similar
Lab accessory	Beaker 150 mL
	Magnetic stirrer bar 30 mm
	Watch glass

Reagents

1	Sodium- or Potassiumchloride – solution 0,1 mol/L
2	Nitric acid 65%
3	Polyvinyl alcohol 0.5%
4	Dimethylglyoxime disodium salt octahydrate
5	Distilled water
All reagents should be of analytical grade or better.	

Titration procedure

Reagents

NaCl – solution 0,1 mol/L

NaCl – solution 0,1 mol/L is available as ready-to-use solution. The titer determination of the NaCl solution is done as described in the application note 201 "Titer determination of NaCl".

Nitric acid 33%

363 mL (508g) HNO₃ 65% are mixed with 400ml dist. Water. After cooling down it is made up to 1L with dist. Water.

Polyvinyl alcohol solution 0.5%

0.5g Polyvinyl alcohol are dissolved in 100mL hot, distilled water.

Dimethylglyoxime disodium salt solution 1%

10g Dimethylglyoxime disodium salt octahydrate are dissolved in 1L dist. Water.

Cleaning and storage of the electrode

The electrode is rinsed with distilled water. Distilled water is suitable for storage of the AgS 62 RG.

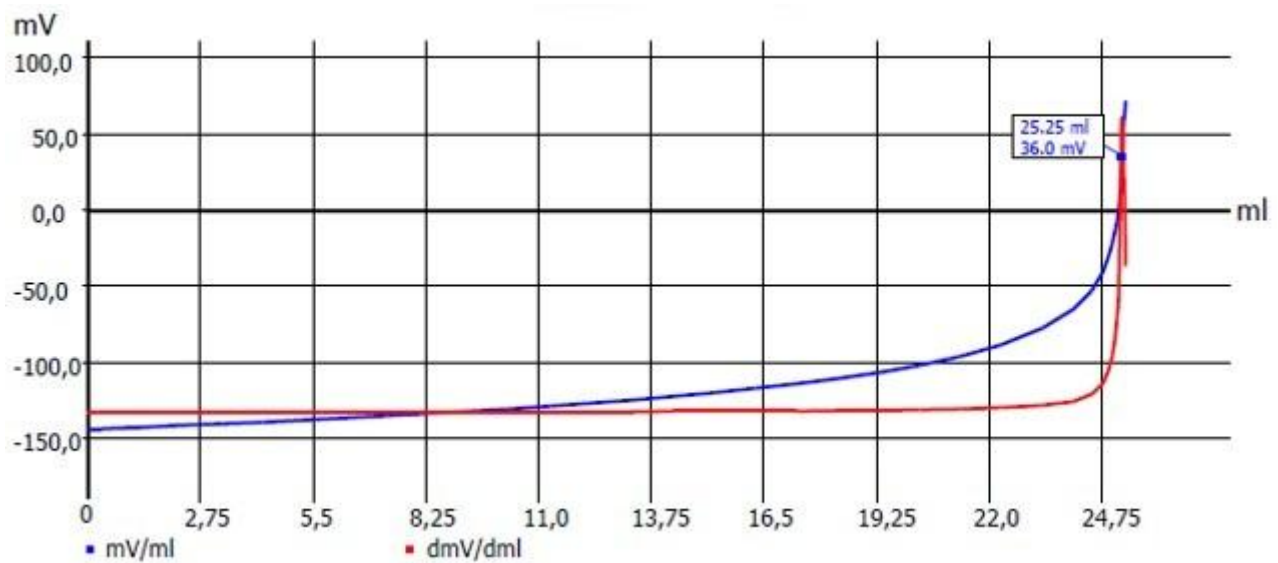
Sample preparation

The sample, which should contain approximately 200–500 mg of Ag, is placed in a 150 mL beaker and 5 mL of 33% HNO₃ are added. The beaker is covered with a watch glass and heated until the sample has dissolved and no more nitrous gases are produced. If necessary, a little more HNO₃ may be required.

The volume is then made up to approximately 60–80 mL with distilled water, approximately 0.5 mL of 0.5% Polyvinyl alcohol solution are added, and the solution is titrated to 1 EQ with 0.1 mol/L NaCl solution.

If the sample contains Palladium, approx. 50 mL of 1% disodium Dimethylglyoxime solution are added after dissolution and made up to approximately 60–80 mL with distilled Water. Approx. 0.5 mL of 0.5% polyvinyl alcohol solution are added and titrated with 0.1 mol/L NaCl solution to 1 EQ.

Titration parameter



Default method	---		
Method type	Automatic titration		
Modus	Dynamic		
Measured value	mV		
Measuring speed / drift	Individual	Minimum holding time	3 s
		Maximum holding time	15 s
		Measuring time	3 s
		Drift	10 mV/min
Initial waiting time	5 s		
Dynamic	steep	Max step size	1.0 ml
		Slope max ml	15
		Min. step size	0.02 ml
		Slope min. ml	230
Damping	none	Titration direction	increase
Pretitration	off	Delay time	0 s
End value	off		
EQ	On (1)	Slope value	400
Max. titration volume	50 ml		
Dosing speed	100%	Filling speed	30 s

Calculation:

Silver:

$$Ag [\%] = \frac{(EQ1 - B) * T * M * F1}{W * F2}$$

B	0	Blank value
EQ1		Consumption of titrant at first Equivalence point
T	WA	Actual concentration of the titrant
M	107,87	Molecular weight of Ag
W	man	sample amount [g]
F1	1	Conversion factor
F2	1	Conversion factor