Titration of Chloride

Description

The determination of the chloride content is done by titration with silver nitrate solution 0.001 - 0.1 mol/l.
This determination is not always easy, since first the chloride has to be released from the sample. This determination is suitable for aqueous or water soluble samples with chloride contents below 1 ppm up to 100%.
For very small chloride contents well below 10 ppm, it may be advantageous to carry out the titration in acetic acid with a 0.001-0.002 mol / l silver nitrate solution (dissolved in 90% acetic acid).
The result is calculated as mg / l chloride.

Instruments

Titrator	TL 5000, TL 7000 or higher
Electrode	AgCl 62 or AgCl 62 RG
Cable	L 1 A
Stirrer	Magnetic stirrer TM 235 / TM 50
Lab accessory	Glass beaker 150 ml
	Magnetic stirrer bar 30 mm

Reagents

1	Silver nitrate solution 0.1 mol/l
2	Nitric acid 4 mol/l
3	Polyvinylalkohol – solution 0.5%
4	Electrolyte solution L 2114 (KNO_3 2 mol/l + KCl 0.001 mol/l) for AgCl 62, Ag 6280
5	Distilled Water
All reagents should be of analytical grade or better.	

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Titration procedure

Reagents

The titer determination of the $AgNO_3$ solution is carried out as described in the application report "Titer determination of $AgNO_3$".

Polyvinyl alcohol - solution 0.5%
0.5 g of polyvinyl alcohol are dissolved in 100 ml of distilled water.

The addition of polyvinyl alcohol is recommended for high chloride/salt concentrations. It avoids the agglomeration of the silver chloride.

Cleaning of the electrode

The electrode is rinsed with distilled water. The electrolyte solution L2114 is suitable for storage. The AgCl 62 RG or Ag 62 RG can be stored in water.

Sample preparation

The sample is pipetted into a 150 ml beaker and filled up to about 80 ml with distilled water. 0.5 ml 4mol/l HNO_3 and 0.5 - 1 ml of the polyvinyl alcohol solution are added. The titration is done with 0.1 mol/l $AgNO_3$ solution to an equivalence point. The consumption should be about 5 - 15 ml.

The titration can be carried out with samples with chloride contents of a few ppm - 100%, but the amount of sample has to be adjusted.

Sample amount for titration with 0.1 mol/l $AgNO_3$	
Chloride content [%]	Sample [g]
< 0.1	> 10
0.1 – 1	1 – 10
1 – 10	0.1 – 2
10 – 50	0.05 – 0.1
50 - 100	0.05

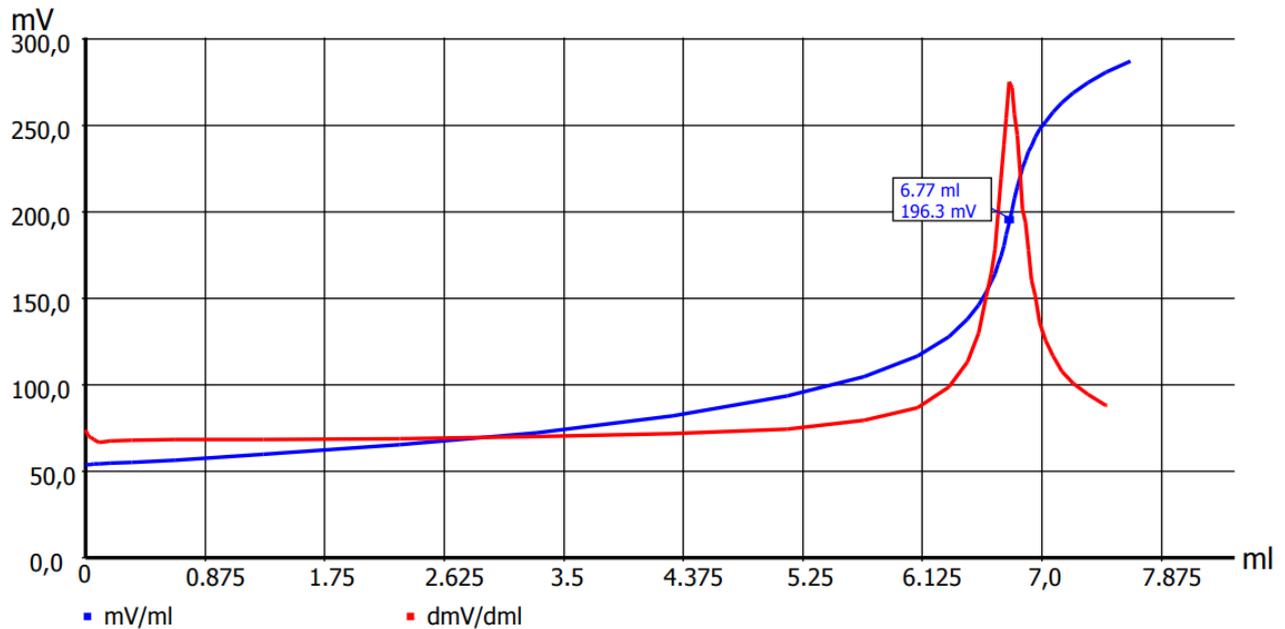### Checking the silver electrode

Calibration with buffers or comparable test solutions as for pH electrodes is not possible, but also not necessary. With a pH electrode, the measured voltage in mV in the pH meter/titrator is converted into pH values using the values determined during the pH calibration such as slope and zero point. In addition, there are many methods where titration to a certain pH value is required, such as the determination of total acidity in beverages.

This is not the case with chloride titration. Here, titration always takes place to an equivalence point (EQ). This means that a certain measuring potential is not important, but the change in the measuring potential during several measuring points.

To check the silver electrode we recommend to titrate a standard such as NaCl and to compare the resulting titration curve with a stored titration curve of a standard at the beginning of use. The potentials should be in the same order of magnitude as at the beginning. More important is the appearance of the curve. It should not be noisy or jagged.

Titration parameter

Sample titration

Graph showing mV on the y-axis (0,0 to 300,0) versus ml on the x-axis (0 to 7.875). Legend: mV/ml (blue), dmV/dml (red). Annotation: 6.77 ml / 196.3 mV.

Default method	Chloride mg/l				
Method type	Automatic titration				
Modus	Dynamic				
Measured value	mV				
Measuring speed / drift	User defined	Minimum holding time	3 s		
		Maximum holding time	15 s		
		Measuring time	3 s		
		Drift	10 mV/min		
Initial waiting time	0 s				
Dynamic	average	Max step size	1.0 ml		
		Slope max ml	10		
		Min. step size	0.02 ml		
		Slope min. ml	120		
Damping	none	Titration direction	increase		
Pretitration	off	Delay time	0 s		
End value	off				
EQ	On (1)	Slope value	150		
Max. titration volume	50 ml				
Dosing speed	100%	Filling speed	30 s		

When titrating very low levels of chloride or titrating in glacial acetic acid, the minimum waiting time should be set to 6 s and the drift to 5 mV/min. In this case, the dynamics should also be set to flat.
For some samples it may happen that the titration curve is very flat and the titrator does not stop the titration at the EQ. In this case, the slope value for the EQ should be reduced.

Calculation:

$$Result\ [mg/l] = \frac{(EQ1 - B) * T * M * F1}{W * F2}$$

B	0	Blank value
EQ1		Consumption of titrant at first Equivalence point
T	WA	Actual concentration of the titrant
M	35.45	Molecular weight of chloride
W	man	sample weight in g
F1	1000	Conversion factor
F2	1	Conversion factor

If the calculation value is not mg/l chloride, but mg/l NaCl, then M is set to the molar mass of NaCl 58.44 g/mol.