

Titration of Chloride

CHEMICAL SERIES



Introduction

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 calculation is made as mg / l chloride.



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Instrument	
Titrator	TL 5000 or higher
Stirrer	Magnetic stirrer TM 235 or similar
Electrode, Cable, and Electrolyte	
Electrode	AgCl 62 or AgCl 62 RG
Cable	L1A
Lab Accessories	
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	Polyvinyl alcohol - solution 0.5%
4	Electrolyte solution L2114 (KNO ₃ 2 mol/l + KCl 0.001 mol/l) for AgCl 62, Ag 6280
5	Distilled Water
All reagents should be in analytical grade or better.	

Titration Procedure

Reagents

The titer determination of the AgNO₃ solution is carried out as described in the application report "Titer determination of AgNO₃".

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

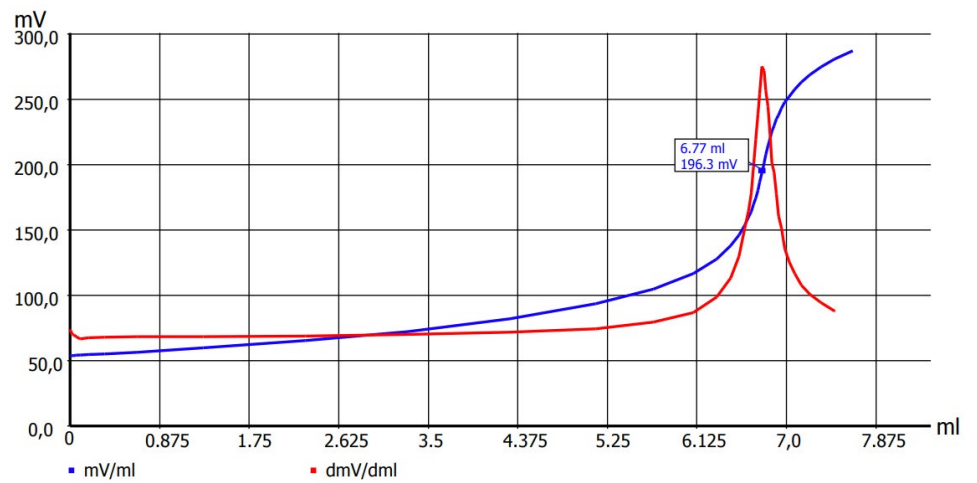
The sample is pipetted into a 150 ml beaker and filled up to about 80 ml with distilled water. 0.5 ml 4mol/l HNO₃ and 0.5 - 1 ml of the polyvinyl alcohol solution are added.

The titration is done with 0.1 mol/l AgNO₃ 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 - 0.2
10 - 50	0.05 - 0.1
50 - 100	0.05

Titration parameter - Sample Titration



Default Method Chloride mg/l			
Method type	Automatic Titration		
Mode	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 Detection	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:

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

EQ1		Consumption of titrant at the first Equivalence point
B	0	Blank value
T	WA	Actual concentration of the titrant
M	35.45	Molecular weight of chloride
W	manual	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.

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