

Titer determination of Perchloric acid in glacial acetic acid

Description

This application report describes the general procedure for the titer determination of Perchloric acid in glacial acetic acid.

The titer is a dimensionless number about 1 for correcting the indicated concentration. In the software of the titration devices and application reports from SI Analytics®, the term “Titer” describes the exact concentration in mol/l and not the dimensionless factor.

Instruments

Titrator	TL 7000 or higher
Exchangeable Unit	WA 20
Electrode	N 6480 Eth, N 6480 Eis, N 61 Eis
Cable	L 1 A
Stirrer	TM 235
Titration tip	TZ 1643
Lab accessoires	Glas beaker 150 ml
	Magnetic stirrer bar 30 mm

Reagents

1	Perchloric acid in glacial acetic acid
2	Glacial acetic acid
3	Potassiumhydrogenphthalate (KHPht) – certified reference material, volumetric standard
4	Electrolyte L 5034 (LiCl/ethanol) or L 5014 (LiCl/glacial acetic acid)
All reagents should be in analytical grade or better.	

Titration procedure

Reagents

The KHPht volumetric standard is dried as described in the corresponding certificate of analysis.

Cleaning and storage of the electrode

Use Ethanol or Isopropylalcohol for cleaning the electrode. For storage use the same electrolyte solution used in the electrode, L 5034 (N6480 Eth) or L 5014 (N 6480/N 61 Eis).

Sample preparation

The amount of volumetric standard depends on the size of the burette and the concentration of the acid. The amount should be chosen so that about half of the burette volume is consumed. The most common is the 20 ml burette. The required quantity of KHPht can be estimated according to this rule of thumb:

$$W [g] = 2 * Concentration[mol/l]$$

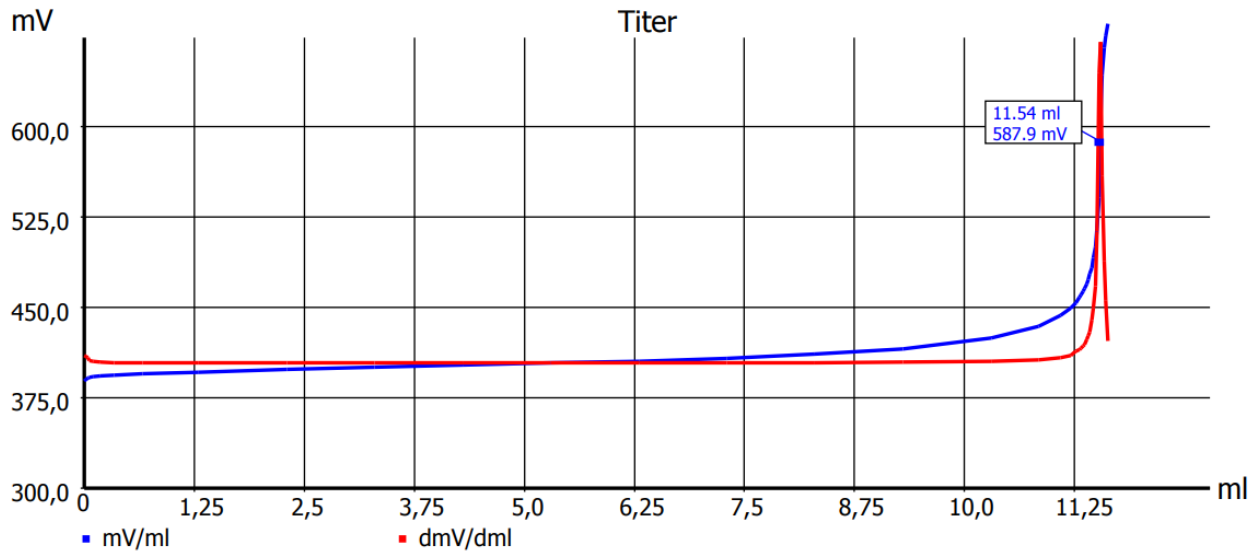
To determine the titer of a 0.1 mol/l Perchloric acid, about 0.2 g KHPht volumetric standard are weighed into a 150 ml beaker and filled up to 60 ml with glacial acetic acid. When the KHPht is completely dissolved, it is titrated with the perchloric acid to an EQ.

If the specified assay of the volumetric standard is significantly different from 100%, the weight for calculating the concentration must be corrected:

$$W = \frac{Weight * specified assay \%}{100}$$

The density of the perchloric acid in glacial acetic acid depends strongly on the temperature. It is recommended to measure and document the temperature at which the titration was carried out. The temperature at the titer determination should be identical to the temperature at the sample titration.

Titration parameter



Default method	Titer Perchloric acid		
Method type	Automatic titration		
Modus	Dynamic		
Measured value	pH		
Measuring speed / drift	Normal	Minimum holding time	3 s
		Maximum holding time	15 s
		Measuring time	2 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	average	Titration direction	Increase
Pretitration	off	Delay time	0 s
End value	off		
EQ	On(1)	Slope value	300
Max. titration volume	20 ml		
Dosing speed	100%	Filling speed	30 s

Calculation:

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

B	0	Blank value
W	man	Weight of the sample [g]
F2	1000	Conversion factor ml - l
EQ1		Consumption of titrant until first Equivalence point
M	204,22	Molecular mass of KHPht
F1	1	Conversion factor

We recommend to store the exact concentration T to the exchangeable Unit (WA) automatically.