

Titration of Bases with perchloric acid (non-aqueous)

Description

The most common method for the determination of pharmaceutical bases is the direct titration with Perchloric acid in Glacial acetic acid. A pH electrode with a filling of LiCl in ethanol or glacial acetic acid is used as electrode.

For some weak bases it is advantageous to use Formic acid, a mixture of Glacial acetic acid or Formic acid with Acetic anhydride or pure Acetic anhydride as solvent. In this case we recommend the electrode N 61 eis, electrodes with an alcoholic electrolyte cause too high results.

For pharmaceutical bases, information to the solvent can be found the pharmacopeia.

The result is calculated as % of the base.

Instruments

Titration	TL 7000 or higher
Electrode	N 6480 Eth (electrolyte L 5034) or N 6480 eis or N 61 eis (electrolyte L 5014 for both options)
Cable	L1A
Stirrer	Magnetic stirrer TM 235 or similar
Titration tip	Long version TZ 1643 required
Lab accessoires	Glas beaker 250 ml tall form without spout
	Watch glass or Parafilm
	Magnetic stirrer bar 30 mm

Reagents

1	Perchloric acid in glacial acetic acid 0.1 mol/l
2	Glacial acetic acid
3	Acetic anhydride
4	Formic Acid
5	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 titer determination of the HClO₄ solution is carried out as described in the application report "Titer determination of Perchloric acid in glacial acetic acid".

Cleaning and storage of the electrode

Use Ethanol or Iso propyl alcohol 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 sample is weighted out in a 150 ml beaker and filled up to 60 ml with glacial acetic acid. Then it is titrated with HClO₄ 0.1 mol / l. The consumption should be about 5 - 15 ml.

For pharmaceutical samples: Please refer to the Pharmacopeia, regarding the recommended sample amounts and the equivalent weights.

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. If the temperature is different, the volume can be corrected according to the European pharmacopeia:

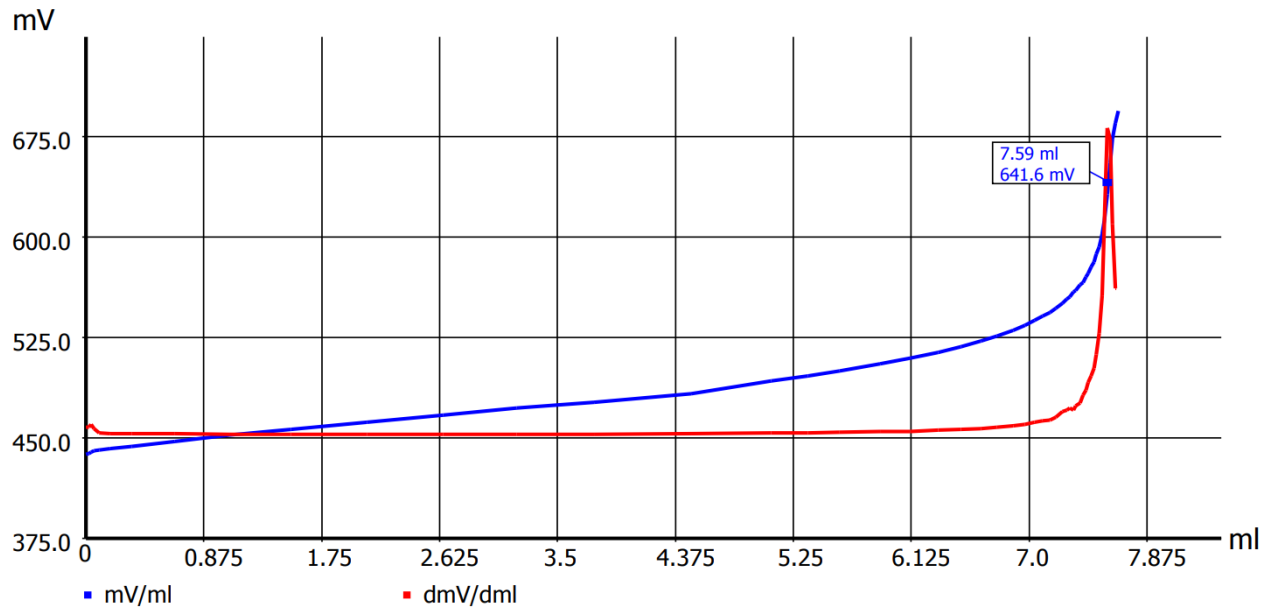
$$EQ_{corr} = EQ1 * (1 + (T_1 - T_2) * 0.0011)$$

EQ1	Consumption of titrant at first Equivalence point
EQ _{corr}	Corrected Volume of titrant
T ₁	Temperature at titer determination
T ₂	Temperature at sample titration

For some weak bases Formic acid, a mixture of Glacial acetic acid or Formic acid with Acetic anhydride or pure Acetic anhydride must be used as solvent. The ratio of acid : Acetic anhydride strongly depends on the sample. If mixtures with Acetic anhydride are used, the mixture heats up strongly during the titration and should be cooled.

Titration parameter

Sample titration



Default method	Bases (Non-Aqueous)		
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	2 s
		drift	10 mV/min
Initial waiting time	0 s		
Dynamic	User-defined	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:

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

B	0	Blank value
EQ1		Consumption of titrant at first Equivalence point
T	WA	Actual concentration of the titrant
M		Molecular mass of the base
W	man	Weight of the sample
F1	0.1	Conversion factor
F2	1	Conversion factor

Any questions? Please contact the application team:

Xylem Analytics Germany Sales GmbH & Co. KG, SI Analytics
Hattenbergstraße 10
D-55122 Mainz, Germany
Telefon: + 49 6131 66 5126
Fax: + 49 6131 66 5101
E-Mail: titration@si-analytics.com

SI Analytics
a xylem brand

Xylem Analytics Germany Sales GmbH & Co. KG · Hattenbergstr. 10 · D-55122 Mainz · Germany
Telefon: +49 6131.66. 5111 · E-Mail: Info.si-analytics@Xyleminc.com · www.si-analytics.com

Alle Namen sind eingetragene Handelsnamen oder Warenzeichen der Xylem Inc. oder eines seiner Tochterunternehmen. Technische Änderungen vorbehalten.
© 2018 Xylem Analytics Germany Sales GmbH & Co. KG.