Determination 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

Titrator	TL 7000, TL 7750 or TL 7800
Exchange unit	WA 10
Electrode	N 6480 Eth, N 6480 eis or N 61 eis
Cable	L1A
Stirrer	Magnetic stirrer TM 235 or similar
Titration tip	Long version TZ 1643 required
Lab accessoires	Glas beaker 150 mL
	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 in Ethanol) or L 5014 (LiCl in glacial acetic acid, only for eis-electrodes)
All reagents should be in analytical grade or better.	

*Depends on the sample!

Titration procedure

Reagents

$HClO_4$ 0.1 mol/L
Perchloric acid 0.1 mol/L is available as ready-to-use solution. The titer is determined as described in our application note "Titer determination of $HClO_4$".

Cleaning and storage of the electrode

The electrode is cleaned with Ethanol or Isopropanol. For storage, the same electrolyte solution with which the electrode is filled is used.
The electrolyte L 5014 (LiCl in glacial acetic acid) may only be used in electrodes with the suffix "eis".

Sample preparation

The sample is weighted in a beaker and made up to 60 - 80 ml with glacial acetic acid. Then it is titrated with $HClO_4$ 0.1 mol/L. The consumption should be about 5 - 15 mL.

For pharmaceutical bases, information on the recommended weight and solvent can be found in the pharmacopoeia.

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:

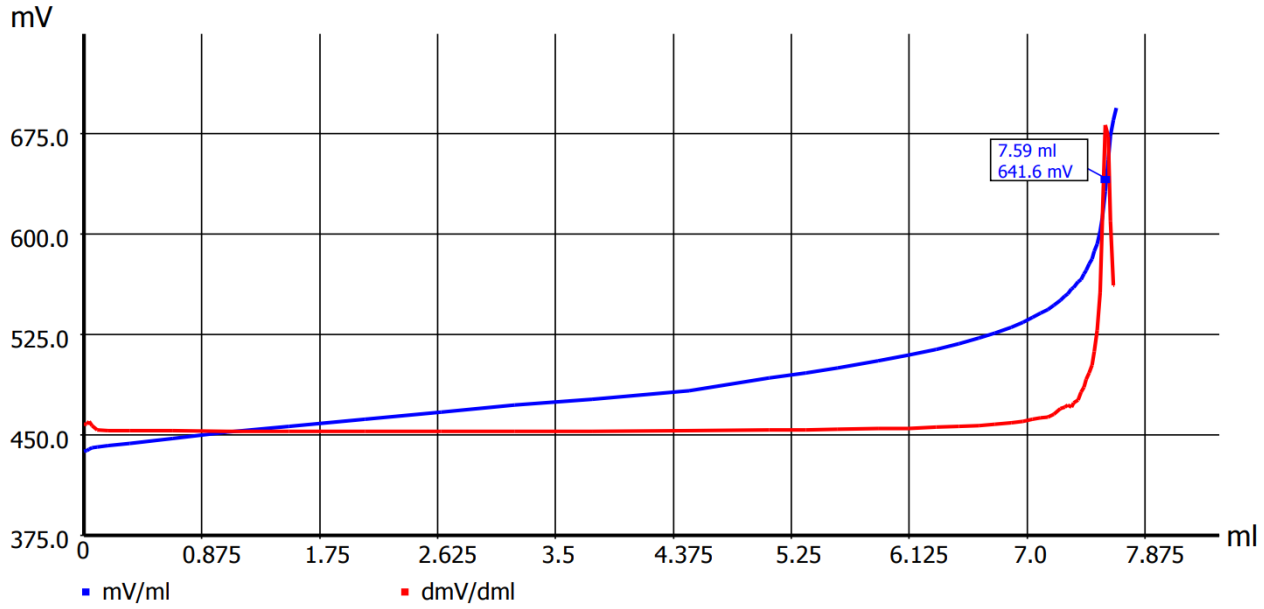
$$EQ_{corr} = EQ1 * (1 + (T_1 - T_2) * 0.0011$$

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	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:

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

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	Sample amount [g]
F1	0.1	Conversion factor
F2	1	Conversion factor

Any questions? Please contact the application team:

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