Determination of bases with Perchloric acid titration

Application

Use

This application note describes the non-aqueous titration of different bases with perchloric acid in glacial acetic acid. This application note do not describes the use of other solvents

Appliances

- Titrator: TL 7000/TL 7750 M1/10 or 20
- Basic device
- Magnetic stirrer TM 235
- 10/20 mL exchange unit WA 10/20, with amber glass bottle for the titrant, complete

Electrodes

- Electrode: N 6480 eth + TZ 1643 titration tip/also suitable is the N 6480 eis
- Electrolyte: L 5034 (LiCl/ethanol) or L 5014 (LiCl/glacial acetic acid)
- Calibration: n.a.

Reagents

- Titrant: perchloric acid in acetic acid 0.1 mol/l
- Titer determination: Potassium hydrogen phtalate
- Solvents: Acetic acid (100 %, glacial acetic acid)

Description

Determination of the exact concentration of the Perchloric acid titrant with Potassium hydrogen phthalate

We recommend ready to use a “ready to use” titrant. The Potassium hydrogen phthalate (volumetric standard) should be dried at 120 °C for at least two hours before use and stored in desiccator.

0.2 g of the standard are weighed accurately (0.1 mg) in a 100 mL beaker and dissolved in 60 mL of glacial acetic acid. Stir at room temperature until the substance is completely dissolved.

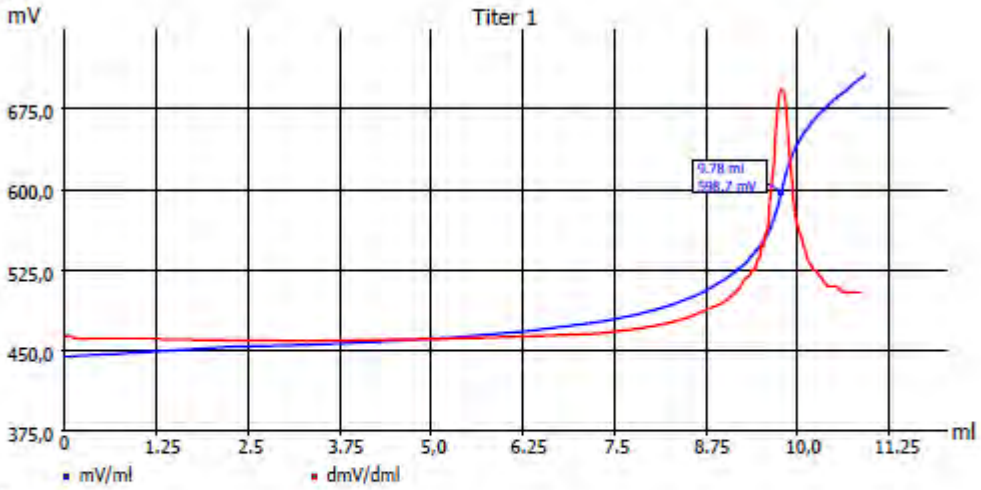
Use the standard method which is installed in the TitroLine 7000/7750. The method is ready to use and must be only saved as a user method.

Repeat the standardization two times. The average value is stored automatically in the exchangeable unit.

Page 1: Curve and result: Titer determination

GLP documentation

Titration graph



Method data

Method name:	Titer Perchloric acid	Titration duration:	5 m 49 s
End date:	08.11.12	End time:	14:16:28

Titration data

Sample ID:	Titer 1	Weight:	0.2022 g
Start mV:	443.8 mV	End mV:	707.5 mV
EQ:	9.779 ml / 598.7 mV	Titer:	0.1015 mol/l
Mean value:	---	RSD:	---

Calculation formula

Titer:	(W*F2)/((EQ1-B)*M*F1) -> M103
Mol (M):	204.22000

Weight (W):	man	Factor 2 (F2):	1000.0000
Blank value (B):	0.0200 ml (M01)	Factor 1 (F1):	1.0000
Statistics:	3		

Device information

Device: TitroLine 7000
Serial number: 00012
Software version: 1230k

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Page 2: Method parameters Titer determination:

Method data overall view

Method name:	Titer Perchloric acid	Created at:	11/08/12 12:22:19
Method type:	Automatic titration	Last modification:	11/08/12 14:07:34
Measured value:	mV	Damping settings:	average
Titration mode:	Dynamic	Documentation:	GLP
Dynamic:	average		
Measuring speed / drift:	Normal:	minimum holding time:	03 s
		maximum holding time:	15 s
		Measuring time:	02 s
		Drift:	10 mV/min
Initial waiting time:	0 s		
Titration direction:	Increase		
Pretitration:	Off		
End value:	Off		
EQ:	On (1)		
Slope value:	User-defined	Value:	350

Dosing parameter

Dosing speed:	100 %	Filling speed:	30 s
Maximum dosing volume:	15.00 ml		

Unit values

Unit size:	20ml
Unit ID:	10039168
Reagent:	HClO4 0.1 mol/L
Batch ID:	no entry
Concentration [mol/l]:	0.10000
Determined at:	11/08/12 20:16:03
Expire date:	--
Opened/compounded:	--
Test according ISO 8655:	05/03/12
Last modification:	11/08/12 12:16:04

Device information

Device:	TitroLine 7000
Serial number:	00012
Software version:	1230k

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Application

Titration in glacial acetic acid

Dissolve the prescribed quantity of the substance in glacial acetic acid and titrate with c(HClO4) = 0.1 mol/L until the first equivalence point (EQ1) is reached

Result calculation

% Sample (e.g. Dichlofenac-Na, Trapidil…)

% Sample = (EQ1-B) * M * T * F1/(W*F2)

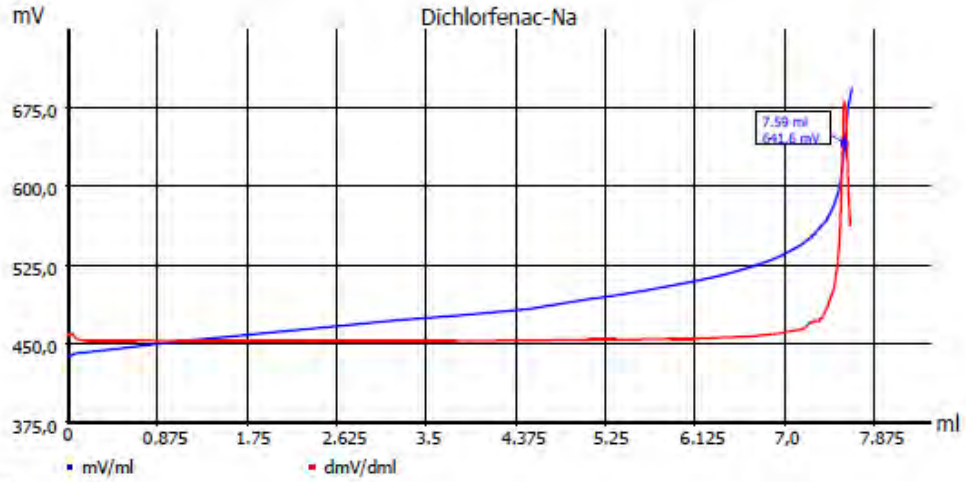
EQ1:	ml consumption at the equivalence point
B:	ml consumption for the blank titration (if required, otherwise = 0)
M:	Equivalent weight of the sample (e.g. 205.3 g/mol for Trapidil)
T:	concentration of the $HClO_4$ titrant (e.g.0.1002 mol/l)
W:	sample weight in g
F1,	100
F2	1000

Application

Example : Dichlofenac-Na
Page 1: Curve and result

GLP documentation

Titration graph



Method data

Method name:	Dichlorfenac Na	Titration duration:	3 m 37 s
End date:	24.01.13	End time:	17:56:55

Titration data

		Weight:	0.24490 g
Start mV:	437.0 mV	End mV:	693.9 mV
EQ:	7.588 ml / 641.6 mV	Dichlorfenac Na:	99.94 %
Mean value:	99.91 %	RSD:	---

Calculation formula

Dichlorfenac Na:	(EQ1-B)*T*M*F1/(W*F2)	Mol (M):	318.10000
Blank value (B):	0.0000 ml	Titre (T):	0.10140000 (a)
Factor 1 (F1):	0.1000	Weight (W):	0.2449 g (m)
Factor 2 (F2):	1.0000	Statistics:	3

Device information

Device:	TitroLine 7750
Serial number:	10018602
Software version:	1304

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Example : Dichlofenac-Na
Page 2: Method parameter

Method data overall view

Method name:	Dichlorfenac Na	Created at:	01/24/13 16:40:14
Method type:	Automatic titration	Last modification:	01/24/13 16:44:41
Measured value:	mV	Damping settings:	Average
Titration mode:	Dynamic	Documentation:	GLP
Dynamic:	Average		
Measuring speed / drift:	Normal:	minimum holding time:	02 s
		maximum holding time:	15 s
		Measuring time:	02 s
		Drift:	20 mV/min
Initial waiting time:	0 s		
Titration direction:	Increase		
Pretitration:	Off		
End value:	Off		
EQ:	On (1)		
Slope value:	User-defined	Value:	350

Dosing parameter

Dosing speed:	100 %	Filling speed:	30 s
Maximum dosing volume:	20.00 ml		

Unit values

Unit size:	20ml
Unit ID:	10039168
Reagent:	HClO4 0.1 mol/L
Batch ID:	no entry
Concentration [mol/l]:	0.10140
Determined at:	11/22/12 1:08:25
Expire date:	--
Opened/compounded:	--
Test according ISO 8655:	05/03/12
Last modification:	01/24/13 17:41:23

Device information

Device:	TitroLine 7750
Serial number:	10018602
Software version:	1304

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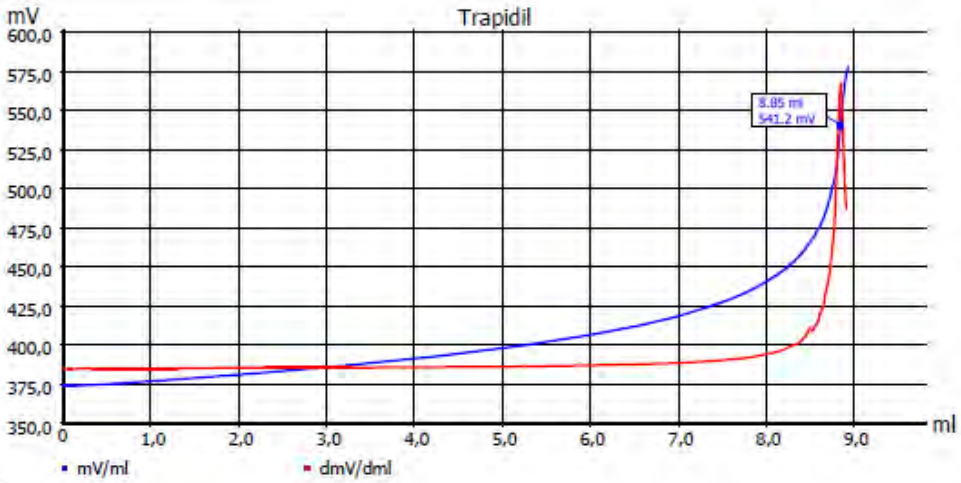
Example : Trapidil
Page 1: Curve and result

GLP documentation

Titration graph



Method data

Method name:	Trapidil	Titration duration:	3 m 27 s
End date:	25.01.13	End time:	17:23:12

Titration data

		Weight:	0.18410 g
Start mV:	374.1 mV	End mV:	578.2 mV
EQ:	8.851 ml / 541.2 mV	Trapidil:	100.08 %
Mean value:	100.08 %	RSD:	0.01 %

Calculation formula

Trapidil:	(EQ1-B)*T*M*F1/(W*F2)	Mol (M):	205.30000
Blank value (B):	0.0000 ml	Titre (T):	0.10140000 (a)
Factor 1 (F1):	0.1000	Weight (W):	0.1841 g (m)
Factor 2 (F2):	1.0000	Statistics:	3

Device information

Device:	TitroLine 7750
Serial number:	10018602
Software version:	1304

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Application

Example : Trapdil
page 2: Method parameter

Method data overall view

Method name:	Trapidil	Created at:	01/25/13 17:01:24
Method type:	Automatic titration	Last modification:	01/25/13 17:05:13
Measured value:	mV	Damping settings:	Average
Titration mode:	Dynamic	Documentation:	GLP
Dynamic:	Average		
Measuring speed / drift:	Normal:	minimum holding time:	02 s
		maximum holding time:	15 s
		Measuring time:	02 s
		Drift:	20 mV/min
Initial waiting time:	0 s		
Titration direction:	Increase		
Pretitration:	Off		
End value:	Off		
EQ:	On (1)		
Slope value:	User-defined	Value:	350

Dosing parameter

Dosing speed:	100 %	Filling speed:	30 s
Maximum dosing volume:	20.00 ml		

Unit values

Unit size:	20ml
Unit ID:	10039168
Reagent:	HClO4 0.1 mol/L
Batch ID:	no entry
Concentration [mol/l]:	0.10140
Determined at:	11/22/12 1:08:25
Expire date:	--
Opened/compounded:	--
Test according ISO 8655:	05/03/12
Last modification:	01/25/13 16:59:16

Device information

Device:	TitroLine 7750
Serial number:	10018602
Software version:	1304

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Application

Notes

Please refer to the Pharmacopea, regarding the recommended sample amounts and the equivalent weights.

If you have any questions on the application, you can feel free to contact us..

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