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Application Note

Determination of Total Base Number (TBN)

ACCORDING TO ASTM D2896/ISO 3771

Introduction

Determination of basic constituents in petroleum products by potentiometric titration with perchloric acid in glacial acetic acid. The total base number (TBN) is the quantity of perchloric acid, expressed in terms of equivalent number of milligrams of potassium hydroxide (or alternatively in milli-equivalents of hydroxide per gram), that is required to neutralize all basic constituents present in 1 g of sample when titrated under the prescribed conditions.

Apparatus

- TitroLine® 7000 or higher
- Magnetic stirrer (TM 235)
- 10 or 5 mL Exchange unit WA 10/WA 5, with amber glass bottle for the titrant, TZ 1643 titration tip

Electrode and Electrolyte

- **Electrode:** N 6480 eis with cable L 1 A
- **Electrolyte:** L 5014 (LiCl/acetic acid)

Reagents

- **Solvent:** Glacial acetic acid/chlorobenzene (1/2)
- **Standardization:** Potassium hydrogen phthalate (KHC8H404) or "Tris"
- **Titrant:** Perchloric acid 0.1 mol/L in glacial acetic acid

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Procedure

Preparation of the perchloric acid solution

Please use a “ready to use” titration solution 0.1 mol/L.

Standardization

Potassium hydrogen phthalate must be dried in an oven (110-120 °C) and then cooled in a desiccator. Take 0.15 to 0.2 g of the potassium hydrogen phthalate or Tris weighed to the nearest 0.1 mg and dissolve it in 40 ml acetic acid under heating. Add 80 ml chlorobenzene. Use method -> **Titer perchloric acid.**

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

Blank value of the solvent mixture

Add 120 mL of the titration solvent into the beaker. Place the beaker on the magnetic stirrer and start the titration method. After titration, rinse the electrode and burette tip with solvent, then with water, then again with solvent in a beaker for approximately 1 minute. Use method **BLANK_TBN.**

Repeat the blank titration one time. The average value can be stored in a global memory e.g. M01 (TBN blank) which must be created beforehand.

Titration of sample

Calculate the quantity of the sample required from expected total base number (TBN) from the equation:

Approximate mass, in gram, of sample = 28/expected TBN

Weigh the sample in a 250 mL beaker and add 120 ml of the titration solvent to the sample. Place the beaker on the magnetic stirrer and start the titration method. After titration rinse the electrode and burette tip with solvent, then with water, then again with solvent in a beaker for approximately 1 minute. Use method **TBN ISO ASTM.**

Maintenance of Electrodes

If you use a combination electrode such as N 6480 store the electrode in the LiCl/glacial acid electrolyte.

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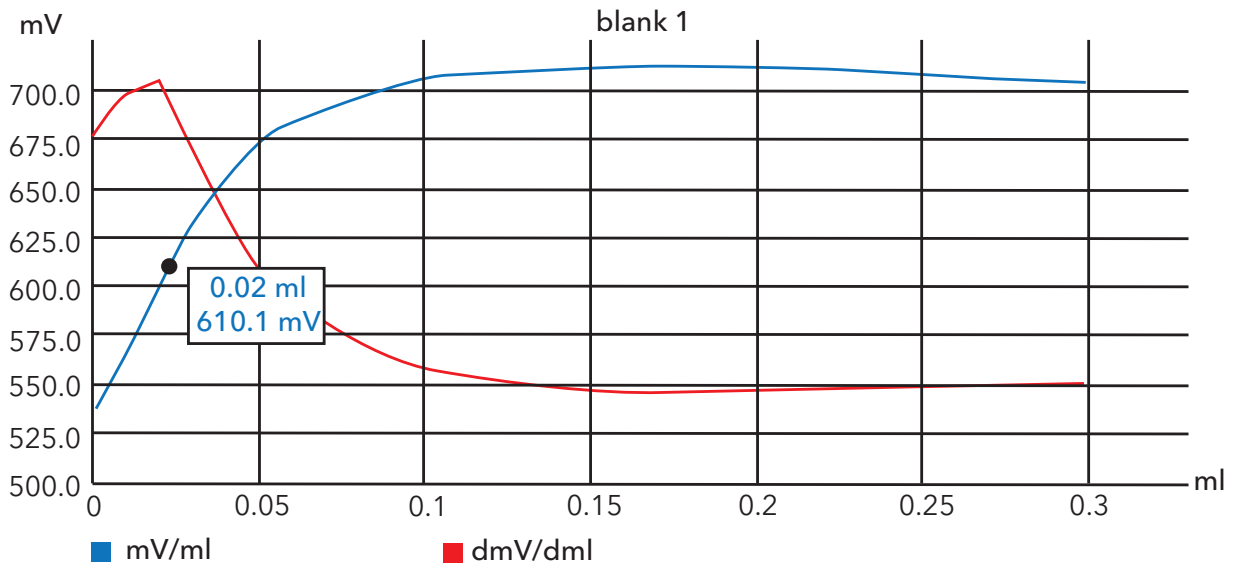
Results

GLP documentation

Titration graph



Method data

Method name:	Blank TBN 2896	Titration duration	5 m 11 s
End date:	21.04.16	End time:	12:42:21

Titration data

Sample ID:	blank 1		
Start mV:	533.2 mV	End mV:	704.6 mV
EQ:	0.02 ml / 610.1 mV	Blank:	0.02 ml

Calculation formula

Blank:	EQ-> M02
Statistics:	Off

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Method Data

Method data overall view

Method name:	Blank TBN	Created at:	11/08/12 12:40:17
Method type:	Automatic titration	Last modification:	11/08/12 12:47:13
Measured value:	mV	Damping settings:	strong
Titration mode:	Linear	Documentation:	GLP
Linear steps	0.010 ml		
Measuring speed / drift	10s		
Initial waiting time:	10 s		
Titration direction:	Increase		
Pretitration:	Off		
End value:	Off		
EQ:	On (1)		
Slope value:	Flat	Value	120

Dosing parameter

Dosing speed:	100 %	Filling speed	30 s
Maximum dosing volume:	0.30 ml		

Unit values

Unit size:	20 ml
Unit ID:	10039168
Reagent:	HCl04 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

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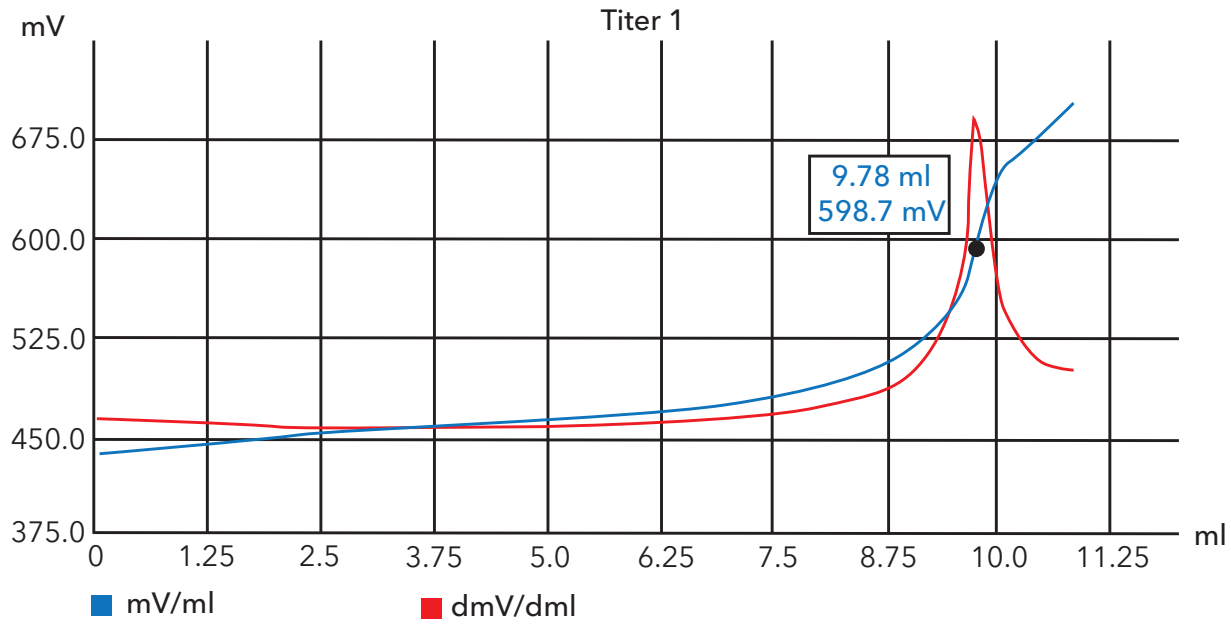
Standardization (Page 1)

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		

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Standardization (Page 2)

Method data

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:	20 ml
Unit ID:	10039168
Reagent:	HCl04 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

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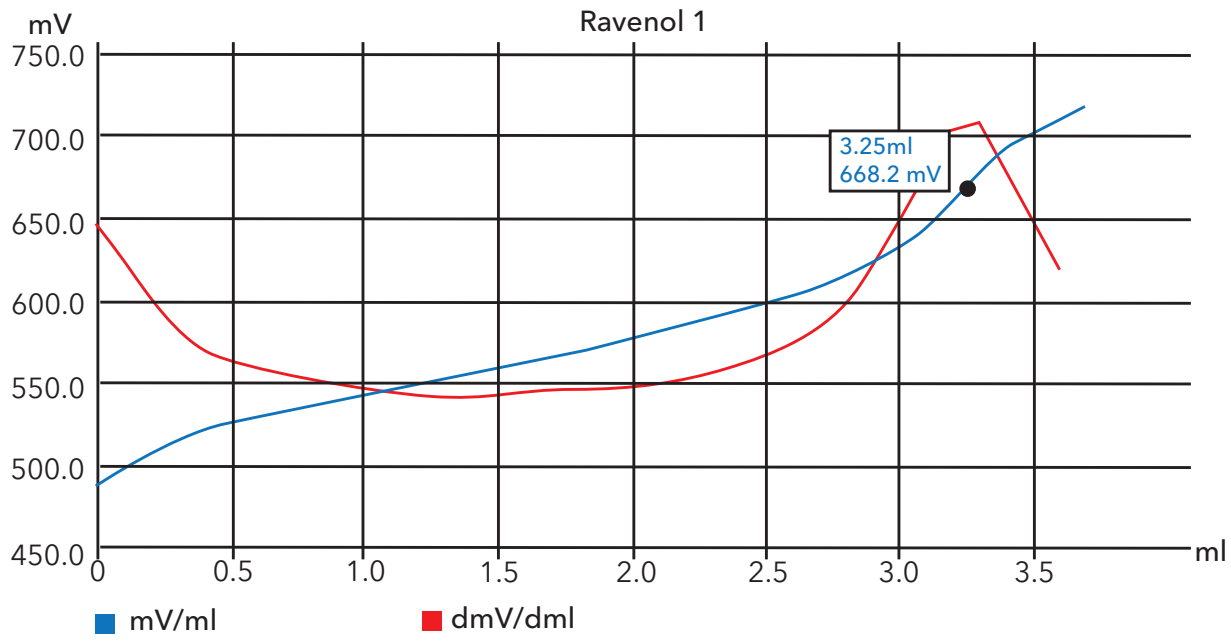
Sample Titration (Page 1)

GLP documentation

Titration graph



Method data

Method name:	TBN ISO 3771	Titration duration	4 m 46 s
End date:	08.11.12	End time:	14:24:44

Titration data

Sample ID:	Ravenol 1	Weight:	1.5102 g
Start mV:	486.8 mV	End mV:	718.8 mV
EQ:	3.255 ml / 668.2 mV	TAN mg KOH/g:	12.197

Calculation formula

TAN mg KOH/g:	(EQ1-B)*T*M*F1/(W*F2)		
Mol (M):	56.10000		
Blank value (B):	0.0200 ml (M01)	Titre (T):	0.10150000 (a)
Factor 1 (F1):	1.0000	Weight (W):	man
Factor 2 (F2):	1.0000	Statistics:	Off

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Sample Titration (Page 2)

Method data

Method data overall view

Method name:	TBN ISO 3771	Created at:	11/08/12 14:17:55
Method type:	Automatic titration	Last modification:	11/08/12 14:18:45
Measured value:	mV	Damping settings:	strong
Titration mode:	Linear	Documentation:	GLP
Linear steps:	0.100 ml		

Measuring speed / drift	User-defined:	minimum holding time:	07 s
		maximum holding time:	20 s
		Measuring time:	04 s
		Drift:	10 mV/min

Initial waiting time:	10 s		
Titration direction:	Increase		
Pretitration:	Off		
End value:	Off		
EQ:	On (1)		
Slope value:	Flat	Value:	120

Dosing parameter

Dosing speed:	100 %	Filling speed	30 s
Maximum dosing volume:	6.00 ml		

Unit values

Unit size:	20 ml
Unit ID:	10039168
Reagent:	HCl04 0.1 mol/L
Batch ID:	no entry
Concentration [mol/l]:	0.10150
Determined at:	11/08/12 20:16:03
Expire date:	--
Opened/compounded:	--
Test according ISO 8655:	05/03/12
Last modification	11/08/12 14:16:29

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Literature

International Standard ISO 3771 or ASTM 2896.

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Xylem Inc.
1725 Brannum Lane
Yellow Springs, Ohio 45387

Application/Technical Support:
+ 1-845-258-1200
titration.ysi@xyleminc.com

Ordering:
+1-937-767-7241
orders@ysi.com

ysi.com

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