

# Determination of Total Acid Number (TAN)

ACCORDING TO ASTM 664

## Introduction

For the determination of acidic constituents in petroleum products and lubricants by potentiometric titration. The total acid number TAN is the quantity of base, expressed in milligrams of potassium hydroxide, that is required to neutralize all acidic constituents present in 1 g of sample.

## 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 eth with cable L 1 A
- **Electrolyte:** L 503 4 (LiCl/Ethanol)

## Reagents

- **Solvent:** Toluene/isopropyl alcohol/water (500/495/5)
- **Standardization:** Potassium hydrogen phthalate standard
- **Titrant:** KOH 0.1 mol/L in isopropyl alcohol



## Procedure

### Preparation and standardization of the alcoholic KOH solution

Add 6 g of KOH to approximately 1 L of anhydrous isopropyl alcohol. Boil gently for 10 min to effect solution. Allow the solution to stand for 2 days and then filter through a fine sintered-glass funnel. Store the solution in a chemical resistant bottle and protect the solution for CO<sub>2</sub> with a guard tube containing soda lime. Standardize with exact weighed quantities of 0.2 g of potassium hydrogen phthalate. Add 80 ml CO<sub>2</sub> free water. **Ready to use solutions are recommended.** Use the method Titer KOH.

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

### Blank value of the solvent mixture

Add 125 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 each. Use method **BLANK TAN**.

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

### Titration of sample

Weigh the sample in a 250 mL beaker and add 125 mL of the titration solution to the sample. The sample weight should be calculated and selected so that the titration amount is not more than 4 ml because of the long titration time.

Place the beaker on the magnetic stirrer and start the titration method (TAN). After the titration, rinse the electrode and burette tip with solvent, then with water (5 min), then again with solvent (1 min).

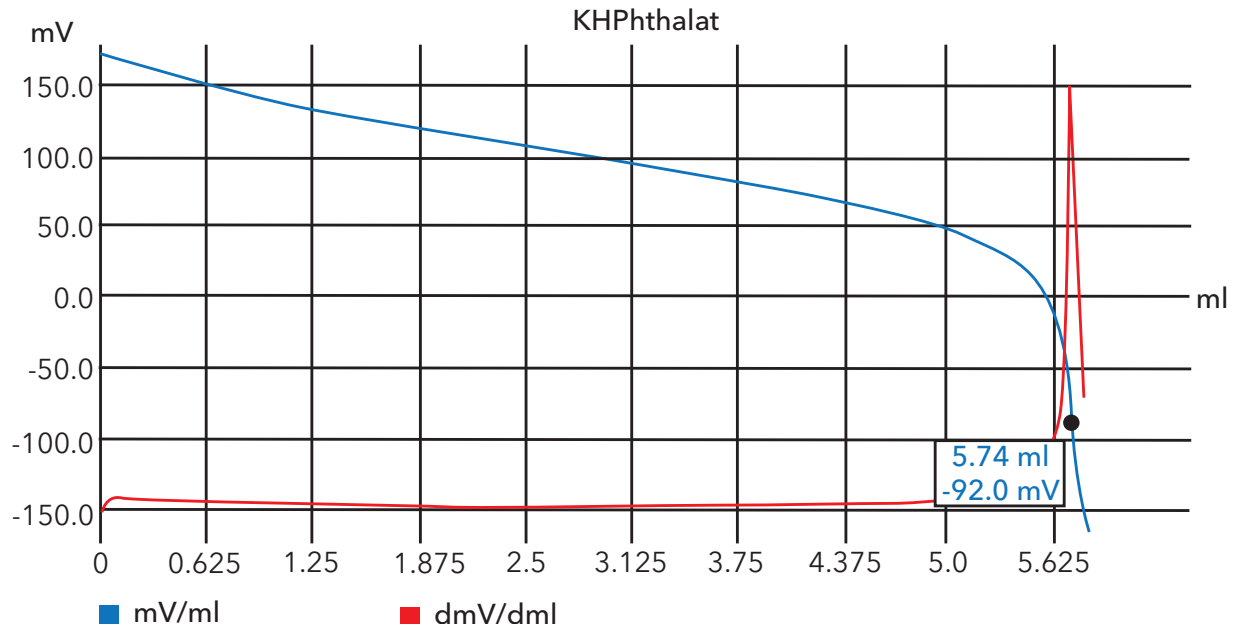
### Maintenance of Electrodes

If you use a combination electrode like N 6480, store the electrode in the LiCl/Ethanol electrolyte. If you use a separate pH glass electrode and reference electrode, then store the glass electrode in water or KCl solution and the reference electrode in LiCl/Ethanol electrolyte.

Standardization (Page 1)

GLP documentation

Titration graph



Method data

Method name:	Titer KOH	Titration duration	3 m 31 s
End date:	21.09.12	End time:	15:13:54

Titration data

Sample ID:	KHPthalat	Weight	0.1209 g
Start mV:	167.9 mV	End mV:	-167.2 mV
EQ:	5.739 ml / -92.0 mV	Titer:	0.1032 mol/l

Calculation formula

Titer:	$(W \cdot F2) / ((EQ1 - B) \cdot M \cdot F1) \rightarrow M103$
Mol (M):	204.22000

Weight (W):	man	Factor 2 (F2):	1000.0000
Blank value (B):	0.0000 ml	Factor 1 (F1):	1.0000
Statistics:	Off		

## Standardization (Page 2)

### Method data

#### Method data overall view

Method name:	Titer KOH	Created at:	09/19/12 17:05:06
Method type:	Automatic titration	Last modification:	09/19/12 17:32:02
Measured value:	mV	Damping settings:	None
Titration mode:	Dynamic	Documentation:	GLP

Dynamic: Steep

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: Decrease  
Pretitration: Off  
End value: Off  
EQ: On (1)  
Slope value: Steep

Value 700

#### Dosing parameter

Dosing speed:	100 %	Filling speed	30 s
Maximum dosing volume:	50.00 ml		

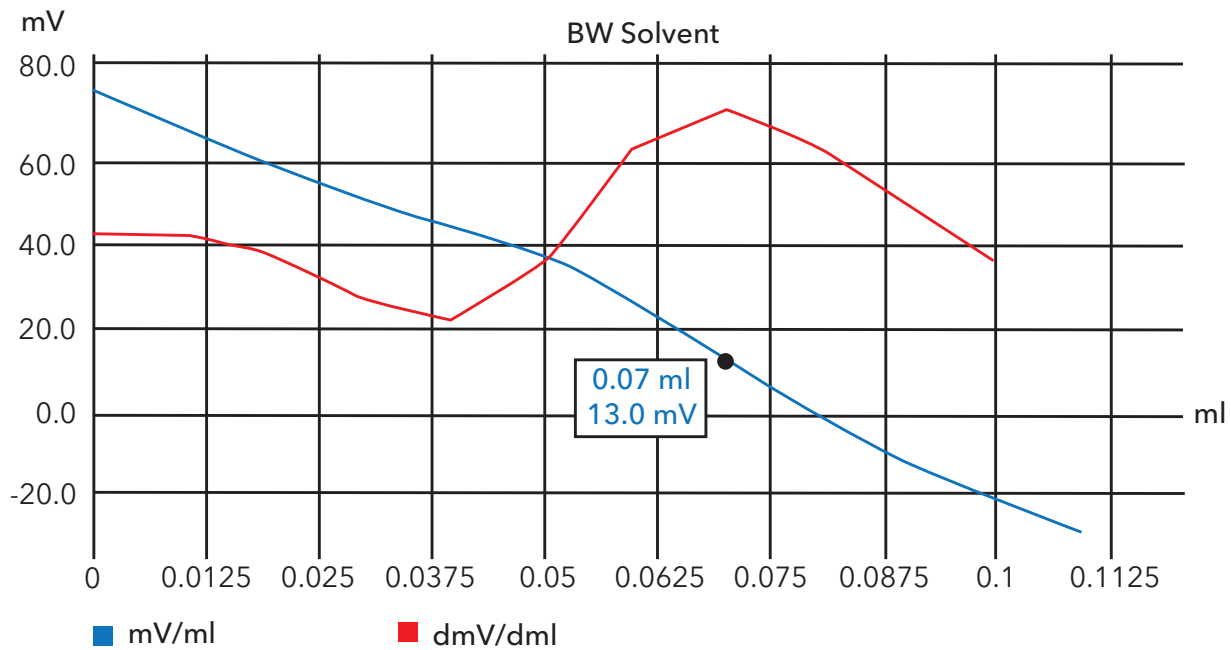
#### Unit values

Unit size: 10 ml  
Unit ID: 00072696  
Reagent: TBA Hydroxid  
Batch ID: 1.0265  
Concentration [mol/l]: 0.10365  
Determined at: 09/20/12 0:57:27  
Expire date: 04/12/12  
Opened/compounded: 10/19/11  
Test according ISO 8655: 12/01/10  
Last modification: 09/21/12 15:06:50

## Blank Value (Page 1)

### GLP documentation

Titration graph



#### Method data

Method name:	Blank TAN-TBN	Titration duration	3 m 1 s
End date:	21.09/12	End time:	16:27:20

#### Titration data

Sample ID:	BW Solvent		
Start mV:	82.7 mV	End mV:	-29.1 mV
EQ:	0.070 ml / 13.0 mV	Blank:	0.070 ml

#### Calculation formula

Blank:	EQ1 -> M01
Mol (M):	1.00000

Statistics:	Off
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## Blank Value (Page 2)

### Method data

#### Method data overall view

Method name:	Blank TAN-TBN	Created at:	09/21/12 15:29:51
Method type:	Automatic titration	Last modification:	09/21/12 16:22:36
Measured value:	mV	Damping settings:	strong
Titration mode:	Linear	Documentation:	GLP
Linear steps:	0.010 ml		

Measuring speed / drift 15 s

Initial waiting time: 10 s  
Titration direction: Decrease  
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:	0.20 ml		

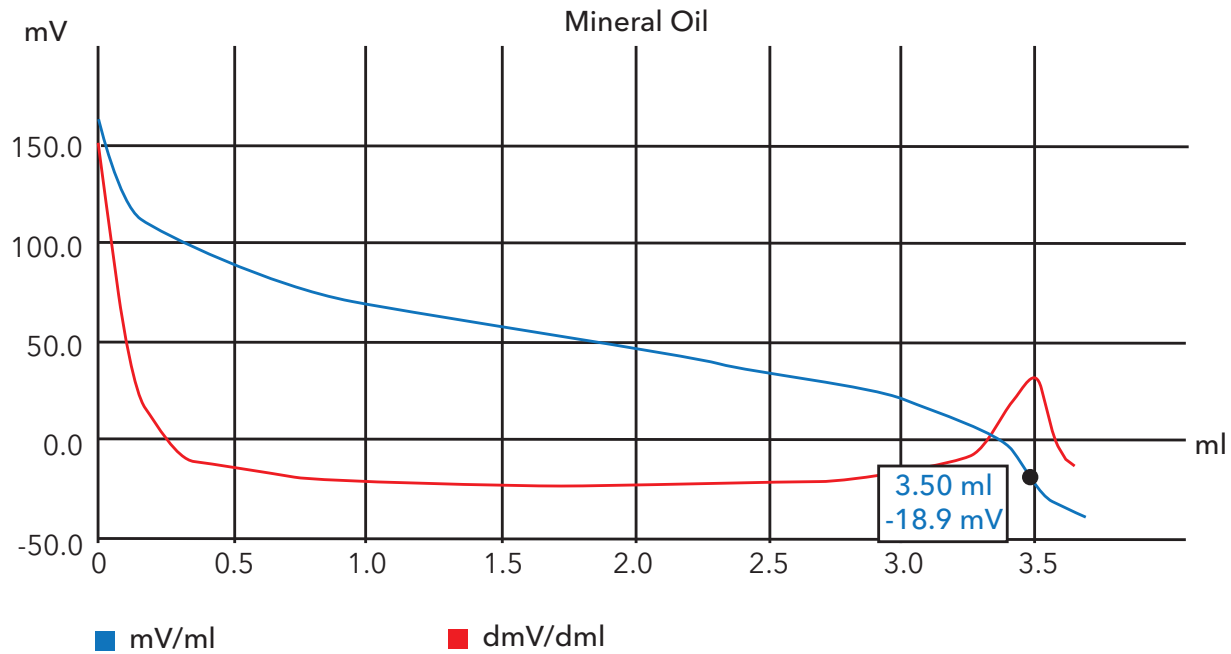
#### Unit values

Unit size: 10 ml  
Unit ID: 00072696  
Reagent: TBA Hydroxid  
Batch ID: 1.0265  
Concentration [mol/l]: 0.10350  
Determined at: 09/21/12 22:27:50  
Expire date: 04/12/12  
Opened/compounded: 10/19/11  
Test according ISO 8655: 12/01/10  
Last modification: 09/21/12 15:28:02

## Sample Titration (Page 1): Example with high or normal TAN value >1

### GLP documentation

Titration graph



#### Method data

Method name:	TAN ASTM 664	Titration duration	10 m 9 s
End date:	21.09.12	End time:	16:48:29

#### Titration data

Sample ID:	Mineral Oil	Weight:	4.0225 g
Start mV:	161.9 mV	End mV:	-39.4 mV
EQ:	3.496 ml / -18.9 mV	TAN mg KOH/g:	4.95

#### Calculation formula

TAN mg KOH/g:	$(EQ1-B)*T*M*F1/(W*F2)$
Mol (M):	56.10000

Blank value (B):	0.0700 ml (M01)	Titre (T):	0.10350000 (a)
Factor 1 (F1):	1.0000	Weight (W):	man
Factor 2 (F2):	1.0000	Statistics:	Off

## Sample Titration (Page 2): Example with high or normal TAN value >1

### Method data

#### Method data overall view

Method name:	TAN ASTM 664	Created at:	09/19/12 16:27:55
Method type:	Automatic titration	Last modification:	09/21/12 16:31:53
Measured value:	mV	Damping settings:	strong
Titration mode:	Linear	Documentation:	GLP
Linear steps:	0.050 ml		

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

Initial waiting time:	10 s		
Titration direction:	Decrease		
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:	5.00 ml		

#### Unit values

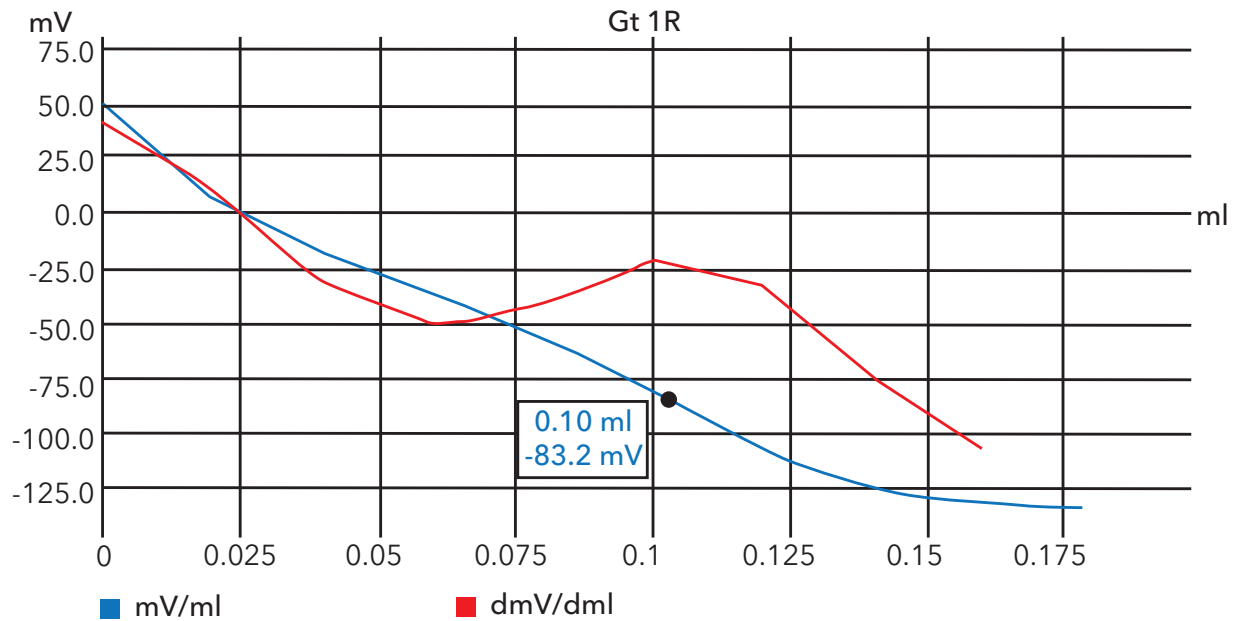
Unit size:	10 ml
Unit ID:	00072696
Reagent:	TBA Hydroxid
Batch ID:	1.0265
Concentration [mol/l]:	0.10350
Determined at:	09/21/12 22:27:50
Expire date:	04/12/12
Opened/compounded:	10/19/11
Test according ISO 8655:	12/01/10
Last modification:	09/21/12 15:28:02



## Sample Titration (Page 1): Example with low TAN value <1

### GLP documentation

Titration graph



#### Method data

Method name:	TAN ASTM 664	Titration duration	2 m 49 s
End date:	12.10.12	End time:	10:01:49

#### Titration data

Sample ID:	Gt 1R	Weight:	19.1539 g
Start mV:	49.6 mV	End mV:	-135.0 mV
EQ:	0.103 ml / -83.2 mV	TAN mg KOH/g:	0.009

#### Calculation formula

TAN mg KOH/g:	$(EQ1-B)*T*M*F1/(W*F2)$
Mol (M):	56.10000

Blank value (B):	0.0740 ml (M01)	Titre (T):	0.10580000 (a)
Factor 1 (F1):	1.0000	Weight (W):	man
Factor 2 (F2):	1.0000	Statistics:	Off

## Sample Titration (Page 2): Example with low TAN value <1

### Method data

#### Method data overall view

Method name:	TAN ASTM 664	Created at:	10/12/12 9:56:06
Method type:	Automatic titration	Last modification:	10/12/12 9:58:13
Measured value:	mV	Damping settings:	strong
Titration mode:	Linear	Documentation:	GLP
Linear steps:	0.020 ml		

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

Initial waiting time:	10 s		
Titration direction:	Decrease		
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:	1.00 ml		

#### Unit values

Unit size:	10 ml
Unit ID:	10035468
Reagent:	KOH 0.1 mol/L
Batch ID:	no entry
Concentration [mol/l]:	0.10580
Determined at:	10/12/12 16:16:25
Expire date:	---
Opened/compounded:	---
Test according ISO 8655:	---
Last modification	10/12/12 9:44:44

ASTM 664



Xylem Inc.  
1725 Brannum Lane  
Yellow Springs, Ohio 45387

Application/Technical Support:  
+ 1-845-258-1200  
titration.yisi@xylem.com

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