# Application Bulletin 405/3 e

# Total base number titration of petroleum products

# **Industry sector**

Chemical; petroleum & petrochemicals

# Keywords

Titration; nonaqueous titration; potentiometric titration; Solvotrode easyClean; dSolvotrode; Optrode; Thermoprobe; dThermoprobe; photometric titration; thermometric titration; TET; TBN; total base number; oil; petroleum products; 6.0229.010; 6.00203.300; 6.1115.000; 6.01117.300; 6.9011.020; 6.0916.040; 6.00921.040; 6.00922.040; ASTM; D2896; D974; DIN; ISO; ISO 3771; DIN ISO 6618; IP 400; S01; S010; S05; S050

# Summary

The determination of the base number plays a significant part in the analysis of petroleum products. This is manifested in the numerous standard procedures in use worldwide (e.g., internal specifications of multinational companies, national and international specifications of ASTM, DIN, IP, ISO, etc.). These procedures differ mainly in the composition of the solvents and titrants used.

This Application Bulletin describes the determination of the total base number (TBN) in petroleum products by applying different types of titration.

The potentiometric determination is described according to ISO 3771 as well as ASTM D2896, and the photometric method is adapted from ASTM D974. For the thermometric titration an ASTM standard method is in planning. Furthermore, the conductometric determination according to IP 400 is described.

# Potentiometric determination according to ISO 3771 and ASTM D2896

# Instruments and accessories

- Titrator with the mode DET U
- Stirrer
- Buret(s)

## **Electrodes**

dSolvotrode	6.00203.300
Solvotrode easyClean	6.0229.010

#### Reagents

- Potassium hydrogen phthalate (KHP), p.a.
- 2-propanol (IPA) anhydrous, p.a.
- Toluene, p.a.
- Glacial acetic acid, p.a.
- Acetone, p.a.
- CO<sub>2</sub>-free H<sub>2</sub>O

#### Solutions

Titrant	HClO <sub>4</sub> in acetic acid; c(HClO <sub>4</sub> ) = 0.1 mol/L; if possible this solution should be bought from a supplier
Solvent	600 mL toluene + 300 mL glacial acetic acid + 100 mL acetone
Electrolyte for electrode	Lithium chloride, c(LiCl) = 2 mol/L in ethanol

# Standard

Potassium	Potassium hydrogen phthalate is
hydrogen	dried at 120 °C for 2 h and cooled
phthalate	down in a desiccator for at least 1 h.



# Sample preparation

No sample preparation is required for new oils or for used oils visibly free of sediments.

For used oils containing sediments, see ISO 3771 procedure.

# Analysis

#### Titer

Approximately 100–200 mg dried potassium hydrogen phthalate is weighed into a titration vessel and 60 mL glacial acetic acid is added. The solution is then titrated using  $c(HCIO_4) = 0.1 \text{ mol/L}$  as titrant until after the equivalence point.

#### Blank

A blank titration is performed using between 60–100 mL of solvent and  $c(HClO_4) = 0.1 \text{ mol/L}$  as titrant.

The same amount of solvent must be used for the blank and sample determination.

#### Sample

An appropriate amount of well-mixed sample according to **Table 1** is weighed into a titration vessel and 60-100 mL solvent is added. The sample is then stirred until it is completely dissolved. Afterward, the titration proceeds using c(HClO<sub>4</sub>) = 0.1 mol/L until after the equivalence point.

After the titration, the electrode and buret tip are rinsed first with the solvent mixture followed by IPA and then  $CO_2$ -free  $H_2O$ . In order to rehydrate the membrane, the electrode is placed for three to five minutes in distilled  $H_2O$ . The electrode is rinsed with IPA before the next measurement.

Table 1.Suggested sample size, dependent on the expectedTBN.

TBN in mg KOH/g sample	Sample weight in g	Weighing accuracy in mg
3–15	2.00	1.0
15–30	1.00	1.0
30–45	0.25	0.1

# Parameters

#### Titer

Mode	DET U
Stirring rate	8
Meas. point distance	4
Min. volume increment	50 μL
Max. volume increment	100 µL
Signal drift	10 mV/min
Max. waiting time	60 s
Stop EP	off
EP criterion	10
EP recognition	Greatest

#### Blank

Mode	DET U
Stirring rate	8
Meas. point distance	4
Min. volume increment	10 μL
Max. volume increment	50 µL
Signal drift	10 mV/min
Max. waiting time	60 s
EP criterion	5
EP recognition	All



## Sample

Mode	DET U
Stirring rate	8
Meas. point distance	4
Min. volume increment	10 μL
Max. volume increment	0.5 mL
Signal drift	10 mV/min
Max. waiting time	20 s
Stop EP	Off
EP criterion	15
EP recognition	All

# Calculations

#### Titer

 $Titer = \frac{m_s}{V_{EP1} \times c_{HCIO4} \times M_A}$ 

- Titer: Titer of the selected titrant
- m<sub>s</sub>: Mass of standard in mg
- $V_{\text{EP1}}: \qquad \text{Titrant consumption until the first equivalence} \\ \text{point in mL}$
- $c_{HCIO4}$ : Concentration of the selected titrant in mol/L; here  $c(HCIO_4) = 0.1 \text{ mol/L}$
- M<sub>A</sub>: Molecular weight of the analyte; here 204.2 g/mol

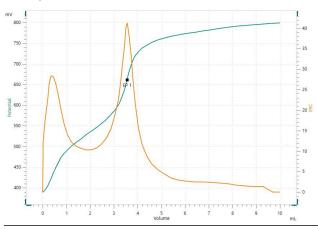
# Sample

$$TBN = \frac{(V_{last EP} - V_{blank}) \times c_{HClO4} \times f \times M_A}{m_s}$$

TBN: Total base number in mg KOH/g sample

- V<sub>last EP</sub>: Titrant consumption in mL to reach the last equivalence point (EP). Normally one EP is obtained, but in the presence of strong bases there may also be several EPs. Always use the last EP for the calculation of the base number.
- V<sub>blank</sub>: Blank value consumption for the used quantity of solvent
- $c_{HCIO4}$ : Concentration of titrant in mol/L; here  $c(HCIO_4) = 0.1 \text{ mol/L}$
- f: Correction factor (titer), dimensionless
- M<sub>A</sub>: Molecular weight of KOH; here 56.106 g/mol
- ms: Sample weight in g

# Example



**Figure 1**. Potentiometric determination of TBN (green = titration curve, orange = ERC).

#### Comments

Electrostatic charges of the electrodes and titration vessels can strongly interfere with nonaqueous titrations. These interferences are reduced to a minimum when using a Solvotrode, which was specially developed for this type of titration.

To replace the electrolyte of the electrode, first all electrolyte is drained from the electrode. The electrode is then rinsed several times with the new electrolyte before replacing the flexible sleeve diaphragm. Finally, the electrode is filled with the new electrolyte. When refitting the sleeve, make sure a free flow of the electrolyte is possible.

A performance test of the electrode can be done as follows:

The electrode is thoroughly rinsed, first with solvent then with distilled  $H_2O$ . The electrode is then placed in a pH 7.00 buffer solution (Metrohm 6.2307.110) and after stirring for one minute, the voltage (in mV) is read off. After rinsing the electrode, the same procedure is repeated in pH 4.00 buffer solution (Metrohm 6.2307.100). For a good electrode, the mV difference will be >162 mV (at 20 to 25 °C). If the difference is smaller than 162 mV, release some electrolyte and repeat the measurements.

As used oil can change appreciably when in storage, samples should be tested as soon as possible after collection. The dates of sampling and testing should be noted.



The norm ASTM D2896 is similar to ISO 3771. The differences are as follows:

 Solvent mixture of glacial acetic acid and chlorobenzene (1/2, (v/v))

As chlorinated solvents are harmful to the environment, it is not recommended to use them for analysis. ASTM D2896 therefore suggests replacing the chlorobenzene with xylene.

# References

## ASTM D2896

Standard test method for base number of petroleum products by potentiometric perchloric acid titration

#### ISO 3771

Petroleum products – determination of base number – perchloric acid potentiometric titration method

Monograph: Electrodes in Potentiometry, 8.015.5013

Leaflet: LL Solvotrode, 8.109.8046

# Photometric determination according to DIN ISO 6618 and ASTM D974

## Instruments and accessories

- Titrator with the mode MET U
- Stirrer
- Buret(s)

#### Electrode

Optrode 6.1115.000

#### Reagents

- Tris(hydroxymethyl) amino methane (TRIS), p.a.
- 2-propanol (IPA) anhydrous, p.a.
- Toluene, p.a.
- Methyl orange, puriss
- CO<sub>2</sub>-free H<sub>2</sub>O

#### Solutions

Titrant	HCl in IPA; c(HCl in IPA) = 0.1 mol/L; if possible this solution should be bought from a supplier
Solvent	500 mL toluene + 495 mL IPA + 5 mL CO <sub>2</sub> -free H <sub>2</sub> O
Methyl orange indicator solution	0.1 g methyl orange is dissolved in 100 mL CO <sub>2</sub> -free $H_2O$ .

#### Standard

TRIS	TRIS is dried at 105 °C for 1 h and
	cooled down in a desiccator for at
	least 1 h.

# Sample preparation

No sample preparation is required for new oils or for used oils visibly free of sediments.

For used oils containing sediments, see ASTM D974 procedure.



# Analysis

#### Titer

Approximately 50–60 mg dried TRIS is weighed into a titration vessel to which 100 mL CO<sub>2</sub>-free H<sub>2</sub>O and 0.5 mL methyl orange indicator are added. After waiting for 30 s, the solution is then titrated using c(HCI in IPA) = 0.1 mol/L as titrant until after the equivalence point.

## Blank

As the solvent mixtures contains weak acidic impurities, the operator must perform an acid number blank according to Application Bulletin 404.

#### Sample

An appropriate amount of well-mixed sample according to **Tables 2 and 3** is weighed into the titration vessel and 100 mL of solvent and 0.5 mL methyl orange indicator are added. The solution is stirred for 30 s in order to dissolve the sample. The sample is then titrated with c(HCI in IPA) = 0.1 mol/L until after the equivalence point.

After titration, the Optrode and buret tip are both rinsed with the solvent mixture.

**Table 2.** Suggested sample size, dependent on the expectedTBN for new or light-colored oil.

TBN in mg KOH/g sample	Sample weight in g	Weighing accuracy in mg
≤3.0	20 ± 2.0	50
>3.0 to 25	2.0 ± 0.2	10
>25 to 250	$0.2 \pm 0.02$	1.0

**Table 3.**Suggested sample size, dependent on the expectedTBN for used or dark-colored oil.

TBN in mg KOH/g sample	Sample weight in g	Weighing accuracy in mg
≤25	2.0 ± 0.2	10
>25 to 250	$0.2 \pm 0.02$	1.0

# Parameters

# Titer

Mode	MET U
Wavelength	520 nm
Stirring rate	8
Pause	30 s
Start volume	3 mL
Signal drift	20 mV/min
Max. waiting time	38 s
Volume increment	50 μL
EP criterion	30 mV
EP recognition	Greatest

#### Sample

Mode	MET U
Wavelength	520 nm
Stirring rate	8
Pause	60 s
Signal drift	20 mV/min
Max. waiting time	38 s
Volume increment	50 μL
Break point evaluation	
Selection of the type	Regular
BP criterion	0.5
Min. deflection	0.9
Max. expected BPs	16
BP recognition	First

# Calculations

## Titer

 $Titer = \frac{m_s}{V_{EP1} \times c_{HCI} \times M_A}$ 

Titer: Titer of the selected titrant

m<sub>s</sub>: Mass of standard in mg

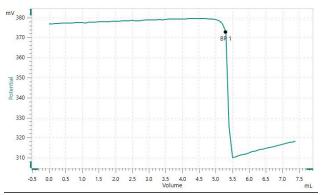
- $V_{\text{EP1}}: \qquad \text{Titrant consumption until the first equivalence} \\ \text{point in mL}$
- $c_{HCI}$ : Concentration of the selected titrant in mol/L; here c(HCl in IPA) = 0.1 mol/L
- M<sub>A</sub>: Molecular weight of the analyte; here 121.14 g/mol

# Sample

 $TBN = \frac{(V_{BP1} - V_{blank}) \times c_{HCl} \times f \times M_A}{m_s}$ 

- TBN: Total base number in mg KOH/g sample
- $V_{\text{BP1}}: \qquad \text{Titrant consumption in mL to reach the first break} \\ \text{point}$
- V<sub>blank</sub>: Blank value consumption for the used quantity of solvent
- $c_{HCI}$ : Concentration of titrant in mol/L; here c(HCI in IPA) = 0.1 mol/L
- f: Correction factor (titer), dimensionless
- MA: Molecular weight of KOH; here 56.106 g/mol
- ms: Sample weight in g

# Example





# Comments

The titration solvent usually contains weak acid impurities which react with the strongly basic components of the sample. To correct the base number for the sample, it is thus necessary to determine an acid number blank of the solvent (e.g., according to AB-404).

Titrations should be carried out at temperatures below 30 °C.

As used oil can change appreciably when in storage, samples should be tested as soon as possible after collection. The dates of sampling and testing should be noted.

The light intensity of the LED must stabilize sufficiently before use. Each time the Optrode is switched on or the wavelength is changed, wait at least five minutes before starting a determination.

The standard DIN ISO 6618 is similar to ASTM D974. For the determination of the base number, no differences are observed.

The differences to ASTM D974 are as follows:

- Methyl orange as indicator for the titration
- Titer determination with a standard instead of a standardized solution

# References

#### ASTM D974

Standard test method for acid and base number by color-indicator titration

DIN ISO 6618

Petroleum products and lubricants – determination of acid or base number – color-indicator titration method

Monograph: Complexometric (chelatometric) Titrations, 8.108.5042

Leaflet: Manual for Optrode, 8.109.8054



# Thermometric determination

# Instruments and accessories

- Titrator with the mode TET
- Rod stirrer
- Buret(s)

# Electrodes

dThermoprobe	6.01117.300
Thermoprobe	6.9011.020

# Reagents

- Potassium hydrogen phthalate (KHP), p.a.
- Isobutyl vinyl ether
- Glacial acetic acid, p.a.
- Toluene, p.a.

# **Solutions**

Titrant	Trifluoromethanesulfonic acid c(TFMSA) = 0.1 mol/L in glacial acetic acid or perchloric acid; if possible this solution should be bought from a supplier
Solvent	Toluene
Standard	

Potassium	Potassium hydrogen phthalate is
hydrogen	dried at 120 °C for 2 h and cooled
phthalate	down in a desiccator for at least 1 h.

# Sample preparation

No sample preparation is required for new oils or for used oils visibly free of sediments.

For used oils containing sediments, see ASTM D974 procedure.

## Analysis

## Titer

Between 0.04–0.07 g KHP is weighed into the titration vessel and dissolved in 20 mL glacial acetic acid. After the sample is homogenized for 2 minutes, 40 mL toluene and 1 mL isobutyl vinyl ether (or *n*-butyl vinyl ether) are added while stirring. The solution is stirred thoroughly for 20 s before titration with c(TFMSA) = 0.1 mol/L to a single endothermic end point.

Titrate at least three different values of KHP to the first endothermic end point.

#### Blank

An appropriate aliquot of the sample according to **Table 4** is weighed directly into the titration vessel, 1 mL isobutyl vinyl ether (or *n*-butyl vinyl ether) and 40 mL solvent are added. The solution is stirred thoroughly for 30 s before titration with c(TFMSA) = 0.1 mol/L to a single endothermic end point.

Titrate at least three different aliquots of the sample to the first endothermic end point.

# Sample

An appropriate aliquot of the sample according to **Table 4** is weighed into the titration vessel to which 1 mL isobutyl vinyl ether (or *n*-butyl vinyl ether) and 40 mL solvent are added. The solution is stirred thoroughly for 30 s before titration with c(TFMSA) = 0.1 mol/L to a single endothermic end point.

Table 4.Suggested sample size, dependent on the expectedTBN.

Expected TBN in mg KOH/g sample	Sample weight in g	Weighing accuracy in mg
0.05-0.99	10 ± 2.0	100
1.00-4.99	5 ± 0.5	20
5–19	1 ± 0.1	5
20–99	0.25 ± 0.02	1
100–250	0.1 ± 0.01	0.5



# Parameters

#### Titer

Mode	TET	
Stirring rate	15	
Pause	20 s	
Dosing rate	3.00 mL/min	
Stop volume	10.00 mL	
Stop slope mode	Off	
Filtering factor	70	
Damping until	0.10 mL	
Evaluation start	0.20 mL	
Sort equivalence points by	Volume (ascending)	
Define EP conditions		
End point(s)	1	
Reaction type	Endothermic	
EP criterion	1000	

# Blank and sample

Mode	TET	
Stirring rate	15	
Pause	30 s	
Dosing rate	3.00 mL/min	
Stop volume	10.00 mL	
Stop slope mode	Off	
Filtering factor	40*	
Damping until	0.20 mL	
Evaluation start	0.50 mL	
Sort equivalence points by	Volume (ascending)	
Define EP conditions		
End point(s)	1	
Reaction type	Endothermic	
EP criterion	500	

\* The filter factor depends on the sample and thus may vary. It is important that the same filter factor is used for both the blank and the sample determination.

# Calculations

# Titer

$$Titer = \frac{1}{a \times c_{TFMSA} \times M_A}$$

Titer: Titer of the selected titrant

- a: Slope of the linear regression
- $c_{TFMSA}$ : Concentration of the selected titrant in mol/L; here c(TFMSA) = 0.1 mol/L
- M<sub>A</sub>: Molecular weight of the analyte; here 204.22 g/mol

A linear regression of the different sizes of the sample (in g) against the volume of titrant consumed (in mL) is plotted automatically by OMNIS or *tiamo<sup>TM</sup>*. The titer is calculated from the slope.

# Blank

A linear regression of the different sizes of the sample (in g) against the volume of titrant consumed (in mL) is plotted automatically by OMNIS or *tiamo*<sup>TM</sup>. The blank is defined as the intercept of the linear regression line with the y-axis.

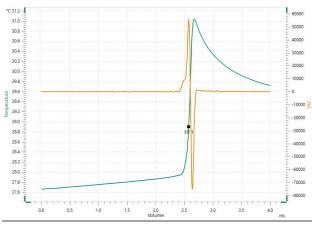
# Sample

$$TBN = \frac{(V_{EP1} - V_{blank}) \times c_{TFMSA} \times f \times M_A}{m_s}$$

- TBN: Total base number in mg KOH/g sample
- V<sub>EP1</sub>: Titrant consumption in mL to reach the first endothermic endpoint
- V<sub>blank</sub>: Blank value consumption for the used quantity of solvent
- $c_{TFMSA}$ : Concentration of the selected titrant in mol/L; here c(TFMSA) = 0.1 mol/L
- f: Correction factor (titer), dimensionless
- M<sub>A</sub>: Molecular weight of KOH; here 56.106 g/mol
- ms: Sample weight in g



# Example



**Figure 3**. Thermometric titration curve for TBN (green = titration curve, orange = ERC).

# Comments

The linear regression for the titer and the blank can be determined automatically from the results using appropriate software such as OMNIS or *tiamo*<sup>TM</sup>.

The thermometric TBN titration is a catalytic indication titration – isobutyl vinyl ether (resp. *n*-butyl vinyl ether) is used for the endpoint indication. Isobutyl vinyl ether has a strongly exothermic reaction with an excess of titrant, resulting in an endothermic endpoint.

Instead of TFMSA, perchloric acid (HClO<sub>4</sub>) can be used as titrant. If  $HClO_4$  is used as titrant, the temperature jump is not as steep as with TFMSA and the ERC becomes smaller. When using  $HClO_4$ , the obtained TBN might be smaller as it is a weaker acid than TFMSA (pKa -9 vs. pKa -20).

For more information about the titer and blank determination in *tiamo*<sup>™</sup>, see Metrohm Application Note H-131.

In a thermometric titration, the titrant reacts with the analyte in the sample either exothermically or endothermically. The Thermoprobe measures the temperature of the titrating solution. When all of the analyte in the sample has reacted with the titrant, the temperature of the solution will change, and the endpoint of the titration is revealed by an inflection in the temperature curve.

The amount of sample determined is not related to the change in temperature of the solution. Therefore, it is not necessary to use insulated titration vessels.

Thermometric titrations are conducted under conditions of constant titrant addition rate. In this respect, they differ from potentiometric titrations where the titrant addition rate may be varied during the titration according to the electrode response. In thermometric titrations, a constant addition rate of titrant equates to a constant amount of heat being emitted or absorbed, and hence a more or less constant temperature change up to the endpoint.

#### References

#### ASTM D974

Standard test method for acid and base number by colorindicator titration

AN-H-131

Determination of titer and method blank for thermometric titrations using *tiamo*<sup>™</sup>

Monograph: Practical thermometric titrimetry, 8.036.5003

Brochure: Thermometric Titration with OMNIS, 8.000.5446

Leaflet: Sensor dThermoprobe, 8.0109.8018

Leaflet: Thermoprobe, 8.109.8055



# Conductometric determination according to IP 400

# Instruments and accessories

- Titrator with the mode MET Cond
- Stirrer
- Buret(s)

# Electrodes

Conductivity measuring cell, $c = 0.1 \text{ cm}^{-1}$ with Pt1000	6.0916.040
Conductivity measuring cell, $c = 0.1 \text{ cm}^{-1}$ with Pt1000	6.00921.040
Conductivity measuring cell, $c = 0.1 \text{ cm}^{-1}$ with Pt1000	6.00922.040
5-ring conductivity measuring cell, c = 0.7 cm <sup>-1</sup> with Pt1000	6.00925.100
5-ring conductivity measuring cell, $c = 0.7 \text{ cm}^{-1}$ with Pt1000	6.0920.100

# Reagents

- Sodium carbonate, p.a.
- 2-propanol, isopropanol, IPA, p.a.
- Ethanol, p.a.
- Toluene, p.a.
- CO<sub>2</sub>-free H<sub>2</sub>O

# **Solutions**

Titrant	HCl in IPA; c(HCl in IPA) = 0.1 mol/L; if possible this solution should be bought from a supplier
Solvent	500 mL toluene + 495 mL IPA + 5 mL $CO_2$ -free $H_2O$

# Standard

Sodium	Sodium carbonate is dried at
carbonate	120 °C for 2 h and cooled down in
standard	a desiccator for at least 1 h.
solution	A standard solution is prepared by dissolving approximately 1 g Na <sub>2</sub> CO <sub>3</sub> in 100 mL CO <sub>2</sub> -free H <sub>2</sub> O.

# Sample preparation

No sample preparation is required for new oils or for used oils visibly free of sediments.

For used oils containing sediments, see IP 400 procedure.

# Analysis

# Titer

Between 1–3 mL of the standard solution and 75 mL of  $CO_2$ -free H<sub>2</sub>O are dosed into a titration vessel. The solution is stirred for 30 s, then the conductivity cell is dipped three times into the sample solution in order to avoid air bubbles before titrating with c(HCl) = 0.1 mol/L.

After the titration, the buret tip and conductivity measuring cell are rinsed with solvent until there is no more visible oil present. The conductivity measuring cell is then rinsed with CO<sub>2</sub>-free  $H_2O$ , followed by the solvent.

#### Sample

An appropriate amount of sample according to the following calculation is weighed into the titration vessel. Then 75 mL solvent is added and the conductivity cell is placed in the titration vessel. The solution is stirred for 30 s to dissolve the sample, then the conductivity cell is dipped three times into the sample solution before titrating with c(HCI) = 0.1 mol/L.

After the titration, the buret tip and conductivity measuring cell are rinsed with solvent until there is no more oil visible. The conductivity measuring cell is then rinsed with CO<sub>2</sub>-free  $H_2O$ , followed by solvent.

$$m_s = \frac{10}{\text{TBN}_{\text{expected}}}$$

ms:	Sample weight in mg
10:	Conversion factor
TBN <sub>expected</sub> :	Expected TBN in mg KOH/g sample



# Parameters

#### Titer

Mode	MET COND
Stirring rate	8
Pause	30 s
Signal drift	off
Min. waiting time	10 s
Max. waiting time	10 s
Volume increment	0.1000 mL
Temperature compensation	Constant factor
Reference temperature	25.0 °C
Temperature coefficient	2.00 %/°C
Stop volume	10.00 mL
Evaluation start	0 mL
Smoothing factor	6
Extension of the linear range	10 %
Weighting factor	5
EP criterion	100
EP recognition	All

# Sample

Mode	MET COND
Stirring rate	8
Pause	30 s
Signal drift	off
Min. waiting time	10 s
Max. waiting time	10 s
Volume increment	0.1000 mL
Temperature compensation	Constant factor
Reference temperature	25.0 °C
Temperature coefficient	2.00 %/°C
Stop volume	10.00 mL
Evaluation start	0 mL
Smoothing factor	6
Extension of the linear range	10 %
Weighting factor	5
EP criterion	100
EP recognition	All

# Calculations

# Titer

# $Titer = \frac{V_{Std} \times c_{Std} \times 2}{V_{EP1} \times c_{HCI}}$

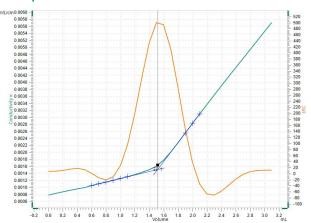
- Titer: Titer of the selected titrant
- V<sub>std</sub>: Added volume of standard solution in mL
- cstd: Exact concentration of standard solution in mol/L
- 2: Stoichiometric factor
- $V_{\text{EP1}}: \qquad \text{Titrant consumption until the first equivalence} \\ \text{point in mL}$
- $c_{HCl}$ : Concentration of the selected titrant in mol/L; here c(HCl) = 0.1 mol/L

# Sample

 $TBN = \frac{V_{EP1} \times c_{HCI} \times f \times M_A}{m_s}$ 

- TBN: Total base number in mg KOH/g sample
- $V_{\text{EP1}}: \qquad \text{Titrant consumption in mL to reach the first} \\ \text{equivalence endpoint}$
- $c_{HCI}$ : Concentration of the selected titrant in mol/L; here c(HCI) = 0.1 mol/L
- f: Correction factor (titer), dimensionless
- MA: Molecular weight of KOH; here 56.106 g/mol
- ms: Sample weight in g

# Example



**Figure 4**. Coulometric titration curve for TBN (green = titration curve, orange = ERC, blue = tangents).

# Comments

This method may be used for samples which have a base number of up to 40 mg KOH/g. However, it will be necessary to dilute these samples in order to obtain a suitable sample weight. This will only give an approximate base number because this method is only validated in the range of 1 to 20 mg KOH/g.

The titration parameters correspond to the IP 400 norm where a manual titration is performed. With OMNIS and the 5-ring conductivity measuring cell, the titration can be sped up considerably without any loss of accuracy. A waiting time of one to two seconds between titrant additions is sufficient.

The stirring speed should be selected in such a way that no air is introduced into the titration solution. This results in a turbulent titration curve.

# References

#### IP 400/01

Determination of base number of petroleum products – conductometric titration method

Monograph: Conductometry – Conductivity measurement, **8.109.5021** 

Leaflet: Conductivity measuring cells, 8.109.8051

# Author

Competence Center Titration

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