

Determination of Total base number (TBN) according to ISO 3771

Description

Determination of basic compounds in petroleum products and lubricants by potentiometric titration with Perchloric acid in Glacial acetic acid.

The total base number TBN is the quantity of HClO_4 , which is required to neutralize all basic constituents in 1 g of sample. The TBN is not expressed in $\text{mg}_{(\text{HClO}_4)} / \text{g}$ but expressed in milligrams of potassium hydroxide (or alternatively in milliequivalents of hydroxide) per gram sample. The titration is done in a mixture of Glacial acetic acid and Chlorobenzene with Perchloric acid in Glacial acetic acid.

The result is calculated as $\text{mg}_{(\text{KOH})} / \text{g}$.

Instruments

Titration	TL 7000 or higher
Electrode	N 6480 eth or N 6480 eis
Cable	L 1 A
Stirrer	Magnetic stirrer TM 235 or similar
Titration tip	TZ 1643 (180 mm)
Lab accessory	glass beaker 150 ml
	Magnetic stirrer bar 30 mm

Reagents

1	HClO_4 0,1 mol/l in Glacial acetic acid
2	Glacial acetic acid
3	Chlorobenzene
4	Distilled water
All reagents should be of analytical grade or better.	

Titration procedure

Reagents

HClO₄ in Glacial acetic acid 0.1 mol/l

It is recommended to use a ready-to-use 0.1 mol/l solution.

The titer determination is done as described in the application note "Titer HClO₄".

Solvent mixture

1 Part Glacial acetic acid and 2 Parts Chlorobenzene are mixed in a bottle.

Cleaning of the electrode

For cleaning and conditioning of the electrode 3 steps are necessary:

First the electrode is rinsed with the solvent mixture to remove residues of the sample.

Then it is conditioned in water.

After the conditioning step the electrode is rinsed with solvent mixture to remove the water.

The electrode is stored in a solution of 1.5 mol/l LiCl in Ethanol (or, if another electrolyte is used, in this electrolyte solution).

Blank value

For blank titration 120 ml solvent are placed in a 250 ml beaker and titrated with 0.1 mol/l HClO₄. The Blank should be below 0.3 ml.

Sometimes the Blank is very low so that no EQ is found. Then the Blank must be estimated and set manually.

Sample preparation

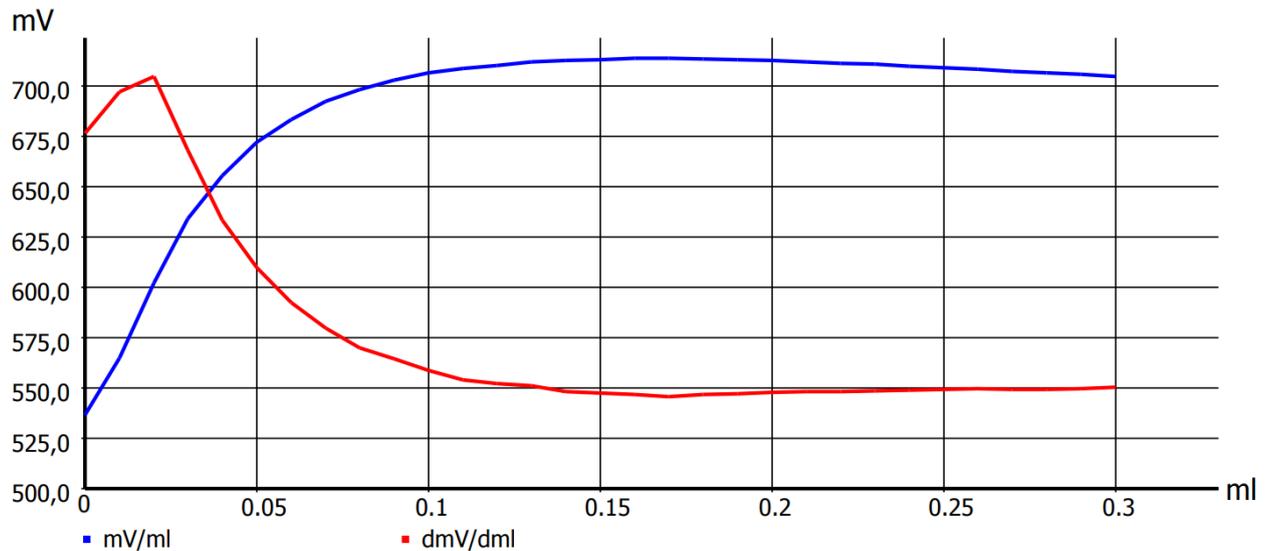
The sample is weighed into a 250 ml beaker, dissolved in 120 ml of the solvent and titrated with 0.1 mol/l HClO₄.

The required amount of sample can be estimated according to this rule of thumb:

$$W(g) = \frac{28}{\text{expected TBN}}$$

Titration parameter

Blank titration



Default method	Blank TAN-TBN		
Method type	Automatic titration		
Modus	linear		
Measured value	mV		
Measuring speed / drift	User defined	Fixed delay time	12 s
Initial waiting time	10 s		
Linear Steps	0.01 ml		
Damping	strong	Titration direction	increase
Pretitration	off	Delay time	0 s
End value	off		
EQ	On (1)	Slope value	60
Max. titration volume	0.3 ml		
Dosing speed	100%	Filling speed	30 s

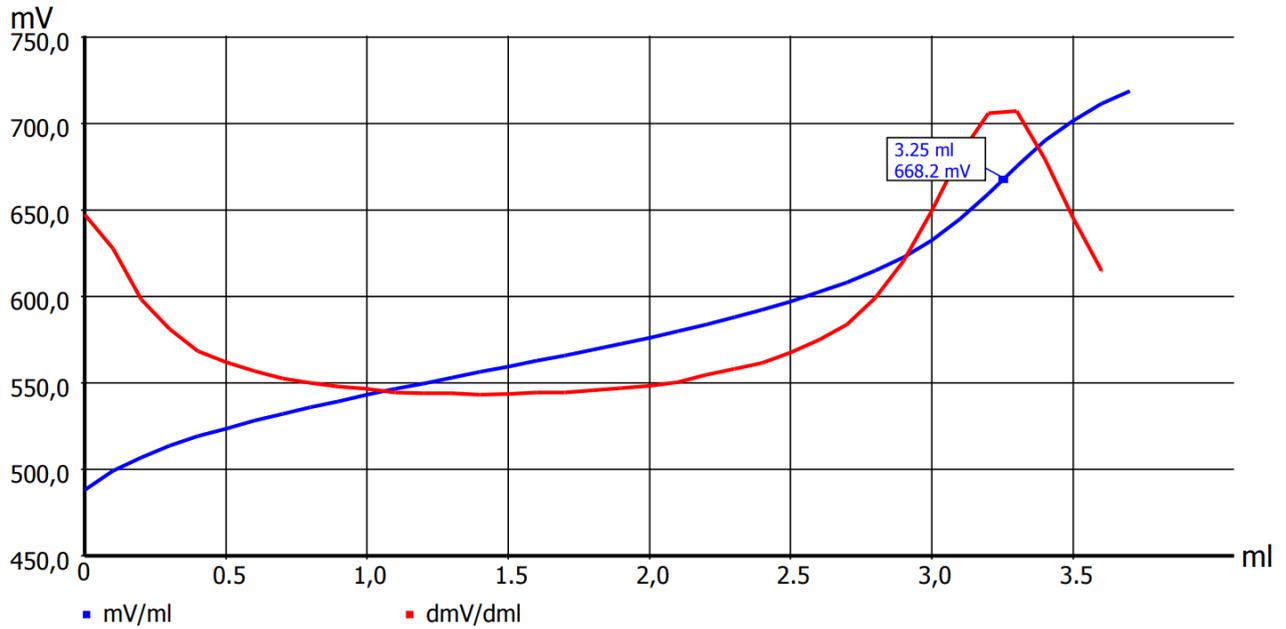
Calculation:

$$ml = EQ1$$

The result is saved in a global memory, e.g. M01. We recommend to use statistics = 3.

Sometimes the Blank is very low so that no EQ is found. Then the Blank must be set manually. In our example, one would estimate a blank of about 0.02ml.

Sample titration



Default method	TBN ISO 3771		
Method type	Automatic titration		
Modus	linear		
Measured value	mV		
Measuring speed / drift	User defined	Minimum holding time	7 s
		Maximum holding time	20 s
		Measuring time	4 s
		Drift	10 mV/min
Initial waiting time	10 s		
Linear Steps	0.1 ml		
Damping	strong	Titration direction	increase
Pretitration	off	Delay time	0 s
End value	off		
EQ	On (1)	Slope value	120
Max. titration volume	6 ml		
Dosing speed	100%	Filling speed	30 s

Calculation:

$$TBN [mg(KOH)/g] = \frac{(EQ1 - B) * T * M * F1}{W * F2}$$

B	M01	Blank value, saved in global Memory M01
EQ1		Consumption of titrant at first Equivalence point
T	WA	concentration of the titrant
M	56.11	Molecular mass
W	man	Weight of the sample in g
F1	1	Conversion factor
F2	1	Conversion factor

Any questions? Please contact the application team:

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