Metrohm

Thermo. Titr. Application Note No. H-096

Title:	Determination of Total Base Number of Lubricating Oils

Scope:	Determination of Total Base Number in lubricating oils. The titration is suitable for full automation using an 814 Sample Processor.

Principle:	Dissolution of oil in toluene, and titration with standard 0.1mol/L trifluoromethanesulfonic acid in acetic acid using isobutyl vinyl ether as a thermometric endpoint indicator. (*Ref.*: E. J. Greenhow and L. E. Spencer (1973) *Ionic polymerisation as a means of endpoint indication in non-aqueous thermometric titrimetry. Part 1. The determination of organic bases.* Analyst, **98**, 81-89)

Reagents:	*Titrant:* 0.1mol/L trifluoromethanesulfonic acid in glacial acetic acid – Riedel de Haën (Sigma-Aldrich) cat. no.35317 *Endpoint indicator:* Isobutyl vinyl ether Aldrich cat. no. 278351 *Solvent:* Toluene *Test samples:* Customer-supplied lubricating oils

Method:	Basic Experimental Parameters:
	Titrant delivery rate (mL/min.) 4
	No. of exothermic endpoints 1
	Data smoothing factor (DSF) 70
	Stirring speed (802 stirrer) 12
	2-3g lubricating oil is weighed accurately into a titration cup and placed in the sample rack of the 814 Sample Processor. The titration program first adds 35mL toluene from a Dosino, then 1mL isobutyl vinyl ether from another Dosino. The stirrer starts, and after a programmed delay to allow full sample dissolution, the titration commences. The titration endpoint is marked by a strong upsurge in temperature.
	In the case of marine grade lubricating oils with high TBN values (70-80 mg KOH/g), a sample mass of 0.3–0.4g is indicated. For highest precision and accuracy, 3-4g is

weighed accurately into a 200mL volumetric flask, made to volume with anyhydrous toluene and mixed well. A 20mL aliquot is taken, and the titration program adjusted to pre-dose just 15mL toluene to take the total volume to 35mL.

For highest accuracy, it is recommended that separate blank determinations are made for oils of different type and specification, and titration methods created for these oils are created, differing only in the titration blank factor.

Examples:	*Samples of customer-supplied lubricating oils*	
	Sample No.	**Total Base Number, mg KOH/g oil**
	LU 0801	8.49±0.03 (n=10)
	LU 0705	8.00±0.01 (n=6)
	LU 0601	9.50±0.04 (n=5)
	LU 0609	7.82±0.01 (n=7)
	"Used oil"	4.78±0.01 (n=7)
	"Fresh marine cylinder oil BN 70"	71.17±0.13 (n=7)

Calculation:

$$\mathrm{mg\,KOH/g} = \frac{((\mathrm{Titre, mL} - \mathrm{blank, mL}) \times CF_3SO_3H\,\mathrm{mol/L} \times 56.11)}{(\mathrm{sample\,mass, g})}$$

Thermometric Titration Plot:

Legend:
Red = solution temperature curve
Black = second derivative curve (for endpoints)

TBN by F3CSO3H - C:\Program Files\Titrotherm\MDF\TBN with IBVE-5.MDF

EP1: 3.348ml
IP1,D2 Min -G

Ω Metrohm
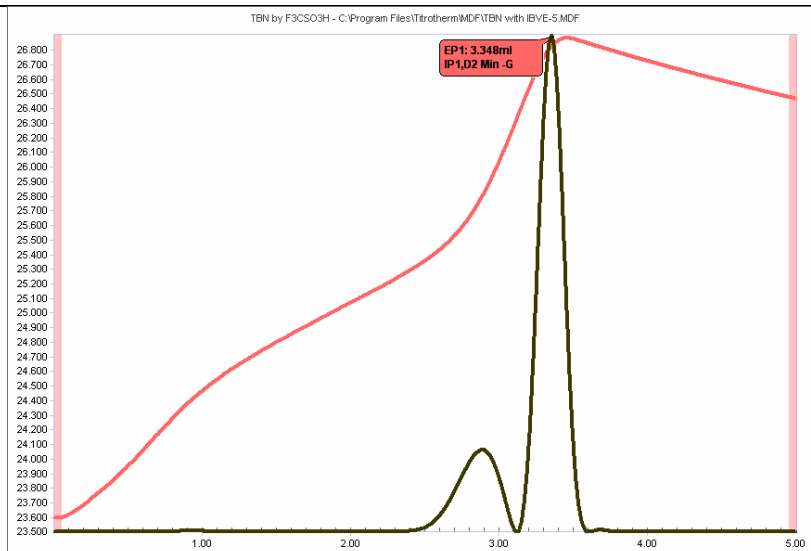
Determination of blank: Example:

Blank determination - oil sample LU 0609

$y = 1.3944x + 0.2761$
$R^2 = 0.9999$

(plot: mL CF$_3$SO$_3$H vs g sample)

Titration blank =
y-intercept = 0.0.276mL

Determination of the titration blank is not necessary for every sample – only for each specific type and specification
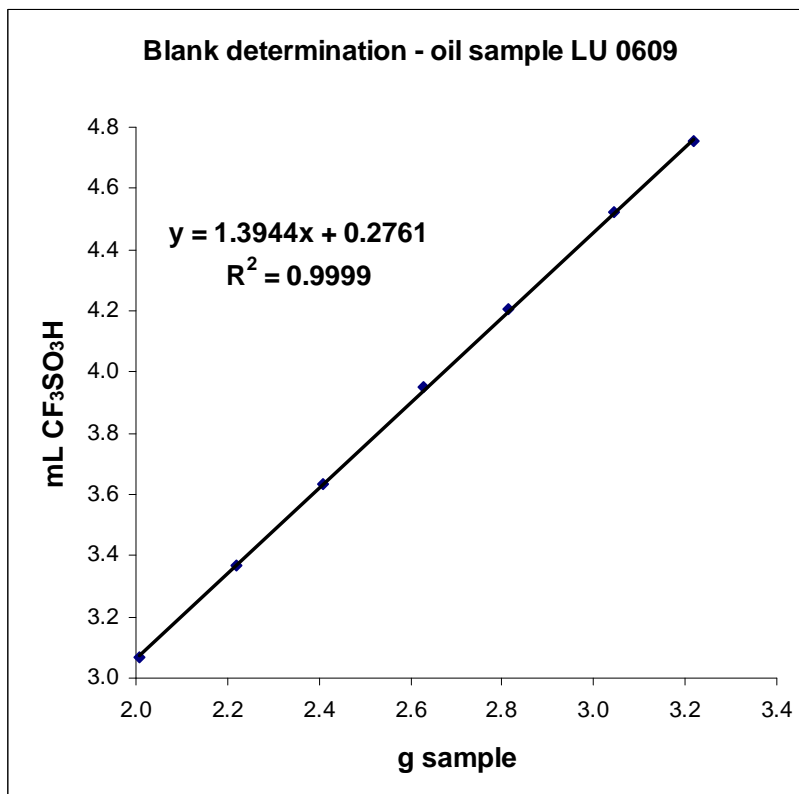
Sample	Titration Blank, mL
LU 0801	0.303
LU 0705	0.336
LU 0601	0.674
LU 0609	0.276
"Used oil"	0.052
"Fresh marine cylinder oil BN 70"	0.113