Determination of Acidity in Insulating Oils according IEC 62021-1

Description

This application note describes the determination of the acidity of unused and used electrical mineral insulating oils. The sample is dissolved in 2-propanol and titrated with alcoholic KOH 0.05 mol/l to a pH value of 11.5.

Instruments

Titrator	TL 7000 or higher
Interchangeable unit	WA 10
Electrode	N 64 or N 6480 with aqueous KCl electrolyte
Stirrer	Magnetic stirrer TM 235
Lab accessory	Glass beaker 100 ml
	Magnetic stirring bars
	Balance

Reagents

1	KOH in 2-propanol, 0.1 mol/L
2	2-Propanol (isopropanol, IPA)
3	Potassium acid phthalate, volumetric standard
4	Deionized water
5	KCl electrolyte solution 3 mol/l
6	Buffer solutions pH 4, pH 7 and pH 10.01 (or pH 11)
All reagents should be of reagent grade or better.	

Standardization of Potassium Hydroxide Solution

Standard volumetric alcoholic solution potassium hydroxide 0.05 mol/l.

Add 3,0 g of potassium hydroxide to 1 000 ml ± 10 ml of 2-propanol. Boil gently for 10 min to effect solution. Cool and stopper the flask. Allow the solution to stand in the dark for 2 days and then filter the supernatant liquid through a 5 µm membrane filter. Store in a suitable amber glass bottle.

We recommend to use either ready to use alcoholic KOH 0.05 mol/l titrant solution or prepare it from a KOH 0.1 mol/l in 2-propanol by diluting 1/1 with 2-propanol

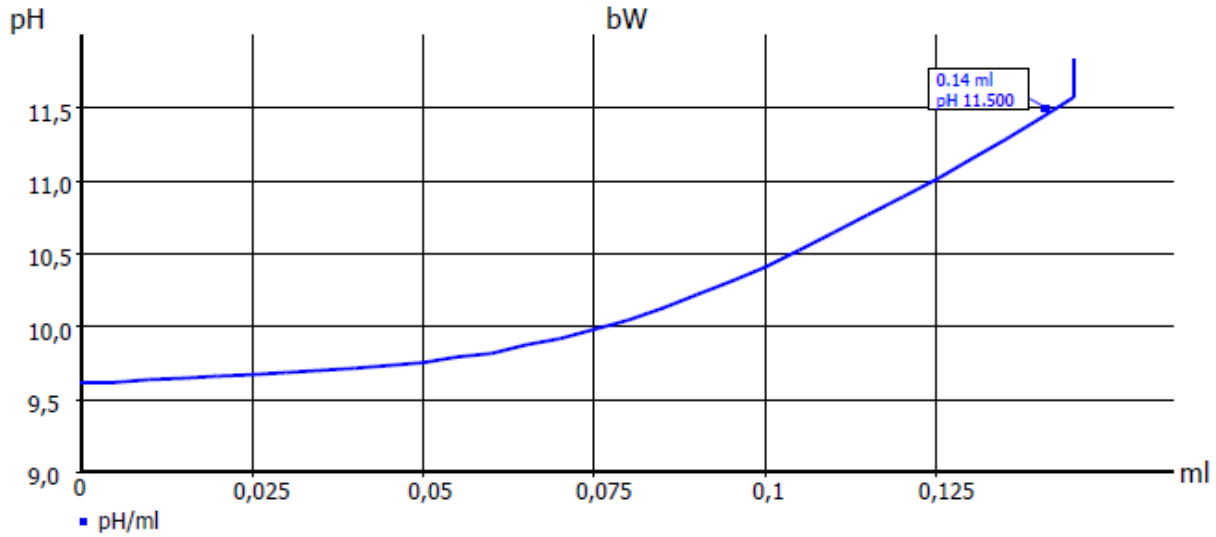
Please refer to the standard method Titer KOH and the application note “Titer strong bases”.

Titration solvent

2-propanol (isopropanol, IPA).

Blank value determination

Perform a blank titration on 40 ml titration solvent. Open the refilling hole of the N 64 and carefully move the ground joint of the electrode so that 1-2 drops of the KCl solution flow out. Rinse the electrode with water and then immerse the electrode and the titration tip into the solvent. Start stirring and then start the blank method.



Default method	-		
Method type	Automatic Titration		
Modus	Linear		
Measured value	pH		
Measuring speed / drift	User defined	Minimum holding time	5 s
		Maximum holding time	15 s
		Measuring time	2 s
		Drift	10 mV/min
Initial waiting time	20 s		
Linear steps	0.01 or 0.005ml		
Damping	average	Titration direction	increase
Pretitration	off ml	Delay time	0 s
End value	pH 11.5		
EQ	Off	Slope value	-
Max. titration volume	0.3 ml		
Dosing speed	100%	Filling speed	30 s

Calculation:

$$Result\ ml = X\ at\ Y\ [11.5]$$

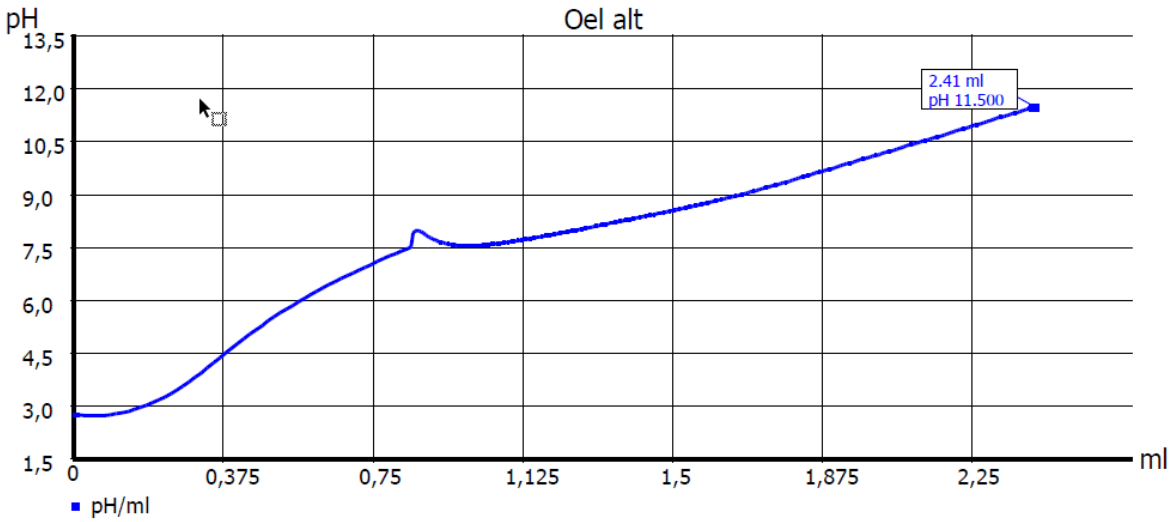
ml x at y		Consumption of titrant at a fixed pH value 11.5

The result is stored as global variable M01.

When the titration is finished rinse the electrode and titration tip with fresh solvent and then with water. After a couple of titrations it is recommend to put the electrode in KCl solution for re-hydration for 1-2 hours.

Sample titration

Weigh 5 g into the 100 ml beaker and add 40 ml titration solvent. Add a stirrer bar, and swirl until the sample is completely dissolved. Open the refilling hole of the N 64 and carefully move the ground joint of the electrode so that 1-2 drops of the KCl solution flow out. Rinse the electrode with water and then immerse the electrode and the titration tip into the solvent. Start stirring and then start the sample method.



Default method	-		
Method type	Automatic Titration		
Modus	Linear		
Measured value	pH		
Measuring speed / drift	User defined	Minimum holding time	5 s
		Maximum holding time	15 s
		Measuring time	2 s
		Drift	10 mV/min
Initial waiting time	20 s		
Linear steps	0.02 – 0.05 (new and used oil)		
Damping	average	Titration direction	increase
Pretitration	off ml	Delay time	0 s
End value	pH 11.5		
EQ	Off	Slope value	-
Max. titration volume	10 ml		
Dosing speed	100%	Filling speed	30 s

Maybe use the manual stop!

Calculation:

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

B	M01	Blank value
ml at y		Consumption of titrant at fixed pH 11.5
T	WA	Exact concentration of the titrant in mol/l
M	56.1	Molecular weight
W	man	Sample weight in g
F1	1	Conversion factor = 1
F2	1	Conversion factor = 1

Any questions? Please contact the application team:

Xylem Analytics Germany Sales GmbH & Co. KG, SI Analytics
Hattenbergstraße 10
D-55122 Mainz, Germany
Telefon: + 49 6131 66 5126
Fax: + 49 6131 66 5101
E-Mail: titration@si-analytics.com

SI Analytics
a xylem brand

Xylem Analytics Germany Sales GmbH & Co. KG ·Hattenbergstr. 10 · D-55122 Mainz · Germany Telefon: +49 6131.66. 5111 · E-Mail: Info.si-analytics@Xyleminc.com · **www.si-analytics.com**

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