Determination of Acid Number by Color-Indicator Titration (ASTM D 974)

Description

The determination described in this report is based on the standard ASTM D 974.

Instruments

Titrator	TL 7000 or higher
Interchangeable unit	WA 10
Electrode	OptiLine 6
Stirrer	Magnetic stirrer TM 235
Lab accessory	Glass beaker 150 ml
	Magnetic stirring bars
	Balance

Reagents

1	KOH in 2-propanol, 0.1 mol/L
2	Toluene
3	2-Propanol
4	Deionized water
5	p-Naphtholbenzein indicator solution
6	Potassium acid phthalate
8	Phenolphthalein indicator solution
All reagents should be of reagent grade or better.	

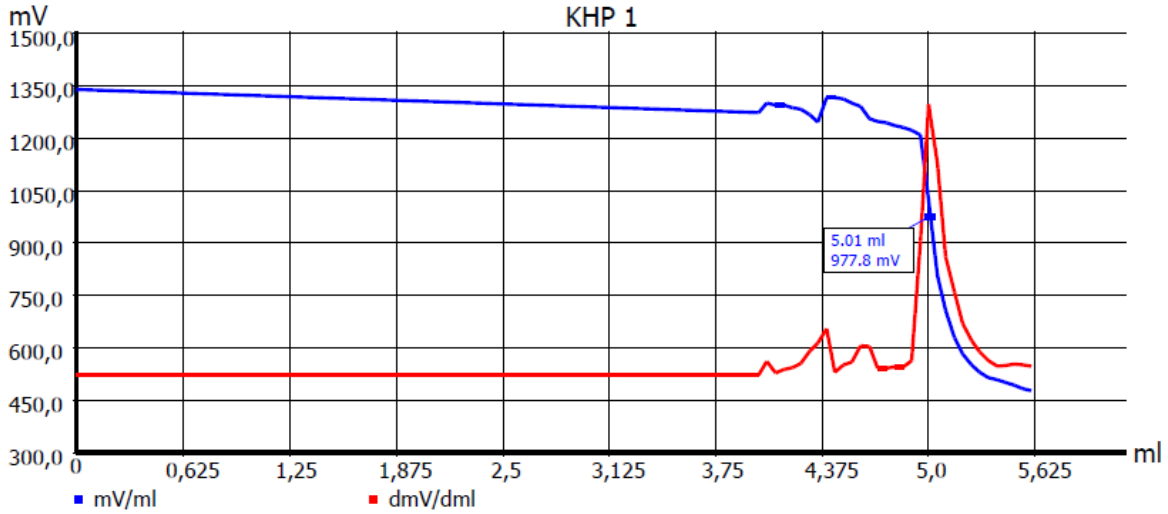
Standardization of Potassium Hydroxide Solution

The standardization can be carried out either potentiometric or colorimetric.

If a pH electrode is available. Please refer to the standard method Titer KOH and the application note “Titer strong bases”.

The colorimetric indication is possible with phenolphthalein indicator solution. Weigh to the nearest 0.1 mg approximately 0.1 g of potassium acid phthalate, which has been dried for at least 1 h at 110 °C and dissolve in 80-100 mL of water, free of CO_2. Add six drops of the phenolphthalein indicator solution and titrate with linear titration steps to the inflection point. For highest accuracy repeat the titer determination three times and calculate the mean value using the statistic function of the titrator

See the titration curve and method below.



Default method	-		
Method type	Automatic Titration		
Modus	Linear		
Measured value	mV-E, 520 nm		
Measuring speed / drift	User defined	Minimum holding time	05 s
		Maximum holding time	15 s
		Measuring time	3 s
		Drift	10 mV/min
Initial waiting time	10 s		
Linear steps	0.05 ml		
Damping	None	Titration direction	decrease
Pretitration	6.00 ml	Delay time	30 s
End value	Off		
EQ	Off	Slope value	-
Max. titration volume	12 ml		
Dosing speed	100%	Filling speed	30 s

Calculation:

$$T\ [mol/l] = \frac{W * F2}{(EQ - B) * M * F1}$$

B	0	Blank value
W	man	Weight of the sample [g]
F2	1000	Conversion factor ml - l
EQ1		Consumption of titrant until first Equivalence point
M	204,22	Molecular mass
F1	1	Conversion factor

We recommend to store the exact concentration T into the exchangeable Unit (WA) automatically.

Titration solvent

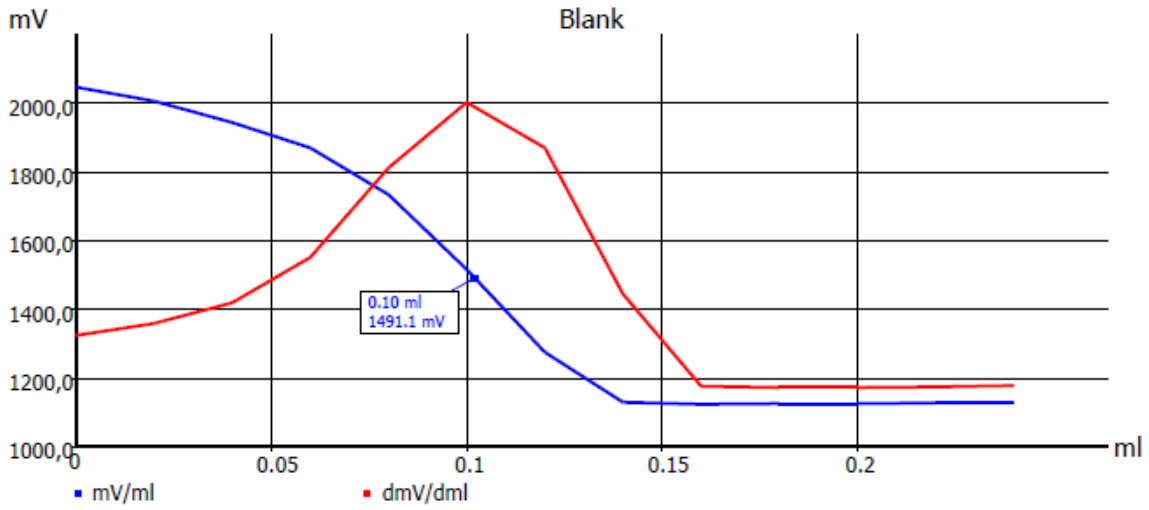
Prepare by mixing toluene, water, and anhydrous isopropyl alcohol in the ratio 100/1/99.

Indicator solution

Prepare a solution of p-naphtholbenzein in titration solvent equal to 10 g/L.

Blank value determination

Perform a blank titration on 100 ml titration solvent and 0.5 mL of the p-Naphtholbenzein indicator solution.



Default method	-		
Method type	Automatic Titration		
Modus	Linear		
Measured value	mV-E, 625 nm		
Measuring speed / drift	User defined	Minimum holding time	10 s
		Maximum holding time	40 s
		Measuring time	4 s
		Drift	20 mV/min
Initial waiting time	10 s		
Linear steps	0.02 ml		
Damping	None	Titration direction	decrease
Pretitration	off ml	Delay time	0 s
End value	Off		
EQ	Off	Slope value	-
Max. titration volume	0.3 ml		
Dosing speed	100%	Filling speed	30 s

Calculation:

$$Result\ ml = EQ1$$

EQ1		Consumption of titrant at the first Equivalence point

The result is stored as global variable M01.

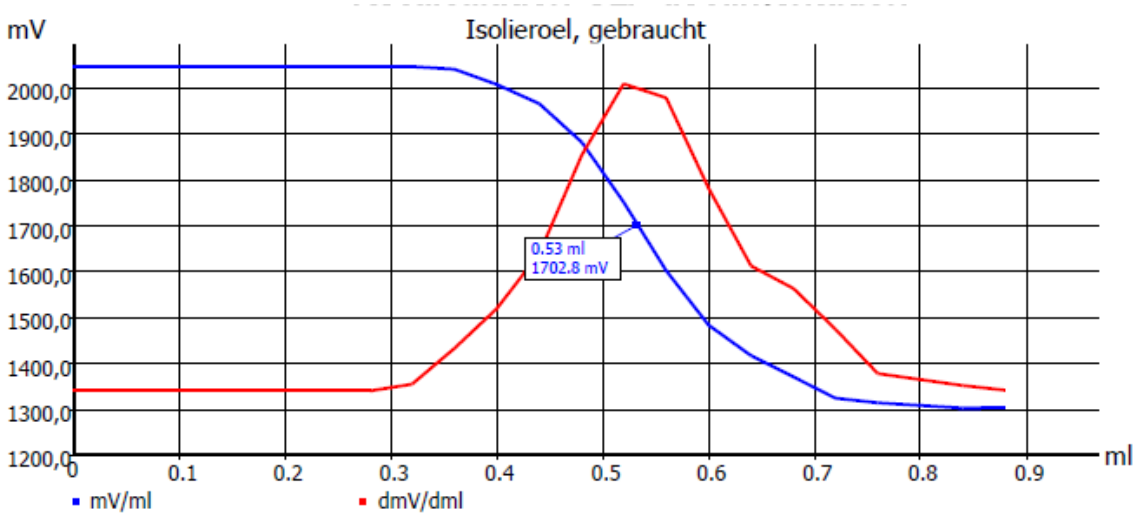
Sample titration

The sample is weight in into a 150 ml beaker. The amount of sample weight is adjusted to the expected acid value.

Acid number (new and Light oils)	Size of sample [g]	Sensitivity of Weighing [g]
0 – 3	20.0 +/- 2.0	0.05
Over 3 to 25	2.0 +/- 0.2	0.01
Over 25 to 250	0.2 +/- 0.02	0.001
Used or Dark-Colored Oils		
0 to 25	2.0 +/- 0.2	0.01
Over 25 to 250	0.2 +/- 0.02	0.001

To the sample 100 ml of solvent and 0.5 ml of the indicator are added. Swirl until the sample is completely dissolved. The color of the indicator must be yellow-orange. If the mixture assumes a green or green-black color then the sample does not have any acid number and the base number can only be determined.

Immerse the OptiLine 6 and the titration tip (if not already happened) and start the titration method.



Default method	-		
Method type	Automatic Titration		
Modus	Linear		
Measured value	mV-E, 625 nm		
Measuring speed / drift	User defined	Minimum holding time	10 s
		Maximum holding time	40 s
		Measuring time	4 s
		Drift	20 mV/min
Initial waiting time	10 s		
Linear steps	0.04 – 0.05 ml		
Damping	None	Titration direction	decrease
Pretitration	off ml	Delay time	0 s
End value	Off		
EQ	Off	Slope value	-
Max. titration volume	6 ml		
Dosing speed	100%	Filling speed	30 s

Maybe use the manual stop!

Calculation:

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

B	M01	Blank value
EQ1		Consumption of titrant at the first Equivalence point
T	WA	Actual Concentration of the titrant
M	56.1	Molecular weight
W	man	Sample weight in g
F1	0.1	Conversion factor 1
F2	1	Conversion factor 1

Any questions? Please contact the application team:

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