

Determination of Acid number and free fatty acids (FFA) in fats and oils

Description

Free fatty acids (FFA) in vegetable oils and fats (e.g. edible oils and fats) influence the quality of these fats. Fats with a high content of free fatty acids can have a bad smell and taste, they are rancid. The free acids are formed during the ageing of the fats by splitting the fatty acid glycerol esters by hydrolysis or oxidation. The determination of FFA in oils and fats according to DIN ISO 660 is done by potentiometric titration in ethanol/diethyl ether with KOH in isopropyl alcohol.

The method is suitable for edible fats and oils such as butter, olive, palm or sunflower oil. The acid number is the quantity of base, expressed in milligrams of potassium hydroxide, which is required to neutralize all acidic constituents present in 1 g of sample. The calculation of % FFA depends on the type of titrated sample and the fatty acid to which the result is to be calculated.

The result is calculated as $\text{mg}_{(\text{KOH})} / \text{g}$ or as $\%_{\text{fatty acid}}$ (mainly as % Oleic acid with $M_{\text{oleic acid}} = 282,47 \text{ g/mol}$).

Instruments

Titration	TL 7000 or higher
Exchangeable head	WA 10
Electrode	N 6480 eth
Cable	L1A
Stirrer	Magnetic stirrer TM 235
Titration tip	TZ 1643
Lab accessory	glass beaker 150 ml
	Magnetic stirrer bar 30 mm

Reagents

1	KOH 0,1 mol/L in Isopropylalcohol (or KOH in Methanol or Ethanol)
2	Ethanol, absolute or Isopropylalcohol.
3	Diethylether, free of Peroxide. alternatively: tert-Butylmethylether oder Toluene
4	Distilled water
5	Electrolyte L 5034
All reagents should be of analytical grade or better.	

Titration procedure

Reagents

KOH in Isopropyl alcohol (IPA) 0.1 mol/l

It is recommended to use a ready 0.1 mol/L solution. KOH in methanol or ethanol can also be used. The solution must be protected against CO₂ with a CO₂ absorbent like soda lime. The titer determination is done as described in the application note "Titer KOH".

Solvent mixture

500 ml absolute Ethanol and 500 ml Diethyl ether are mixed in a bottle. Isopropanol can also be used instead of Ethanol. Instead of Diethyl ether, tert-Butyl methyl ether or Toluene can also be used.

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 rinsed 1min with water for conditioning.
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 the blank titration about 70 ml solvent are added in a 150 ml beaker and titrated with 0.1 mol/L KOH in IPA to one EQ. The blank value should be below 0.3 ml.

Sample preparation

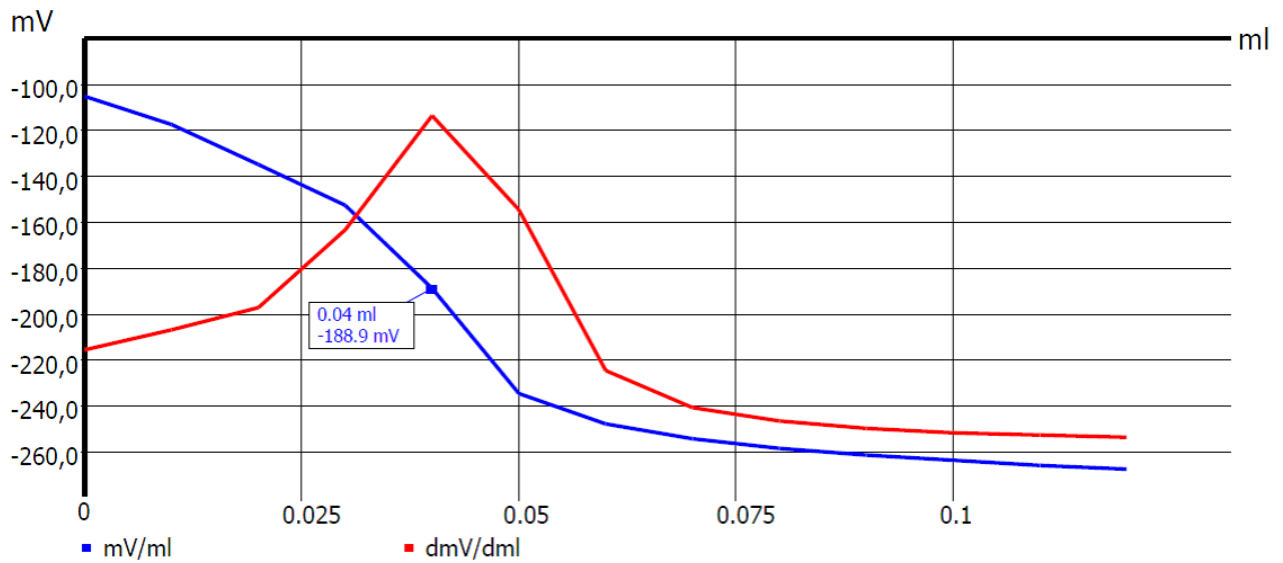
The sample is weighed into a 150 ml beaker and dissolved in 70 ml of the solvent. It can be necessary to heat the mixture to increase the solubility of the oil/fat, especially with solid fats (e.g. coconut fat). After a complete dissolution, the sample is titrated with 0.1 mol/L KOH.

The sample weight should be selected that the titration amount is not more than 4-5 ml because of the long titration time. The required amount of sample depends on the expected Acid number (mg_{KOH}/g).

Expected acid value (mg _{KOH} /g)	Sample amount [g]
0 - 1	10 - 20
1 - 4	2,5 - 10
>15	0,5 – 2,5

Titration parameter

Blank titration



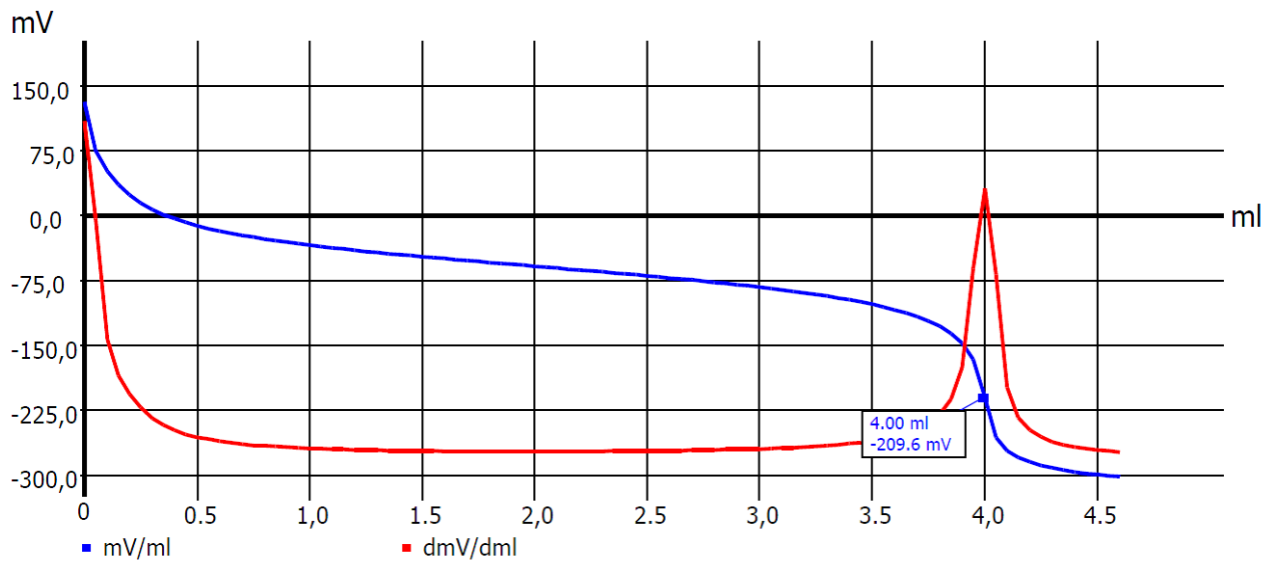
Default method	Blank TAN-TBN		
Method type	Automatic titration		
Modus	linear		
Measured value	mV		
Measuring speed / drift	User defined	Fixed delay time	12 s
Initial waiting time	10 s		
Linear Steps	0.01 ml		
Damping	strong	Titration direction	decrease
Pretitration	off	Delay time	0 s
End value	off		
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.

Sample titration



Default method	--		
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	0.05 ml		
Damping	strong	Titration direction	decrease
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

For samples with very low FFA values the linear steps can be reduced to 0.02 or 0.01 ml.

Calculation:

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

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

$$FFA [\%] = \frac{(EQ1 - B) * T * M * F1}{W * F2}$$

B	M01	Blank value, saved in global Memory M01
EQ1		Consumption of titrant at first Equivalence point
T	WA	concentration of the titrant
M	282,47	Molecular mass of oleic acid
W	man	Weight of the sample in g
F1	0.1	Conversion factor
F2	1	Conversion factor

If the result is to be expressed as a fatty acid other than oleic acid, the molecular weight of this fatty acid must be used for M.