xylem

Application Note
YSI, a Xylem Brand • XA00080

Determination of Acid Number and Free Fatty Acids (FFA) in Fats and Oils

FOOD & BEVERAGE SERIES

Introduction

Free fatty acids (FFA) in plant oils and fats (e.g. edible oils and fats) are a quality feature for these fats. Fats with high levels of FFA are more susceptible to oxidative aging, they become rancid more quickly. The FFA should be removed during a refining process.

Determination of the FFA in Oils and fats is done by potentiometric titration in Ethanol / Diethyl ether as solvent 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 $mg_{(KOH)}$ / g or as $\%_{fatty\ acid}$ (mainly as $\%_{oleic\ acid}$ with Moleic acid = 282,47 g/mol).

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Instrument
TL 7000 or higher
Magnetic stirrer TM 235 or similar

Electrode, Cable, and Electrolyte
N 6480 eth Electrode
L 1 A Cable

Lab Accessories
Glass Beaker 150 ml
Magnetic stirrer bar 30 mm

Reagents	
1	KOH 0,1 mol/l in Isopropylalcohol
2	Ethanol, absolute
3	Diethylether
4	Distilled water
All reagents should be in analytical grade or better.	

Procedure

Reagents

KOH in Isopropyl alcohol 0.1 mol/l
It is recommended to use a ready 0.1 mol/l solution. The solution must be protected against CO_2 with a CO_2 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.

Cleaning of the electrode

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

1. First the electrode is rinsed with the solvent mixture to remove residues of the sample.
2. Then it is conditioned in water.
3. 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 70 ml solvent are placed in a 150 ml beaker and titrated with 0.1 mol/l KOH. The Blank should be below 0.3 ml.

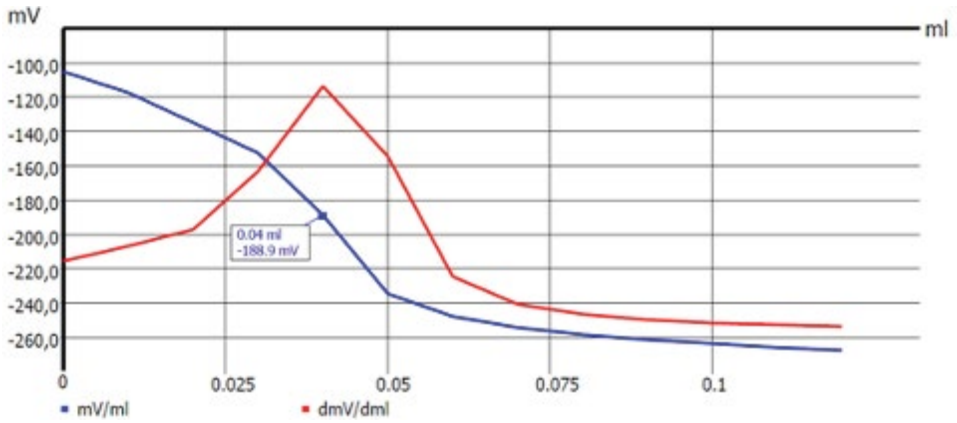
Sample Preparation

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]
0.2 - 1	10 - 20
1 - 10	1 - 3

Titration parameter - Blank Titration

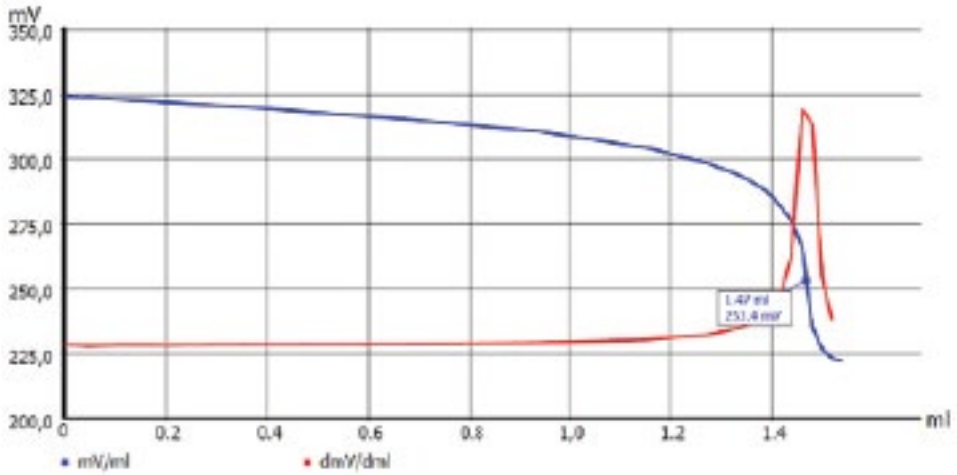


Default Method – Blank TAN-TBN			
Method type	Automatic Titration		
Modus	Linear		
Measured Value	mV		
Measuring Speed / Drift	User Defined	Fixed Delay Time	12 s
Initial Waiting Time	10 s		
Linear Steps	0.01 ml		
Damping	Strong	Titration Direction	Decrease
Pretitration	off	Delay Time	0 s
End Value	off		
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	Dynamic		
Measured Value	mV		
Measuring Speed / Drift	User Defined	Minimum Holding Time	07 s
		Maximum Holding Time	20 s
		Measuring Time	04 s
		Drift	10 mV/min
Initial Waiting Time	10 s		
Linear Steps	0.05 ml		
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

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

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 another fatty acid, M must be the molecular weight of this fatty acid.

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