



Acidity of Edible Oils

(According to standard NF.EN.ISO 660-1999)

Introduction

In the edible oils industry, the degree of acidity is an important parameter for classifying the various oils. Acidity also has an influence on the product taste. As the titration is performed in nonaqueous media, it is recommended to use a titration with inflection point detection.

Principle

The sample is dissolved in methylisobutyl ketone (methyl4-pentanone2) and titrated with KOH (generally 0.1M) in isopropanol. The result is expressed as mg/g of KOH for acid number and also as a % of oleic (or lauric or palmitic) acid (see results).

Electrode & Reagents

As the titration occurs in nonaqueous media, it is recommended to work with separate electrodes and a three-electrode system (see **electrode maintenance and storage notes**).

pHG311 Glass Electrode (part no. E11M004) with a CL114 cable (part no. A94L114) as measuring electrode

REF361 Reference Electrode (part no. E21M003) filled with LiCl 1M in isopropyl alcohol as reference electrode

M241Pt Metal Electrode (part no. E31M001) as cell grounding KOH 0.1M in isopropyl alcohol as titrant solution

Add 6 g of KOH to approximately 1000 ml of isopropyl alcohol. Boil gently for 10 minutes. Allow the solution to rest for 2 days, filter, store in a chemically resistant bottle and standardise versus potassium hydrogen phthalate.

This titrant is also commercially available.

Ethyl alcohol as cleaning solution for the glass electrode

Titration solvent: methyl-isobutyl ketone (methyl4-pentanone2)

Filling solution for reference electrode: Dissolve 4.2 g of LiCl in 100 ml of ethyl alcohol

Buffer solutions pH 4.00 (part no. S11M012) and pH 10.00 (part no. S11M014)

Warning: Reagents used in this application note are flammable. They can cause severe burns and are hazardous if swallowed, breathed or come into

contact with the skin or eyes. Always respect laboratory health and safety regulations when using these reagents.

Electrode & Reagents

CONTINUOUS ADDITION MODE (CONTINUOUS IP)

Cell grounding:	M241Pt
Measure:	mV
Blank:	YES
Min. ordinate:	-185 mV (see notes)
Max. ordinate:	100 mV
Stirring speed:	550 rpm
Stirring delay:	30 s
Burette volume:	10 ml
Maximum volume:	10 ml
Stop point:	-300 mV
Smoothing parameter:	7
Inflection point number:	1
Minimum speed:	0.2 ml/min
Maximum speed:	1 ml/min
Direction:	Decreasing mV
Stop at last IP:	YES
Inflection1	
Min. ordinate:	-220 mV
Max. ordinate:	-50 mV
Sample unit:	g
Sample amount:	see working range
Results:	
Number of results:	2
Result 1	
Result unit:	mg/g
Molar weight:	56.11
Reaction:	1 smp + 1 titr
Calculate with IP:	1
Result 2	
Result unit:	%
Molar weight:	282 (see results)
Reaction:	1 smp + 1 titr
Calculate with IP:	1

Procedure

It is strongly recommended to work under a hood.

When performing the application for the first time, prepare the REF361 Reference Electrode.

The REF361 is delivered filled with aqueous KCl solution, empty this solution, rinse the electrode with isopropyl alcohol and fill it with the LiCl solution in isopropyl alcohol.

Weigh and dissolve the necessary amount of sample in 50 ml of titration solvent.

Dip electrodes and delivery tip in the solution.

Run the titration.

a) When a titration is finished, rinse the electrodes with titration solvent, then with ethyl alcohol and distilled water and dip them in the pH 4.00 buffer solution for 30/60 seconds. Before starting a new titration rinse electrodes with ethyl alcohol.

b) After a cycle corresponding to 5/10 titrations, change the measuring glass electrode. Clean it with titration solvent, ethyl alcohol and distilled water and store it in pH 4.00 buffer solution for one day.

c) Every morning or before starting a new titration cycle, check the electrode system. Measure the potentials reached by the electrodes dipped first in the pH 4.00 and then in the pH 10.00 buffer solutions. The difference between the two measurements should be at least 165 mV.

d) Once a week clean the glass electrode using the Radiometer Analytical GK ANNEX Electrode Maintenance Kit (part no. S91M001).

Results

As indicated before results can be expressed in 2 different ways: **expressed as acid number in mg/g of KOH**

$$R(\text{mg/g}) = V_{\text{titr}} * C_{\text{titr}} * 56.11/W_{\text{smp}}$$

V_{titr} = Total volume of titrant used in ml

C_{titr} = Concentration of titrant in mol/l

W_{smp} = Sample weight in g

56.11 = molecular weight of KOH expressed as acidity as a % of acid

$$R(\%) = V_{\text{titr}} * C_{\text{titr}} * M * 100/1000 * W_{\text{smp}}$$

V_{titr} = Total volume of titrant used in ml

C_{titr} = Concentration of titrant in mol/l

W_{smp} = Sample weight in g

M = Molar weight of organic acid used for result expression (see below)

Depending on the oil, three different organic acids are used for result expression:

Oil	Acid	Acid formula	Molar Weight
Coconut oil	Lauric acid	$\text{CH}_3-(\text{CH}_2)_{10}-\text{COOH}$	200 g/mol
Palm oil	Palmitic acid	$\text{CH}_3-(\text{CH}_2)_{14}-\text{COOH}$	256 g/mol
Other oils	Oleic acid	$\text{CH}_3-(\text{CH}_2)_{10}-\text{CH}=\text{CH}-(\text{CH}_2)_7-\text{COOH}$	

Results on olive oil (3 determinations)

Acid number

Mean: 0.5124 mg/g

Standard deviation: 0.0056 mg/g

Acidity

Mean: 0.275%

Standard deviation: 0.0028%

Working Range

Depending on the expected result, take a sample amount as indicated opposite:

Acid # (mg/g)	Acidity (%) (*)	Sample in g	Titrant volume in ml (**)
<1	<0.5	20	<3.6
1-4	0.5-2	10	1.8-7.2
4-15	2-7.5	2.5	1.8-6.75
15-75	7.5-3.75	0.5	1.3-6.75
>75	>37.5	0.1	>1.35

Notes

Note regarding ordinates

The indicated values for the different ordinates are experimentally measured using the above-mentioned electrodes and methyl-isobutyl ketone as titration solvent. These values may change depending on electrode behaviour (especially the reference electrode) and solvent quality.

Note regarding titrant standardisation

If necessary, standardise the KOH 0.1M in isopropyl alcohol against weighed potassium acid phthalate (KOOCC₆H₄COOH with a molar weight of 204.22 g/mol and 1smp + 1 titrant) and dissolved in CO₂-free distilled water.

Note regarding neutralisation of titration solvent

As the titration solvent has a slight acid reaction it is necessary to run a blank for every new batch of solvent.

Note regarding some particular oils

As indicated in standard ISO 660-1999, some oils do not give detectable inflections. In this case, it is possible to run an end point titration with an end point value corresponding to the equivalent point of oleic acid dissolved in the titration solvent. Using the above-mentioned electrodes, this end point value is close to -179 mV.

To determine this value, weigh around 100-120 mg of purified oleic acid and run a titration with KOH 0.1M in isopropyl alcohol as titrant solution.

Curves

