



Application Note AN-T-112

Acid value and free fatty acids in edible oils

Fully automated determination according to the current EN ISO, Ph. Eur, and USP standards

The acid value (or acid number), together with the free fatty acid value, are important parameters used for the characterization and quality assessment of edible fats and oils. These parameters are also used to monitor the production process of fats and oils. By definition, the acid value in oils and fats refers to the weight in milligrams of the amount of potassium hydroxide (KOH) or sodium hydroxide (NaOH)

required to neutralize one gram of this sample. Acid value and free fatty acid determination by titration are among the best-known analysis methods.

This Application Note describes both the acid value titration as well as the free fatty acid titration in different edible oils. The method is based on the standards EN ISO 660, USP<401>, and Ph.Eur. 2.5.1.

Find more information in the video:

INTRODUCTION

Free fatty acids (FFAs) are triglyceride-free fatty acids. They are only found in very small quantities in virgin (native) vegetable oils. The more refined or processed the oil is, the higher the free fatty acid content. The acid value and free fatty acid value may only be present up to a certain level in edible oils; therefore, both values are used for quality classification purposes. In general, the acid value increases with the age of an oil as triglycerides decompose into smaller fatty acids and glycerol as an effect of time.

In short, FFAs are caused by the hydrolysis of long chain oils into shorter fatty acid chains. The higher the

acid value and FFA content, the lower the quality and stability of the oil against oxidation.

FFA analysis is used for purity testing. In certain cases, it allows conclusions to be drawn about the pretreatment or ongoing decomposition reactions—in particular for saturated fatty acids and unsaturated fatty acids in fat or oil samples.

Using the acid value titration method or the free fatty acid titration method by potentiometric indication, very accurate results can be achieved for a wide range of edible oils, animal fats, or waxes, up to products with high saponification.

SAMPLE AND SAMPLE PREPARATION

The analysis is demonstrated on canola oil (rapeseed oil), palm oil, sunflower oil, and olive oil. No sample

preparation is required.

EXPERIMENTAL

This analysis is performed on an automated system consisting of an OMNIS Advanced Titrator and an OMNIS Sample Robot S with Dis-Cover equipped with a dSolvotrode (**Figure 1**).

To a reasonable amount of sample, a solvent mixture consisting of ethanol and diethyl ether is automatically added, and the solution is stirred for one minute to dissolve the sample. Afterwards, the sample is titrated with standardized ethanolic potassium hydroxide (KOH) until after the equivalence point is reached.

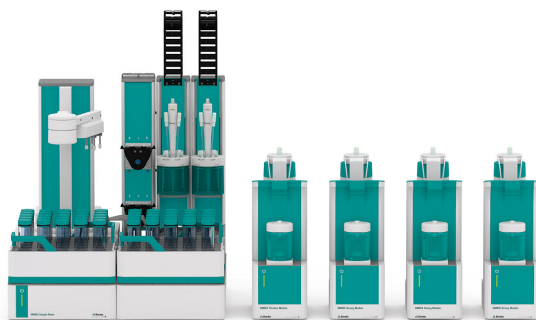


Figure 1. OMNIS Sample Robot S with Dis-Cover functionality, Dosing module, and OMNIS Advanced Titrator equipped with dSolvotrode for the determination of acid value and free fatty acids.

RESULTS

This method demonstrates acceptable results and well-defined titration curves for acid value and free fatty acids displayed in **Table 1** and **Figure 2**. The SD(rel) is apparently high with max. 5.3%, however, this corresponds to a SD(abs) of approx. 8.5 µg KOH/g sunflower oil or 4.4 µg KOH/g rapeseed oil, respectively.



Figure 2. Titration curve of the determination of the acid value of palm oil.

Table 1. Results for acid value (AV) and free fatty acids (FFA) expressed as oleic acid (canola oil, olive oil, sunflower oil) or palmitic acid (palm oil) for different edible oils.

Sample (n = 5)	Acid value in mg KOH/g	Free fatty acids in %	SD(rel) in %
Canola oil	0.11	0.05	4.0
Olive oil	0.41	0.21	2.0
Palm oil	11.6	5.3	0.2
Sunflower oil	0.16	0.08	5.3

Acid and free fatty acid titration with NaOH or KOH is a precise and reliable method to determine these values in various edible oils according to several international standards.

Using an OMNIS Sample Robot with Dis-Cover functionality allows the fully automated determination of up to four samples in parallel,

freeing up valuable time of the operator and thus increasing the productivity in the lab. The OMNIS system offers the opportunity to customize the system according to your needs and expand it for other required titration applications on edible oils, such as the peroxide value or iodine value.

Internal reference: AW TI CH1-1278-062019

CONTACT

Metrohm ANZ
56 Buffalo Road
2111 Gladesville

info@metrohm.com.au

CONFIGURATION



OMNIS Sample Robot S Pick and Place

OMNIS Sample Robot S with a "Peristaltic" (2-channel) pump module and a Pick&Place module in addition to extensive accessories for the direct transition to fully automatic titration. The system provides space in two sample racks for 32 sample beakers of 120 mL each. This modular system is supplied completely installed and can thus be put into operation in a very short time.

The system can also be extended upon request to include two additional peristaltic pumps and another Pick&Place module, thus doubling the throughput. If additional workstations are required, then this Sample Robot is already able to be expanded to become an L-sized OMNIS Sample Robot, thus enabling samples from seven racks to be processed in parallel on up to four Pick&Place modules and quadrupling the sample throughput.



OMNIS Dosing Module without stirrer

Dosing module for connection to an OMNIS Titrator for extending the system to include an additional buret for titration/dosing. Can be supplemented with one magnetic stirrer or rod stirrer for use as separate titration stand. Freely selectable cylinder unit with 5, 10, 20 or 50 mL.



OMNIS Advanced Titrator without stirrer

Innovative, modular potentiometric OMNIS Titrator for endpoint titration and equivalence point titration (monotonic/dynamic). Thanks to 3S Liquid Adapter technology, handling chemicals is safer than ever before. The titrator can be freely configured with measuring modules and cylinder units and can have a stirrer added as needed. If required, the OMNIS Advanced Titrator can be additionally equipped for parallel titration with a corresponding software function license.

- Control via PC or local network
- Connection option for up to four additional titration or dosing modules for additional applications or auxiliary solutions
- Can be supplemented with magnetic stirrer and/or rod stirrer
- Various cylinder sizes available: 5, 10, 20 or 50 mL
- Liquid Adapter with 3S technology: Safe handling of chemicals, automatic transfer of the original reagent data from the manufacturer

Measuring modes and software options:

- Endpoint titration: "Basic" function license
- Endpoint and equivalence point titration (monotonic/dynamic): "Advanced" function license
- Endpoint and equivalence point titration (monotonic/dynamic) with parallel titration: "Professional" function license



dSolvotrode

Digital, combined pH electrode for OMNIS for all non-aqueous acid/base titrations. The glass membrane is optimized for poorly conducting solutions and thanks to the flexible ground-joint diaphragm, the electrode is suitable for contaminated samples.

This electrode can be used with non-aqueous reference electrolytes (lithium chloride or tetraethylammonium bromide).

Storage in corresponding reference electrolyte.
dTrodes can be used on OMNIS Titrators.