Metrohm

Oxidation stability of fats and oils in solid foodstuffs – Rancimat method

Branch

Food, stimulants, beverages, flavors

Keywords

Oxidative stability; oxidation stability; rancidity; 892; oils; fats; sample preparation; extraction; butter cookies; muffins; potato chips; potato crackers; peanut curls; almonds; peanuts; instant noodles; soup pearls; hazelnuts; instant noodles; Kitsune Udon noodles; Yakisoba noodles; branch 7

Summary

The determination of the oxidation stability of fats and oils is an important means for quality control in the food industry. Not only liquid samples can be measured by the Rancimat method, but also solid foodstuffs. The oxidation stability of fats and oils in solid foodstuffs is directly measured if possible or using the isolated fat after cold extraction if the samples cannot be measured directly.

Sample

Oil- and fat-containing products

Instruments

892 Professional Rancimat	2.892.0010
Equipment for determination of temperature correction	6.5616.100
StabNet PC software	6.6068.xxx
Auxiliary instruments for sample preparation	

- Mortar and pestle
- Laboratory balance (resolution ± 0.01 g)
- Magnetic stirrer
- Rotary evaporator (with cooling trap)

Reagents

- Deionized water (ISO 3696 Type II)

For sample preparation

- Petroleum ether, low boiling, boiling point 30 – 40 °C, puriss. p.a., CAS 101216-46-5

Sample preparation

Direct measurement

Solids with a high amount of fat, such as nuts and oil seeds (e.g., hazelnuts, almonds, sunflower seeds, sesame seeds, etc.), can be measured directly. Before the sample is weighed in, it has to be crushed to small grains, e.g., by a mortar. Care has to be taken that the sample is not overheated and not contaminated by traces of transition metals. Additionally it is important that the grain size distribution of the crushed sample is approximately the same between measurements since it has an influence on the induction time.

Cold extraction

Fat from samples with low fat content, e.g., powdered milk, or processed foodstuffs such as salad dressings, has to be extracted before the determination. This is preferably done by cold extraction, since heating would alter the fat. Before the extraction the sample has to be crushed if it is not already liquid or powdered. Enough sample to extract approx. 15 g of fat (sufficient for four measurements) is weighed into a conical flask. Approx. 3 times the sample volume of low boiling petroleum ether is added. The extraction is performed by stirring for at least 1 hour. The petroleum ether phase is then separated from the residues by either filtering in case of solid samples or by a separating funnel in case of liquid samples and transferred into a round bottom flask. The petroleum ether is distilled off at 20 – 30 °C under vacuum, e.g., with a rotary evaporator.

Analysis

Preparation of the Rancimat

The heating block is heated up to the respective temperature.

Preparation of the measuring vessel

The measuring vessel is filled with 60 mL deionized water and placed on the Rancimat together with the measuring vessel cover.

Preparation of the reaction vessel

For each determination, a new reaction vessel is used. To remove particles (e.g., from the cardboard box) the reaction vessel is air-cleaned inside and outside by a sharp stream of nitrogen. Then sample is weighed directly into the reaction vessel. For extracted fat that is liquid or melts at elevated temperatures a sample size of 3.0 g ± 0.1 g is used. Ensure that the air inlet tube always immerses in the sample. Solid samples which do not melt should only cover the bottom of the reaction vessel in order to avoid a temperature gradient within the sample. In this case 0.5 ± 0.05 g of the powdered sample is weighed into the reaction vessel. The reaction vessel is closed with a reaction vessel cover assembled with an air inlet tube.

Determination

Before the determination can be started, the temperature of the heating block has to be stable. The two tubings between Rancimat and reaction vessel and between reaction vessel and measuring vessel are connected. Then the reaction vessel is placed in the heating block and the measurement is started immediately.

Parameters

| Sample size	Direct measurement: 0.5 ± 0.05 g Extracted fat: 3.0 g ± 0.1 g
Measuring solution	60 mL deionized water
Gas flow	20 L/h
Temperature	120 °C
Temperature correction	Automatic
Stop criteria	Endpoints Conductivity: 100 µS/cm
Evaluation	Induction time
Evaluation sensitivity	1.0

Results

Direct measurements

Sample	Induction time / h
Butter cookies	24.25
Muffins (Magdalenas)	10.23
Potato chips (crackers)	28.47
Peanut curls	8.19
Almonds	7.56
Peanuts	3.10
Instant noodles	16.81
Kitsune Udon noodles	15.12
Yakisoba noodles	21.02
Soup pearls	8.31
Hazelnuts	20.60
Potato chips (crackers, 140 °C)	9.20

Cold extraction

Sample	Induction time / h
Butter cookies	28.09
Popcorn grains	3.82
Olive paste	19.82
Salad dressing	1.23
Hazelnuts	10.63
Potato chips (crackers, 140 °C)	1.30

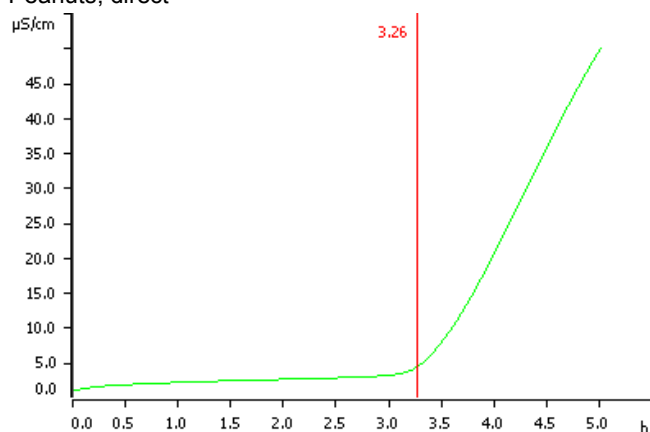## Example determinations

Potato chips (crackers), direct, 120 °C
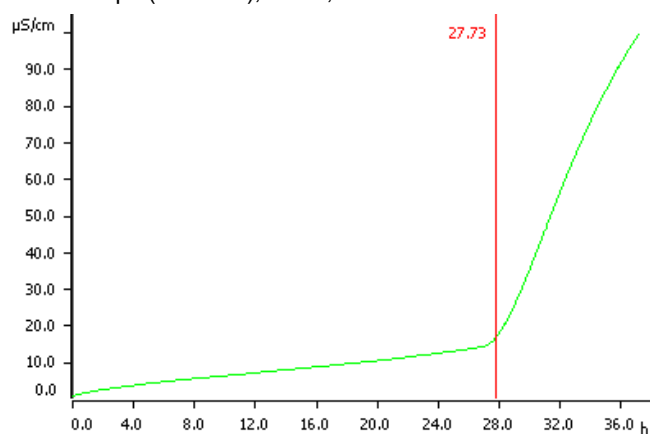
Butter cookies, extracted
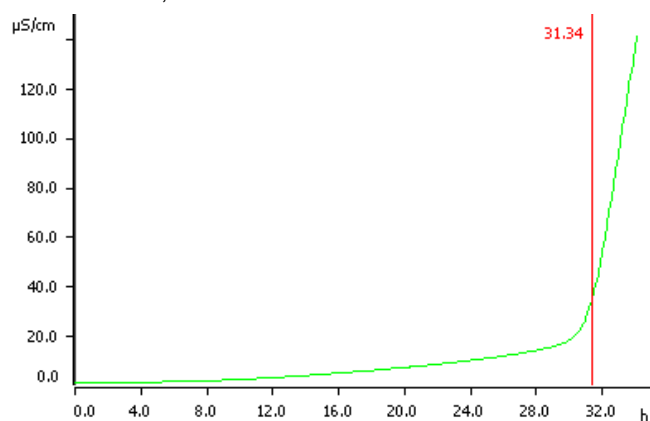
Peanuts, direct

Comments

- Some samples (e.g. butter cookies extract) produce foam during the measurement. To avoid the foam to be transferred into the measuring vessel a foam barrier (6.1451.010) and additional oil separator (6.2753.200) can be installed.

- Samples can be determined both by direct measurement and after cold extraction. Keep in mind that the results between direct measurement and cold extraction differ due to influences of the sample matrix on the oxidation reaction.

- Since the particle size distribution of the solid samples has an influence on the induction time it is recommended to prepare the sample as a fine powder since with small grain sizes the induction time does not vary strongly and the induction times are shorter. Additionally the resulting measuring curve is usually steeper than with bigger particle sizes.

- The induction time is evaluated automatically by the maximum of the second derivative of the measured curve. If the induction time is not evaluated automatically the evaluation sensitivity might be too high and has to be adjusted in the method. In case there are still problems with the automatic evaluation manual evaluation with tangents may be used instead.