

Application Bulletin



Of interest for:
Factories in the edible oil and edible fat processing sector,
foodstuff industry, pharmaceuticals and cosmetics

No. 204/1 e

Oxidative stability of oils and fats – Rancimat method

Summary	<p>The Rancimat allows the automatic determination of the oxidative stability of oils and fats without the need for expensive and environmentally hazardous chemicals and time-consuming titrations. Six samples can be determined simultaneously side by side. Whether the oil- or fat-containing samples originate from the foodstuff sector, from edible fats or edible oils, or are creams, ointments and lotions from the pharmaceutical and cosmetic industries - the Rancimat helps save time and money.</p> <p>This Bulletin provides a detailed description, above all of the requisite sample preparation steps. To exemplify a determination, the analysis of fresh and used palm oil is also described.</p>
Apparatus	<ul style="list-style-type: none">▶ 2.679.001X Rancimat▶ Auxiliary units for sample preparation (see below)
Reagenzien	<ul style="list-style-type: none">▶ Petroleum ether, low-boiling puriss p.a. b.p. 30 ... 40°C▶ Sodium sulphate, anhydrous puriss p.a.
Sample preparation	<p>Fat-containing substances in solid or powder form can not be used directly. The fats must first be isolated by extraction.</p> <ul style="list-style-type: none">▶ Edible oils, skin oils These can be measured directly. A disposable syringe without needle is used for this.▶ Edible fats Are carefully melted in the sample vessel on a water bath and then weighed into the reaction vessel.▶ Water-containing fats (butter, margarine) Can also be weighed in directly. In many cases, when water separation has been specified for the AOM test, proceed as follows: Sample (ca. 20 g) is added to a centrifuge tube and warmed to 50°C in a water bath. After 5 min centrifuging at 3000 rpm, the upper fat phase is filtered through glass wool coated with sodium sulphate in a drying oven at 50°C.▶ Mayonnaise and mustard Sample (ca. 20 g) is added to a conical flask with ground-glass joint and covered with a 1-cm layer of petroleum ether. A magnetic stirrer bar is added, the flask stoppered, wrapped in aluminum foil and placed on a magnetic stirrer. Extraction is now performed for 1 ... 3 h with stirring and the petroleum ether phase filtered through glass wool into a round-bottom flask. The petroleum ether is then evaporated on a rotary evaporator and the flask contents predried for 30 min at 12 mbar in an initial aftertreatment. Final drying with sodium sulphate is followed by another filtration in the drying oven through glass wool at 50°C. If the extracts so obtained are not to be used immediately, they should be stored in a refrigerator under nitrogen.

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Sample preparation (continued)

▶ **Powdered milk, powdered baby food**

In principle, the fat can be extracted with a soxhlet apparatus. However, as the fats could be thermally damaged by the warm extraction, particularly in the case of baby food, the following cold extraction should be given preference.

Powdered sample (ca. 150 g) is weighed into a conical flask with ground-glass joint and 350 mL petroleum ether added. Extraction is performed with stirring for at least 1 h, the mixture is then filtered into a round-bottom flask and the petroleum ether distilled off at 20 ... 30°C under vacuum (e.g. rotary evaporator).

▶ **Nuts and seeds**

(e.g. hazel nuts, almonds, sunflower seeds, coffee beans, rice, cocoa beans, etc.)

The sample material is pulverized as gently as possible (do not overheat, no contamination by heavy metals). Extraction is performed in a soxhlet apparatus or in a fat extractor. Enough sample to provide ca. 10 g fat is weighed in.

The cold extraction described above can also be used.

▶ **"Solid" foodstuffs, fodder**

(e.g. chocolate, biscuits, cakes, animal feed, etc.)

After mechanical comminution, enough sample is weighed out to give approx. 10 g fat. Extraction is performed with petroleum ether in a soxhlet apparatus or a fat extractor. In some cases, the cold extraction is also suitable.

Method

▶ **Preparation of the Rancimat**

- The temperature calibration uses the same amount of paraffin oil at the same temperature and with the same air flow as is employed in the subsequent analysis of the samples (see 679 Instructions for use).
- Normally, 2.5 g oil/fat are weighed into the reaction vessel without contamination of the side walls. With samples containing large amounts of water (creams, lotions), 4 ... 8 g are used as the water evaporates during the determination and hence the air inlet tube would no longer be immersed in the sample.
- Dist. or deion. water (60 ... 75 mL) is added to the absorption vessels and the measuring cells installed ensuring freedom from air bubbles and coverage of the top lateral holes (rotate vessel gently). The measuring cells are then closed.
- The reaction vessels filled with sample are now placed in the heating block for 5 ... 10 min, the air flow (normally 20 L/h) and the absorption vessels attached and the Rancimat started with "GO".
- The temperature to be selected depends on the oxidative stability of the sample. For the type of sample described here, it is usually between 70 ... 160°C (50 ... 220°C possible). Work is mainly carried out at 120°C (lower stability – lower temperature). The following rule of thumb holds: *An increase in the temperature by 10°C lowers the induction time by a factor of two.*

Note: *The reproducibility of the results obtained (induction times) is very heavily dependent on the cleanliness of the reaction vessels used and the purity of the samples. Solid particles, traces of heavy metals, carbon particles, as well as acids and bases catalyse the determination.*

Literature

- ▶ METROHM AG
Instructions for Use for 679 Rancimat
- ▶ Läubli, M.W. / Bruttel, P.A.
Determination of the oxidative stability of fats and oils: Comparison between the active oxygen method (AOCS Cd 12-57) and the Rancimat method.
JAOCS 63, (1986) 792-795
- ▶ Läubli, M.W. / Bruttel, P.A. / Schalch, E.
A modern method of determining the oxidative stability of fats and oils.
Int. Food Marketing & Technology 1, (1988) 16-18
- ▶ Warner, K. / Frankel, E.N. / Mounts, T.L.
Flavor and oxidative stability of soybean, sunflower and low erucic acid rapeseed oils.
JAOCS 66, (1989) 558-564

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Curve examples

The samples are warmed on a water bath at 50°C and 2.5 g are weighed into the reaction vessels. Determination in triplicate in each case.

Positions 1, 2, 3: Fresh palm oil $\bar{x}(3) = 11.40 \pm 0.35$ h induction time
 Positions 4, 5, 6: Used palm oil $\bar{x}(3) = 8.88 \pm 0.10$ h induction time

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METROHM 679 RANCIMAT                      METHOD 0
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RESULTS
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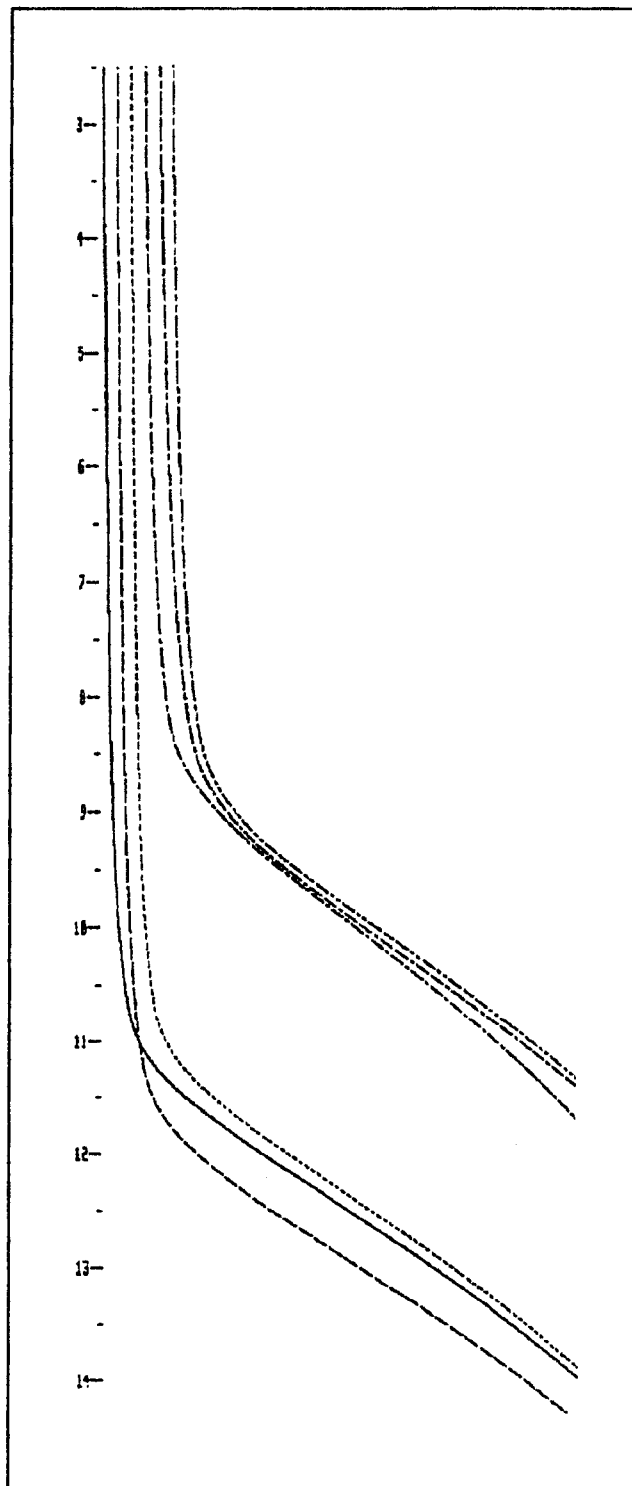
ch	smpl.ident	eval.1	eval.2	eval.3
1	1.1	11.2 h		
2	1.2	11.8 h		
3	1.3	11.2 h		
4	2.1	8.77 h		
5	2.2	8.95 h		
6	2.3	8.93 h		

eval.1: induction time

DATE 90-12-04 TIME 07:08

PARAMETERS
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temperature                      120    Cel
temperature correction           2.2    Cel
conductivity range               200    uS/cm
evaluation modes                 1/-/-
delay time                        0      h
paper feed                        2      cm/h
cell constants: channel 1        0.83   /cm
                  channel 2        0.88   /cm
                  channel 3        0.86   /cm
                  channel 4        0.80   /cm
                  channel 5        0.87   /cm
                  channel 6        0.79   /cm
measuring time                    INF    h
end mode:    EP stop              OFF
              heater stop         OFF
              air stop              ON
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Table of investigated sample materials with Induction times

The tables make no claim to completeness.

The temperatures and above all the induction times are approximate values.

a) Animal oils and fats, direct determination

Product	Temperature	Induction times
Butter	120°C	3 ... 6 h
Fish oil	80°C	ca. 15 min
Fish oil and other fats	100°C	10 min ... 5 h
Chicken fat	110°C	ca. 30 min
Kidney fat	110°C	3 ... 4 h
Beef suet	120°C	3 ... 8 h
Lard	100°C	1 ... 3 h
Pigeon fat	110°C	ca. 20 min

b) Samples after extraction

Product	Temperature	Induction times
Baby food	120°C	1 ... 2 h
Peanuts	120°C	1 ... 2 h
Hazel nuts	120°C	7 ... 13 h
Mayonnaise	120°C	1 ... 4 h
Powdered milk	120°C	4 ... 32 h

c) Vegetable oils and fats

Product	Temperature	Induction times
Cottonseed oil	120°C	2 ... 3 h
Citrus oil	90°C	ca. 30 min
Thistle oil	120°C	1 ... 2 h
Peanut oil	120°C	3 ... 4 h
Peanut fat	120°C	9 ... 10 h
Hazel nut oil	120°C	7 ... 11 h
Hazel nut fat	120°C	10 ... 12 h
Coffee oil	110°C	ca. 15 h
Cocoa butter	120°C	9 ... 15 h
Coconut oil	120°C	up to 33 h
Linseed oil	110°C	ca. 40 min
Margarine	120°C	2 ... 6 h
Olive oil	120°C	6 ... 11 h
Orange oil	90°C	ca. 2 h
Palm oil	120°C	7 ... 12 h
Rape-seed oil	130°C	12 ... 17 h
Rap-seed oil, hydrogenated	140°C	10 ... 11 h
Soja bean oil	120°C	1 ... 7 h
Sunflower oil	120°C	1 ... 2 h