Application SOXTHERM

B.1.9.1.a. Free Fat in Potato Chips

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1 Principle

The sample is extracted with petroleum ether or with n-hexane. After extraction, the solvent is distilled and the residue is dried and weighed. The content of free fat is calculated as the difference between the weight at the start and the end of the analysis.

2 Method

This application note is meant to be a guideline for the operation of your C. Gerhardt analysis system and has to be adapted to your sample matrix and the local peculiarities in your laboratory.

This document is based on the following german official method:

- § 64 LFGB (before § 35 LMBG) L 17.00-4 Determination of Fat in Bread and Bread Products May 1982
  (without hydrolysis)

3 Chemicals and Accessories

Quality p. a.

1. Petroleum ether, boiling range 40 to 60 °C
2. N-Hexane p.a.
3. Extraction thimble 33 x 80 mm, Art. No. 13-0054
4. Extraction thimble 33 x 94 mm, Art. No. 13-0057
5. Cotton wool, chemically pure and degreased

4 Instruments

- Mechanical blender, e.g. mincer with breaker plate, diameter of breaker < 4 mm or universal mixer, e. g. Moulinex "Moulinette"
- Analytical balance (reading accuracy 0.1 mg)
- Desiccator, with a desiccant, e.g.: Silica gel orange / Sorbsil C 2.5
- Extraction unit SOXTHERM Micro/Macro with MULTISTAT, Art. 13-0011 or SOXTHERM Manager, Art. 13-0012
- Drying cabinet, electrically heated, with natural ventilation and automatic temperature control

5 Sample Type and Preparation

The extraction beakers are loaded with 3 - 5 boiling stones, dried for 1 h in the drying oven at 100 ± 2 °C, cooled to room temperature in the desiccator and then weighed to 1 mg on point.
The sample is to be stored sealed at 2 - 6 °C in such a way that spoilage and changes in its composition are prevented.
The sample material is homogenized in the mechanical blender (4.1.).
5 to 6 g are weighed into an extraction thimble (0.1 mg precision) and are covered with cotton wool (3.5.).

Sample Potato Chips

Sample Potato Chips crushed

6 Extraction

The thimble holder and the thimble containing the sample are inserted into the extraction beaker.
After adding 150 ml (SOXTHERM macro) or 100 ml (SOXTHERM micro) extraction solvent (3.1 or 3.2.), the sample is extracted after the following program:

Table 2: Instrument configuration SOXTHERM

Parameter	Note / Cat.-no.
Solvent	Petroleum ether / n-hexane
Boiling point/range	40-60 °C / 68,9°C
Solvent amount	100 ml SOXTHERM micro
	150 ml SOXTHERM macro
Sealing type	Viton / 1000578
Extraction thimbles	33 x 80 mm /13-0054 33 x 94 mm /13-0057
Thimble holders	SHK2 /13-0062
Boiling stones	13-0047
Compressor / Connection compressed air	13-0010, minimum 4.5 bar
Water connection / Chiller	minimum 0.5 bar

Table 3: SOXTHERM Program Fat

Program step	Petrolether	n-Hexane	Note
T-classification	200 °C	200°C	
Extraction temperature	150 °C	180°C	
Reduction interval	4 min.	4 min	
Reduction pulse	1 s		
Hot extraction	30 min	30 min	Sample must be immersed completely
Evaporation A	4-5 x interval	4-5x interval	After phase A the solvent level should be at least 10 mm below the thimble
Rinsing time	80 min.	80 min	
Evaporation B	3-4 x Interval	3-4x interval	After B the extraction beaker should be widely free of extraction agent.
Evaporation C	4 min.		

At the end of the program, the extraction beakers are dried for 30 minutes at 103 ± 2 °C in the drying cabinet. The extraction beakers are then placed in the desiccator, cooled down to room temperature and weighed as precisely as possible to 1 mg. To check the weight consistency, the beakers are dried again for 30 minutes and weighed again after cooling down.
This procedure is repeated until two successive weighings do not differ more than 1 mg. In case of an increase in weight, the previous low value is to be taken.
Extraction, drying and weighing must follow each other immediately.

Note: At extraction temperatures above 150 °C, the extraction beakers should be cooled down slowly, e.g. in a drying cabinet. That is an important step to avoid extreme thermal stress, especially for the bottom of the hydrolysis beaker, and an associated formation of fine glass cracks.

7 Evaluation

7.1. Calculation
The crude fat content w in g/100 g (equivalent to %) of the sample is calculated according to the following equation:

$$w = \frac{(m_2 - m_1)\ 100}{m_0}$$

m_1: Mass of the empty extraction beaker with boiling stones [g].
m_2: Mass of the extraction cup with fat after drying [g].
m_0: Weighing-in [g]

The result is rounded to one decimal place.

8 Samples of a result

Sample	Mass of dried Residue after Extraction [g]	Content of free Fat[%]
normal quality	2.13	35.2
normal quality	2.18	34.9
normal quality	2.13	35.1
mean value:		35.1
relative standard deviation:		0.1
light quality	1.27	23.8
light quality	1.06*	20.1
light quality	1.33	24.8
mean value:		22.9
relative standard deviation:		2.5

* outlier, problems during analysis

9 Troubleshooting Extraction

9.1 Result too high

Cause	Remedy
Water in the sample, water drops swim on the fat surface, fat can't be concentrated within the usual drying time	Filter or sample must be dry when they are put in
Too high temperature in the drying oven, fat is oxygenized and mass increases	Check temperature of the drying oven
Drying time too long, fat is oxygenized and mass increases	Observe needed drying time
Extraction beaker is dirty	Work as clean as possible in drying, weighing and cooling off process
Solvent is used several times; it has taken up fat which is detected, too.	Check blank value of the solvent
Parts of the thimble or sample residue are left in the extraction beaker	Check whether the fat film is clear Check whether the thimble is porous Check whether the grinding of the sample is too fine. Use a second filter Thimble is too impermeable, sample spills over

9.2 Result too low

Cause	Remedy
Incomplete extraction	Extract thimble again
Incomplete extraction	Flowrate cannot be achieved because of too low permeability of the thimble. Change thimble
Incomplete extraction	Sample sticks together, enlarge the surface e.g. by using sodium sulphate
Cooling off time too short	Follow correct weighing procedure
Boiling delay during concentration process, fat splashes can be found in the thimble	Wrong or missing boiling stones, glass beads cannot be used. Heating power too high, reduce the extraction temperature

9.3 Result fluctuates

Cause	Remedy
Uneven extraction due to solvent losses	Check for leakages in the apparatus at O-rings and connectors
Delayed boiling during extraction, sample residues may stick to the teflon connector	Use boiling stones
Delayed boiling because of usage of glass beads	Use standard boiling stones
Filtration problems, paper filter was not evenly wetted, fat losses because of fat running through. Can be seen from fat residues in the washing solution	Make sure filter in HYDROTHERM is wetted evenly

COMPREHENSIVE APPLICATION DATA BASE

C. Gerhardt offers a wide range of application notes for many methods and procedures. Please contact our application lab team via application@gerhardt.de for further information on:

- Nitrogen in food and feed samples according to Kjeldahl and Dumas
- Crude fibre, ADF and NDF in feed
- Fat in food and feed
- Alcohol determination
- Total cyanide in water
- Trace metal in soil and sludge
- COD determination in water
- Total nitrogen determination in water, soil and plants
- Many more application notes on request.

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