Fat Determination in Dairy Products according to Weibull-Stoldt Method

FatExtractor E-500: Soxhlet Extraction after hydrolysis with the HydrolEx H-506

A simple and reliable procedure for fat determination of food and feed products according to Weibull-Stoldt is introduced. The sample is hydrolyzed with the HydrolEx H-506. The Soxhlet extraction is performed with the FatExtractor E-500. Calculation of total fat content follows gravimetrically after the extract has been dried to a constant weight. This application follows official methods (EN 98/64/EG, AOAC 963.15, ISO 14156:2001, ISO 1443:1973, AOAC 945.16). The combination of the new HydrolEx H-506 and the FatExtractor E-500 increases the sample throughput.

1. Introduction

Fat determination is one of the key analysis performed in the food industry. The samples require a hydrolysis step with hydrochloric acid to break the chemically bound and naturally encased fat from the matrix. Afterwards, the fat is extracted with a suitable solvent according to Soxhlet. After the extract has been dried to a constant weight the total fat content is determined gravimetrically.

2. Experimental

Equipment: HydrolEx H-506, FatExtractor E-500 Soxhlet

Samples: Milk powder LUV No. 17-4b with a certified fat content of 24.27 g/100 g (+/- 0.542 g/100 g), Yoghurt muva-jo-1422 with a certified fat content of 3.76 g/100 g (+/- 0.13 g/100 g).

Determination: 20 g of quartz sand was added to a glass sample tube and 2 g Celite® 545 was placed on top. The samples were weighed into a hydrolysis vessel containing 2 g of Celite®. After adding 2 x 50 mL hydrochloric acid (4 M) into each vessel the samples were hydrolyzed for 30 min using the H-506. The hydrolyzate was transferred and the vessels washed with warm (50 °C) deionised water, until a neutral pH was obtained. The glass sample tubes were dried in a vacuum oven, drying oven or microwave oven. After cooling down in a desiccator another layer of quartz sand (20 g) was added to the sample tube. The extraction was performed using the E-500 (Picture 1) applying the parameters specified in Table 1.

Table 1: Parameters for the extraction with the FatExtractor E-500 SOX

Method parameters	
Solvent	Petroleum ether / Hexane / Diethyl ether / Chloroform
Extraction step	20 cycles (heating level 5 - 9[1])
Rinse step	5 min (heating level 5 - 9[1])
Drying step	10 - 13 min (heatinge level 5 - 7[1])
Solvent volume	100 mL

The samples were extracted in triplicate. The extracts were dried to a constant weight in a drying oven at 102 °C and the total fat content was calculated.

Picture 1: FatExtractor E-500 SOX

3. Results

The determined fat contents are presented in Table 2. The results correspond to the certified values of the reference materials. The determinations show low relative standard deviations.

Table 2: Determined fat content in dairy products, fat in g/100 g (relative standard deviation in brackets), n=3

Solvent	Milk powder	Youghrt
Petroleum ether	24.35 (0.26)	3.80 (1.07)
Hexane	24.45 (0.28)	3.70 (0.75)
Diethyl ether	24.50 (0.22)	3.75 (0.26)
Chloroform	24.70 (0.41)	3.81 (1.29)

4. Conclusion

The determination of fat in different dairy products using the HydrolEx H-506 and the FatExtractor E-500 provides reliable and reproducible results. These results correspond well to the labelled values, with low relative standard deviations (rsd).

With the FatExtractor E-500 Soxhlet, the time per cycles is reduced significantly. The programmed 20 cycles are accomplished in approx. 70 min.

5. References

[1] EN 98/64/EG Commission Directive 98/64/EC Fat in feedingstuffs
[2] ISO 14156:2001 Milk and milk products -- Extraction methods for lipids and liposoluble compounds
[3] ISO 1443:1973 Meat and meat products -- Determination of total fat content
[4] AOAC 963.15 Fat in Cacao Products
[5] AOAC 945.16 Oil in Cereal Adjuncts

For more detailed information and safety considerations please refer to the Application Note No. 348/2019.

[1] Heating level proposed by the system depending on the selected solvent.