

Fat Determination in Bakery Product and Chocolate by Economic Continuous Extraction

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

The Economic Continuous Extraction (ECE) is a continuous extraction where the sample is constantly kept in hot solvent vapor whilst efficiently rinsed with freshly distilled solvent. The total fat content is determined gravimetrically after the extract has been dried to a constant weight. This method is also called Twisselmann and follows several standards, like ISO 22630:2015, ISO 6492:1999 and §64 LFGB L 17.00-4:1982-05.

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. 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 Economic Continuous Extraction

Samples: Cookie LUV No. 17-11 with a certified fat content of 27.47 g/100 g (+/- 0.311 g/100 g), Chocolate LVU No. 17-13 with a certified fat content of 30.93 g/100 g (+/- 0.356 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 ECE

Method parameters

Solvent	Petroleum ether / Hexane / Diethyl ether / Chloroform
Extraction step	60 min (heating level 5 - 8 ¹)
Drying step	6 - 12 min (heating level 4 - 7 ¹)
Solvent volume	70 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 ECE

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 bakery product and chocolate, fat in g/100 g (relative standard deviation in brackets), n=3

Solvent	Cookie	Chocolate
Petroleum ether	27.41 (0.46)	30.94 (0.14)
Hexane	27.50 (0.47)	30.95 (0.04)
Diethyl ether	27.46 (0.62)	31.04 (0.25)
Chloroform	27.57 (0.57)	31.05 (1.10)

4. Conclusion

The determination of fat in different bakery product and chocolate 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).

5. References

- [1] ISO 22630:2015 Oilseed meals -- Determination of oil content -- Rapid extraction method
- [2] ISO 6492:1999 Animal feeding stuffs -- Determination of fat content
- [3] §64 LFGB L 17.00-4:1982-05 Bestimmung des Gesamtfettgehaltes in Brot einschliesslich Kleingebäck aus Brotteigen

For more detailed information and safety considerations please refer to the Application Note No. 355/2019.

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