

*Extraction Unit E-816 ECE:  
Fat Determination in Food using Twisselmann Extraction*

The determination of fat in food is a routine procedure used for quality assurance and labelling. Below, a simple and easy procedure for fat determination in food, according to §64 LFGB L 17.00-4 and ISO 1444:1996 is introduced. The sample is hydrolyzed with hydrochloric acid using the Hydrolysis Unit E-416, followed by a Twisselmann extraction with the Extraction Unit E-816 ECE (Economic Continuous Extraction). The determined fat contents correspond well to the labelled values.

### 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 Twisselmann. With this extraction technique the sample is constantly kept in hot vapor whilst efficiently rinsed by fresh distilled solvent. After the extract has been dried to a constant weight the total fat content is determined gravimetrically.

### 2. Experimental

Equipment: Hydrolysis Unit E-416, Extraction Unit E-816 ECE

Samples: Madeira Cake LGC7107 with a labelled fat content of  $13.4 \pm 0.7$  g/100 g; Processed meat ERM®-BB501b, labelled fat content  $11.57 \pm 0.44$  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 homogenized samples were weighed into a digestion 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 E-416. The hydrolyzate was transferred and the vessels washed with warm (40-50 °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-816 ECE (Figure 1) applying the parameters specified in Table 1.

*Table 1: Parameters for the extraction with the Extraction Unit E-816 ECE*

#### Method parameters

Solvent	Petroleum ether / Diethyl ether / Hexane / Chloroform
Extraction step	50 min (Heater 100 %)
Drying step	10 min (Heater 100 %)
Solvent volume	70 mL

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



*Figure 1: Extraction Unit E-816 ECE (Economic Continuous Extraction)*

### 3. Results

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

*Table 2: Determined fat content in food samples, fat in g/100g (relative standard deviation in brackets), n=6*

Solvent	Maderia Cake	Processed meat
Petroleum ether	12.82 (1.13)	11.22 (1.70)
Diethyl ether	13.24 (1.36)	11.62 (0.42)
Hexane	12.97 (0.51)	11.36 (1.42)
Chloroform	13.16 (0.56)	11.68 (1.70)

### 4. Conclusion

The determination of fat content in different hydrolyzed food samples using the Twisselmann extraction on the E-816 ECE provides reliable and reproducible results that correspond well to the labelled values of the certified reference materials.

### 5. References

§64 LFGB L 17.00-4:1982-05 Bestimmung des Gesamtfettgehaltes in Brot einschliesslich Kleingebäck aus Brotteigen  
ISO1444:1993 Meat and meat products – Determination of fat content

- Operation Manual of Hydrolysis Unit E-416
- Operation Manual of Extraction Unit E-816 ECE

For more detailed information and safety considerations please refer to the Application Note no. 173/2014.