

**Fat determination of calcium soap** *FatExtractor E-500: Total fat determination of calcium soap from palm fatty acid distillate (PFAD) using E-500 with simultaneous hydrolysis*

A simple, reliable and unique procedure for the determination of fat in calcium soap is introduced. Calcium soap has become a common supplement to feed since 1980 for high milkyielding dairy ruminants, such as cows, goats and sheeps, to increase the milk yield [1-3]. In the presented application, the total fat content of two samples was determined. The hydrolysis is crucial for the total fat recovery. Therefore, a unique method that combines hydrolysis and extraction in one working step is introduced. The extraction with simultaneous hydrolysis was performed using the FatExtractor E-500 Soxhlet. The total fat content was determined gravimetrically. The fat recovery was within the expected value with low relative standard deviations.

**1. Introduction**

Fat determination is one of the key analysis performed in the feed industry. The samples consist of calcium salts of fatty acids, which are still highly hydrophobic yet insoluble in most hydrocarbons (e.g. n-hexane). The hydrolysis step includes the formation of fatty acids by protonation and removal of the calcium-ions. In this simple and unique procedure, the sample is simultaneously hydrolyzed and extracted. After the extract has been dried to a constant weight the total fat content is determined gravimetrically.

**2. Experimental**

Equipment: FatExtractor E-500 Soxhlet

Sample 1: Calcium soap from palm fatty acid distillates (PFAD), expected fat content: 84%, consists of pellets.  
Sample 2: Calcium soap from palm fatty acid distillates (PFAD), expected fat content: 84%, consists of coarse powder.

Determination: The samples were weighed into a glass fibre thimble placed in a glass sample tube with frit. Quartz sand was added to the glass fibre thimble and stirred well. The sample was covered with glass wool. The extraction was performed using the FatExtractor E-500 (Figure 1) applying the parameters specified in Table 1.

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

Method parameters	
Solvent	n-Hexane :HCOOH mixture
Extraction step	360 min (heating level 8)
Rinse step	5 min (heating level 8)
Drying step	SmartDrying
Solvent volume	100-120 mL

An initial n-hexane:HCOOH mixture was prepared which formed two phases. From the upper phase, 100-120 mL were transferred into the beaker (for example by using a separating funnel). 5 mL of the lower phase were transferred using a 5 mL volumetric pipette into the beaker.

After the extraction, the recovered solvent from the drying process can be reused in the next extraction.

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



Figure 1: Calcium soap

**3. Results**

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

Table 2: Determined fat content of calcium soap samples, fat in g/100 g (relative standard deviation in brackets), n=3

Samples	Fat content (g/100g)
Sample 1 – powder from pellets	<b>82.67</b> (0.80%)
Sample 2 – powder from coarse powder	<b>85.20</b> (0.70%)

**4. Conclusion**

The fat determination of calcium soap samples using the unique procedure and the FatExtractor E-500 provides reliable and reproducible results.

**5. References**

[http://www.lipico.com/processes\\_calcium-soap-production.html](http://www.lipico.com/processes_calcium-soap-production.html), 16.07.2021  
Handojo L. et al., Calcium soap from palm fatty acid distillate for ruminant feed: Ca(OH)<sub>2</sub> as calcium source, MATEC Web of Conferences, 159, 2018.  
Block E. et al, Calcium salts are highly digestible, Feedstuffs, 30, p. 1-5, 2005.

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