

APPLICATION SOXTHERM

Cerhardt

BLATT 1 VON 2

OIL AND FAT IN WATER AND WASTE WATER

1. Principle

Determination of the fat content in water and waste water.

2. Methode

based on Standard Methods for the Examination of Water and Wastewater, 19th Edition APHA, AWWA, WBF, 1995

Type of Sample	Water or waste water
Amount of Sample	11

3. Chemicals

Quality p.a. Water: demineralised oder distilled

3.1. Kieselgur

3.2. Petroleum ether, boiling range 40 - 60 °C

4. Instruments

- 4.1. Shaker LS 500, catalogue no. 4055 or THL 500/1, catalogue no. 4371 with accessories for separating funnel
- 4.2. Glass funnel, measuring cylinder
- 4.3. Whatman Filter No.1
- 4.4. Extraction unit SOXTHERM micro or macro, with MULTISTAT, cat. no. 13-0011 or SOXTHERM Manager, cat. no. 13-0012
- 4.5. Electric drying chamber with natural aeration and automatic control of the temperature with a precision of ± 2 K
- 4.6. Desiccator with drying agent, e.g. Blaugel
- 4.7. Balance (0.0001 g)
- 4.8. Cotton wool, chemically clean and fat-free

5. Sample Preparation

A teaspoon of Kieselgur is added to the sample and shaken vigorously for one minute; higher fat quantities might require higher quantities of Kieselgur. The suspension obtained is filtered using a moistened Whatman filter no 1. The filtrate is collected and the amount is written down in mg.

5.1. Drying Process

The filter is taken out carefully of the funnel and put openly on a watch glass to be dried for about 10 - 15 minutes at 103 °C in the drying chamber (4.5.). Then it is put into the extraction thimble and closed with cotton wool (4.8.). Any fat traces remaining on the watch glass are taken up with this cotton. Drying is effected in the drying chamber at 103 °C for 1 - 1.5 hours.

5.2. Extraktion

The extraction beakers with the boiling stones are dried at 103 °C for 1 hour in the drying chamber and then left in the desiccator to cool down for 30 minutes. After that they are weighted to 1 mg exactly. The extraction thimbles with holders are then placed into the extraction beakers and the solvent is added. The prepared beakers are put into the SOXTHERM and the program is started with the following parameter:

Solvent: Boiling Point: Amount of Solvent: Petroleum ether 40/60 Boiling range 40 - 60 °C SOXTHERM micro 100 ml / SOXTHERM macro 150 ml





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Parameters for the Instrument and Connections

Size of the extraction beakers:	Micro, cat. no. 13-0051 / macro, cat. no. 13-0050		
Type of seal:	Viton		
Extraction thimbles:	Type SE33A, 33 x 80 mm - cat. no. 13-0054, or		
	Type SE33B, 33 x 94 mm - cat. no. 13-0057		
Holder for thimbles:	SHK2, cat. no. 13-0062		
Boiling stones:	Cat. no. 1000774		
Compressor / connection for compressed air:	Cat. no. 13-0010 or 4.5 bar minimum		
Water connection			
or recirculating condenser:	0.5 bar minimum		

Program Parameters

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Program Step	Parameter	Comment
Safety Temperature	200 °C	
Extraction Temperature	150 °C	
Reduction Interval	4 min	
Reduction Pulse	2 s	
Hot Extraction	30 min	
Evaporation A	4 - 5 x Intervall	After A the level of solvent has to be at least 10 mm below the thimble
Rinsing Time	60 min	
Evaporation B	3 - 4 x Intervall	After B the extraction beaker should be nearly empty
Evaporation C	4 min	

The beakers are removed from the SOXTHERM using tongs and the thimbles are taken out of the extraction beakers. The beakers are left in the fume hood to evaporate any remaining solvent residue. Then the extraction beakers are dried for 30 minutes at 103 °C and left to cool down in the desiccator. After that the beakers are weighed.

6. Calculation

The oil and fat content is given in mg per litre

Mass concentration r [mg/l] = filtrate volume [I]

No.	Mass extraction	Mass extraction	Filtrate volume	Weight at the end of
	beaker before	beaker after	[ml]	analysis [mg]
	extraction [g]	extraction [g]		
1				
2				
3				
4				
5				
6				

