



# Application Note

## No. 306/2017, Version B

Oil and grease in waste water

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Extraction Unit E-816 SOX:  
Determination of oil and grease in waste water according to Standard  
Methods AWWA 5520



## 1. Introduction

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This Application Note describes the extraction and determination of oil and grease from waste water samples following Standard Methods 5520 part D (Soxhlet Extraction Method). "Oil and grease" is defined as any material recovered as a substance soluble in the solvent [1]. Therefore, compounds with similar physical properties will be determined as well as oil and grease (e.g. sulfur compounds, organic dyes, chlorophyll).

The waste water sample is acidified with hydrochloric acid and extracted using a Soxhlet extraction method on the Extraction Unit E-816 SOX. After the extract has been dried to a constant weight, the amount of oil and grease is determined gravimetrically.

This Application Note is suitable for biological lipids and mineral hydrocarbons, as well as for most industrial waste water. It is not applicable to low-boiling point fractions which vaporize below 85 °C [1].

For this Application Note distilled water was spiked with

- i) sunflower seed oil (certified reference material)
- ii) hexadecane and stearic acid

A blank was determined as well.

## 2. Equipment

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- Extraction Unit E-816 SOX
- Recirculation chiller F-308
- Water jet pump
- Analytical balance (accuracy  $\pm 0.1$  mg)
- Drying oven

## 3. Chemicals and materials

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Chemicals:

- n-Hexane, AnalaR NORMAPUR, VWR (2460.321)
- Acetone, ACS reagent,  $\geq 99.5$  %, Sigma Aldrich (179124)
- Hydrochloric acid, 37 %, Fluka (84422)
- Celite Hyflo Supercel, VWR (1.02688.1000)

For safe handling please pay attention to all corresponding MSDS.

Materials:

- Extraction cellulose thimble, 33 x 94, BUCHI (11058983)
- Buchner funnel, for filter paper diameter 11 cm
- Filter flask that holds at least 1 L
- Filter paper, Whatman No. 40, diameter 11 cm, Sigma Aldrich (Z241288 ALDRICH)
- Muslin cloth disk, 100 % cotton, diameter 15 cm, solvent extracted
- Glass stick
- Cottonwool
- Desiccator

Samples:

- Distilled water
- For spiking:
  - i) Sunflower seed oil, certified reference material, Supelco (47123)
  - ii) Hexadecane, analytical standard, Supelco (442679)  
Stearic acid, puriss  $\geq 98.5$  %, Fluka (85680)  
Weigh 200 mg  $\pm$  2 mg hexadecane and 200 mg  $\pm$  2 mg stearic acid in a 100 mL volumetric flask and fill to the mark with acetone [2]

## 4. Procedure

The determination of oil and grease in waste water includes the following steps:

- Cleaning of sample container
- Acidification of the waste water sample
- Filtration and sample transfer
- Drying of sample
- Extraction of oil and grease using Extraction Unit E-816 SOX
- Drying of the extract
- Calculation of the result

### 4.1. Cleaning of sample container

1. Wash the glass bottle with soap and rinse with water.  
Rinse with solvent to remove residual oil and grease.

As an alternative to solvent rinsing, the bottle can be capped with alumina foil and baked at 200-250 °C for 1 h.

### 4.2. Acidification of the waste water sample

2. Weigh the empty bottle, fill with the water samples and weigh again. As an alternative, you can mark the sample volume in the bottle and determinate the volume later.
3. Acidify the waste water sample with 5 mL hydrochloric acid (1:1) to pH 2 or lower.
4. For spiking:
  - i) Add 100 mg of sunflower seed oil to the water sample to produce a 100 ppm oil and grease sample.
  - ii) Add 10 mL of the hexadecane / stearic acid spike solution to the water sample to produce a 40 ppm oil and grease sample.

### 4.3. Filtration and sample transfer

5. Assemble the extraction thimbles with the holders for paper thimbles.
6. Prepare the Buchner funnel for filtration: place a muslin cloth disk and a filter paper in it as shown in Figure 1. Wet both with distilled water.
7. Pass 100 mL Celite Hyflo Supercel solution (10 g/L) through it by applying vacuum. Wash with 1 L distilled water and apply vacuum until no further water passes through. The obtained Celite filter bed is shown in Figure 2.



*Fig. 1: Filtration flask with Buchner funnel, muslin cloth and filter.*



*Fig. 2: Buchner funnel, muslin cloth and filter after passing Celite Hyflo Supercel solution through.*



*Fig. 3: Wiping the sample container with cotton wool soaked in solvent.*

8. Filtration of acidified sample: add the water sample to the filter by guiding with a glass stick. Apply vacuum until no more water is passes through.
9. Wipe the sample container and the cap thoroughly with cotton wool soaked in solvent and add to filter as shown in Figure 3.



10. Wipe the glass stick with cottonwool twice and add it to the filter.
11. Remove the muslin cloth and filter using tweezers, roll it and place it in the extraction thimble.
12. Wipe the Buchner funnel with cottonwool and add it to the extraction thimble.

#### 4.4. Drying of sample

13. Dry the extraction thimble containing the sample in a drying oven at 103 °C for 30 min.

#### 4.5. Extraction of oil and grease using the Extraction Unit E-816 SOX

##### 4.5.1. Preparation of the beakers

14. Use clean and dry beakers for the extraction: add three boiling stones and dry them for at least 30 min at 103 °C in a drying oven.
15. Let the beakers cool down to ambient temperature in a desiccator for at least 1 h.
16. Record the exact weight prior to extraction.

##### 4.5.2. Soxhlet extraction

17. Place the thimble in the extraction chamber.
18. Adjust the optical sensor to the height of the samples.
19. Fill the beakers with solvent and place them on the heating plate.
20. Activate the corresponding positions in the menu.
21. Close the safety shield and move the rack down.
22. Open the cooling water or switch on the connected chiller.
23. Start the extraction according to the parameters listed in Table 1.

Table 1: Parameters for the extraction using the Extraction Unit E-816 SOX

Solvent	<i>n</i> -Hexane	
Extraction steps :		
1 Extraction	240 min	heater 120 %
2 Rinse	5 min	heater 120 %
3 Drying	20 min	heater 120 %
Solvent volume	120 mL	

#### 4.6. Drying of the extracts

24. Dry the beakers for at least 30 min at 103 °C in a drying oven.
25. Let the beakers cool down to ambient temperature in a desiccator for at least 1 h. Make sure that the cooling down time of the beakers in the desiccator is the same before and after extraction. Differences in beaker temperature falsify the results. Record the exact weight after the extraction.

#### 4.7. Calculation

The results are calculated according to equation (1).

$$\text{Blank} = \frac{(m_{\text{Total}} - m_{\text{Beaker}}) * 1000}{V_{\text{Sample}}} \quad (1)$$

$$\text{Oil and grease} = \frac{(m_{\text{Total}} - m_{\text{Beaker}}) * 1000}{V_{\text{Sample}}} - \text{blank} \quad (2)$$

$$\text{Recovery} = \frac{\text{oil and grease}}{m_{\text{spike}}} * 100 \quad (3)$$

Blank	: residue of extraction without sample [mg/L]
$m_{\text{Total}}$	: beaker + extract [g]
$m_{\text{Beaker}}$	: empty beaker weight [g]
$V_{\text{Sample}}$	: initial sample volume [L]
Oil and grease	: amount of oil and grease in the sample [mg/L]
Recovery	: recovery [%]
$m_{\text{spike}}$	: amount of spike [mg]

## 5. Result

The oil and grease content in waste water samples were determined with a

- Sun flower seed oil spike
- Hexadecane / stearic acid spike

### 5.1. Sunflower seed oil spike

Table 2: Blank content in water sample.

$m_{\text{Beaker}}$ [g]	$m_{\text{total}}$ [g]	$m_{\text{Extract}}$ [mg]	$V_{\text{sample}}$ [L]	Blank [mg/L]
98.6128	98.6185	5.7	0.9470	6.02
101.1315	101.1280	6.5	0.9351	6.95
98.6606	98.6666	6.0	0.9801	6.12
<b>Mean value [%]</b>				<b>6.54</b>
rsd [%]				<b>7.82</b>

Table 3: Oil and grease content in water sample; sample spiked with 100 mg of sunflower seed oil. The blank is taken into consideration.

$m_{\text{Beaker}}$ [g]	$m_{\text{total}}$ [g]	$m_{\text{Extract}}$ [mg]	$m_{\text{spike}}$ [mg]	$V_{\text{sample}}$ [L]	Oil and grease [mg/L]	Recovery [%]
99.0459	99.1507	104.8	99.5	0.9779	100.64	101.14
100.1253	100.2098	84.5	82.9	0.9792	79.76	96.21
99.1792	99.2854	106.2	102.7	0.9292	107.75	104.92
<b>Mean value</b>						<b>100.76</b>
sd						4.37
rsd [%]						<b>4.34</b>

### 5.2. Hexadecane / stearic acid spike

Table 4: Blank content in water sample.

$m_{\text{Beaker}}$ [g]	$m_{\text{total}}$ [g]	$m_{\text{Extract}}$ [mg]	$V_{\text{sample}}$ [L]	Blank [mg/L]
98.1216	98.1257	4.1	0.9803	4.18
97.2786	97.2837	5.1	0.9571	5.33
97.9207	97.9250	4.3	0.9692	4.44
<b>Mean value [%]</b>				<b>4.88</b>
rsd [%]				<b>12.33</b>

Table 5: Oil and grease content in water sample; sample spiked with 40 mg of hexadecane / stearic acid. The blank is taken into consideration.

$m_{\text{Beaker}}$ [g]	$m_{\text{total}}$ [g]	$m_{\text{spike}}$ [mg]	$V_{\text{sample}}$ [L]	Oil and grease [mg/L]	Recovery [%]
98.1633	98.2057	42.4	0.9775	38.49	96.24
99.2489	88.2909	42.0	1.0065	36.85	92.12
98.6802	98.7204	40.2	1.0067	35.05	87.63
<b>Mean value</b>					<b>91.99</b>
sd					4.30
rsd [%]					<b>4.68</b>

## 6. Remarks

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- a) Muslin cloth disk

The muslin cloth has to be 100 % cotton. It is mandatory to clean it using solvent before use.

- b) Extraction Unit E-816 SOX

The Extraction Unit E-816 SOX is dedicated to the fat determination. As alternative the determination of oil and grease in waste water samples can be performed using the Extraction System B-811 which is dedicated to environmental determinations.

## 7. Conclusion

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The oil and grease content of waste water samples can be determined using the Extraction Unit E-816 SOX following Standard Methods AWWA 5520. The mean recovery of the certified reference material sunflower seed oil was 100.8 %.

## 8. References

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- [1] Standard Methods AWWA 5520 Oil and Grease; approved by Standard Methods Committee, 2001.
- [2] EPA Method 1664, Revision B: n-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated n-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry; February 2010.