Application Note
No. 374/2019

Oil and grease in waste water

UniversalExtractor E-800:
Determination of oil and grease in waste water according to Standard Methods AWWA 5520

1. Introduction

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 UniversalExtractor E-800. 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 sunflower seed oil (certified reference material). A blank was determined as well.

2. Equipment

- UniversalExtractor E-800 Pro (with chamber heater)
- Water jet pump
- Analytical balance (accuracy ± 0.1 mg)
- Drying oven

3. Chemicals and materials

Chemicals:

- n-Hexane, AnalaR NORMAPUR, VWR (2460.321)
- Acetone, ACS reagent, ≥ 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 150, BUCHI (11067446)
- 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: Sunflower seed oil, certified reference material, Supelco (47123)

4. Procedure

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

- Cleaning of sample container and muslin cloth
- Acidification of the waste water sample
- Filtration and sample transfer
- Drying of sample

- Extraction of oil and grease using UniversalExtractor E-800
- Drying of the extract
- Calculation of the result

4.1. Cleaning of sample container and muslin cloth

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.

2. Boil the muslin cloth in hexane for 20 min.

4.2. Acidification of the waste water sample

3. 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.
4. Acidify the waste water sample with 5 mL hydrochloric acid (1:1) to pH 2 or lower.
5. For spiking:
   - Add 100 mg of sunflower seed oil to the water sample to produce a 100 ppm oil and grease sample.
6. Measure a blank sample as well.

4.3. Filtration and sample transfer

7. Assemble the extraction thimbles with the holders for paper thimbles.
8. 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.
9. 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 cottonwool soaked in solvent.

10. 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.
11. Wipe the sample container and the cap thoroughly with cottonwool soaked in solvent and add to filter as shown in Figure 3.
12. Wipe the glass stick with cottonwool twice and add it to the filter.
13. Remove the muslin cloth and filter using tweezers, roll it and place it in the extraction thimble.
14. Wipe the Buchner funnel with cottonwool and add it to the extraction thimble.

4.4. Drying of sample

15. 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 UniversalExtractor E-800

4.5.1. Preparation of the beakers

16. Use clean and dry beakers for the extraction. Dry them for at least 30 min at 103 °C in a drying oven.
17. Let the beakers cool down to ambient temperature in a desiccator for at least 1 h.
18. Record the exact weight prior to extraction.

4.5.2. Soxhlet extraction

19. Place the thimble in the extraction chamber.
20. Adjust the optical sensor to the height of the sample.
21. Fill the beakers with solvent and place them on the heating plate.
22. Activate the corresponding positions in the menu.
23. Close the safety shield.
24. Start the extraction according to the parameters listed in Table 1.

Table 1: Extraction method for UniversalExtractor E-800.

Parameter	Value		
Extraction method	Soxhlet		
Solvent	n-Hexane		
Solvent volume	150 mL		
Extraction:			
Time	240 min		
Heating level	11		
Rinse:			
Time	5 min		
Heating level	11		
Dry:			
Time 1	Analyte protection	☑	0 min
Heating level	11		
Time 2	Analyte protection	☐	5 min
Heating level	5		
Time 3	Analyte protection	☐	5 min
Heating level	0		

4.6. Drying of the extracts

25. Dry the beakers for at least 30 min at 103 °C in a drying oven.
26. 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.

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

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

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

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

Table 2: Blank content in water sample.

m_{Beaker} [g]	m_{total} [g]	$m_{Extract}$ [mg]	V_{sample} [L]	Blank [mg/L]]
110.7754	110.7803	4.9	0.9805	5.0
110.8154	110.8194	4.0	0.9843	4.0
110.2852	110.2900	4.8	0.9693	4.9
Mean value [%]				**4.6**
rsd [%]				**11.27**

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_{Beaker} [g]	m_{total} [g]	$m_{Extract}$ [mg]	m_{spike} [mg]	V_{sample} [L]	Oil and grease [mg/L]]	Recovery [%]
111.1160	111.2364	120.4	116.1	0.9817	117.98	101.62
111.2416	111.3466	105.0	100.8	0.9925	101.13	100.33
111.3614	111.4693	107.9	100.5	0.9769	105.78	105.25
Mean value						**102.40**
sd						2.56
rsd [%]						**2.50**

6. Remarks

a) Muslin cloth disk

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

7. Conclusion

The oil and grease content of waste water samples can be determined using the UniversalExtractor E-800 following Standard Methods AWWA 5520. The mean recovery of the certified reference material sunflower seed oil was 102.40 %.

8. References

[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.