

Oil and grease in waste water

Determination of oil and grease in waste water according to Standard Methods AWWA 5520

This Short 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).

1. Introduction

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 Short 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 Short Note distilled water i) spiked with sunflower seed oil (certified reference material) or ii) spiked with ii) hexadecane and stearic acid was used. A blank was determined as well.

2. Experimental

1 L water sample was spiked with i) sunflower seed oil or with ii) hexadecane and stearic acid. The sample was acidified with hydrochloric acid and filtrated through a muslin cloth and a filter paper covered with a Celite Hyflo Supercel suspension. Prior to extraction the filter was dried in a drying oven. The extraction was carried out on the Extraction Unit E-816 SOX using a Soxhlet extraction method. After the extract has been dried to a constant weight, the amount of oil and grease is determined gravimetrically. A blank was determined as well.



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

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

| Solvent | n-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 | |

3. Results

Table 2: Blank content in water sample.

| m _{Beaker} [g] | m _{total} [g] | m _{Extract} [mg] | V _{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 |
| Mean value [mg/L] | | | | 6.54 |
| rsd [%] | | | | 7.82 |

Table 3: Oil and grease content in water sample; sample spiked with 100 mg of sunflower seed oil (blank value: 6.54 mg/L).

| m _{Extract} [g] | m _{spike} [g] | V _{sample} [L] | Oil and grease [mg/L] | Recovery [%] |
|--------------------------|------------------------|-------------------------|-----------------------|---------------|
| 104.8 | 99.5 | 0.9779 | 100.64 | 101.14 |
| 84.5 | 82.9 | 0.9792 | 79.76 | 96.21 |
| 106.2 | 102.7 | 0.9292 | 107.75 | 104.92 |
| Mean value [%] | | | | 100.76 |
| rsd [%] | | | | 4.34 |

Table 4: Blank content in water sample for determination with hexadecane and stearic acid spike.

| m _{Beaker} [g] | m _{total} [g] | m _{Extract} [mg] | V _{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 |
| Mean value [mg/L] | | | | 4.88 |
| rsd [%] | | | | 12.33 |

Table 5: Oil and grease content in water sample; sample spiked with 40 mg of hexadecane and stearic acid (blank value 4.88 mg/L).

| m _{spike} [g] | V _{sample} [L] | Oil and grease [mg/L] | Recovery [%] |
|------------------------|-------------------------|-----------------------|--------------|
| 42.4 | 0.9775 | 38.49 | 96.24 |
| 42.0 | 1.0065 | 36.85 | 92.12 |
| 40.2 | 1.0067 | 35.05 | 87.63 |
| Mean value [%] | | | 91.99 |
| | | | 4.68 |

4. Conclusion

The oil and grease content of waste water samples can be determined using the Extraction Unit E-816 SOX following Standard Methods AWWA 5520.

5. References

[1] Standard Methods AWWA 5520 Oil and Grease; approved by Standard Methods Committee, 2001.

For more detailed information and safety considerations please refer to the Application Note No. 306/2017 Version B.