Determination of Mineral Oil Hydrocarbons by Means of Thin-Layer Chromatography – Analytical Directions

C. Reimers
Technical University of Hamburg-Harburg, Department of Waste Management, Harburger Schloßstraße 37, 21079 Hamburg, Germany

38.1 Range of Application

The detection limit of thin layer chromatography is at 0.1 µg absolute for squalane and lubricating oil respectively, and at 0.4 µg absolute for diesel fuel. Under the conditions indicated below, this equates to detection levels of 170 mg kg⁻¹ squalane and lubricating oil, and 670 mg kg⁻¹ diesel fuel. Smaller contents can be determined by spreading greater volumes. At a spread volume of 20 µl 8 mg kg⁻¹ lubricating oil and 30 mg kg⁻¹ diesel fuel can be detected.

38.2 Principle of the Method

The determination of medium and low volatile mineral oil hydrocarbons (aliphatics and naphthene) in soil materials is based on their separation from other substance classes such as aromatic hydrocarbons, lipids and other compounds and subsequent determination by summation. The extraction of the soil samples is carried out using a mixture of n-hexane and acetone (1:1) with ultrasonic support. The total petroleum hydrocarbons are developed on silica gel 60 phases in n-hexane and appear after colouring as one spot at a hRf-value of about 80. Polycyclic aromatics are recognisable distinctly from the aliphatic compounds. Prerequisite for the in-situ-detection of the aliphatics on silica gel layers is their visibility caused by suitable colouring agents.

As detection reagents suitable are anilinnaphalinsulfonic acid (Gitler 1972), bromothymolblue (Goebgen and Brockmann 1977), sodiumfluoresceinate (Mamlock 1981), berberinechloride (Jork et al. 1990), such as acid violet (Engelhardt and Engel 1997). The evaluation can be carried out visually or densitometrically by means of a thin-layer-scanner.
38.3 Chemical Reagents

- Hexane,
- acetone,
- diesel fuel (1): 200 mg in 10 ml hexane or heptane/acetone, 1:100 to be diluted (0.2 μg μl⁻¹),
- lubricating oil (2): 200 mg in 10 ml hexane or heptane/acetone, 1:200 to be diluted (0.1 μg μl⁻¹),
- methanol,
- ethanol,
- citric acid monohydrate,
- anilinonaphthalenesulfonic acid: 100 mg 8-anilinonaphthalene-1-sulfonic acid as ammoniumsalt are dissolved in the following mixture and well shaken: a) 20 ml of a 0.2 M NaOH-solution and 20 ml ethanol, b) 57 ml of a solution of 2.1 g citric acid monohydrate and 0.8 g NaOH-flakes in 100 ml water.

38.4 Equipment

- Ultrasonic device,
- spreading device with syringe,
- centrifuge glasses: 50 ml with screw lid and PTFE-coated lid inlays,
- laboratory centrifuge,
- HPTLC-plates: silica gel 60 phases with concentration zone, clean-up in hexane or heptane, subsequently in methanol or i-propanole, then 10 min of air-drying,
- TLC-development chambers,
- UV-observation device,
- TLC-device for plate immersion,
- hair-dryer,
- plate heater,
- TLC-scanner.

38.5 Extraction

- 5–10 g homogenised naturally humid soil to be accurately weighed into a 50 ml centrifuge glass with screw lid (Teflon sealing). Drying of the soil is not necessary.
- 10 or 20 ml of a mixture consisting of hexane (or heptane) /acetone 1/1 are to be added. n-Hexane, i-hexane, cyclohexane can be used but n-heptane is less neurotoxic.
- Shake vigorously and extract in an ultrasonic bath (30 min, full performance). Force fields, water level and temperature of the ultrasonic bath are to be care-