

Mineral oils

Application Note

Environmental

Authors

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Introduction

The determination of hydrocarbons and lubrication oil by GC and FID detection according to DIN H53 is a common method used in environmental labs. The DIN refers to sample preparation and the determination of hydrocarbons as a sum parameter. The difficulty for this application is the separation of the lubrication oil from hydrocarbons C_{10} - C_{40} . On most GC columns, the lubrication oil coelutes with the later eluting hydrocarbons. This application shows a sample preparation method and determination of the extract by GC/FID using an Agilent FactorFour low bleed column.



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Conditions

Technique	: GC-capillary
Column	: Agilent FactorFour VF-5ms, 0.20 mm x 12 m fused silica (df = 0.33 μm) (Part no. CP8935)
Temperature	: 50 $^{\circ}\text{C}$ / 1 min with 10 $^{\circ}\text{C}/\text{min}$ to 200 $^{\circ}\text{C}$, with 30 $^{\circ}\text{C}/\text{min}$ to 300 $^{\circ}\text{C}/5$ min
Carrier Gas	: Nitrogen, 40 kPa, 0.4 bar
Injector	: Splitless, splitless time, 1 minute T = 350 $^{\circ}\text{C}$
Detector	: FID T = 350 $^{\circ}\text{C}$
Sample Size	: 1 μL
Courtesy	: Dipl.-Ing. J. Horst, Prof. Dr. Harald Weber Niederrhein, University of Applied Sciences, Krefeld Dipl.-Ing. P. Gerhards, Agilent Deutschland, GmbH, Darmstadt

Sample preparation

For the extraction, 20 g of soil are extracted in 50 mL of cyclohexene. The method used for this application is based on a cold extraction, where the soil sample is added to a glass shaker and then shaken for 1 hour. Afterwards, the sample is evaporated close to dryness and made up to 5 mL with cyclohexene.

For clean up, an Agilent Bond Elut 500 mg, 120 μm , 10 mL silica cartridge (p/n 14113036) was used. The sample was applied to the cartridge and washed with 5 mL cyclohexene, to remove polar substances. Then the sample is again evaporated and made up to 5 mL. 1 mL of this solution is placed into a glass vial and 1 μL is injected splitless into the GC.

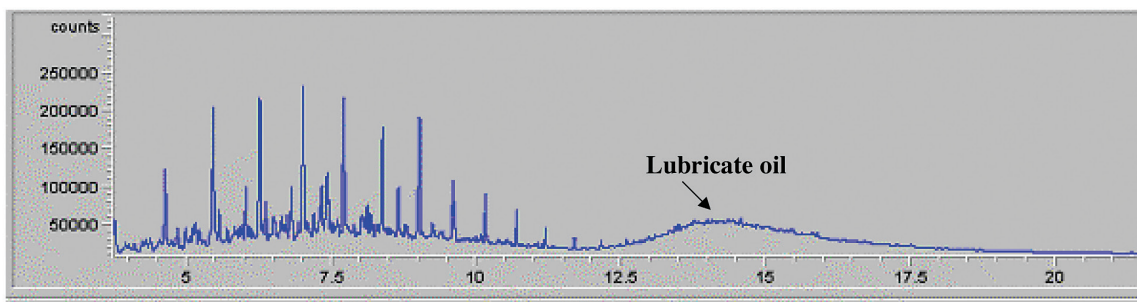


Fig. 1 Diesel and lubricate oil standard 50 $\mu\text{g}/\text{ml}$ Diesel and 50 $\mu\text{g}/\text{ml}$ lubricate oil.

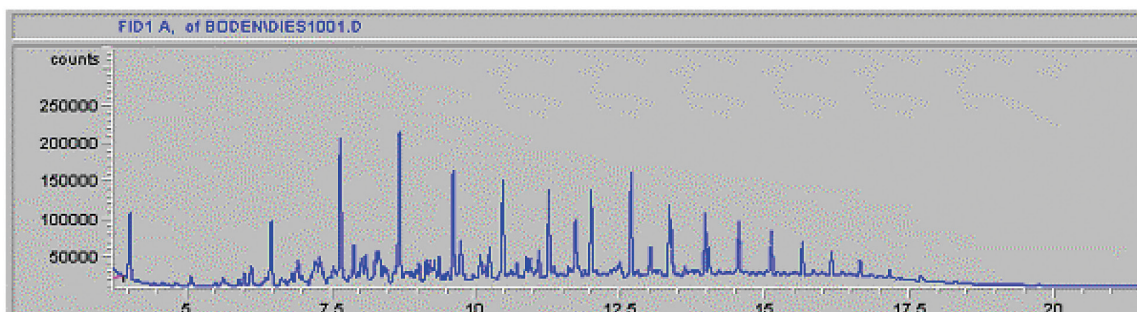


Fig. 2 Real soil sample extracted with Cyclohexene and clean up via Bond Elut Si cartridge; The real sample contained 200 ppm Diesel

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