Hydrocarbons C_3 – C_{32}

Thermal desorption of rock sample

Application Note

Energy & Fuels

Authors

Agilent Technologies, Inc.

Introduction

Gas chromatography using Agilent columns separates 32 components from the thermal desorption of a potential oil-bearing rock.

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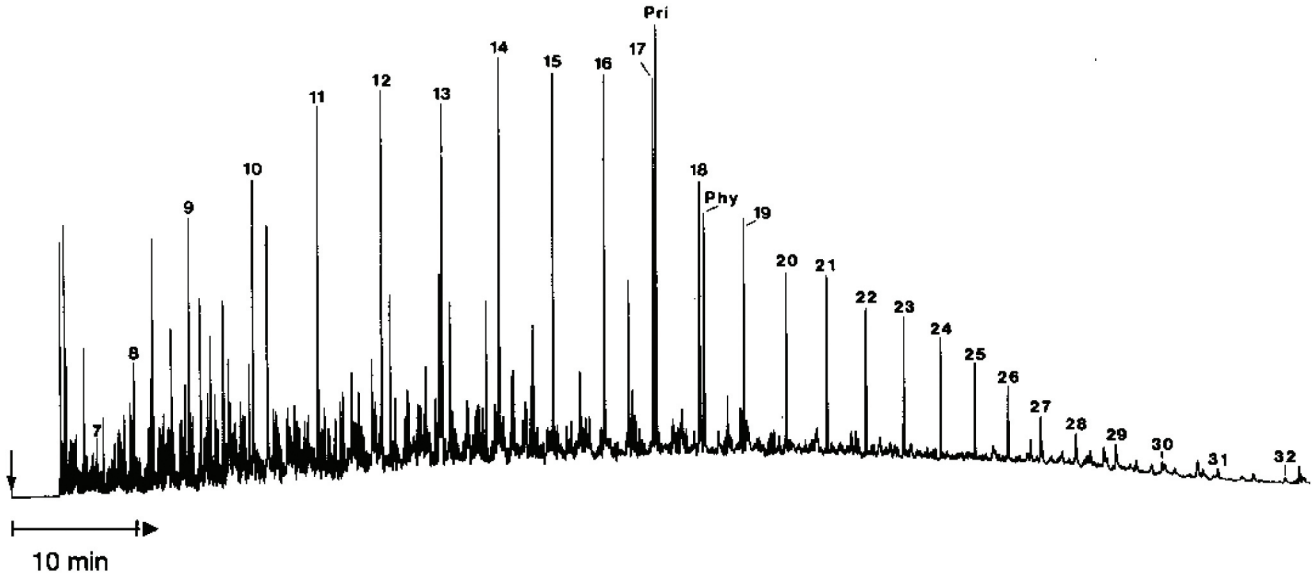
Conditions

Technique	:	GC-capillary
Column	:	Agilent, 0.32 mm x 50 m fused silica WCOT SE54 (0.2 µm) (Part no. CP6047)*
Cold Trap	:	0.5 mm fused silica coated with CP-Sil 5 CB (5 µm)
Desorption Flow	:	desorption : 300 °C (15 min) cryofocussing : → 120 °C injection : 200 °C (15 min) oven : 30 °C → 280 °C; 3 °C/min
Injector	:	200 °C
Detector	:	FID
Carrier Gas	:	He
Courtesy	:	Dr. Schaefer (Institut für Erdöl und Organische Geochemie, KFA Jülich GmbH, Jülich, Germany) (1)



* CP-Sil 8 CB is advised as chemically bonded equivalent of SE-54

Chromatogram 1

Analysis of hydrocarbons in sedimentary rocks with the TCT

The problem with the determination of the oil and gas potential in rocks is the extremely wide volatility range of the hydrocarbons: between one and more than 30 carbon atoms. Solvent extraction followed by LC and GC analysis is suitable for compounds > C_{11}, together with headspace analysis (<C_7) and thermo-vaporization (C_8-C_{14}). The Thermal Desorption Cold Trap injector (combined with a capillary GC) provides an attractive approach for the one-step analysis of the complete range of hydrocarbons found in rock samples and coals.

Method

The finely ground sample (1-5 mg) is placed in the desorption tube. After desorption (250-300 °C), the components are trapped at -120 °C. By heating the cold trap to 200 °C the components are injected into the capillary column.

Results

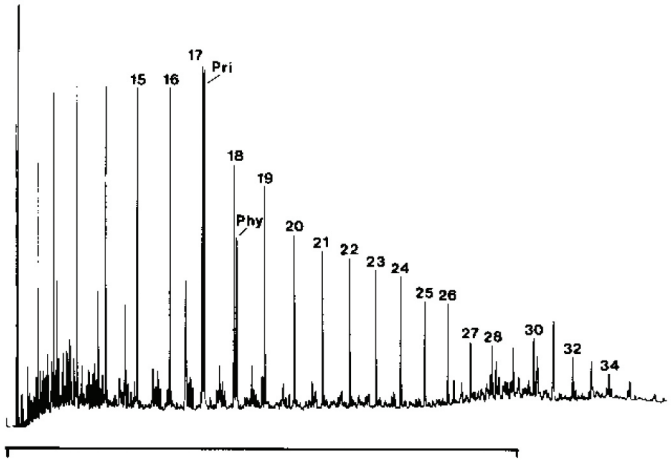
Two techniques are shown in the chromatogram; 1 shows a thermal desorption run of a potential oil source rock, 2 shows the analysis of the same rock after extraction with dichloromethane and fractionation with MPLC. It is clear that the range covered by the thermal desorption method is far greater than with solvent extraction. In view of the pore structure of the rock matrix and the very high boiling points in the C_{30} range it is not surprising that thermal desorption tends to be slightly less effective in this range than exhaustive extraction. This could be improved by increasing the desorption time and/or temperature.

Significant amounts of a-olefins (common pyrolysis products of sedimentary organic matter) did not form. Use of the TCT will provide useful parameters for various aspects of (petroleum) geochemistry, such as generation and migration of hydrocarbons, mass balance calculations on a molecular basis, and source rock/crude oil correlations.

Conditions

Technique	: GC-capillary
Column	: Agilent, 0.32 mm x 25 m fused silica WCOT methyl-phenyl (5 %) silicone (0.1 µm) *
Temperature	: 90 °C (2 min) ⟶ 285 °C, 3 °C/min
Injector	: Splitter, 1:50 ⟶ 275 °C
Detector	: FID
Carrier Gas	: He

* Note: the corresponding column is 0.32 mm x 25 m fused silica WCOT CP-Sil 8 CB (0.12 µm) (Part no. CP7741)



Chromatogram 2

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