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REPORT

TITLE

Analysis of headspace and occluded gas (C1-C9) in nine canned cuttings

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SUMMARY

This report contains tables and figures with data from gas chromatographic analysis of headspace and occluded gas from nine canned cutting samples from an undisclosed location. The yields (in $\mu\text{l}/\text{kg}$ dry rock) and relative proportions (in volume %) of 23 hydrocarbon compounds ranging from C1 to C9 are tabulated. Some geochemically relevant peak ratios are also listed and plotted.

KEYWORDS ENGLISH

Gas analysis
Organic geochemistry

KEYWORDS NORWEGIAN

Gassanalyse
Organisk geokjemi

1. Introduction

Ten canned cutting samples were received from Hydro Oil & Energy (Contract No.: 5313623) for gas chromatographic analysis of the C1 to C9 hydrocarbons contained in the headspace and occluded gas.

One of the samples could not be analyzed due to an open lid by arrival, and the report will therefore contain results from nine samples. The hydrocarbon concentrations are expressed as μl gas per kg of dried cuttings. The hydrocarbon composition is expressed in volume percent of all recorded hydrocarbons.

2. Experimental

2.1 Headspace gas

A septum was attached to the can and a sample of headspace gas was taken and injected into a gas chromatograph for analysis of C1 to C9 hydrocarbons.

The can was opened and the volume of the headspace was determined. The cuttings were washed with water (ca. 30 °C) on 4.0, 1.0 and 0.125 mm sieves in order to remove the drilling mud, and were then weighed and dried.

2.2 Occluded gas

Prior to drying, an aliquot of the 1-4 mm fraction was crushed in water for 10 minutes using a gas-tight ball mill. An aliquot of the evolved gas was injected into a gas chromatograph for analysis of C1 to C9 hydrocarbons.

2.3 Gas chromatographic analysis

The gas was analysed on a gas chromatograph fitted with a gas injector. The GC temperature program started at 35 °C, since separation of alkenes from alkanes was of no interest in this project. The instrument was fitted with a capillary column connected to an FID for hydrocarbon detection. Details of the instrumentation are listed in Table 2.1.

Table 2.1 Analytical equipment

| | |
|-----------------------------|---|
| Gas chromatograph | Agilent 6890 |
| Injector | Gas injector connected to a 1.0 ml loop |
| Columns | HP-PONA column: 50 m x 0.20 mm i.d, 0.5 μm film thickness. |
| Carrier gas | Helium |
| Detector | FID (250 °C) |
| Temperature program | 35 °C (5 min.) - 8 °C/min. - 180 °C (10 min.) |
| Chromatographic data system | HP ChemStation Rev. A.10.01 |

2.4 Identification

Peaks were identified based on three Supelco Reference Standards, guidelines in “The Norwegian Industry Guide to Organic Geochemical Analyses” (Edition 4.0) and internal procedures. Figure 2.1 shows an example of a gas chromatogram with annotation from the method used in this project. Identified compounds, retention indices and comments are listed in Table 2.2.

Table 2.2 Identified compounds with retention indices (RI) and comments. For peak labels see explanation below the table.

| Peak label | RI (Kováts) | Comments |
|-----------------------------------|-------------|------------------------------|
| C1 | 100 | |
| C2 | 158 + 200 | Includes ethane and ethene |
| C3 | 300 | Includes propane and propene |
| iC4 | 354 | |
| C4ene | 385 | |
| nC4 | 400 | |
| 2,2-DMC3 | 410 | |
| RI=431 | 431 | Unknown |
| iC5 | 467 | |
| RI=485 | 485 | Unknown |
| RI=493 | 493 | Unknown |
| nC5 | 500 | |
| RI=529 | 529 | Unknown |
| 2,2-DMC4 | 531 | Not found in these samples |
| CyC5 + 2,3-DMC4 | 561 | Not found in these samples |
| 2-MC5 | 565 | Not found in these samples |
| 3-MC5 | 581 | |
| nC6 | 600 | |
| 2,2-DMC5 | 624 | Not found in these samples |
| MCyC5 | 627 | Not found in these samples |
| 2,4-DMC5 | 630 | Not found in these samples |
| 2,2,3-TMC4 | 637 | Not found in these samples |
| Benzene | 651 | Not found in these samples |
| 3,3-DMC5 | 656 | |
| CyC6 | 661 | Not found in these samples |
| 2-MC6 | 668 | Not found in these samples |
| 2,3-DMC5 | 670 | Not found in these samples |
| 1,1-DMCyC5 | 674 | Not found in these samples |
| 3-MC6 | 676 | Not found in these samples |
| c-1,3-DMCyC5 | 684 | Not found in these samples |
| t-1,3-DMCyC5 | 687 | Not found in these samples |
| t-1,2-DMCyC5 + 3-EC5 + 2,2,4-TMC5 | 690 | |
| nC7 | 700 | |
| MCyC6 + c-1,2-DMCyC5 | 725 | Not found in these samples |
| 2,2-DMC6 + 1,1,3-TMCyC5 | 727 | Not found in these samples |
| 2,5-DMC6 + 2,2,3-TMC5 | 734 | Not found in these samples |
| 2,4-DMC6 | 736 | Not found in these samples |
| 3,3-DMC6 + t-1,c-2,4-TMCyC5 | 744 | Not found in these samples |
| t-1,c-2,3-TMCyC5 | 751 | Not found in these samples |
| 2,3,4-TMC5 | 754 | Not found in these samples |
| Toluene + 2,3,3-TMC5 | 759 | Not found in these samples |
| 2,3-DMC6 | 763 | Not found in these samples |
| 2-M,3-EC5 | 765 | Not found in these samples |
| 2-MC7 | 768 | Not found in these samples |
| 4-MC7 + 3-M,3-EC5 | 769 | Not found in these samples |
| 3,4-DMC6 | 773 | Not found in these samples |
| 3-MC7 + c-1,t-2,3-TMCyC5 | 775 | Not found in these samples |

| Peak label | RI (Kováts) | Comments |
|---------------------------|-------------|----------------------------|
| RI=781 (DMCyC6) | 780 + 781 | Not found in these samples |
| nC8 | 800 | |
| 2,3,5-TMC6 + c,1,2-EMCyC5 | 819 | Not found in these samples |
| E-Benzene | 855 | |
| m+p-Xylene | 863 | |
| 4-MC8 + 2-MC8 | 874 | Not found in these samples |
| o-Xylene | 886 | |
| nC9 | 900 | |

Explanations:

| Structural groups | Parent structures | Numbers of functional groups | Names of functional groups | Steric configurations |
|-------------------|-------------------|------------------------------|----------------------------|-----------------------------------|
| n = normal | C1 = methane | D = di | M = methyl | c = cis |
| i = iso | C2 = ethane | T = tri | E = ethyl | t = trans |
| Cy = cyclo | etc. | | P = propyl | o = ortho m = meta p = para |

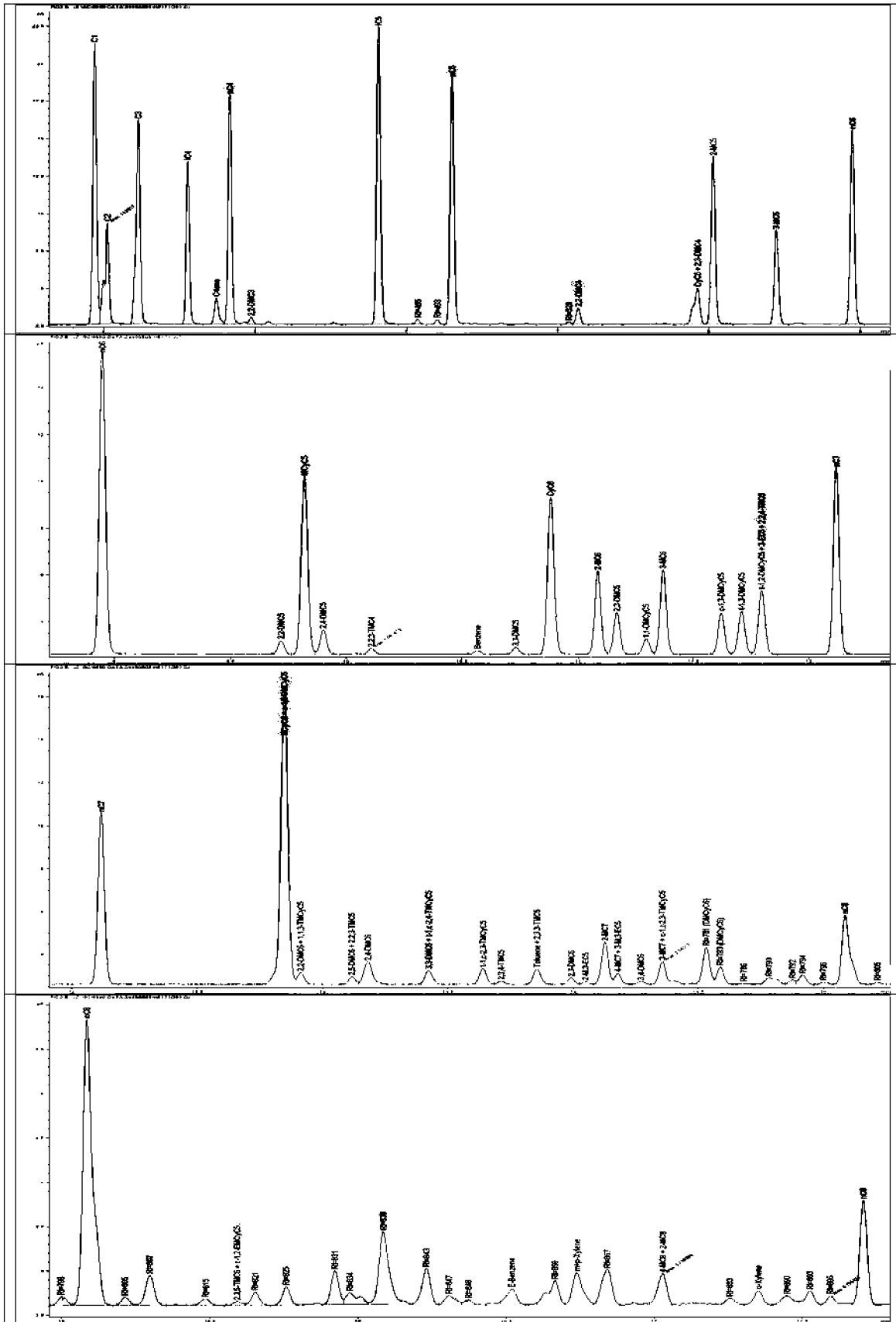


Figure 2.1 An example of a gas chromatogram with annotation. Expanded views of C1 to n-C6, n-C6 to n-C7, n-C7 to n-C8 and n-C8 to n-C9 ranges.

2.5 Quantification

A 1000 ppm ($\mu\text{l/l}$) standard gas sample containing methane, ethane, propane, i-butane, n-butane, n-pentane and n-hexane was used for quantification. The variation between the standards analysed was small (Table 2.3), and the average response factors were used for quantification of all samples. The response factor for n-C5 was used for all compounds eluting between n-C4 and n-C5 and so on. Response factors for the C6-C7, C7-C8 and C8-C9 component groups were extrapolated based on molecular mass.

A 100 ppm ($\mu\text{l/l}$) standard gas sample containing methane, ethane, propane, n-butane, n-pentane and n-hexane was used for control of linearity.

Rock-related concentrations (in $\mu\text{l/kg}$) for compounds having a concentration of less than 0.2 ppm ($\mu\text{l/l}$) in the analysed gas are not reported, as this corresponds to an area of about 0.08, which is the lowest reliable area in the gas chromatograms.

Table 2.3 Results from analyses of 1000 ppm standard (based on peak area)

| | C1 | C2 | C3 | iC4 | nC4 | nC5 | nC6 |
|-----------------------|-----|-----|-----|-----|-----|-----|------|
| Average ppm | 994 | 991 | 991 | 992 | 993 | 996 | 1003 |
| Stdev. | 17 | 16 | 16 | 16 | 15 | 16 | 20 |
| Stdev. (% of average) | 1.7 | 1.6 | 1.6 | 1.6 | 1.5 | 1.6 | 2.0 |
| n | 6 | 6 | 6 | 6 | 6 | 6 | 6 |

2.6 Concentrations and ratios

The yields of hydrocarbons ($\mu\text{l/kg}$ dry rock) in headspace (H) and occluded (O) gas and the sum of H + O are given in Table 4.3, and the hydrocarbon composition (volume %) is given in Table 4.4. Selected summary data and peak ratios are calculated and given in Table 4.5. Abbreviations used in Table 4.5 are explained in Table 2.4.

Table 2.4 Explanations of the variables listed in Table 4.5.

| Abbreviation | Explanation |
|---|--|
| Hydrocarbon yields for selected C-number ranges | |
| CncC1 | Concentration of C1 ($\mu\text{l/kg}$ dry rock) |
| CncC2C4 | Concentration of C2 through n-C4 ($\mu\text{l/kg}$ dry rock) |
| CncC5C9 | Concentration of C5 through n-C9, i.e. all peaks eluting after n-C4 ($\mu\text{l/kg}$ dry rock) |
| CncC1C9 | Sum of these concentrations |
| Hydrocarbon composition for selected C-number ranges | |
| PctC1 | Fraction of C1 (% volume of all C1-C9 HC) |
| PctC2C4 | Fraction of C2 through n-C4 (% volume of all C1-C9 HC) |
| PctC5C9 | Fraction of C5 through n-C9, i.e. all peaks eluting after n-C4 (% volume of all C1-C9 HC). |
| PctC1C9 | Sum of these fractions (= 100 vol %) |
| Wetness | |
| Wetness | 100* (Sum C2 to n-C4 / (Sum C1 to n-C4) (volume %) |

2.7 Water content

The water content of the cuttings was determined by weighing the fractions before and after drying at 35 °C for at least 24 hours. The dry weight of the rock used for occluded gas analysis was determined using the wet weight of this rock and the water content of the remaining 1-4 mm fraction. Water contents for the three individual grain size fractions are listed in Table 4.2.

3. Comments on samples and analytical data

The wet cutting samples were received in pressure-lid cans of 1 l volume at ambient temperature. Secondary modification of the gas composition by microbial activity cannot be completely ruled out.

The samples were labelled '1A' to '1J'. One of the lids, '1F', was open by arrival and all the sample material had leaked out.

Some of the samples contained soft material and had to be cleaned extremely carefully to prevent loss of sample material. The adhering (and foaming) mud could hence not be removed completely. The mud in the samples '1C', '1D', '1E', '1G', '1H', '1I' and '1J' also contained a black, fine-grained material with almost graphite-like lustre. The material floated on the water and was decanted from the samples. However, a significant part remained in the 0.125 – 1 mm fractions.

Sample descriptions are listed in Table 4.1. Those of samples '1C', '1E', '1G', '1H' and '1I' were done on the 0.125 – 1 mm and the >4 mm fraction as all material from the 1-4 mm fraction was used for the occluded gas analyses.

Only 23 hydrocarbon compounds were identified in these samples. Some of the peak ratios in Table 4.5 and Figure 4.1 show erratic values. This is typically due to low concentrations. Care should, therefore, be taken in the interpretation of these ratios.

4. Results

Table 4.1
Sample description

Table 4.1 Sample description. Lithologies in order of decreasing abundance.

| Sample ID | External sample ID | Lithology (description on 1 – 4 mm fraction if not specified otherwise. numbers represent roughly estimated percentages) | Gas volume headspace (ml) | Gas volume occluded (ml) | Rock weight total sample dry (g) | Rock weight occluded sample dry (g) |
|-----------|--------------------|---|---------------------------|--------------------------|----------------------------------|-------------------------------------|
| H6174 | 1A | 100 marl?, ltgy; tr fragments of crystalline rocks (quartz-rich, with euhedral garnets) | 40 | 14.1 | 235.5 | 12.1 |
| H6175 | 1B | 90 marl?, ltgy; 10 fragments of crystalline rocks (euhedral garnets etc.); tr metal flakes | 20 | 19.5 | 168.3 | 15.3 |
| H6176 | 1C | >4 mm: 95 rounded "mud lumps" containing numerous spherical inclusions, ltgy; 5 basalt?, tr metal; 0.125 - 1.0 mm: Quartz sand + black additive | 174 | 32.5 | 268.3 | 7.5 |
| H6177 | 1D | 50 "mud lumps" containing spherical inclusions, ltgy; 30 marl? and carbonates; 20 crystalline rocks; tr plastic and metal flakes; 0.125 - 1.0 mm: Quartz sand + black additive | 194 | 29 | 329.2 | 12.0 |
| H6178 | 1E | >4 mm: (5 pieces) ltgy "mud lumps", black chert?, metal flake; 0.125 - 1.0 mm: Quartz sand + black additive | 396 | 24.4 | 449.5 | 13.2 |
| H6179 | 1F | ----- No analysis ----- | - | - | - | - |
| H6180 | 1G | >4 mm: (3 pieces) ltgy "mud lump", basalt, metal flake; 0.125 - 1.0 mm: Quartz sand + black additive | 170 | 27.2 | 802.3 | 4.7 |
| H6181 | 1H | 0.125 - 1.0 mm: Quartz sand + black additive | 120 | 34.4 | 260.0 | 9.9 |
| H6182 | 1I | >4 mm: "mud lumps" containing spherical inclusions, ltgy; 0.125 - 1.0 mm: Quartz sand + black additive | 155 | 29.5 | 136.0 | 11.3 |
| H6183 | 1J | very small and poorly washed sample: mostly crystalline (metamorphic?) rocks; >4 mm: 95 rounded "mud lumps" containing numerous spherical inclusions, ltgy; 5 various crystalline rock fragments; 0.125 - 1.0 mm: Quartz sand + black additive. | 160 | 20.2 | 280.2 | 15.9 |

Key: SH = shale, SST = sandstone, glauc = glauconitic, ADD = mud additive, tr = trace, v = very, fgr = fine grained, gy = grey, dk = dark, m = medium, lt = light.

Table 4.2
Water contents of different grain size fractions

Table 4.2 Water contents of different grain size fractions.

| Sample ID | External sample ID. | Water content >4 mm fraction (wt% of wet fraction) | Water content 4-1 mm fraction (wt% of wet fraction) | Water content 1-0.125 mm fraction (wt% of wet fraction) | Water content >0.125 mm (wt% of wet fraction) | |
|-----------|---------------------|--|---|---|---|--|
| H6174 | 1A | 42.2 | 36.5 | 30.6 | 34.3 | |
| H6175 | 1B | 31.5 | 24.2 | 21.2 | 22.2 | |
| H6176 | 1C | 50.0 | 30.0 ¹ | 21.8 | 23.4 | |
| H6177 | 1D | 44.8 | 30.4 | 19.3 | 21.7 | |
| H6178 | 1E | 25.0 | 30.0 ¹ | 19.7 | 20.0 | |
| H6179 | 1F | ---- No analysis ---- | | | | |
| H6180 | 1G | 28.6 | 30.0 ¹ | 16.9 | 17.0 | |
| H6181 | 1H | 0.0 | 30.0 ¹ | 18.3 | 18.9 | |
| H6182 | 1I | 20.8 | 30.0 ¹ | 18.2 | 19.3 | |
| H6183 | 1J | 20.9 | 23.7 | 19.0 | 19.5 | |

Comment:

- 1) Estimated value due to a very small sample amount

Table 4.3
Yield of hydrocarbons in headspace and occluded gas
($\mu\text{l}/\text{kg}$ dry rock)

Table 4.3 Yield of hydrocarbons in headspace and occluded gas ($\mu\text{l}/\text{kg}$ dry rock).

| Sample-ID | H6174 | H6174 | H6174 | H6175 | H6175 | H6175 | H6176 | H6176 | H6176 |
|-----------------------------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| Gas fraction | H | O | H+O | H | O | H+O | H | O | H+O |
| External ID. | 1A | 1A | 1A | 1B | 1B | 1B | 1C | 1C | 1C |
| Compound | | | | | | | | | |
| C1 | 549 | 72 | 621 | 1253 | 216 | 1469 | 992 | 606 | 1598 |
| C2 | 3 | 4 | 7 | 6 | 10 | 16 | 4 | 79 | 83 |
| C3 | 2 | 2 | 4 | 1 | 3 | 4 | 2 | 34 | 37 |
| iC4 | 0 | 0 | 1 | 0 | 0 | 0 | 0 | 0 | 0 |
| C4ene | 0 | 1 | 1 | 0 | 1 | 1 | 1 | 9 | 10 |
| nC4 | 0 | 1 | 1 | 0 | 1 | 1 | 0 | 7 | 7 |
| 2,2-DMC3 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| RI=431 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| iC5 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| RI=485 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| RI=493 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| nC5 | 0 | 1 | 1 | 0 | 0 | 0 | 0 | 6 | 6 |
| RI=529 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 3-MC5 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| nC6 | 0 | 1 | 1 | 0 | 1 | 1 | 0 | 10 | 10 |
| 3,3-DMC5 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| t-1,2-DMCyC5 + 3-EC5 + 2,2,4-TMC5 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| nC7 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| nC8 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| E-Benzene | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| m+p-Xylene | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| o-Xylene | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| nC9 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Sum FID | 554 | 81 | 636 | 1260 | 233 | 1493 | 1000 | 750 | 1750 |

| Sample-ID | H6177 | H6177 | H6177 | H6178 | H6178 | H6178 | H6180 | H6180 | H6180 |
|-----------------------------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| Gas fraction | H | O | H+O | H | O | H+O | H | O | H+O |
| External ID. | 1D | 1D | 1D | 1E | 1E | 1E | 1G | 1G | 1G |
| Compound | | | | | | | | | |
| C1 | 1372 | 559 | 1932 | 942 | 626 | 1568 | 182 | 1351 | 1533 |
| C2 | 8 | 36 | 44 | 4 | 38 | 42 | 2 | 67 | 70 |
| C3 | 2 | 13 | 15 | 5 | 13 | 19 | 2 | 24 | 27 |
| iC4 | 0 | 2 | 2 | 1 | 2 | 2 | 1 | 4 | 5 |
| C4ene | 1 | 4 | 4 | 2 | 4 | 6 | 1 | 4 | 5 |
| nC4 | 0 | 4 | 4 | 0 | 3 | 3 | 0 | 6 | 6 |
| 2,2-DMC3 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| RI=431 | 0 | 0 | 0 | 1 | 0 | 1 | 0 | 0 | 0 |
| iC5 | 0 | 1 | 1 | 1 | 1 | 2 | 0 | 3 | 3 |
| RI=485 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| RI=493 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| nC5 | 0 | 1 | 2 | 1 | 1 | 2 | 0 | 3 | 3 |
| RI=529 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 3-MC5 | 0 | 1 | 1 | 0 | 1 | 1 | 0 | 0 | 0 |
| nC6 | 0 | 1 | 1 | 1 | 1 | 2 | 0 | 2 | 2 |
| 3,3-DMC5 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| t-1,2-DMCyC5 + 3-EC5 + 2,2,4-TMC5 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| nC7 | 0 | 1 | 1 | 0 | 1 | 1 | 0 | 0 | 0 |
| nC8 | 0 | 1 | 1 | 0 | 1 | 1 | 0 | 0 | 0 |
| E-Benzene | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 |
| m+p-Xylene | 1 | 1 | 2 | 0 | 0 | 0 | 0 | 6 | 6 |
| o-Xylene | 0 | 1 | 1 | 0 | 0 | 0 | 0 | 3 | 3 |
| nC9 | 0 | 1 | 1 | 0 | 0 | 0 | 0 | 0 | 0 |
| Sum FID | 1384 | 626 | 2010 | 956 | 692 | 1649 | 189 | 1474 | 1663 |

Table 4.3 Continued

| Sample-ID | H6181 | H6181 | H6181 | H6182 | H6182 | H6182 | H6183 | H6183 | H6183 |
|-----------------------------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| Gas fraction | H | O | H+O | H | O | H+O | H | O | H+O |
| External ID. | 1H | 1H | 1H | 1I | 1I | 1I | 1J | 1J | 1J |
| Compound | | | | | | | | | |
| C1 | 556 | 520 | 1076 | 2338 | 463 | 2801 | 1634 | 262 | 1896 |
| C2 | 3 | 40 | 42 | 12 | 43 | 56 | 9 | 26 | 35 |
| C3 | 2 | 18 | 19 | 7 | 20 | 26 | 3 | 13 | 16 |
| iC4 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 |
| C4ene | 1 | 7 | 8 | 3 | 7 | 10 | 1 | 5 | 6 |
| nC4 | 0 | 3 | 3 | 0 | 2 | 2 | 0 | 2 | 2 |
| 2,2-DMC3 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 |
| RI=431 | 1 | 0 | 1 | 3 | 0 | 3 | 2 | 0 | 2 |
| iC5 | 0 | 1 | 1 | 0 | 1 | 1 | 0 | 1 | 1 |
| RI=485 | 0 | 1 | 1 | 0 | 1 | 1 | 0 | 1 | 1 |
| RI=493 | 0 | 1 | 1 | 0 | 1 | 1 | 0 | 1 | 1 |
| nC5 | 0 | 1 | 1 | 0 | 0 | 0 | 0 | 1 | 1 |
| RI=529 | 0 | 1 | 1 | 0 | 0 | 0 | 0 | 1 | 1 |
| 3-MC5 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| nC6 | 0 | 1 | 1 | 0 | 0 | 0 | 0 | 1 | 1 |
| 3,3-DMC5 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| t-1,2-DMCyC5 + 3-EC5 + 2,2,4-TMC5 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| nC7 | 0 | 1 | 1 | 0 | 0 | 0 | 0 | 0 | 0 |
| nC8 | 0 | 1 | 1 | 0 | 0 | 0 | 0 | 0 | 0 |
| E-Benzene | 0 | 1 | 1 | 0 | 0 | 0 | 0 | 0 | 0 |
| m+p-Xylene | 0 | 6 | 7 | 0 | 0 | 0 | 0 | 0 | 0 |
| o-Xylene | 0 | 4 | 4 | 0 | 0 | 0 | 0 | 0 | 0 |
| nC9 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Sum FID | 563 | 606 | 1169 | 2363 | 539 | 2902 | 1651 | 312 | 1963 |

Table 4.4
Composition of hydrocarbons in headspace and occluded gas
(volume %)

Table 4.4 Composition of hydrocarbons in headspace and occluded gas (volume %).

| Sample-ID | H6174 | H6174 | H6174 | H6175 | H6175 | H6175 | H6176 | H6176 | H6176 |
|-----------------------------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| Gas fraction | H | O | H+O | H | O | H+O | H | O | H+O |
| External-ID | 1A | 1A | 1A | 1B | 1B | 1B | 1C | 1C | 1C |
| Compound | | | | | | | | | |
| C1 | 99.0 | 88.6 | 97.7 | 99.5 | 92.6 | 98.4 | 99.2 | 80.8 | 91.3 |
| C2 | 0.5 | 5.3 | 1.1 | 0.5 | 4.5 | 1.1 | 0.4 | 10.5 | 4.7 |
| C3 | 0.3 | 2.3 | 0.6 | 0.0 | 1.4 | 0.3 | 0.2 | 4.6 | 2.1 |
| iC4 | 0.0 | 0.5 | 0.1 | 0.0 | 0.2 | 0.0 | 0.0 | 0.0 | 0.0 |
| C4ene | 0.0 | 0.7 | 0.1 | 0.0 | 0.4 | 0.1 | 0.1 | 1.2 | 0.6 |
| nC4 | 0.0 | 0.6 | 0.1 | 0.0 | 0.3 | 0.1 | 0.0 | 0.9 | 0.4 |
| 2,2-DMC3 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| RI=431 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| iC5 | 0.0 | 0.0 | 0.0 | 0.0 | 0.2 | 0.0 | 0.0 | 0.0 | 0.0 |
| RI=485 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| RI=493 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| nC5 | 0.0 | 0.7 | 0.1 | 0.0 | 0.2 | 0.0 | 0.0 | 0.7 | 0.3 |
| RI=529 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| 3-MC5 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| nC6 | 0.0 | 1.3 | 0.2 | 0.0 | 0.2 | 0.0 | 0.0 | 1.3 | 0.6 |
| 3,3-DMC5 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| t-1,2-DMCyC5 + 3-EC5 + 2,2,4-TMC5 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| nC7 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| nC8 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| E-Benzene | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| m+p-Xylene | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| o-Xylene | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| nC9 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| Sum FID | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 |

| Sample-ID | H6177 | H6177 | H6177 | H6178 | H6178 | H6178 | H6180 | H6180 | H6180 |
|-----------------------------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| Gas fraction | H | O | H+O | H | O | H+O | H | O | H+O |
| External-ID | 1D | 1D | 1D | 1E | 1E | 1E | 1G | 1G | 1G |
| Compound | | | | | | | | | |
| C1 | 99.1 | 89.4 | 96.1 | 98.5 | 90.5 | 95.1 | 96.4 | 91.6 | 92.2 |
| C2 | 0.6 | 5.8 | 2.2 | 0.4 | 5.5 | 2.6 | 1.2 | 4.6 | 4.2 |
| C3 | 0.1 | 2.1 | 0.7 | 0.6 | 1.9 | 1.1 | 1.2 | 1.7 | 1.6 |
| iC4 | 0.0 | 0.3 | 0.1 | 0.1 | 0.2 | 0.1 | 0.4 | 0.3 | 0.3 |
| C4ene | 0.0 | 0.6 | 0.2 | 0.2 | 0.6 | 0.4 | 0.5 | 0.3 | 0.3 |
| nC4 | 0.0 | 0.6 | 0.2 | 0.0 | 0.4 | 0.2 | 0.0 | 0.4 | 0.3 |
| 2,2-DMC3 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| RI=431 | 0.0 | 0.0 | 0.0 | 0.1 | 0.0 | 0.0 | 0.3 | 0.0 | 0.0 |
| iC5 | 0.0 | 0.2 | 0.1 | 0.1 | 0.2 | 0.1 | 0.0 | 0.2 | 0.2 |
| RI=485 | 0.0 | 0.0 | 0.0 | 0.0 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 |
| RI=493 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| nC5 | 0.0 | 0.2 | 0.1 | 0.1 | 0.2 | 0.1 | 0.0 | 0.2 | 0.2 |
| RI=529 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| 3-MC5 | 0.0 | 0.2 | 0.1 | 0.0 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 |
| nC6 | 0.0 | 0.2 | 0.1 | 0.1 | 0.2 | 0.1 | 0.0 | 0.1 | 0.1 |
| 3,3-DMC5 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| t-1,2-DMCyC5 + 3-EC5 + 2,2,4-TMC5 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| nC7 | 0.0 | 0.1 | 0.0 | 0.0 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 |
| nC8 | 0.0 | 0.1 | 0.0 | 0.0 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 |
| E-Benzene | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.1 | 0.1 |
| m+p-Xylene | 0.0 | 0.2 | 0.1 | 0.0 | 0.1 | 0.0 | 0.0 | 0.4 | 0.4 |
| o-Xylene | 0.0 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.2 | 0.2 |
| nC9 | 0.0 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| Sum FID | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 |

Table 4.4 Continued

| Sample-ID | H6181 | H6181 | H6181 | H6182 | H6182 | H6182 | H6183 | H6183 | H6183 |
|---------------------------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| Gas fraction | H | O | H+O | H | O | H+O | H | O | H+O |
| External-ID | 1H | 1H | 1H | 1I | 1I | 1I | 1J | 1J | 1J |
| Compound | | | | | | | | | |
| C1 | 98.8 | 85.7 | 92.0 | 98.9 | 86.0 | 96.5 | 99.0 | 83.9 | 96.6 |
| C2 | 0.4 | 6.6 | 3.6 | 0.5 | 8.0 | 1.9 | 0.5 | 8.3 | 1.8 |
| C3 | 0.3 | 2.9 | 1.6 | 0.3 | 3.7 | 0.9 | 0.2 | 4.2 | 0.8 |
| iC4 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.2 | 0.0 |
| C4ene | 0.2 | 1.1 | 0.7 | 0.1 | 1.3 | 0.3 | 0.1 | 1.4 | 0.3 |
| nC4 | 0.0 | 0.4 | 0.2 | 0.0 | 0.4 | 0.1 | 0.0 | 0.5 | 0.1 |
| 2,2-DMC3 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.2 | 0.0 |
| RI=431 | 0.3 | 0.0 | 0.1 | 0.1 | 0.0 | 0.1 | 0.1 | 0.0 | 0.1 |
| iC5 | 0.0 | 0.2 | 0.1 | 0.0 | 0.1 | 0.0 | 0.0 | 0.2 | 0.0 |
| RI=485 | 0.0 | 0.2 | 0.1 | 0.0 | 0.2 | 0.0 | 0.0 | 0.2 | 0.0 |
| RI=493 | 0.0 | 0.2 | 0.1 | 0.0 | 0.2 | 0.0 | 0.0 | 0.3 | 0.0 |
| nC5 | 0.0 | 0.2 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 | 0.2 | 0.0 |
| RI=529 | 0.0 | 0.1 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 | 0.2 | 0.0 |
| 3-MC5 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| nC6 | 0.0 | 0.2 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 | 0.3 | 0.0 |
| 3,3-DMC5 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| t-1,2-DMC5 + 3-EC5 + 2,2,4-TMC5 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| nC7 | 0.0 | 0.2 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| nC8 | 0.0 | 0.2 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| E-Benzene | 0.0 | 0.2 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| m+p-Xylene | 0.0 | 1.1 | 0.6 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| o-Xylene | 0.0 | 0.6 | 0.3 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| nC9 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| Sum FID | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 |

Table 4.5

Ratios and summary data

(See Table 2.4 for explanations and comments;
-1 = ratio cannot be calculated)

*Table 4.5 Ratios and summary data. (See Table 2.4 for explanation and comments;
-I = ratio cannot be calculated*

| Sample-ID | H6174 | H6174 | H6174 | H6175 | H6175 | H6175 | H6176 | H6176 | H6176 |
|-----------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| Gas fraction | H | O | H+O | H | O | H+O | H | O | H+O |
| External ID. | 1A | 1A | 1A | 1B | 1B | 1B | 1C | 1C | 1C |
| Compound | | | | | | | | | |
| CncC1 | 549 | 72 | 621 | 1253 | 216 | 1469 | 992 | 606 | 1598 |
| CncC2C4 | 5 | 8 | 13 | 7 | 16 | 23 | 7 | 129 | 136 |
| CncC5C9 | 0 | 2 | 2 | 0 | 1 | 1 | 1 | 15 | 16 |
| CncC1C9 | 554 | 81 | 636 | 1260 | 233 | 1493 | 1000 | 750 | 1750 |
| PctC1 | 99.0 | 88.6 | 97.7 | 99.5 | 92.6 | 98.4 | 99.2 | 80.8 | 91.3 |
| PctC2C4 | 1.0 | 9.4 | 2.0 | 0.5 | 6.8 | 1.5 | 0.7 | 17.2 | 7.8 |
| PctC5C9 | 0.1 | 2.0 | 0.3 | 0.0 | 0.6 | 0.1 | 0.1 | 2.0 | 0.9 |
| PctC1C9 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 |
| Wetness | 1.0 | 9.6 | 2.0 | 0.5 | 6.9 | 1.5 | 0.7 | 17.5 | 7.8 |

| Sample-ID | H6177 | H6177 | H6177 | H6178 | H6178 | H6178 | H6180 | H6180 | H6180 |
|-----------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| Gas fraction | H | O | H+O | H | O | H+O | H | O | H+O |
| External ID. | 1D | 1D | 1D | 1E | 1E | 1E | 1G | 1G | 1G |
| Compound | | | | | | | | | |
| CncC1 | 1372 | 559 | 1932 | 942 | 626 | 1568 | 182 | 1351 | 1533 |
| CncC2C4 | 11 | 58 | 69 | 12 | 60 | 72 | 6 | 106 | 112 |
| CncC5C9 | 1 | 8 | 10 | 2 | 6 | 9 | 0 | 18 | 18 |
| CncC1C9 | 1384 | 626 | 2010 | 956 | 692 | 1649 | 189 | 1474 | 1663 |
| PctC1 | 99.1 | 89.4 | 96.1 | 98.5 | 90.5 | 95.1 | 96.4 | 91.6 | 92.2 |
| PctC2C4 | 0.8 | 9.3 | 3.4 | 1.3 | 8.6 | 4.4 | 3.4 | 7.2 | 6.7 |
| PctC5C9 | 0.1 | 1.3 | 0.5 | 0.2 | 0.9 | 0.5 | 0.3 | 1.2 | 1.1 |
| PctC1C9 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 |
| Wetness | 0.8 | 9.5 | 3.5 | 1.3 | 8.7 | 4.4 | 3.4 | 7.3 | 6.8 |

| Sample-ID | H6181 | H6181 | H6181 | H6182 | H6182 | H6182 | H6183 | H6183 | H6183 |
|-----------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| Gas fraction | H | O | H+O | H | O | H+O | H | O | H+O |
| External ID. | 1H | 1H | 1H | 1I | 1I | 1I | 1J | 1J | 1J |
| Compound | | | | | | | | | |
| CncC1 | 556 | 520 | 1076 | 2338 | 463 | 2801 | 1634 | 262 | 1896 |
| CncC2C4 | 5 | 67 | 72 | 22 | 72 | 94 | 13 | 46 | 59 |
| CncC5C9 | 2 | 20 | 22 | 4 | 3 | 7 | 3 | 5 | 8 |
| CncC1C9 | 563 | 606 | 1169 | 2363 | 539 | 2902 | 1651 | 312 | 1963 |
| PctC1 | 98.8 | 85.7 | 92.0 | 98.9 | 86.0 | 96.5 | 99.0 | 83.9 | 96.6 |
| PctC2C4 | 0.9 | 11.0 | 6.1 | 0.9 | 13.4 | 3.2 | 0.8 | 14.6 | 3.0 |
| PctC5C9 | 0.4 | 3.2 | 1.8 | 0.2 | 0.6 | 0.2 | 0.2 | 1.5 | 0.4 |
| PctC1C9 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 |
| Wetness | 0.9 | 11.4 | 6.3 | 0.9 | 13.5 | 3.2 | 0.8 | 14.8 | 3.0 |