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Wax analysis of condensate

30/2-1 DST no. 2B

STATOIL EXPLORATION & PRODUCTION LABORATORY

by

Hans Petter Rønningsen

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#### 1. SUMMARY.

The wax content and the carbon number distribution of the wax was determined for one condensate sample. It contained 3.0% by weight of wax. The most abundant n-alkanes in the wax sample, as determined by gas chromatography, were  $n-C_{24} - n-C_{25}$ . The amounts of other n-alkanes decreased rapidly below and slowly above these carbon numbers. Density, kinematic viscosity, pour point and wax appearence point of the sample was also measured.

#### 2. SAMPLE.

The sample used for the analysis was:

Condensate 30/2-1 DST no. 2B (separator sample), sampled 250982.

The sample had been kept in a 20 l can from the date of sampling, and was kept at  $20^{\circ}$ C for about two weeks prior to the analysis. The can was shaken thoroughly before a small sample was taken out.

#### 3. EXPERIMENTAL.

#### 3.1 Wax precipitation.

A modification of UOP method 46-64(1), as described by Burger et. al. (2), the acetone precipitation technique, was used to precipitate the waxy material in the sample. The sample was dissolved in petroleum ether, acetone added, and the mixture kept at  $-25^{\circ}$ C for two hours. The sample-solvent mixture was then filtered using a Büchner-funnel with glass fiber filter. The filter was placed in a preweighed bottle, and wax crystals on the funnel was washed into the bottle with toluene. The toluene was evaporated to dryness and the bottle reweighed to determine the weigth of waxy material.

The precipitated material was slightly coloured, indicating minor contribution from heavy polar/aromatic material. It was not further purified prior to gas chromatographic analysis.

#### 3.2 Gas chromatographic analysis.

To determine the carbon number distribution of the waxy material, on-column injection with  $n-C_{16}$  as internal standard was used (about 10% internal standard).

Column : Chemically bonded SE 54, 0.3 mm i.d., 0.30 µm filmthickness.

Carrier gas : Helium.

Detector : Flame ionization, temp. 350<sup>o</sup>C, make-up gas N<sub>2</sub>.

Injection : 1.5 µl of a 1% toluene solution.

Temp. program:  $70^{\circ}$ C isothermal 1 min,  $10^{\circ}$ /min to 200  $^{\circ}$ C,  $6^{\circ}$ /min from 200  $^{\circ}$ C to 340  $^{\circ}$ C.

3.3 Measurement of kinematic viscosity.

Kinematic viscosity was measured at 37.8 $^{\circ}$ C using a KPG  $^{\textcircled{R}}$  reverse-flow viscometer.

3.4 Pour point measurement.

Pour point was measured according to ASTM method D-97(1980).

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### 3.5 Measurement of wax appearance point.

Wax appearance point was measured optically with polarized light using a LABORLUX 12 microscope with heattable for temperatureregulation. "As received" samples were first heated to  $50^{\circ}$ C on the heattable to remove all visible waxcrystals. Then the temperature was decreased slowly (about  $10-12^{\circ}$  per hour) until the first wax crystals appeared. The measurements seem to be repeatible within  $2^{\circ}$ , but the result may depend on the pretreatment of the sample.

## 4. RESULTS AND DISCUSSION.

The condensate was found to contain 3.0% by weight of waxy material. This represents the potentially depositable material. What actually can be expected to deposit during production/transportation will certainly be less than this, and depends on a lot of factors, as temperature, pressure, flowrate, solvents used etc.

The pour point is low  $(-18^{\circ}C)$ , while the wax appearance point is unexpectedly high  $(+16^{\circ}C)$  in comparison. But because the pour point is quite an unreliable value, being dependent on sample pretreatment, it may be that the measured value is unrealistic low.

In table 1 some physical data of the samples are given, as are the wax precipitation results. The wax is divided into two groups of compounds, n-alkanes and "other hydrocarbons", which may contain both iso-alkanes, naphtenes and aromatics. Above  $C_{40}$  it is not possible to differentiate between the two groups. The sum of hydrocarbons above  $C_{50}$  is determined as the difference between 100% and the recovery of sample, as determined by the internal standard. 20.9% of waxy material could not be identified by the gas chromatographic method.

In fig. 1 the carbon number distribution of the wax sample is given graphically.

Explanation of symbols:

- o gives % by weight of n-alkanes within one carbon number fraction.
- □ gives % by weight of hydrocarbons other than n-alkanes within one carbon number fraction.
- △ gives % by weight of total wax sample within one carbon number fraction.
- Table 1. Physical data and wax precipitation results of condensate 30/2-1 DST no. 2B.

Density (g cm-3,  $15^{\circ}$ C) 0.795 Kinematic viscosity  $(mm^2 s^{-1}, 37.8^{\circ}C)$ 2.0 Pour point (<sup>O</sup>C) -18 Wax appearance point(<sup>O</sup>C) +16Precipitated material (wt%) 3.0  $\Sigma$  n-alkanes  $C_{17}-C_{40}$  (wt%) 60.2  $\Sigma$  other HC C<sub>17</sub>-C<sub>40</sub> (wt%) 11.7  $\Sigma = HC C_{40} - C_{50}$  (wt%) 7.2 нс с<sub>50</sub>+ 20.9 Σ (wt%)

HC = hydrocarbons

Ι



Fraction	Melting	wt% of wax		wt% of fraction	
	point ( <sup>O</sup> C)	n-alkanes	other HC	n-alkanes	other HC
C17		0.1	0.0	100.0	0.0
C18	-	0.1	0.0	100.0	0.0
C19	-	0.1	0.0	100.0	0.0
C20	36.8	0.3	0.0	100.0	0.0
C21	40.2	1.6	0.0	100.0	0.0
C22	44.0	3.9	0.0	100.0	0.0
C23	47.5	6.5	0.0	100.0	0.0
C24	50.6	7.1	0.0	100.0	0.0
C25	53.5	7.0	0.0	100.0	0.0
C26	56.3	6.3	0.0	100.0	0.0
C27	58.8	5.4	0.3	99.6	0.4
C28	61.2	4.5	0.4	92.1	7.9
C29	63.4	3.9	0.6	86.5	13.5
C30	65.4	3.3	0.8	81.0	19.0
C31	67	2.8	0.9	75.2	24.8
C32	69	2.2	0.9	72.0	28.0
C33	71	1.6	0.9	64.5	35.5
C34	73	1.2	1.0	54.4	45.6
C35	74.6	0.8	1.0	44.9	55.1
C36	75.9	0.5	1.0	34.6	65.4
C37	77	0.4	1.0	26.4	73.6
C38	78.5	0.3	1.0	20.4	79.6
C39	80	0.2	1.0	16.0	84.0
C40	81	0.1	0.9	12.8	87.2
C41-C50	>81	7.2	2**	-	
>C50	-	20.9	)**	-	

Table 2. Carbon number distribution of wax material from condensate 30/2-1 DST no. 2B.

\* Melting point of n-alkanes

**\*\*** Sum of all hydrocarbons

## REFERANCES.

- "Laboratory Test Methods for Petroleum Oils", Universal Oil Products, Des Plaines, IL (1969).
- 2. Burger, E.D., Perkins, T.K., Striegler, J.H., Journal of Petroleum Technology, 1075-1086, june 1981.

Fig. 2. Gas chromatogram of condensate 30/2-1 DST no. 2B

-c<sub>10</sub> n C<sub>15</sub> n ł n-C<sub>20</sub>

Column : Chrompack Cp sil 5, 0.2mm i. d., filmthickness 0.14µm Carrier gas : Helium Detector : FID Temp. program : 10°C 4min., 4°/min to 300°C

F Fig. 3. Gas chromatogram of wax from condensate 30/2-1 DST no. 2B - C <sub>25</sub> n - c<sub>30</sub> n Manna

n-C<sub>16</sub>

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