

Evaluation of the Wax distillate from the Egyptian land – Balayiem Crude

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Summary

This paper deals with a deep evaluation of the constituents of the Wax distillate in the land Balayiem Crude. Combination of different methods of analysis was applied, (distillation, adsorption chromatography, urea addition, n. d. M.-method and elementary analysis). By dewaxing processes using ABT-mix., Paraffinic Wax was obtained. The chain length of the n-Paraffinic constituents of the Waxes was found.

Methods

1. To find the percentages of the different fractions in the Crude, 200 g of the crude was distilled in a distillation apparatus, which had a column of 50 cm height, 2.5 cm diameter, filled with saddle packings of 0,3 cm diameter. It was first atmospheric distilled up to a temperature of 231 °C. 5 cm³-fractions were taken. Before the vacuum distillation was carried out, the apparatus was cooled. The temperature rose again slowly by a pressure of 1 mm Hg. (Results in table 1).

2. The lubricating oil base stocks were obtained in the form of a wax distillate, which was delivered from Suez Comp. (U. A. R.). It was first in 9 fractions vacuum distilled at a vacuum of 3 mm Hg. The physical constants (solidification point at rotary thermometer¹), molecular weight²), n_D^{70} , d_{70} , kinematic Viscosity at 70 °C and the elemen-

Table 1
Percentage of the different products
in the land Balayiem crude

Product	Boiling range °C	Percent-weight
Benzine	60—180	8.25
Kerosine	180—240	6.00
Diesel oil	240—350	14.50
Spindle oil	350—420	9.25
Neutral oil I	420—480	9.00
Neutral oil II	480—520	7.30
Residue	over 520	42.0

¹) C. ZERBE, Mineralöle (1952), p. 383.

²) VAN NEZ - VAN WESTEN, Aspects of the Constitution of mineral oils, p. 461.

Table 2
Physical Properties and Elementary Analysis of the 9 Wax Distillate-Fractions

Fr. No.	Boiling range	% Weight	Solidification Point °C	M	n_D^{20}	d_4^{20}	V_k 70°C	C %	H %	S %
1	170—280	10.5	0.3	276	1.4648	0.8490	2.66	85.04	12.8	2.4
2	280—311	10.8	76.7	266	1.4753	0.8682	4.66	84.92	12.59	2.68
3	311—330	9.8	21.7	275	1.4792	0.8719	5.90	84.88	12.58	7.65
4	330—350	10.1	25.6	302	1.4819	0.8773	7.40	84.76	12.37	2.60
5	350—355	9.5	28.8	323	1.4838	0.8813	8.80	84.71	12.49	2.60
6	355—360	10.1	31.7	335	1.4859	0.8860	10.60	84.43	12.51	2.65
7	360—370	9.0	35.8	361	1.4881	0.8904	13.80	84.55	12.04	2.27
8	370—410	10.1	43.4	390	1.4899	0.8940	17.90	84.79	12.37	2.5
9	410—450	11.5	46.8	430	1.4922	0.8976	26.20	85.32	12.54	2.58
10	Residue	5.3						83.82	11.68	3.2

tary analysis C, H, S % were determined for the nine fractions and the results were given in Table 2.

3. Chemical Structure of the 9 distillate fractions

3.1. Structural Group Separation with Adsorption Chromatography³⁾

A column of 3 cm diameter and 100 cm filling height was used. It was filled by sedimentation in the column using Petroleum ether 30/50°C with activated silica gel of particle size 0.1—0.4 mm. One gram of the oil fraction was dissolved in few cm³ petroleum ether 30/50°C and given to the column. For elution of the saturated hydrocarbons, 1000 cm³ Petroleum ether were used, for aromatic hydrocarbons, 500 cm³ Benzene were used, and for resins 500 cm³ Methanol were used. By means of a siphon, 76 cm³ fractions were received. The Petroleum ether-eluate was colourless, only the last three fractions were weak yellow in colour. After the elution with Petroleum ether, the upper part of the silica gel in the column was dark brown in colour. By the elution with benzol, a brown ring of 5 cm height moved downwards in front of benzol. The benzol eluate had a strong blue green fluorescence. After the evaporation of benzol (Infra red lamp was used), the benzol eluate gave a dark brown, viscous syrupy mass.

After the elution with benzol, the upper part of the column remained dark brown in colour. This rest wandered down by elution with methyl alcohol, in the form of dark brown ring, which had no fluorescence. After the evaporation of methanol, it gave a half solid black brown residue. (For results see Table 3.)

³⁾ a) M. LEDERER, *Chromatography*, Elsevier Publ. Corp. 1955, p. 67. b) *Die chromatographischen Adsorptionsmethoden*. Springer-Verlag, Wien 1938, 2. Aufl.

Table 3
Adsorption Analysis of the 9 Fractions and n-Paraffin Content
(urea adduct)

Fr. No.	Saturate % Wt.	Aromatics % Wt.	n-Paraffin % Wt.	Naphthenes + Iso Paraffin % Wt.	Resins % Wt.
1	64.7	31.3	14.8	49.9	4.05
2	60.7	34.7	24.4	36.3	4.7
3	62.1	34.3	28.0	34.1	4.4
4	59.7	35.8	23.3	36.4	4.5
5	63.8	29.5	22.3	41.5	7.0
6	67.5	29.0	22.2	45.34	6.5
7	68.2	28.5	19.86	48.34	7.5
8	64.0	32.0	15.8	49.2	7.6
9	57.8	37.0	13.7	44.1	6.3

3.2. Normal-Paraffin-Separation by Urea addition⁴⁾

50 g of the oil fraction were dissolved in 100 cm³ benzol. The solution was warmed to 60 °C and slowly added at this temperature to 80 g of urea dissolved in 160 cm³ methanol. The two solutions were vigorously stirred and slowly cooled to 15 °C. After one hour the reaction was at the end and the urea adduct with the n-Paraffin was precipitated. The adduct was filtered, washed with 50 cm³ cold benzol and given to a one litre beaker, which contains 700 cm³ water. 2 drops of conc. HCl was added to water and the slurry heated to 90 °C, at which temperature the adduct was decomposed. After cooling the paraffin layer was removed and weighed. The filtrate was washed 3 times with water, dried, the benzol distilled and the rest (non adduct) was weighed (see Table 3).

3.3. Combination of the elementary analysis with the n-d-M-Method⁵⁾

The sum-formula of the average molecule in every fraction was calculated from

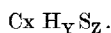
1. n-d-M-method-values.
2. molecular weight.
3. elementary analysis for C, H and S.

$$\text{The number of carbon atoms} = \frac{\% C \cdot M}{12 \cdot 100} = X$$

$$\text{The number of Hydrogen atoms} = \frac{\% H \cdot M}{1 \cdot 100} = Y$$

$$\text{The number of sulphur atoms} = \frac{\% S \cdot M}{38 \cdot 100} = Z$$

From the above 3 formulas, the sum-formula is,



⁴⁾ a) M. BENGEL, *Angew. Chem.* **63**, 207 (1951). b) W. SCHLENK, *Liebigs Ann. Chem.* **565**, 204 (1949). c) W. HESSLER u. U. G. MEINHARDT, *Fette und Seifen* **55**, 441, 786, 855 (1953). d) SCHLIEF, *Chem. Techn.* **6**, 456 (1954).

⁵⁾ S. GIPP, Dissertation Karl-Marx-Universität Leipzig 1961.

From the n-d-M-method values; the following were calculated:

$$\text{Carbon atoms in Aromatics} = \frac{X}{100} \cdot C_A = X_A$$

$$\text{Carbon atoms in Naphthens} = \frac{X}{100} \cdot C_N = X_N.$$

The amount of paraffinic carbon atoms was calculated from

$$X = X_A + X_N + X_P,$$

$$X_P = X - X_A - X_N.$$

This means that X carbon atoms of the average molecule includes X_A aromatic, X_N naphthenic, and X_P paraffinic carbon atoms.

When X_A has a value of 6 and R_A a value one, so the average molecule is a benzol ring; when $X_N = 10$ and $R_N = 2$ so in every molecule there is a decalin ring.

So the number of carbon atoms in every aromatic ring

$$= \frac{X_A}{R_A} = A$$

and the number of carbon atoms in every naphthenic ring

$$= \frac{X_N}{R_N} = N$$

$A = 6$ means Benzol $A = 5$ naphthalin,

$A = 4.67$ Anthracene,

$N = 6$ means cyclo hexans and $N = 5$ decalin.

(Results for the 9 fractions see Table 4).

4. Dewaxing

4.1. Distillation

59 kg of the wax distillate were first distilled in a pilot unit in three fractions: A diesel oil cut from initial boiling temperature up to 350 °C, a spindle oil cut 350/420 °C and a neutral oil I cut 420/480 °C.

The physical constants of the fractions were given in Table 5.

4.2. Solvent dewaxing⁶⁾

Dewaxing of the 3 fractions (from 4.1.) was carried out by the use of A B T (Aceton, Benzene, Toluene) mixture in the ration 3:3.5:3.5. The ratio of the oil to the solvent A B T was 1:3 and the dewaxing Temperature was -10 °C. The oil fraction was first dissolved in the mixture of solvents at a temperature of about 50 °C and then slowly cooled to normal temperature. The wax was then slowly in a refrigerator over night crystallised at the dewaxing temperature (see Table 6).

⁶⁾ S. GIPP, Dissertation Karl-Marx-Universität Leipzig 1961.

Table 4
Combination of the n.-d.-M.-Method Values with the Elementary Analysis Values

Fr. No.	N	X	Y	Z	Sum formula	X _A	X _N	X _P	R _A	R _N	A
1	5.7	16	28	0.17	C ₁₆ H ₃₀ S 0.17	2	8	6	0.23	1.4	6
2	6.0	19	34	0.23	C ₁₆ H ₃₄ S 0.23	2	9	8	0.35	1.6	5.7
3	6	20	34.6	0.22	C ₂₀ H _{31.6} S 0.22	3	9	8	0.46	1.42	6
4	6	21	37.4	0.24	C ₂₁ H _{37.4} S 0.24	3	9	9	0.48	1.52	6
5	5.8	23	40.4	0.26	C ₂₃ H _{40.4} S 0.26	3	10	10	0.49	1.7	6
6	5.5	24	42	0.27	C ₂₄ H ₄₂ S 0.27	3	10	11	0.5	1.8	6
7	5.2	25	43.8	0.29	C ₂₅ H _{43.8} S 0.29	3	11	11	0.4	1.9	6
8	5.7	27	46.4	0.3	C ₂₇ H _{46.4} S 0.3	3	11	13	0.54	2.13	5.5
9	4.5	30	53.7	0.34	C ₃₀ H _{53.7} S 0.34	3	12	15	0.59	2.8	5

Table 5
Physical Constants and Paraffins in the 3 Wax Distillate Fractions

Fraction	Yield % Wt.	d ₂₀ ^o	n _D ²⁰	Pour pt. C	VI	Flash point °C	Conrads C% Wt.	Acid No.	Iodine No.	n-Paraffin % Wt.	Solid. point of n-Paraffin	Paraffin content
Diesel oil	7.8	0.8466	1.4658	2—	—	147	0.06	0.2	30.7	17.2	24.2	8.8
Spindle oil	42.0	0.8724	1.4790	+22	73.5	196	0.1	0.16	30.0	22.7	40.2	16.6
Neutral oil	36.5	0.8966	1.868	+37	65.3	228.5	0.4	0.09	21.5	17.8	55.0	16.3
Residue	13.7	—	—	—	—	—	—	—	—	8.3	64.4	—

After Dewaxing the Spindle oil had a Pourpoint — 5°C and VI 56.2; the Neutral oil — 9.5°C, 50.0 Respectively.

Table 6
Dewaxing At -10°C (no deoiling)

Fraction	Slack Wax % Wt.	S_p $^{\circ}\text{C}$	Oil content	n_D^{70}
Diesel oil	2.4	41.3	—	1.4292
Spindle oil	14.6	45.5	10.2	1.4356
Neutral oil	17.2	55.8	12.3	1.4427

Table 7
Dewaxing at -10°C , $\pm 0^{\circ}\text{C}$ and $10 + ^{\circ}\text{C}$ of the 3 Wax Distillate Fractions

Fraction	Dewaxing Temp. $^{\circ}\text{C}$	% Wt.	S_p $^{\circ}\text{C}$	Oil content % Wt.	n_D^{70}
Diesel oil	-10	2.4	41.3	—	1.4292
	from -10 to -30	5.7	30.2		1.4261
Spindle oil	$+10$	3.73	51.7	9.9	1.4368
	± 0	10.0	48.4	8.3	1.4339
	-10	14.0	45.5	10.2	1.4356
Neutral oil I	$+10$	11.73	59.2	10.8	1.4418
	± 0	15.80	56.0	10.3	1.4403
	-10	17.20	55.3	12.3	1.4427

Table 8
Fractionating Dewaxing of neutral Oil I

Dewaxing Temp. $^{\circ}\text{C}$	Wax % Wt.	S_p $^{\circ}\text{C}$	Oil content	n_D^{70}
$+10$	10.3	60.2	10.8	1.4430
± 0	2.6	57.2	11.45	1.4395
-10	4.26	44.4	21.5	1.4497
-20	0.8	41.0	—	1.4422
Oil rest	80.0	—		

Table 9
Deoiling of Slack Wax

	Wax-Fraction	% Wt.	Oil content % Wt.	S_p C	n_D^{70}
Before deoiling	Spindle oil	12.3	0.5	47.6	1.4234
	Neutral oil	14.3	2.7	57.4	1.4344
After deoiling	Spindle oil	11.3	0.0	49.0	1.4280
	Neutral oil	13.2	0.0	58.8	1.4328

In other experiments the 3 fractions were dewaxed with A B T at temperatures $+10^{\circ}\text{C}$, $\pm 0^{\circ}\text{C}$ and -10°C , the Neutral oil fraction also at -20°C (see results given in Table 7 and 8). The deoiling took place as by the dewaxing process, but washings in both ases was with 100% A B T, cooled to the dewaxing or deoiling-temperature (see Table 9).

5. Chain length and contents of the n-paraffin⁷⁾ in the dioled pure white wax

5.1. The physical constants of the waxes obtained from the 3 fractions were determined (Table 10).

5.2. The n-Paraffins were isolated, using urea as in 3.2., but using 50 g wax in 500 cm³ benzene, 400 g urea in 800 cm³ Methanol.

With the help of the sum value of adducted and non-adducted material we could control the purity of the n-paraffins. The sum value (S_v) is evaluated by GROSS and GRODDE⁸⁾, given in the following formula:

$$\text{Sum value } S_v = 2 \cdot 10^3 \cdot (n_D^{60} - 1.400) - 0.84 \text{ S. P.},$$

$$S_v = 0 \text{ for n-paraffins,}$$

$$S_v < 0 \text{ for iso-paraffins,}$$

$$S_v \gg 0 \text{ for cyclic paraffins.}$$

Two or three additions of the material was performed to obtain an n-paraffin of high purity with a low sum-value (see Tables 11, 12, 13).

5.3 Distillation of the n-Paraffins obtained in 5.2

100 g of the n-Paraffin was distilled in 5 cm³ fractions. n_D^{70} and solidification point at rotary thermometer were determined. The molecular weight was calculated from the formula using the solidification point S_p , and from the molecular weight the chain length or the number of C-atoms of the n-Paraffinic fractions was found (see Table 14)

$$S_p = 415 \frac{M}{M + 95} = 273.$$

Discussion

The Balaiem Land crude is an intermediate base crude⁹⁾. By its distillation up to 520°C , it gave

⁷⁾ W. HAGER, Dissertation Karl-Marx-Universität Leipzig 1954.

⁸⁾ H. GROSS u. K. H. GRODDE, Öl und Kohle 38 419 (1942).

⁹⁾ Special Report, Balaiem Crud Oils from the Egyptian General Petroleum Corporation Cairo/V.A.R., Publ.No. I.

Table 10
Physical Constants of the Waxes Obtained from the 3 Fractions Dieseloil, Spindle Oil and Neutral Oil I

Wax fraction	d^{70}	S_p C	n_D^{70}	Iodine No.	Acid No.	Sapon No.	kin. Visc. KVcSt 70°C	Soft Paraffin Wt. %
Diesel oil	—	41.3	1.4292	4	0.4	0	—	—
Spindle oil	0.7671	49.2	1.4281	1.72	0.0	0.0	4.7	7.5
Neutral oil	0.7766	58.6	1.4329	1.1	0.0	0.0	6.77	0.0

Table 11
Urea Addition of Wax from Diesel Oil, Dewaxing Temp. -10°C
Wax before Addition Has: Sum Value Sv = 7.5, $n_{\text{D}}^{20} = 1.4211$

	1. Addition Wax Wt.	G-Added	% Wt.	n_{D}^{20}	$S_{\text{p}} \text{ C}$	S N
1.	26.0	23.01	92.0	1.4182	43.3	0
2.	21.27	19.50	97.6	1.4182	43.2	0
3.	20.86	19.14	92.0	1.4182	43.3	0
g-not Added						
1.		2.12	8.5	1.4662	- 3	730
2.		1.80	8.4	1.4660	- 3	730
3.		1.64	7.9	1.4660	- 3	730

This Means that this Wax Contained 93.8% n Paraffin and 8.2% Iso Paraffin.

Table 12
Wax urea Addition of Wax from Spindle Oil (Dewaxing Temp. -10°C)
 $n_{\text{D}}^{20} = 1.4273$, $S_{\text{p}} = 49.0^{\circ}\text{C}$, Sv = 1.9 (see Table 9)

	Wax Wt.	1-Addition g-Added	% Wt.	n_{D}^{20}	$S_{\text{p}} \text{ C}$	Sv
1.	51.02	49.14	96.3	1.4205	48.6	0.18
2.	51.69	50.12	96.9	1.4205	48.6	0.18
3.	53.07	51.50	97.1	1.4206	48.9	0.12
not Added						
1.		1.04	2.0	1.4556	22.5	92.3
2.		0.88	1.7	1.4557	22.3	92.7
3.		7.06	2.0	1.4554	22.4	92.0

This Means that this Wax Constained 96.8% Wt. n-Paraffin and 1.9% Wt. Iso Paraffin.

a high percentage of asphaltic residue, which was solid at room temperature (see table 1) (42.0% wt). The aim of this work was the study of the chemical structure of the wax distillate obtained from Balayiem Land crude, by a vacuum distillation unit in the Suez-Refinery in V. A. R. The following methods of separation were good applied to give a picture on the structure of the lubricating oil base stocks, obtained from the wax distillate.

1. Distillation of the wax distillate in nine fractions by which we can separate the fraction-constituents according to the molecular weight which rise in the fractions from 276 to 430 (table 2). Distillation was also applied to separate the n-Paraffinic constituents in different chain lengthes (table 14).

2. Column Chromatographic adsorption analysis gave a good separation of the structural groups, which depend in this separation, not only on the

Table 13
 Urea Addition of Wax from Neutral Oil I (Dewaxing Temp. -10°C)
 (see Table 9) $n_D^{20} = 1.4265$, $S_p = 58.8^{\circ}\text{C}$, $S_v = 4.3$

	1. Addition		% Wt.	n_D^{20}	S_p C	S_v
	Wax Wt.	g-Added				
1.	50.0	47.40	94.8	1.4250	58.0	1.3
2.	51.03	48.45	95.0	1.4250	58.2	1.2
3.	52.67	50.22	95.2	1.4249	58.3	1.0
not Added						
1.		2.36	4.6	1.4531	32.4	79.0
2.		2.54	4.9	1.4491	34.7	69.1
3.		2.42	4.6	1.4486	35.3	69.6

The first added n-Paraffins were again readded, as the S_v was somewhat high.

2. Addition

1.	44.2	43.3	98.0	1.4242	58.3	0
2.	44.2	43.1	97.5	1.4243	58.2	0
3.	44.2	43.2	97.7	1.4242	58.2	0
not Added						
1.		0.84	1.9	1.4369	40.5	39.8
2.		0.80	1.8	1.4370	40.2	40.3
3.		0.86	1.9	1.4369	40.4	39.9

3. Addition Gave 0.3 Wt. % Iso Paraffin. This Means that this Wax Constained 92.8% Wt. n-Paraffin a. 6.9% Wt. isop.

Table 14
 Chain Length of the Different n-Paraffin Constituents

Chain length	% weight in Diesel Oil	% weight in Spindle Oil	% weight in Neutral Oil
$C_{17}-C_{18}$	4.1	—	—
$C_{18}-C_{19}$	4.2	—	—
$C_{19}-C_{20}$	9.0	—	—
$C_{20}-C_{21}$	20.7	3.7	—
$C_{21}-C_{22}$	17.2	17.7	—
$C_{22}-C_{23}$	17.6	20.0	—
$C_{23}-C_{24}$	7.8	19.7	5.6
$C_{24}-C_{25}$	—	13.3	6.4
$C_{25}-C_{26}$	—	13.4	19.4
$C_{26}-C_{27}$	—	6.4	16.1
$C_{27}-C_{28}$	—	—	23.0
$C_{28}-C_{29}$	—	—	20.5
$C_{29}-C_{30}$	—	—	2.0
Residue	6.5	5.3	5.0
Losses	12.9	0.5	2.0

molecular weight, but also separates the constituents according to their structure.

3. Urea addition of the nine wax distillate fractions and also of the waxes obtained by dewaxing. It is an excellent method for the separation of n-Paraffins from the other constituents.

4. Dewaxing with acetone-benzene-toluene mixture, by which it was possible to separate the higher-melting constituents of the wax distillate from the other ones.

By combination of these different separation methods and other methods of analysis, as n-d-M-method, elementary analysis and other physical constants, we could obtain a picture on the structure of the wax distillate. It was shown (table 3), that the naphthenic hydrocarbons gave the highest percentage in the wax distillate, with a mean value in the fractions of 43.6% wt. the aromatic hydrocarbons giving a mean value of 32.4% wt. the n-Paraffins 18% wt. and resins 5.8% wt. This means that the naphthenic and aromatic hydrocarbons constituted the main bulk of the wax distillate. With the help of the elementary analysis the sum formulas of the 9-wax distillate fractions were obtained (table 2 and 4). The decrease in H-content in these fractions, with increase in the molecular weight, was due to the increase in the cyclic structure and condensation of the rings, as was clear from the increase of the value of the number of carbon atoms in aromatic and naphthenic structure (R_A and R_N in table 4). It was also clear that the number of carbon atoms increase from C_{16} to C_{30} .

The wax distillate was distilled in 3 vacuum fractions in order to obtain the lubricating oil base stocks. From the physical constants given in table 5, it was clear, that the luboils which could be obtained, from these base stocks were of high pour points and low viscosity index (VI).

The pour point for spindle oil was $+22^\circ\text{C}$ and its viscosity index was 73.5. The pour point of Neutral oil I was $+37^\circ\text{C}$ and its viscosity index 65.3. After dewaxing of these oils the pour point and viscosity index fell to -5°C and 56.2 respectively for spindle oil and to -9.5 and 50.0 for Neutral oil I. The decrease in VI was due to the removal of some of the n-Paraffinic Hydrocarbons, that have high viscosity index.

The dewaxing of the wax distillate was good carried out with an ABT mixture in a ratio to the oil fraction of 3:1, the dewaxing temperature being at -10°C (table 6). The oil content of the wax fractions before deoiling was low (from 0.5 to 2.7% wt.) (table 9). In other experiments, where no washings of the slack wax took place (table 7, the oil content of these waxes reached 12.3%. After deoiling the pure waxes were white odourless, tasteless and oil free products, that needed no further refining with sulphuric acid or bleaching clays. It was clear (table 7 and 9) that we can obtain three grades of

paraffinic waxes of solidification points at rotary thermometer $Sp = 41.3^{\circ}\text{C}$, 49°C and 58.8°C . These waxes were of good quality (table 10). They have low iodine, acid and saponification numbers. The middle grade product contained some percent of soft paraffin and all 3 grades have n-Paraffin content higher than 90% wt.

The chain length distribution of the n-paraffin-constituents of these waxes was found by their isolations (table 11, 12, 13) with Urea-addition, following by their distillation in different fractions, which differentiated in melting points and molecular weight. The 3 grade waxes contained n-paraffinic constituents of chain lengths varying from C_{16} to C_{30} (table 14).

Conclusion

The wax distillate contained lubeoil base stocks of low grade, as shown from its low viscosity index and high pour points. It was rich in naphthenic and aromatic structures.

The waxes obtained from it have good quality and the chain length range of its high n-Paraffinic constituents offers a good substrate for the production of fatty acids, especially from the diesel oil- and spindle oil paraffins.

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