ENCLOSURE (B) 35

STUDIES ON POUR POINT DEPRESSANTS FOR LUBRICATING OILS

by

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LIST OF TABLES AND ILLUSTRATIONS

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SUMMARY

Turbine oil, obtained from Oha crude oil, contains about 5% wax and has pour points ranging from (+)5 to 15°C.

To depress pour points below 0°C without dewaxing the use of "Paraflow," the best known commercial pour point depressant, was tried. When added to the oil in small quantities such as 0.1 to 0.5%, these compounds effectively depressed the pour point without unfavorable effect on other properties of oil. The synthesis of "Paraflow" was studied and a most effective product was obtained by condensing naphthalene and chlorinated wax at 50-100°C in the presence of 5% of anhydrous aluminum chloride for 3 hours. It was found necessary to control the reaction by the addition of small quantities of water. The need for water was accidentally discovered, and was not mentioned in the U.S. Patent on "Paraflow."

I. INTRODUCTION

A. History of Project

Some of the turbine oils used by the Japanese Navy in 1941 were prepared from Oha crude oil. Oha crude oil had been thought to be a naphthenic base crude, but it sometimes contained a small amount of wax, and the turbine oils prepared from it had high pour points. To conform to the specifications for turbine oil, the pour point had to be below 0°C. The capacities of the dewaxing plants in Japanese refineries were too small to dewax turbine oil, since dewaxing plants were used principally for the preparation of aero engine oil.

Suida, H. and Pöll, H. (U.S.A. Text book) pointed out that the use of "Paraflow" in transformer or turbine oils had been found undesirable, due to its unfavorable effect on oxidation stability. The authors tested "Paraflow" imported from America and found it very effective as a pour point depressant and not deleterious in regard to oxidation stability.

Studies were carried out as to the optimum conditions for preparing the same compound as "Paraflow" from chlorinated wax and naphthalene. These studies began in June, 1943, and satisfactory results were obtained in March, 1944.

B. Key Personnel Working on Project

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II. DETAILED DESCRIPTION

A. An outline showing the steps in the preparation of turbine oil from Oha topped crude oil is shown in Figure 1(B)35. The properties of the turbine oil are given below. The properties of its fractions are given in Table I(B)35.

	Turbine Oil
Density (d_4^{15})	0.9306
Flash Point (°C)	195
Viscosity (R.1. sec.) 30°C	631
50°C	185
80°C	63
(S.U.S.) 100°C	371.2
210°C	49.8
V.I.	26.9
Pour Point (°C)	10
Conradson's Carbon Residue (%)	0.11
Stability Test	Good
Demulsibility	54
Remain Water in Oil (%)	1.82
Acid Value	0.06
Volatility (%)	0.58
Aniline Point	74
Corrosion	Good

This oil was dewaxed by diluting with five volumes of dichlorethane at (-)15°C, and a yield of 95% of dewaxed oil was obtained. Its pour point was (-)18°C, and the yield of wax having a melting point of 39°C was 3%, as shown in Table II(B)35.

B. Properties of "Paraflow"

The properties of the imported "Paraflow," used in these experiments, are tabulated below.

Specific Gravity (d_4^{25})	0.8900
Index of Refraction (n_D^{25})	1.4985
Specific Ref. (r_D^{25})	0.3296
Aniline Pt. (°C)	112
Molecular Weight	962
Elementary C (%)	85.77
Analysis H (%)	12.42
Molecular Formula	$C_{69}H_{119}O$
Acid Value	0.05
Saponification Value	0.26
Iodine Value	14.0
Viscosity (S.U.S.) 100°F	2245
210°F	171.4
Viscosity Index	112.9
Conradson's Carbon Residue (%)	1.20
Pour Point (°C)	-11
Flash Point (°C)	248
Volatility (135°C-6 hrs) (%)	0.047
Stability (175°C-15 hrs)	Good
Oxidation Test... Viscosity Ratio	1.88
Conradson's Carbon (%)	3.41
Color	Greenish Red

~~The results of vacuum distillation are shown in Table IV(B)35.~~ "Paraflow" was dewaxed with five volumes of dichlorethane at (-)20°C, and it was found that only the waxy compound was effective in depressing the pour point of turbine oil, as shown in Table III(B)35.

C. Tests of "Paraflow" in Turbine Oils

"Paraflow" was tested in the laboratory at the First Naval Fuel Depot and also in several oil refineries; the results are given in Table V(B)35 and Figure 2(B)35.

A cold storage test was performed as shown on Table IV(B)35.

A turbine oil (Pour point, 8°C) and the same oil blended with 0.1% "Paraflow" (Pour point (-)10°C) were stored and thermostatically kept at 0°C and changes in their properties were determined. The results are tabulated in Table VI(B)35 and show that the pour point of the turbine oil blended with "Paraflow" was not changed while in storage at low temperatures.

D. Synthesis of "Paraflow"

Referring to U.S. Patent 1,815,022, it was attempted to synthesize a pour point depressant similar to "Paraflow." The properties of sweated wax are shown below.

Color		White
Pour Point (°C)		56
Conradson Carbon (%)		0.09
Viscosity (S.U.S.)	210°F	38.4
Elementary Analysis	C(%)	84.88
	H(%)	14.76
Mean Molecular Weight		461
Molecular Formula		$C_{32}H_{66}$
Distillation Test (at 5mm)	IBP	205
	10%	226
	20%	230
	30%	236
	40%	239
	50%	242
	60%	246
	70%	249
	80%	255
	90%	259
	98%	294

This wax was chlorinated to dichlor paraffin by chlorine gas at 80°C. The results are given in Figure 3(B)35.

A pour point depressant was prepared from this dichlor paraffin and naphthalene by the following procedure: (see Figure 4(B)35). Dichlor paraffin (300 grams) and naphthalene (150 grams) were placed in a three-necked glass flask and heated to 50°C.

After both were dissolved completely, five grams of aluminum chloride were added slowly. After 5-20 minutes a vigorous reaction took place, the reaction gas foaming up to the necks. Then three grams of water were added to control the reaction. Over a period of about 60 minutes, 16 grams of aluminum chloride were added. After maintaining a temperature of 50°C for 90 minutes, the reaction temperature was raised to 100°C and maintained for 120 minutes.

After settling, the sludge was separated and 304 grams of the condensation product were obtained. For dechlorination, nine grams of active clay were added to the condensation product which was then covered with carbon dioxide gas, and heated to 250°C for two hours. In this process 21 grams of unreacted naphthalene were distilled off. The product was then cooled to 150°C. A gas oil (1000 grams) were added for the extraction of the product, and the clay was filtered off.

After recovering the gas oil which had been added, the lighter fraction boiling up to 240°C was distilled off in a vacuum of 2mm Hg.

By adding 0.5% of this product to the turbine oil having a (+)10°C pour point, the pour point was lowered to (-)18°C. The properties of the product are shown in Table VII(B)35. The synthetic product was dewaxed and only the waxy substance was found effective.

E. Addition of Water in the Synthesis of the Pour Point Depressant

The procedure was repeated according to the patent description, but successful results were not obtained. To determine the extent of reaction, the quantity of HCl gas evolved in the condensation of dichlor paraffin with naphthalene was measured by absorption in water.

In these studies, accidentally some water flowed back, and a small quantity of water was introduced into the reaction flask.

A desirable product was thereby obtained. Then the rôle of water in the condensation reaction was studied, and the following results were obtained.

When 300 grams of dichlor paraffin, 150 grams of naphthalene, and 21 grams of aluminum chloride were used in the condensation reaction, the quantity of water was varied from one gram to six grams. The condensation product was tested by adding 0.5% of it to a turbine oil having a pour point of (+)10°C. The results are shown below.

Quantity of Water (grams)	Pour Point (°C)
1	11
2	-6
3	-20
4	-18
5	
6	Filtration was impossible.

Thus, the presence of water was necessary for the synthesis of the pour point depressant, and the addition of water affects the concentration of hydrogen chloride present.

F. The Effect of Pour Point Depressant on High Viscosity Lubricating Oils

The effect of this pour point depressant on high viscosity lubricating oils was tested.

As shown in Table VII(B)35, it was also effective in these oils.

G. Determination of Allowable Range of Wax Content for the Use of the Pour Point Depressant

A 54°C melting point wax was mixed in various proportions to a turbine oil having a pour point of (-)18°C, and the pour points of the oil were determined.

The results are given in Figure 5(B)35 and show that a small amount of wax markedly raised the pour point. The effect of the pour point depressant for this oil was tested and the results are shown in Figure 6(B)35.

Spindle oil was also tested and the results are given in Figure 8(B)35. The 56°C and 46°C melting point waxes were added to turbine oil, fuel oil, or normal heptane, respectively, and their pour points are shown in Figure 8(B)35. From these results it was observed that the lower the viscosity of the oil or the lower the melting point of wax contained, the greater the allowable percentage of wax for a given pour point.

H. Test Procedures Used

1. Stability. When no black sludge is formed after heating 20 grams of a sample of oil at 170°C for 15 hours.

2. Demulsibility. A sample of oil (20cc) is emulsified by means of inducing water vapor until the total volume reaches 40cc, and then settled at 95°C. Demulsibility is calculated from the rate of separation of oil using the following equation.

$$\text{Demulsibility} = \frac{\text{cc of oil separated} \times 5}{\text{time (minutes)}}$$

3. Volatility. Percentage of loss in weight of 65cc of oil after heating at 135°C for six hours.

4. Corrosion. A.S.T.M. copper strip corrosion test for three hours.

III. CONCLUSIONS

It was found that pour point depressant could be prepared which differed in physical properties from the imported "Paraflow" but was effective in depressing the pour points of oils.

It was prepared on an industrial scale and actually used.

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Table I(B)35
PROPERTIES OF EACH FRACTION OBTAINED FROM TOPPED CRUDE OIL

Volume (%)	Distillation (°C/4mm)	Temperature Converted to (°C/760mm)	Density (d_4^{22})	Viscosity 100°F	(S.U.S.) 210°F	Viscosity Index	Pour Point (°C)
0~10	142~190	302~366	0.9091	71.1	36.1		-13
10~20	~198	~378	0.9172	109.8	38.5	-	-9
20~30	~210	~392	0.9205	156.1	41.2	16.0	-2
30~40	~221	~405	0.9227	239.3	44.6	17.5	3
40~50	~231	~416	0.9230	363.1	48.9	13.5	7
50~60	~241	~429	0.9277	560.8	54.5	7.0	10
60~70	~250	~438	0.9350	906.7	63.3	0.3	14
70~80	~261	~455	0.9331	1222.9	72.3	9.1	14
80~90	~278	~478	0.9273	1610.3	82.9	16.7	19
90~100	~302	~508	0.9308	2355.7	102.0	26.3	20

Table II(B)35
THE DEWAXING OF THE TURBINE OIL

Name	Yield (%)	Density (d_4^{15})	Pour Point (°C)	Viscosity 100°F	(S.U.S.) 210°F	Viscosity Index	Aniline Point (°C)
Dewaxed Turbine Oil	95	0.9325	-18	467.2	50.8	-9.9	72
Wax	3	-	+39	-	-	-	-
Loss	2	-	-	-	-	-	-

Table III(B)35
DEWAXING OF IMPORTED "PARAFLOW"

	Yield (%)	Pour Point (°C)	Pour Point Depressing Test For Turbine Oil			
			0	0.1%	0.3%	0.5 %
Dewaxed Oil	31	-6	10	10	8	7
Waxy Compound	62.5	2	10	-5	-12	-18
Loss	6.5	-	-	-	-	-

Table V(B)35

ERIMENTAL RESULTS OF ADDITION OF "PARAFLOW" TO TURBINE OIL

…sity (…wood)		Pour Point (°C)	Corrosion	Volatile Material (%)	Conradson's Carbon (%)	Animal and Vegetable Oil	Acid Value	Demulsibility	Residual Water in Oil	Stability	Amount of "Paraflow"	P.P.
50°C	80°C											
189	63	+2	Good	0.367	0.08		0.06	73	1.062	Good		
187	63	+2	Good	0.362	0.082		0.06	66	1.233	Good	0.02	+2
185	63	-1	Good	0.360	0.088		0.06	60	1.252	Good	0.2	-5
183	63	-7	Good	0.360	0,093		0.06	60	1.246	Good		
183	62.5	+9	Good	0.375	0.055		0.06	73	1.033	Good		
183	62.5	+9	Good	0.368	0.058		0.06	73	1.052	Good	0.02	+8
183	62.5	+5	Good	0.360	0.061		0.06	65	1.326	Good	0.2	+2
183	63	-6	Good	0.358	0.063		0.09	70	1.082	Good		
188	63	+15	Good	0.365	0.032		0.06	60	1.422	Good		
187	63	+10	Good	0.358	0.043		0.06	60	1.365	Good	0.02	+8
187	63	+5	Good	0.363	0.041		0.06	66	1.214	Good	0.2	+2
185	63	-6	Good	0.362	0.063		0.09	65	1.065	Good		
109	50	+3	Good	0.488	0.135	No	0.030	80	0.448	Good		
109	50	-1	Good	0.462	0.135	No	0.030	80	0.448	Good		
		-15	Good									
109	50	-19	Good	0.445	0.140	No	0.03	80	0.500	Good		
		-27	Good									
109	50	-31	Good	0.339	0.145	No	0.035	80	0.645	Good		
180	62.5	+5	Good	0.252	0.159	No	0.035	57	0.898	Good		
180	62.5	+13	Good	0.256	0.159	No	0.035	50	0.898	Good		
		-13										
180	62.5	-17	Good	0.258	0.160	No	0.042	40	1.074	Good		
		-24										
180	62.5	-25	Good	0.260	0.162	No	0.045	33.3	1.193	Good		
184	62.5	+13	Good	0.258	0.160	No	0.028	57	0.988	Good		
184	62.5	+10	Good	0.262	0.160	No	0.028	43	1.151	Good		
		-5										
184	62.5	-12	Good	0.235	0.163	No	0.035	43	1.268	Good		
		-20										
184	63.0	-24	Good	0.277	0.174	No	0.040	40	1.285	Good		

Table V(B)35

EXPERIMENTAL RESULTS OF ADDITION OF "P

	Base Lub.	Amount of "Paraflow" (%)	Reaction	Specific Gravity	Flash Point (°C)	Viscosity (Redwood) 30°C	50°C	80°C	Pour Point (°C)	Corrosion	Volatile Material (%)
Nihon Oil Company Ltd. Yokohama Refinery	No. 1 Turbine Oil	0		0.9277	190	573	189	63	+2	Good	0.367
		0.01		0.9274	190	582	187	63	+2	Good	0.362
		0.1		0.9274	190	590	185	63	-1	Good	0.360
		0.5		0.9274	190	588	183	63	-7	Good	0.360
	No. 2 Turbine Oil	0		0.9306	190	586	183	62.5	+9	Good	0.375
		0.01		0.9297	190	599	183	62.5	+9	Good	0.368
		0.1		0.9294	190	595	183	62.5	+5	Good	0.360
		0.5		0.9294	190	602	183	63	-6	Good	0.358
	No. 3 Turbine Oil	0		0.9297	190	593	188	63	+15	Good	0.365
		0.01		0.9301	190	593	187	63	+10	Good	0.358
		0.1		0.9398	190	598	187	63	+5	Good	0.363
		0.5		0.9298	190	602	185	63	-6	Good	0.362
Nihon Oil Company Ltd. Niigata Refinery	Light Turbine Oil	0	Neutral	0.929	181	313	109	50	+3	Good	0.488
		0.01	Neutral	0.929	181	313	109	50	-1	Good	0.462
		0.05	Neutral						-15	Good	
		0.1	Neutral	0.930	181	313	109	50	-19	Good	0.445
		0.2	Neutral						-27	Good	
		0.5	Neutral	0.930	181	314	109	50	-31	Good	0.339
	No. 1 Turbine Oil	0	Neutral	0.932	189	589	180	62.5	+5	Good	0.252
		0.01	Neutral	0.930	190	589	180	62.5	+13	Good	0.256
		0.05	Neutral						-13		
		0.1	Neutral	0.934	190	589	180	62.5	-17	Good	0.258
		0.2	Neutral						-24		
		0.5	Neutral	0.938	190	592	180	62.5	-25	Good	0.260
	No. 2 Turbine Oil	0	Neutral	0.932	189	610	184	62.5	+13	Good	0.258
		0.01	Neutral	0.933	190	610	184	62.5	+10	Good	0.262
		0.05	Neutral						-5		
		0.1	Neutral	0.934	190	610	184	62.5	-12	Good	0.235
		0.2	Neutral						-20		
		0.5	Neutral	0.935	190	614	184	63.0	-24	Good	0.277

Table V(B)35 Continued

…ULTS OF ADDITION OF "PARAFLOW" TO TURBINE OIL

Pour Point (°C)	Corrosion	Volatile Material (%)	Conradson's Carbon (%)	Animal and Vegetable Oil	Acid Value	Demulsi-bility	Residual Water in Oil	Stability	Amount of "Paraflow"	P.P.
+6			0.073					Good		
+45								Good		
+2								Good		
-55								Good		
-135								Good		
-17			0.073					Good		
+11	Good		0.21		0.029	109		Good		
+9	Good		0.21		0.29	100		Good		
-55	Good		0.20		0.014	92		Good		
-17	Good		0.20			80		Good		
-16	Good	0.12	0.05	No	0.006	66.6	0.72	Good		
-16										
-17										
-17										
-18										
-20	Good	0.12	0.05	No	0.018	66.6	0.65	Good		
+10	Good			No	0.028	66.6	1.30	Good		
+10										
+8										
+6										
0										
-12	Good			No	0.037	66.6	1.30	Good		
+5	Good	0.150	0.05	No		77	0.886	Good		
+2.5	Good	0.152	0.05	No		79.3	0.907	Good		
+1										
-2	Good	0.155	0.05	No		81.0	0.911	Good		
-6										
-24	Good	0.160	0.05	No		83.2	0.913	Good		
+10										
+95										
+9										
+6										
-4										
-22										
-15										
+14										
+12.5										
+12										
+2									0.3	-7
-16									0.4	-13

Table V(B)35 Continued

EXPERIMENTAL RESULTS OF ADDITION OF "PARAFLO

	Base Lub.	Amount of "Paraflow" (%)	Reaction	Specific Gravity	Flash Point (°C)	Viscosity (Redwood) 30°C	50°C	80°C	Pour Point (°C)	Corrosion	Volatile Material (%)
Showa Oil Company Ltd. Niigata Refinery	Turbine Oil	0			180		164		+6		
		0.05							+45		
		0.07							+2		
		0.1							-55		
		0.15							-135		
		0.2			181		165		-17		
Showa Oil Company Ltd. Kawasaki Refinery	Turbine Oil	0	Neutral		195	454	150	58	+11	Good	
		0.01	Neutral		195	460	151	58	+9	Good	
		0.1	Neutral		195	479	154	59	-55	Good	
		0.5	Neutral		193	485	155	59	-17	Good	
Mitsubishi Oil Company Ltd. Kawasaki Refinery	Mitsubishi Turbine Oil No. 4	0	Neutral	0.932	196	605	174	61	-16	Good	0.12
		0.01							-16		
		0.05							-17		
		0.10							-17		
		0.20							-18		
		0.50	Neutral	0.933	196	612	176	61	-20	Good	0.12
	Turbine Oil	0	Neutral	0.928	182	650	190	69	+10	Good	
		0.01							+10		
		0.05							+8		
		0.10							+6		
		0.20							0		
		0.50	Neutral	0.928	184	654	190	68	-12	Good	
Maruzen Oil Company Ltd. Shimazu Refinery	No. 1 Turbine Oil	0	Neutral	0.928	194	604	188	63.5	+5	Good	0.150
		0.01	Neutral	0.928					+2.5	Good	0.152
		0.05							+1		
		0.10	Neutral	0.928					-2	Good	0.155
		0.20							-6		
		0.50	Neutral	0.928					-24	Good	0.160
	No. 2 Turbine Oil	0							+10		
		0.01							+95		
		0.05							+9		
		0.10							+6		
		0.20							-4		
		0.50							-22		
	No. 3 Turbine Oil	0							-15		
		0.01							+14		
		0.05							+12.5		
		0.1							+12		
		0.2							+2		
		0.5							-16		

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Table IV(B)35
VACUUM DISTILLATION OF IMPORTED "PARAFLOW"

	Distilled Temp. (°C/1mm)	Converted to 760mm	Yield (wt. %)	Appearance
1	175-200	382-416	1.2	Pale yellow, liquid
2	-250	-483	11.7	Pale yellow, liquid mixed with crystal
3	-274	-515	16.6	Orangeyellow, liquid
4	-290	-533	17.2	Orangeyellow, liquid
5	-300	-550	4.8	Orangeyellow, liquid
6	300-	550-	48.2	Dense green, liquid

(See pages 386 and 387 for Table V(B)35.)

Table VI(B)35
STORAGE TEST OF TURBINE OILS

Time Elapsed (hours)	Turbine Oil Without Addition of "Paraflow"			Turbine Oil With Addition of 0.1% "Paraflow"		
	Viscosity (Redwood No.2) (0°C)	Pour Point (°C)	Pour Point Preheated to 50°C	Viscosity (Redwood No.2) (0°C)	Pour Point (°C)	Pour Point Preheated to 50°C
45	1357.8	+ 8	+12	1005	-20	-7
110	1590.9	+ 4	+12	995.8	-19	-10
158	1536.5	-	-	985.0	-	-
397	1569.3	-	-	998.2	-	-

Table VII(B)35
PROPERTIES OF SYNTHETIC PRODUCTS

	Specific Gravity (d_4^{15})	Ind. of Refra. (n_D^{20})	Viscosity (S.U.S.) 100°F	Viscosity (S.U.S.) 210°F	Pour Point (°C)	Conradson's Carbon	Acid Value	Saponification Value	Iodine Value	Molecular Weight	Ash	Lowering Test of Pour Point by Addition 0.5% to (+)10°C Turbine Oil
A	-	1.5335	-	423.3	29	1.16	0.62	1.01	9.61	769	-	-18
B	-	1.5400	30,000	1,067	20	0.64	0.65	0.96	5.70	962	-	-18
C	0.9279	-	-	-	25	0.33	0.47	1.30	7.47	-	0.02	-18

Table VIII(B)35
THE EFFECT OF THE POUR POINT DEPRESSANT ON HIGH VISCOSITY OILS

	Specific Gravity (d_4^{15})	Viscosity (S.U.S.) 100°F	Viscosity (S.U.S.) 210°F	Viscosity Index	Carbon Residue (%)	Acid Value	Saponification Value	Stability Test Vis. Ratio	Stability Test Conradson Carbon (%)	Pour Point (°C) Depressant Added 0	0.1	0.5	1.0
Aeroplane Oil	0.9029	1567.8	118.2	100.6	0.59	0.09	-	1.35	1.85	+7	4	-1	-4
Synthetic Lubricating Oil	0.9002	1443.0	124.7	111.7	0.82	0.32	0.58	2.10	2.93	10	10	8	0
Natural Lubricating Oil	0.9342	2849.6	126.2	66.3	1.59	0.11	0.34	2.70	5.27	8	5	3	-3

ENCLOSURE (B)35

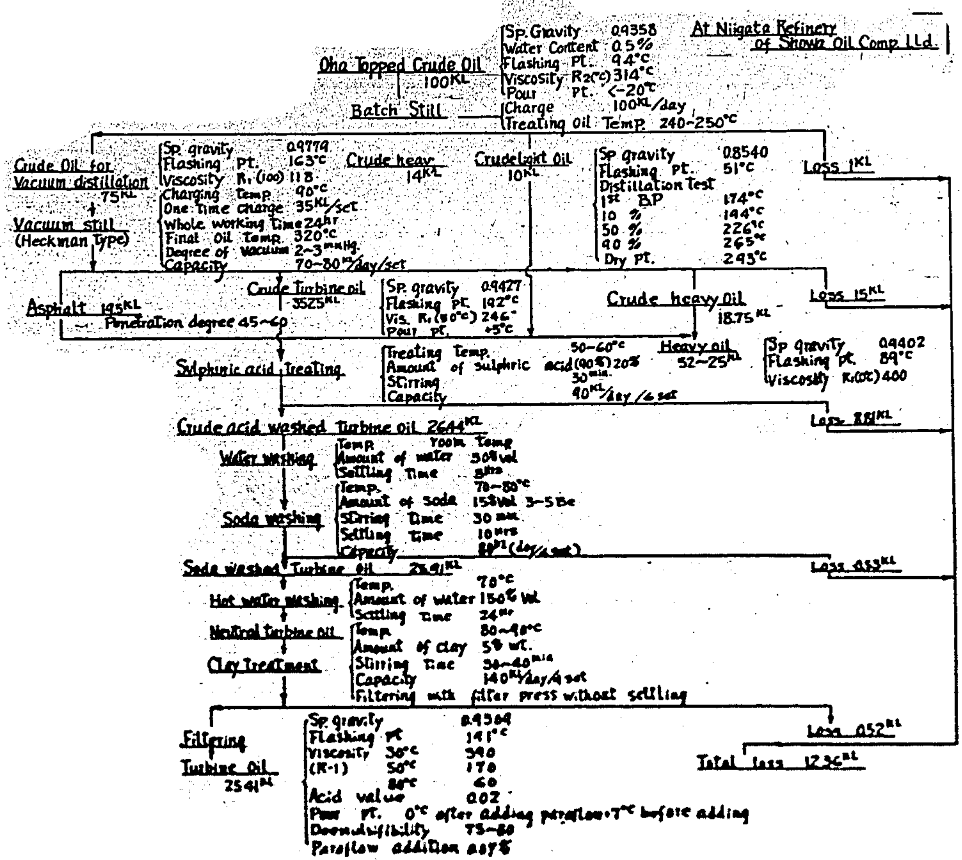

Figure 1(B)35

FLOW SHEET OF PRODUCTION OF TURBINE OIL FROM OHA CRUDE OIL

ENCLOSURE (B)35

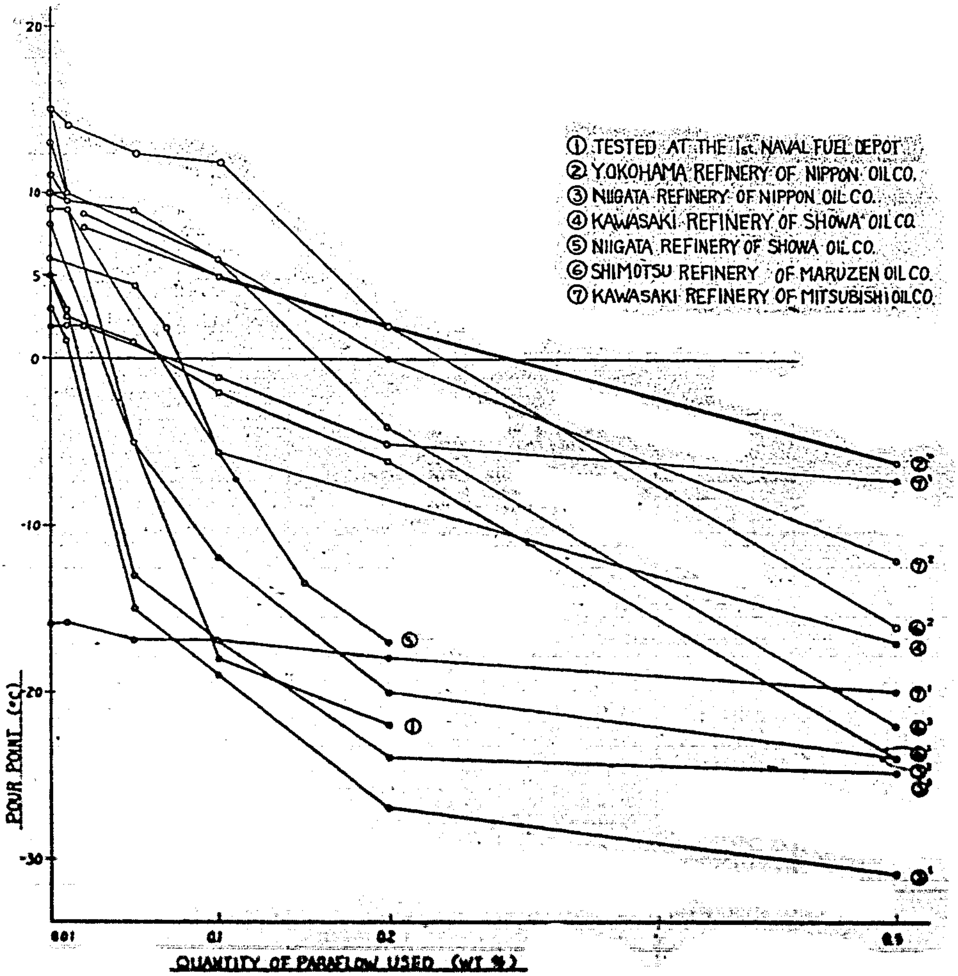

Figure 2(B)35
TEST OF PARAFLOW IN TURBINE OILS

ENCLOSURE (B)35

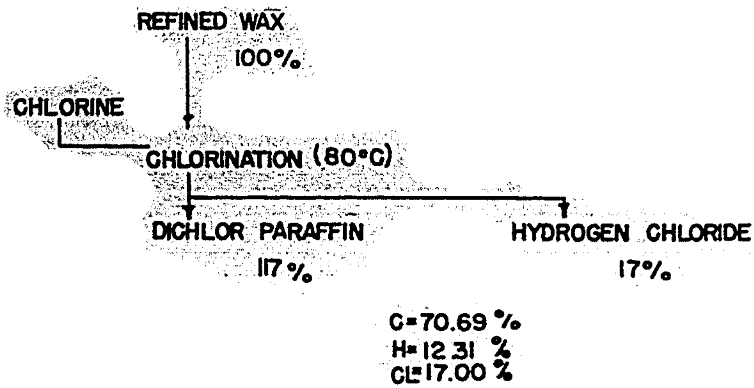

Figure 3(B)35
CHLORINATION OF PARAFFIN WAX

ENCLOSURE (B)35

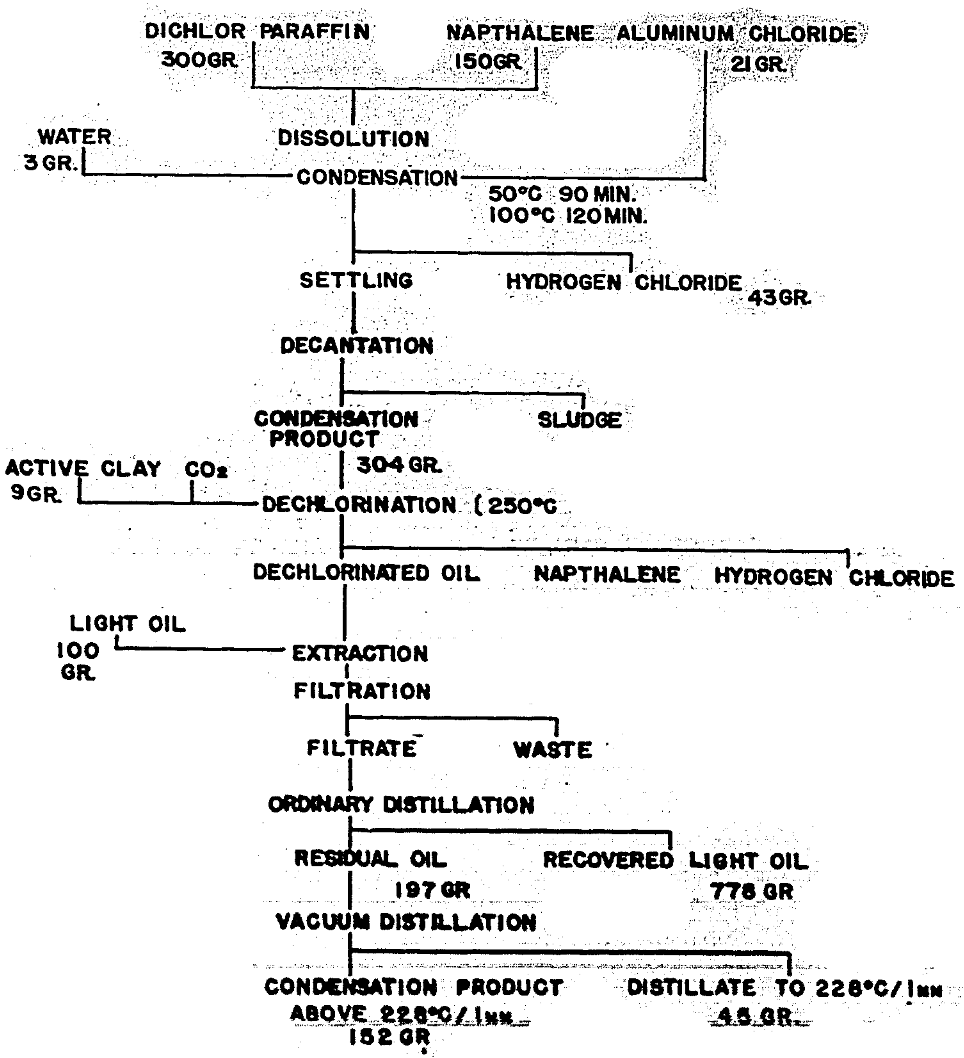

Figure 4(B)35
THE SYNTHETIC METHOD FOR PREPARING POUR POINT DEPRESSANT

ENCLOSURE (B)35

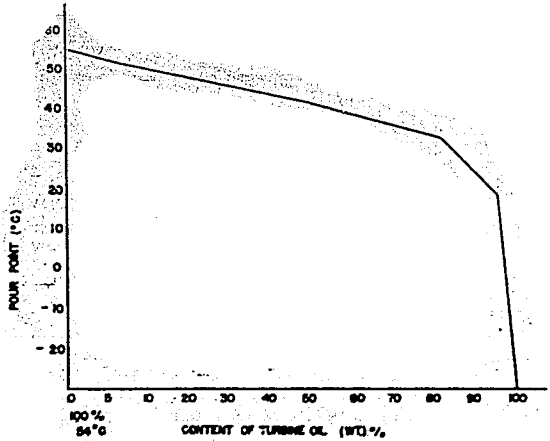

Figure 5(B)35
EFFECT OF WAX IN POUR POINT OF TURBINE OIL

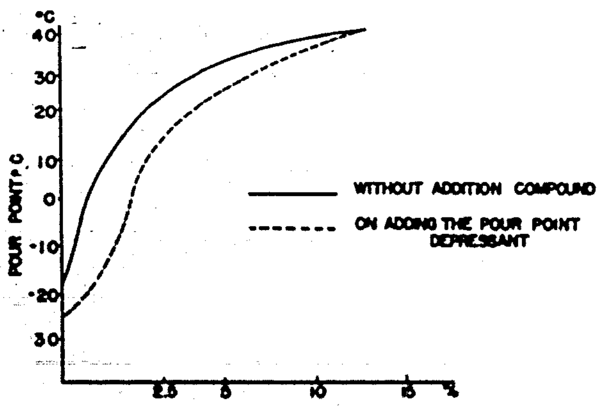

Figure 6(B)35
EFFECT OF THE POUR POINT DEPRESSANT IN TURBINE OIL
(Made at OFUNA)

ENCLOSURE (B)35

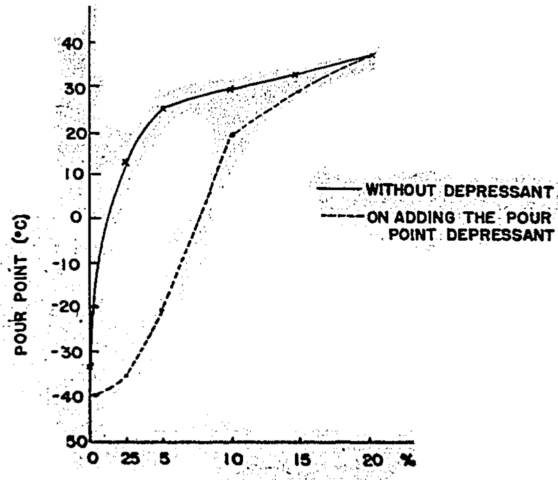

Figure 7(B)35
EFFECT OF POUR POINT DEPRESSANT FOR SPINDLE OIL

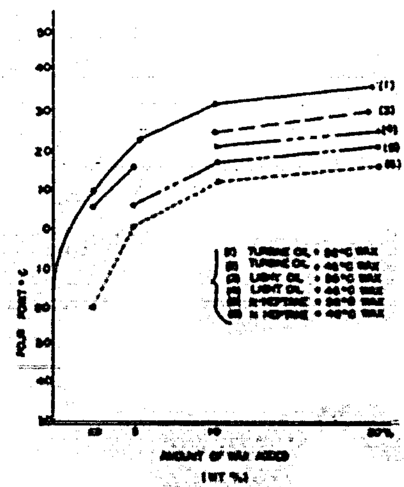

Figure 8(B)35
THE EFFECT OF WAX ON POUR POINT TEST