ENCLOSURE (B) 16

STUDIES ON HYDROCRACKING OF OILS AND TARS

(In Six Parts)

by

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INTRODUCTION

The object of these studies was to investigate the production of high quality aviation gasoline from oils and tars by hydrocracking.

This report consists of six parts as follows:

Part 1. Studies on hydrocracking of high temperature coal tar.

Part 2. Studies on hydrocracking of low temperature coal tar.

Part 3. Studies on hydrocracking of OHA gas oil.

Part 4. Studies on hydrocracking of OMONOGAWA gas oil.

Part 5. Studies on hydrocracking of SUMATRA kerosene.

Part 6. Studies on hydrocracking OHA gas oil in a semi-commercial plant.

The results were as follows:

Raw Material	Catalyst	Reaction Temp. (°C)	Reaction Press. (kg/cm^2)	Yield of Aviation Gasoline Vol. %	Octane Number of Aviation Gasoline Leaded 0.1 %
High Temp. Tar.	MoS_3	470	200	25	92
Low Temp. Tar.	MoS_3	430	200	50	89
Sumatra Kerosene	*	420	200	70	87
Omonogawa Gas Oil	*	430	200	73	89
Oha Gas Oil	*	410	200	70	92

*Nickel-Molybdenum oxide

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PART I

STUDIES ON HYDROCRACKING OF HIGH TEMPERATURE COAL TAR

by

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Research Period: March 1941-December 1942

SUMMARY

The object of these experiments was to study the preparation of aviation gasoline high temperature coal tar by means of high pressure hydrocracking.

To prepare aviation gasoline of satisfactory quality, it was necessary to process in two steps, i.e. primary and secondary hydrocracking, using MoS_3 as a catalyst. In the primary hydrocracking step under conditions of 200kg/cm^2 and 420°C, a fraction boiling at 200 to 300°C (60 vol.% yield) was prepared as feed for secondary hydrocracking.

In the secondary hydrocracking step a 40 vol. % yield of aviation gasoline was obtained under conditions of 200 kg/cm^2 and 470°C. The octane value of this gasoline with 0.1 % of lead was 92.

I. INTRODUCTION

A. History of Project

A large amount of high temperature coal tar is produced as a by-product of coke manufacturing in steel works. Because of limited resources of oil in Japan, consideration was given to the use of such tar as a raw material for preparation of aviation gasoline. These hydrocracking tests on high temperature tar were started in March 1941 and completed in December 1942.

B. Key Research Personnel Working on Project

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II. DETAILED DESCRIPTION

A. Feed Stock

The creosote oil-feed stock was a brownish black, viscous liquid fractionated from crude high temperature coal tar at the Showa Steel Works. Crystals of crude anthracene had been removed from this cut by filtration.

Properties of this oil were as follows:

Properties of Creosote Oil

Ultimate Analysis

C	90.1 wt.%
H	5.6 wt.%
S	0.3 wt.%
N	0.9 wt.%
C/H	16.0 wt.%
Ash	trace
d_{20}^{20}	1.135
Free Carbon	trace

Distillation

Moisture	0.3%
First Drop	230°C
10% point	284°C
20% point	306°C
30% point	324°C
40% point	337°C
50% point	347°C
60% point	357°C
70% point	368°C
80% point	384°C
--300°C fraction	17.0%
--400°C fraction	88.5%
Phenols	4.0 vol.%
Bases	6.6 vol.%
Unsat. hydrocarbon	13.4 vol.%
Arom. hydrocarbon	76.0 vol.%
Sat. hydrocarbon	0 vol.%

B. Autoclave Tests

1. Comparison of Catalysts

200 to 300 gm of oil and 5 wt% of catalyst were introduced in a 2.4lit rotary autoclave and hydrogen was added until the pressure reached 100 kg/cm^2 at normal temperature. The autoclave was heated at the rate of 2.5°C per minute to 450°C and the reaction was continued for 30 to 60 minutes. The relative activities of catalysts were determined by comparison of the properties of oil products and analyses of the residual gases.

Experimental data are summarized in Tables I(B)16 and II(B)16.

Catalyst with good activity showed high H_2 consumption and low density of oil product. Of the various catalysts, MoS_3 demonstrated

the best activity.

2. Influence of Reaction Temperature

In these experiments 200 gm of oil and 10 gm of MoS_3 were charged to the autoclave (2.4lit Can) and H_2 was added until pressure reached 100 kg/cm2 at normal temperature.

Results of these experiments are tabulated in Table III(B)16.

On the basis of these results a temperature of 450 to 470°C was selected for the primary hydrocracking of this oil, because the yield of 200 to 300°C feed stock for secondary hydrocracking was a maximum.

3. Secondary Hydrocracking

A 200 to 300°C fraction made from creosote oil hydrogenated at 450°C over MoS_3 catalyst, was used as the feed stock. Reaction conditions and results of these experiments are given in Tables IV(B)16, V(B)16 and VI(B)16.

On the basis of these data 480° was selected as the optimum reaction temperature for secondary hydrocracking on the basis of producing maximum yield of aviation gasoline is maximum. This temperature is also regarded as the maximum allowable due to increasing coking and gasification difficulties.

C. Experiments in the Continuous Hydrocracking Pilot Plant

1. Primary Hydrocracking

A flow sheet of the pilot plant is given in Figure 1(B)16. This same unit was also used in experiments on hydrocracking of low temperature tar, Oha gas oil, pine root oil and soya bean oil.

MoS_3 was selected as the catalyst for these experiments.

Reaction conditions and results are given in Tables VII(B)16 through XI(B)16. Good yields of oil product and only a small amount of gaseous hydrocarbons were obtained in these experiments. The ratio of feed stock and hydrogen was optimum at 3 m^3/lit.oil.

Yield of the oil fraction boiling at 200 to 300°C (raw material for secondary hydrocracking) was about 60%. CS_2 was added in small amounts to maintain activity of catalyst.

2. Secondary Hydrocracking

Data on secondary hydrocracking in the pilot plant, using MoS_3 catalyst, are given in Tables XII(B)16 through XV(B)16.

D. Material Balance

A material balance for the process under optimum conditions is given in the following diagram.

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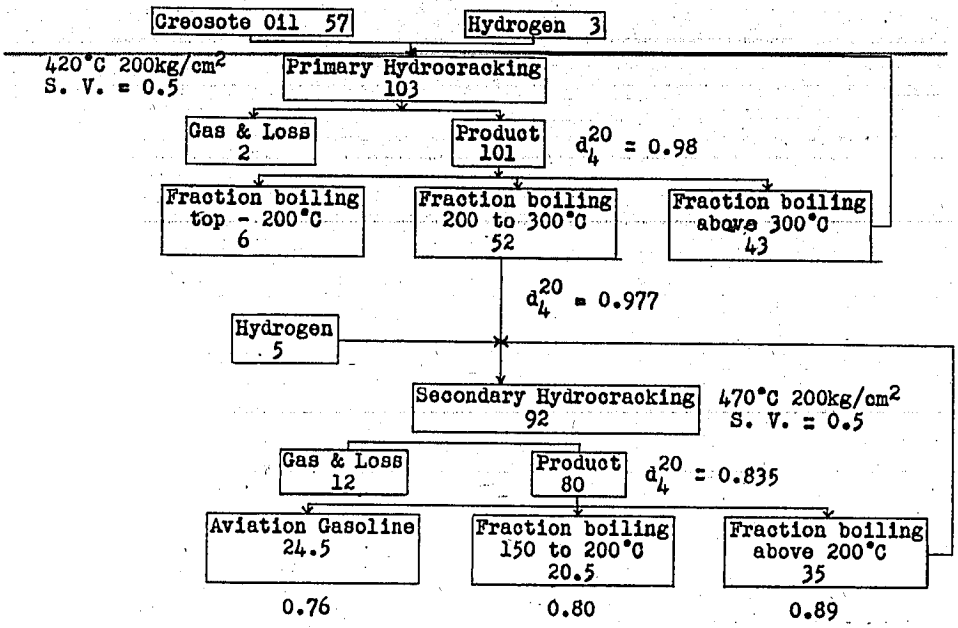
MATERIAL BALANCE



III. CONCLUSIONS

A. MoS_3 showed the best activity for hydrocracking of creosote oil in both autoclave and continuous pilot plant experiments.

B. 470°C was the optimum reaction temperature for primary hydrocracking in autoclave tests, but 420°C was optimum in the continuous pilot plant.

C. By primary hydrocracking in the continuous pilot plant, it was found that the oil product contained 10% of the fraction boiling below 200°C, 60 vol.% of the fraction boiling between 200 to 300°C.

D. By primary hydrocracking in the continuous pilot plant, the yield of aviation gasoline was 40 vol.% under conditions of 200 kg/cm² and 470°C, using MoS_3 catalyst. Octane value of this aviation gasoline was 92 with 0.1% of lead.

APPENDIX A

PROCEDURE FOR DETERMINING HYDROCARBON TYPES

A. Unsaturated Hydrocarbons

Sample used	100cc
H_2SO_4(80% d_4^{15} = 1.732)	200cc
Temperature	below 7°C
Period	15 minutes

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Content of Unsaturated Hydrocarbons (Vol.%) = Percent of Sample Over in Engler Distillation - Percent Residual Oil Distilled Over (After Treatment by 80% H_2SO_4)

B. Aromatic Hydrocarbons

Sample (Oil with unsaturated hydrocarbon removed) used ... 50cc
H_2SO_4 (98%, d_4^{15} = 1.841) added 150cc
Temperature ..... 15°C
Period ..... 30 minutes

Content of Aromatic Hydrocarbons (vol.%) = $(100-U) \times \frac{50-V}{50}$

Where: V = Oil after treatment
U = content of unsaturated hydrocarbons (vol.%)

C. Naphthenic Hydrocarbon

Sample (with unsaturated and aromatic hydrocarbons removed) used 10cc
Aniline added (b.p. 184 ± 2°C, d_4^{15} = 1.026) 10cc

Content of Naphthenic Hydrocarbon = $\frac{70-T}{30}$ (100-U-A) (Vol.%)

Where: T = Aniline point °C of sample
U = Content of unsaturated hydrocarbon (Vol.%)
A = Content of aromatic hydrocarbon (Vol.%)

D. Paraffinic Hydrocarbon

Where: Content of paraffinic hydrocarbon = 100-(U A N) (Vol.%)
N = Content of naphtenic hydrocarbon (Vol.%)

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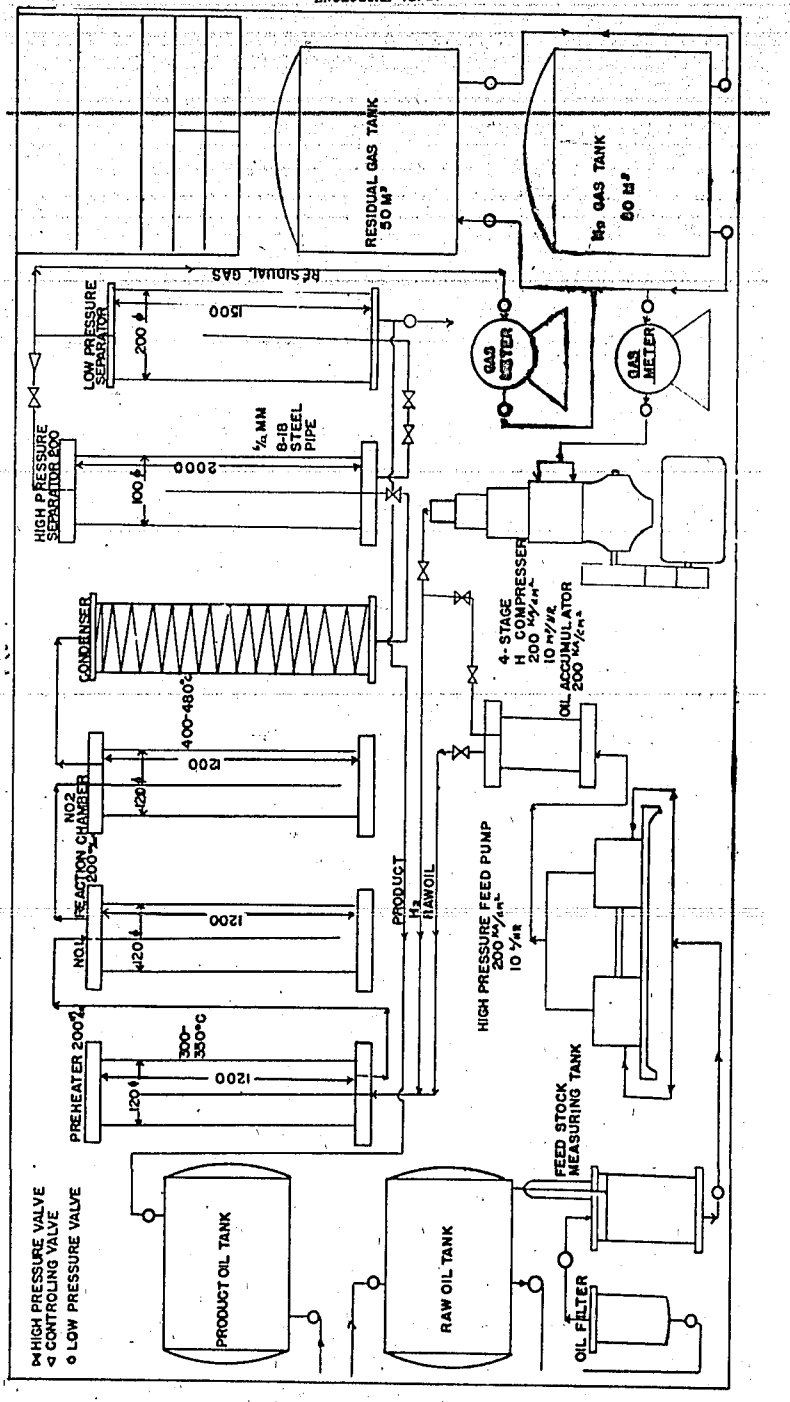

Figure 1(B)16
FLOW SHEET OF HYDROCRACKING PILOT PLANT

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Table I(B)16
CATALYST TESTS IN ATUOCLAVE
REACTION CONDITIONS

Exp. No.	Catalyst	Reaction Conditions			H_2 Consumption		Analysis of Residual Gases (vol. %)							
		Oil (gm)	Catalyst (gm)	Temp. (°C)	Time (min)	Press. Drop (kg/cm²)	Weight (%)	CO_2	O_2	CnH_{2n}	CO	H_2	CnH_{2n+2}	N_2	
1	No Catalyst	300		450	30	7.3	0.7	0.2	0.6	0.3	0.3	92.0		2.8	
2	No Catalyst	300		470	30	9.9	0.9	0.1	0.4		0.1	94.8		4.6	
3	$Cu\ CrO_4$	300	15	450	30	16.4	1.6	0.1	0.7	0.5	0.3	89.2	6.2	2.9	1.2
4	MoO_3	300	15	450	30	35.0	2.4			0.1	0.4	96.2		3.3	
5	MoO_3	300	15	470	30	43.0	2.9	0.1		0.7	0.4	95.5		3.4	
6	MoO_3	200	10	450	60	31.5	3.5		0.2	0.1		93.7		6.2	
7	MoO_3	200	10	470	60	37.6	3.9	0.2			0.3	96.2	2.3	2.0	3.0
8	$Al(CH_3)_3$	300	15	450	30	20.5	1.6	0.3	0.3	0.2	0.8	93.2		5.2	
9	MoO_3-ZnO(3:10.3)	300	15	450	30	23.4	1.4		0.2	0.2	0.5	96.5		2.6	
10	MO_3	300	15	450	30	16.6	1.4		0.3	0.3	0.3	94.0		5.3	
11	$Zn(CH_3COO)_2$	300	15	450	30	9.3	0.7			0.1	0.4	96.9		2.8	
12	$ZnCl_2$	300	15	450	30	18.4	1.3		0.2	0.2	0.4	94.6		4.0	
13	$ZnCl_2$	300	15	450	30	43.3	2.9		0.3		0.5	94.6	3.3	1.3	1.2
14	Al_2O_3	300	15	450	30	21.2	1.7	0.1		0.2	0.4	91.7	4.0	3.3	1.0
15	$ZnCl_2$	300	15	450	30	24.0	1.7	0.4	0.2	0.2	0.5	95.4		3.3	
16	MoO_3-ZnO-S(1:0.1:2)	300	15	450	30	30.5	2.0	0.4		0.1	0.6	95.9		2.0	
17	$(NH_4)_2MoO_4$	300	15	450	30	23.9	1.7	0.1	0.2	0.2	0.3	95.9		3.3	
18	$ZnMoO_4$	300	15	450	30	12.3	0.9			0.4	0.5	97.1		2.0	
19	$(NH_4)_2[Mo_2O_7F_2\cdot S\cdot Al_2O_3$ (1 : 2 : 2 : 2)	300	15	450	30	35.5	2.4			0.2	0.4	95.8		3.6	
20	$(NH_4)_2P_2[Mo_2O_7]\cdot 2H_2O$	200	10	450	60	31.3	3.4	0.1			0.2	94.5	2.7	2.6	1.5
21	$2(NH_4)_2O\cdot SiO_2\cdot 12MoO_3\cdot x\ H_2O$	200	10	450	60	38.8	4.3	0.2	0.1	0.1	0.1	93.0	3.9	2.7	1.5
22	$(NH_4)_3P_6[Mo(Mo_2O_7)_2]\cdot 5H_2O$	200	10	450	60	27.3	3.0		0.1	0.5	0.2	95.9		3.3	
23	$3(NH_4)_2O\cdot P_2O_5\cdot 24MoO_3\cdot x\ H_2O$	200	10	450	60	35.7	3.9		0.1	0.2	0.4	95.2	1.7	2.4	1.0
24	$(NH_4)_3P_6[Mo(Mo_2O_7)_2]\cdot 7H_2O$	200	10	450	60	35.7	2.0	0.1		0.5	0.6	94.2		3.6	
25	$(NH_4)_8[Mo(Mo_2O_7)_2]\cdot 2OH_2O$	200	10	450	60	22.3	2.5			0.2	0.2	95.6		4.0	
26	MoO_3-MgO-KOH(1:1:0.3)	200	10	450	60	13.4	1.7	0.1	0.1	0.2	0.5	94.5		4.6	
27	Fe_2O_3	200	10	450	60	14.9	1.6	0.1		0.1	0.6	97.2		2.0	
28	$FeCl_2$	200	10	450	60	16.2	2.0		0.2	0.6	0.3	94.9		4.0	
29	$Na_2PO_4\cdot 12H_2O$	200	10	450	60	13.0	1.7	0.2	0.3	0.2	0.3	95.0		4.0	
30	$(NH_4)_3PO_4(MoO_3)_{12}\cdot 6H_2O$	200	10	450	60	33.1	3.6	0.1	0.5	0.5	0.6	95.0	0.5	2.3	
31	$3\ KIO\cdot P_2O_5\cdot 24MoO_3$	200	10	450	60	29.0	2.4	0.2	0.4	0.1	0.3	96.4		2.6	
32	MoO_3	200	10	450	60	46.1	4.8			0.3	0.5	94.6	3.0	1.6	2.3

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Table II(B)16
CATALYST TESTS IN AUTOCLAVE
PRODUCT YIELDS AND INSPECTIONS

EXP. No.	Yield of Product		Density	Fractional Distillation (°C)									Fraction boiling up to 360°C (Vol%)
	Wt%	Vol%	20/20 °C	I.B.P.	10%	20%	30%	40%	50%	60%	70%	80%	
1	100.1	101.5	1.119	220	270	292	306	315	330	342	357		75
2	99.5	105.6	1.069	215	254	294	304	321	331	342	356		75
3	98.9	101.9	1.102	215	256	287	307	317	328	342	355		78
4	102.7	110.6	1.054	104	225	262	288	299	312	325	339	355	84
5	97.6	107.5	1.042	95	221	251	269	286	302	314	332	347	88
6	93.5	102.5	1.032	118	224	254	269	290	302	315	327	342	90
7	92.8	104.3	1.010	95	182	231	248	273	289	301	317	333	91
8	98.3	102.8	1.085	114	250	281	300	311	325	336	346		81.5
9	100.1	105.7	1.085	103	255	281	298	313	323	333	345		80.0
10	97.5	100.6	1.100	193	264	284	303	314	325	336	348		79
11	97.1	101.1	1.091	127	276	298	312	320	333	355			75
12	98.0	101.8	1.093	180	262	274	298	313	326	337	349		79
13	100.5	114.0	1.045	104	225	255	278	296	311	324	339	358	83
14	99.4	103.5	1.090	165	260	277	299	316	323	333	347		78
15	96.7	101.2	1.085	145	240	272	290	305	319	330	343	358	82
16	99.8	105.8	1.071	165	242	273	293	299	317	330	343	357	83
17	98.0	102.5	1.085	145	252	273	290	308	322	334	344	359	81
18	98.5	100.8	1.109	214	265	288	307	312	335	342	357		72
19	99.0	105.7	1.063	128	239	265	284	301	315	326	338	353	85
20	96.6	105.8	1.036	116	221	253	271	291	303	315	327	340	90
21	94.1	104.8	1.019	114	216	246	264	279	292	305	318	332	92
22	96.5	104.7	1.046	142	238	268	284	291	311	320	333	348	88
23	95.1	105.4	1.024	128	225	254	271	286	299	314	325	340	90
24	96.4	100.8	1.085	193	254	279	295	313	321	334	343	358	83
25	95.4	101.6	1.065	160	248	272	288	303	316	326	338	352	85
26	96.9	100.8	1.091	188	256	284	296	314	325	334	347	359	81
27	98.4	102.2	1.093	168	252	282	298	311	321	330	342	358	84
28	97.0	102.2	1.077	140	243	267	291	306	317	329	341	356	84
29	99.1	102.7	1.095	186	252	281	298	312	324	335	346	359	81
30	96.0	105.3	1.035	109	226	249	275	292	306	318	330	347	88.5
31	95.9	103.6	1.030	174	241	268	280	296	307	317	329	342	92
32	94.9	108.1	0.9965	92	191	232	252	268	282	296	309	328	92

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Table III(B)16
EFFECT OF TEMPERATURE WITH MoS_3 CATALYST

Exp. No.	1	2	3	4	5	6
Catalyst	MoS_3	MoS_3	MoS_3	MoS_3	MoS_3	MoS_3
Reaction Temperature (°C)	400	430	430	450	470	480
Reaction Time (min)	90	60	90	60	60	60
Initial Pressure H_2 (Kg/cm^2)	100	100	100	100	100	100
Pressure Drop (Kg/cm^2)	35.5	43.3	44.2	46.1	45.6	47.8
H_2 Absorbed (Wt% of oil charge)	3.9	4.6	4.7	4.8	5.1	4.4
Analysis of Residual Gas						
CO_2	0.2		0.1			
O_2	0.3	0.2	0.2		0.1	0.1
C_nH_{2n}	0.2	0.5	0.4	0.3	0.1	0.1
CO	0.4	0.5	0.6	0.5	0.7	0.8
H_2	94.3	94.2	94.1	94.6	89.6	87.2
C_nH_{2n+2}	2.3	3.5	3.3	3.0	5.9	10.6
N_2	2.7	1.1	1.3	1.6	3.3	1.2
n	1.3	1.6	1.0	2.3	2.5	2.3
Yield of Product						
Wt %	99.2	98.5	97.7	94.9	87.7	85.0
Vol%	109.2	110.0	110.0	108.1	100.9	98.0
Density $\left(d_{20}^{20}\right)$	1.031	1.016	1.008	0.997	0.986	0.985
Fractional Distillation (Vol%)						
F.D.	168	132	108	92	85	80
10 %	241	226	222	191	152	142
30 %	282	275	267	252	225	217
50 %	305	298	294	282	259	256
70 %	332	319	316	309	295	297
90 %	358	359	353	357	336	350
Fraction Boiling						
200°C (%)	4.0	7.0	8.0	12.5	20.0	24.0
300°C (%)	47.0	52.5	55.5	63.0	72.5	71.0
360°C (%)	91.0	92.0	92.5	92.0	97.0	94.0

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Table IV(B)16
SECONDARY HYDROCRACKING IN AUTOCLAVE
CONDITION OF REACTION

Exp. No.	Catalyst	Reaction Temp (°C)	Reaction Time (min)	H_2 Initial Press (kg/cm^2)	Press Drop (kg/cm^2)	H_2 Consumption (wt.%)	Yield of Product (wt.%)	Yield of Product (vol%)
1	MoS_3	450	60	100	25.5	2.8	96.7	103.5
2	MoS_3	470	60	100	33.8	3.9	83.9	92.2
3	MoS_3	480	60	100	41.1	6.5	75.7	85.1

Table V(B)16
SECONDARY HYDROCRACKING IN AUTOCLAVE
CONDITION OF REACTION

Exp. No.	CO_2	O_2	C_nH_{2n}	CO	H_2	C_nH_{2n+2}	N_2	n
1	0.2	0.2	0.1	0.8	95.2	2.9	0.6	1.0
2	0.4	0.1	0.4	0.2	91.7	5.9	0.3	2.3
3	0.2	0.2	0.6	0.8	82.5	12.4	3.3	3.2

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Table VI(B)16
SECONDARY HYDROCRACKING IN AUTOCLAVE
PROPERTIES OF FEED STOCK AND PRODUCTS

	Feed Stock	Oil Product			A Fraction Boiling ~150°C of Product (Exp No.3)
		1	2	3	
d_{20}^{20}	0.9886	0.9241	0.9005	0.8800	0.8110
F.D. (°C)	146	96	74	60	64
10 % (°C)	208	168	116	103	82.5
20 % (°C)	222	196	142	119	87
30 % (°C)	229	206	165	137	90.5
40 % (°C)	241	218	185	155	95
50 % (°C)	250	228	200	176	99
60 % (°C)	260	236	210	193	105
70 % (°C)	272	247	220	206	111
80 % (°C)	288	260	235	219	119
90 % (°C)	305	278	263	256	131
D.P. (°C)	348	347	340	334	161
Total Distilled (%)	99.0	98.8	98.5	98.5	98.0
~150°C (%)		6.5	23.0	37.0	
~200°C (%)		22.0	50.0	65.0	
Un Sat. H.C. (%)					1.5
Arom. H.C. (%)					55.2
Naph. H.C. (%)					43.3
Para. H.C. (%)					
Octane Value Clear					83.6
Octane Value Lead					95.2

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Table VII(B)16
CONTINUOUS PILOT PLANT - PRIMARY HYDROCRACKING
REACTION CONDITION AND YIELD

Run No.		9	10	11	12	13	14
Temp of Preheater (°C)		300	300	300	300	300	300
Temp Reaction Chamber No.1		380	380	380	380	380	380
Temp Reaction Chamber No.2		430	430	430	430	430	440
H_2 Pressure (Kg /cm^2)		200	200	200	200	200	200
H_2 Charged (m^3/hr)		4	8	6	4	6	7
Oil Charged (lit/hr)		4	8	6	4	6	7
CS_2 Added (cc/lit oil)		3			3	1	1
Run Period (hr)		6	6	6	72	24	72
Yield of Product (Vol%)		114.7	112.6	118.3	115.3	115.4	116.3
Yield of Product (Wt%)		98.5	101.5	105.7	100.4	102.7	105.1
H_2 Consumption Per Hour	(m^3)	3.49	5.35	3.79	2.99	3.48	3.60
	(lit/lit oil)	845	669	630	724	573	516
	(gm/kg oil))	66.8	52.7	49.8	57.2	45.3	40.8

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Table VIII(B)16
CONTINUOUS PILOT PLANT - PRIMARY HYDROCRACKING
PROPERTIES OF PRODUCTS

Run No.	9	10	11	12	13	14	Raw Material
Properties of Product							
d_4^{20}	0.9771	1.0281	1.0155	0.9897	1.0114	1.0283	1.135
First Drop (°C)	66.0	79.0	78.0	66.0	77.0	74.0	230
10 % point (°C)	156	210	201	150	207	217	284
20 % point (°C)	179	240	229	205	227	243	306
30 % point (°C)	228	264	250	218	252	271	324
40 % point (°C)	245	284	277	248	276	283	337
50 % point (°C)	260	297	290	265	287	300	347
60 % point (°C)	280	313	308	290	308	312	357
70 % point (°C)	296	331	322	310	323	326	368
80 % point (°C)	319	346.5	340	328	345	348	384
90 % point (°C)	350	373	370	366	378	370	
97 % point (°C)	390		390			390	
Dry point (°C)	397	410	395	395	395	402	
Total Distilled (%)	98.5	96.5	97.5	96.5	96.5	98.0	
Residue (%)	0.7	3.0	2.0	3.0	3.0	1.5	
Loss (%)	0.8	0.5	0.5	0.5	0.5	0.5	
~150 °C (%)	0.9						
~200 °C (%)	21.0	6.0	25	19.0	9.0	8.0	
~300 °C (%)	72.0	51.0	55.5	67.5	56.0	50.0	17.0
Analysis of residual gas							
CO_2		0.3			0.1		
C_nH_{2n}	0.2	0.3	0.2	0.1	0.2	0.5	
CO	0.6	0.3	0.6	0.9	0.5	0.4	
H_2	87.6	91.8	93.6	92.0	93.2	91.6	
C_nH_{2n+2}	11.6	6.6	6.0	7.0	5.6	5.0	
N_2		0.7			0.3	1.0	
n	1.1	1.6	1.0	1.6	1.3	2.2	
O_2					0.1	0.2	

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Table IX(B)16
CONTINUOUS PILOT PLANT - PRIMARY HYDROCRACKING
PROPERTIES OF RAW MATERIAL

Run No.	1	2	3	4	5	6	7	8
d_4^{20}	0.9423	0.9960	0.9612	0.9651	0.9842	1.0250	1.0176	1.0167
First Drop (°C)	940	104	82	92	85	95	87	92
10 % point (°C)	170	210	183	209	205	215	218	222
20 % point (°C)	212	240	223	234	235	247	245	255
30 % point (°C)	235	260	240	248	262	262	262	278
40 % point (°C)	256	276	258	263	280	283	280	290
50 % point (°C)	270	291	273	277	292	300	303	308
60 % point (°C)	285	305	284	290	305	315	313	315
70 % point (°C)	300	323	303	300	319	328	335	330
80 % point (°C)	320	338	321	315	341	344	351	343
90 % point (°C)	345	371	345	340	364	380	374	373
Dry point (°C)	378	387	385	382	385	384	395	390
Total Distilled (%)	95.0	95.0	97.0	97.0	96.5	95.0	95.0	97.0
Residue (%)			2.8	2.6	3.1			
Loss (%)			0.2	0.4	0.4			
~200 °C (Vol %)	16.5	8.5	15.0	8.0	8.0	8.0	8.0	7.5
~300 °C (Vol %)	70.0	58.0	68.5	70.0	56.0	50.0	49.5	48.0
Analysis of Residual Gas								
CO_2		0.1				0.4	0.2	
O_2		0.2	0.2		0.2	0.4	0.2	0.1
C_nH_{2n}	0.6	0.2	0.6		0.6	1.2	1.0	0.5
CO	0.6	0.4	0.4			0.6		0.1
H_2	92.0	90.0	90.9		93.9	90.9	91.5	91.3
C_nH_{2n+2}	5.3	4.0	2.0		3.4	4.2	2.0	7.2
N_2	1.5	5.1	5.9		1.9	2.3	5.1	0.8
n	1.7	1.0	1.0		1.0	1.0	1.0	1.0

ENCLOSURE (B)16

Table X(B)16
CONTINUOUS PILOT PLANT - PRIMARY HYDROCRACKING
REACTION CONDITIONS AND YIELDS

Run No.		1	2	3	4	5	6	7	8
Temp of Preheater (°C)		350	350	350	350	350	350	350	350
Temp Reaction Chamber	No.1	380	380	380	380	380	390	390	390
	No.2	420	420	420	420	420	420	430	430
H_2 Pressure (kg/cm^3)		200	200	200	200	200	200	200	200
H_2 Charged (m^3/Hr)		5	10	7	15	5	10	10	10
Oil Charged (lit/Hr)		5	10	5	5	5	10	10	10
CS_2 Added (cc/lit oil)		3	0.1	3	7	4	0.3	3	2
Name of raw material		(A)	(A)	(A)	(A)	(B)	(A)	(A)	(B)
Run Length (Hr)		6	6	6	6	12	10	6	6
Yield of Product	(Vol%)	124.5	119.5	120.2	121.8	113.9	112.7	114.2	110.4
	(Wt %)	103.4	104.9	101.8	103.6	101.8	101.8	102.4	102.0
H_2 Consumption (m^3)		403	5.50	3.78		2.24	5.71	5.01	4.79
(lit/lit vol)		803	548	747		448	571	503	479
(gm/kg oil)		63.6	43.4	59.2		36.6	45.2	39.8	39.1

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Table XI(B)16
CONTINUOUS PILOT PLANT - PRIMARY HYDROCRACKING
PROPERTIES OF PRODUCTS

Raw Material	(A)	(B)
d_{20}^{20}	1.135	1.103
First Drop (°C)	230	230
10 % point (°C)	284	295
20 % point (°C)	306	308
30 % point (°C)	324	321
40 % point (°C)	337	333
50 % point (°C)	347	343
60 % point (°C)	357	353
70 % point (°C)	368	364
80 % point (°C)	384	379
~300 °C (Vol%)	17.0	15.0
~400 °C (Vol%)	88.5	

(A) Creosote oil.

(B) Creosote oil mixed with fraction boiling above 300°C of hydrogenated product in the ratio of 4:6 by Vol.

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Table XII(B)16
CONTINUOUS PILOT PLANT - SECONDARY HYDROCRACKING
PROPERTIES OF FEED STOCK

Raw Material		(A)	(B)	(C)
d_4^{20}		0.9767	0.9574	0.9563
First Drop	(°C)	190	80.5	200.5
10 % point	(°C)	220	178	220.5
20 % point	(°C)	230	214	227
30 % point	(°C)	239	226.5	233.5
40 % point	(°C)	247	235.5	241
50 % point	(°C)	254	247	249.5
60 % point	(°C)	264	257.5	259
70 % point	(°C)	271	269.5	268
80 % point	(°C)	283	282	280
90 % point	(°C)	300	298	296.5
97 % point	(°C)	317	321.5	325
Dry Point	(°C)	323	331	346
Total distilled	(%)	98.5	99	99
Residue	(%)	1.0	0.6	0.5
Loss	(%)	0.5	0.4	0.5

(A) A 200-300°C fraction from primary hydrogenation (Table IV Run 12)

(B) A F.D ~300°C fraction from primary hydrogenation (Table IV Run 12)

(C) Mixture of raw material (A) and a fraction 200°C~ of secondary hydrogenated oil (Table VIII Run 4) in ratio of 2:1 by vol.

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Table XIII(B)16
CONTINUOUS PILOT PLANT - SECONDARY HYDROCRACKING
YIELDS AND REACTION CONDITIONS

Run No.		1	2	3	4	5	6	7
Temp of Reaction Chamber	No.1	380	400	400	400	400	450	450
	No.2	420	440	460	470	470	470	470
H_2 Pressure (kgm/cm^2)		200	200	200	200	200	200	200
H_2 Charged (m^3/hr)		5	5	5	5	5	5	5
Oil Charged (ℓ/hr)		5	5	5	5	5	5	5
CS_2 Added (cc/ℓ oil)		3.5	3.5	3.5	3.5	3.5	3.5	3.5
Name of raw material		(A)	(A)	(A)	(A)	(B)	(B)	(C)
Run Period (hr)		4	4	4	6	6	6	6
Yield of Product	(Vol%)	103.1	113.1	99.0	104.2	101.4	100.9	104.2
	(Wt %)	91.2	99.8	99.0	92.2	96.4	91.9	92.4
H_2 Consumption	(m^3)	2.42		3.42	2.80	2.55	2.73	2.26
	(ℓ/ℓ oil)	474		689	563	504	555	458
	(gm/kg oil)	43.7		63.4	51.8	47.4	52.1	42.9

Catalyst: Reaction chamber No. 1 MoS_3 + active clay 10 lit packed
Reaction chamber No. 2 MoS_3 10 lit packed

ENCLOSURE (B)16

Table XIV(B)16
CONTINUOUS PILOT PLANT - SECONDARY HYDROCRACKING
PROPERTIES OF PRODUCTS

Run No.	1	2	3	4	5	6	7
d_4^{20}	0.8661	0.8620	0.8668	0.8615	0.8655	0.8358	0.8352
First Drop (°C)	64	67	53.0	52	50	49	48
10 % point (°C)	121	150		102	100.5	91.5	90
20 % point (°C)	157	186	138	125	122	100	117
30 % point (°C)	189	204	174.5	152	148	132.5	142
40 % point (°C)	200	213	193.5	187	178	154	165.5
50 % point (°C)	209	221	204	200	198	178.5	188
60 % point (°C)	218.5	233	217	214	208	196	200
70 % point (°C)	229.5	245	228	226	221	206.5	210
80 % point (°C)	243	268	241.5	240	239	224	225
90 % point (°C)	263	300	265	263	265	244	248
97 % point (°C)	287	309	297	312	320	308.5	294
Dry point (°C)	300		375	330	330		313
Total Distilled (%)	98.5	98.5	98.0	98.5	98	98	98.5
Residue (%)	1.2	0.8	0.9	0.9	1.0	0.7	0.8
Loss (%)	0.3	0.7	1.1	0.6	1.0	1.3	0.7
~150 °C (%)	18.0	20.0	24.0	29.0	31.0	39.0	32.0
~200 °C (%)	40.0	38.5	47.0	50.0	52.0	64.5	60.0
Yield of Aviation Gasoline (%)	22.2	24.4	28.9	35.2	37.8	44.0	39.2
Analysis of Residual Gas (%)							
CO_2 (%)	0.1		0.1		0.1	0.1	
O_2 (%)	0.2		0.1	0.1	0.1	0.1	0.1
C_nH_{2n} (%)	0.3		0.4	0.5	0.5	0.6	0.2
CO (%)	0.4		0.5	0.4	0.5	0.5	0.2
H_2 (%)	93.1		89.0	89.8	89.0	86.2	89.0
C_nH_{2n+2} (%)	4.6		8.9	8.3	8.9	12.2	9.6
N_2 (%)	1.3		1.0	0.9	0.9	0.3	0.6
n (%)	1.2		1.4	1.3	1.1	1.7	1.5

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Table XV(B)16
CONTINUOUS PILOT PLANT - SECONDARY HYDROCRACKING
PROPERTIES OF AVIATION GASOLINE

Run No.		1	2	3	4	5	6	7
d_4^{20}		0.7689	0.7645	0.7755	0.7724	0.7710	0.7607	0.7534
First Drop (°C)		52	45.5	50	51	53.5	48	43.5
10 %		69.5	69	72	71	73	68	66.5
20 %		77	76	78	78.5	80	78	73.5
30 %		81.5	81.5	82.5	83.5	85	83	80
40 %		94.5	85	86	87.5	88	87	85
50 %		97.5	89	90.5	91.5	92.5	91	89.5
60 %		102	92.5	95	96.5	96.5	99	95
70 %		107.5	98	101	103.5	102	104	101
80 %		108	108	110	114	110.5	113	111
90 %		123.5	123	127	130	128	132	127
97 %		158	155	155	156.5	158	158	160
Dry Point		168.5	177.5	178	171	172	172	172
Total Distilled		98.5	99	99	98.5	98.5	98.5	99
Residue		0.5	0.6	0.5	0.5	0.6	0.6	0.4
Loss		1.0	0.4	0.5	1.0	0.9	0.9	0.6
Sum of 10 %, 50 %, 90 % point (°C)		280.5	281	289.5	292.5	293	291	282
Constituent Analysis								
Unsat. H.C %		1.5	2.0	2.5	1.5	4.5	3.5	4.5
Arom H.C. %		26.6	23.5	31.5	27.6	25.8	21.2	22.0
Naph H.C. %		70.7	69.5	65.2	69.7	69.7	69.0	64.9
Para H.C. %		1.2	5.0	1.1	1.1		6.3	8.6
Vapor Pressure (kg/cm^2)		0.57	0.57	0.56	0.54	0.51	0.56	0.64
Octane Value	Alone	78.6	78.4	79.0	79.2	78.2	77.7	78.6
	Leaded 0.1 %	92.1	92.2	91.9	91.7	91.6	91.5	91.7