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STUDIES ON THE SYNTHESIS OF  
AERO-ENGINE OILS FROM RUBBER

by

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SUMMARY

The object of these studies was to investigate the method of preparing aero-engine oil from natural rubber and the results obtained were as follows:

1. Pale crepe rubber was extracted with several solvents and the molecular weight, iodine value and composition of the extracts were determined. The results showed that natural rubber consisted of isoprene polymers having different degrees of polymerization.
2. It was impossible to obtain an aero-engine oil from rubber by thermal cracking or catalytic cracking with acid clay.
3. By co-polymerization of the cracked distillate of natural rubber with 2.5 volumes or more of cracked wax in the presence of 5%  $AlCl_3$  at 100°C, a desirable aero-engine oil was obtained.
4. By the high pressure hydrocracking of the paraffins of pale crepe rubber in paraffinic hydrocarbon solvent in the presence of nickel catalysts a desirable aero-engine oil was obtained.

I. INTRODUCTIONA. History

Natural rubber was one of the most important hydrocarbon sources available to Japan. It appeared possible to prepare an aero-engine oil if the rubber molecules could be broken into stable compounds having an average molecular weight of about 1,000. In connection with this problem studies on the composition of pale crepe rubber, the co-polymerization of the cracked rubber with cracked wax, and the high pressure hydrocracking of rubber, were conducted from April, 1943 to August, 1944.

B. Key Research Personnel Working on Project

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II. DETAILED DESCRIPTIONA. Experiments on the composition of pale crepe rubber by solvent extraction method.1. Experimental Method

Pale crepe rubber was cut into rectangular pieces, 2mm X 2mm X 10cm, and extracted in an atmosphere of nitrogen by a Soxhlet apparatus with acetone, alcohol, iso-octane, ethyl ether and benzene in that order.

After removing the solvent, the extracts were dissolved in pure benzene with a concentration of 1.5gm/lit and their molecular weights were determined by Staudinger's method (1).

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The molecular weight was calculated by the following formulas:

$$\frac{S_0 t_0}{S t} - 1 = C \times K_m \times P; \quad M = P \times 68(C_5H_8)$$

in these formulae:

$S_0, S_0$  = density of rubber solution and pure benzene at 20°C

$t_0, t_0$  = viscosity (in sec) of rubber solution and pure benzene by Ostwald's viscometer.

$C$  = gm of rubber hydrocarbon dissolved in 1 liter of solvent.

$P$  = degree of polymerization.

$M$  = molecular weight

$K_m$  = constant

=  $3.8 \times 10^{-4}$ —in the case of unrefined rubber. (2)

=  $3.0 \times 10^{-4}$ —in the case of acetone soluble rubber (3)

=  $1.1 \times 10^{-4}$ —in the case of ethyl ether soluble rubber (4)

=  $1.7 \times 10^{-4}$ —in the case of ethyl ether insoluble and benzene soluble rubber (5)

Iodine value was measured by Wijs' method adding 35% excess of iodine.

## 2. Results:

The results obtained are tabulated in Table I(B)15. The following conclusions were derived from these results:

a. The substance extracted by acetone had high oxygen content, low molecular weight, and high iodine value, and consisted of resinous matter or impurities containing oxygen compounds. (6)

b. The substance extracted by alcohol had low carbon content, high oxygen content, very small molecular weight and iodine value, and was presumed to be composed of saccharides.

c. The substances extracted by iso-octane, ethyl ether and benzene had the same elementary composition, and iodine value as isoprene.

d. Proteins were not extracted by these solvents and the greater part of them remained in the residue of extraction. Based on the nitrogen content, the total content of protein in pale crepe rubber was presumed to be 2.2%.

## B. Depolymerization of Natural Rubber

### 1. Experimental Method

1 cc crepe rubber was dissolved in 4 volumes of Sanga Sanga gas oil,

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boiling from 150°C to 200°C, and depolymerized thermally under various conditions, and with and without the use of acid clay.

2. Results of Experiments

Results obtained are given in Tables II(B)15 and III(B)15, and the following conclusions were obtained.

a. The depolymerization of rubber molecules was affected sharply by the heating temperature and the influence of the acid clay catalyst.

b. The molecular weight decreased by depolymerization to about 1,000, but the product was very volatile and highly unsaturated and could not be used as a high class lubricating oil.

C. Polymerization of the Cracked Distillate of Pale Crepe Rubber

A fraction of cracked distillate of pale crepe rubber boiling from 100°C to 300°C was first washed 3 times with one volume of 10% caustic soda solution and then with water. The treated oil was polymerized in the presence of  $AlCl_3$  or acid clay. Results obtained are given in Table IV(B)15 and it was observed that the products prepared by this method had a very low viscosity index and were unsuitable for use as high class lubricating oils.

The formation of low viscosity index oils was assumed to be due to the existence of cyclic compounds such as limonene in the raw material of the polymerization. The isoprene fraction, therefore, was distilled off and was polymerized. The results are shown in Table V(B)15.

From these experiments the following facts were recognized:

1. A good lubricant can be obtained by polymerization of the isoprene fraction of the cracked distillate of pale crepe rubber at about 0°C.
2. A higher viscosity index oil was obtained by the polymerization in the presence of  $SnCl_4$ .
3. Further studies are necessary for the preparation of an excellent aero-engine oil by this method.

D. Co-polymerization of the Cracked Distillate of Pale Crepe Rubber With the Cracked Distillate of Paraffin Wax

To improve the viscosity index and the viscosity ratio (British Air Ministry Oxidation Test), the cracked distillate of pale crepe rubber was co-polymerized with the cracked distillate of paraffin wax, and the results are shown in Table VI(B)15.

An excellent aero-engine oil could be obtained from the cracked distillate of pale crepe rubber by co-polymerizing with 2.5-4 volumes of the cracked distillate of paraffin wax. This method would be suitable for the synthesis of an aero-engine oil from rubber and paraffin wax.

E. Hydrocracking of Pale Crepe Rubber

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1. Hydrocracking of Pale Crepe Rubber Without Solvent.

The thermally depolymerized products of pale crepe rubber have a high iodine value. Therefore, it is necessary to hydrogenate these products to saturated compounds in order to improve their oxidation stability. Hence, the high pressure hydrocracking of pale crepe rubber in an autoclave without solvent was studied and the results tabulated in Table VII(B)15 were obtained. From these results, it may be concluded as follows:

- a. The higher the temperature and the longer the time of reaction, the greater the content of the lower molecular weight components. The viscosity of the product increased sharply by topping to 150°C, but the flash point was low. However, the viscosity of the product could not be determined after making the British Air Ministry Oxidation Test, since the increase of viscosity with oxidation was too large.
- b. Two-stage hydrogenation decreased the iodine value of the product, but the oxidation stability did not improve.
- c. Sulphuric acid treatment of the product improved the oxidation stability but decreased the viscosity index considerably.
- d. The MoS<sub>2</sub> catalyst was less effective for hydrocracking than the Ni-catalyst.
- e. The topped residue of the product had a low flash point and a high viscosity. These results may be due to the formation of low molecular weight compounds mixed with those of high molecular weight, due to the non-uniform cracking of rubber.

2. Hydrocracking of Pale Crepe Rubber in the Presence of 2 Parts of Solvent

The hydrocracking of pale crepe rubber with 2 parts of various solvents at various temperatures was studied and the results shown in Table VIII(B)15 were observed.

From these results, the following conclusions may be made:

- a. The optimum conditions for the hydrocracking of rubber with 2 parts of solvent were temperature of 300°C and reaction time of 20 hours. The shorter the time, the worse the oxidation stability.
- b. It was found desirable to use a higher boiling solvent in preparing an oil of good oxidation stability.
- c. The type of solvent seemed to have a marked effect on the stability of the products.

3. Hydrocracking of Pale Crepe Rubber With Various Amounts of Solvent

Hydrocracking of pale crepe rubber with various amounts of solvent was studied and the results given in Table IX(B)15 were obtained. The following conclusions may be drawn:

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a. The optimum amount of solvent at the reaction temperature of 300°C was 5 parts of solvent to one part of rubber. The stability and the viscosity index of the product formed under these conditions showed the best results. It had a viscosity ratio of 2.23 in the oxidation test and a flash point of 134°C.

b. The number of aromatic nuclei in the product as determined by (1) bore a close relationship to the viscosity index, as may be seen in Table VIII(B)15 and IX(B)15. To obtain a viscosity index over 100, the number of aromatic nuclei in a molecule must be less than 4. To accomplish this, it is necessary to treat under 300°C, using a short time of reaction.

#### 4. Hydrocracking of Pale Crepe Rubber with 5 Parts of Various Solvents

The effect of various types of solvents such as decalin as a naphthenic compound, dodecane as a pure paraffinic hydrocarbon, and the various light oils were investigated, and the results shown in Table XI(B)15 were obtained (See Table X). The following conclusions may be made from these results:

a. In the case of paraffinic hydrocarbons, hydrocracking was difficult, but the reaction product had a low iodine value without decreasing the viscosity index at reaction temperatures above 350°C: that is, a highly stable lubricating oil was prepared by using this solvent.

b. In the case of the naphthenic solvent, the viscosity index of the reaction product was very low.

c. The higher the content of paraffinic components in the solvent, the higher the viscosity index and the better the oxidation stability of the product.

#### 5. Hydrocracking of Paste of Pale Crepe Rubber

For continuous operation of hydrocracking in a pilot plant, it is necessary to dissolve the rubber in a solvent to make a rubber paste. A pale crepe rubber was dissolved in a solvent at 170°C for 5-8 hours, and the hydrocracked product was compared with that obtained by direct hydrocracking. The results obtained are given in Table XI(B)15 and it was observed that this treatment (Exp. No. 51) yielded a product of lower viscosity index and better stability than the previous treatment (Exp. No. 31).

#### 6. Hydrocracking of Pale Crepe Rubber Paste Using Fischer Oil As A Solvent.

Pale crepe rubber and smoke sheet rubber were dissolved in Fischer oil boiling from 150°C to 250°C and the results shown in Table XII were obtained.

The following conclusions may be drawn:

a. The product of hydrocracking rubber at 300°C in the presence of Fischer oil has a high iodine value and poor oxidation stability.

b. The hydrocracking of smoke sheet rubber was more difficult than that of pale crepe rubber, but two stage hydrocracking for a period of long duration produced an oil of good stability from

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smoke sheet rubber.

**7 Summary of Autoclave Hydrocracking Experiments**

The optimum conditions for preparing a good aero-engine oil having high viscosity index and good stability from natural rubber by hydrocracking are summarized as follows:

- a. Solvent: Paraffinic solvent such as Fischer oil
- b. Volume of solvent: 5 parts to 1 part of rubber.
- c. Conditions for dissolving rubber in the solvent 170°C for 5-8 hours.
- d. Catalyst: Reduced nickel catalyst, consisting of 65% of NiO, 30% of diatomaceous earth, and 5% of graphite.
- e. CONDITIONS OF HYDROCRACKING

1st stage: 100 atmospheres of initial pressure, 300°C reaction temperature.

2nd stage: 300°C reaction temperature.

**8. Procedure for continuous hydrocracking of natural rubber paste in the pilot plant.**

20 parts of pale crepe rubber were dissolved in 100 parts of Fischer oil boiling from 200°C to 250°C at 170°C for 8 hours. This paste was hydrocracked in the continuous hydrocracking pilot plant shown in Figure 1(B)15, under the following conditions:

**Catalyst:** Nickel catalyst, consisting of 65% of NiO, 30% of diatomaceous earth and 5% of graphite, previously reduced in the reaction cylinder.

**Amount of Catalyst:** 3 liter of catalyst in both the reaction and preheating cylinders.

**Pressure of reaction:** 200 kg/cm<sup>2</sup>

**Temperature of preheating cylinder:** 300°C

The results of these experiments are summarized in Table XIII(B)15. From these results it was recognized, that the conditions of hydrocracking in experiment No. 7 were best. These conditions were as follows:

**Catalyst:** Nickel catalyst

**Temperature of preheating cylinder:** 300°C

**Temperature of reaction cylinder:** 400°C

**Pressure of reaction:** 200 kg/cm<sup>2</sup>

**Amount of charge:** 1.5 lit/hr to 3 lit of catalyst in each reaction and preheating cylinder.

**Amount of H<sub>2</sub> gas:** 1.5 m<sup>3</sup>/hr





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Table I(B)15  
SOLVENT EXTRACTION OF PALE CREPE RUBBER

Properties of product	Solvent	Acetone	Alcohol	Iso-octane	Ethyl Ether	Benzene	Residue of Extraction
		Yield of Extract (%)	2.78	3.24	9.32	15.66	28.38
	Mol wt	15,200	2,300	41,000	126,000	157,000	210,000
	Em	$3.8 \times 10^{-4}$	$3.8 \times 10^{-4}$	$1.1 \times 10^{-4}$	$1.1 \times 10^{-4}$	$1.7 \times 10^{-4}$	$3.0 \times 10^{-4}$
	Yodine Value	535.6	11.4	296.4	370.5	368.5	318
	Elementary Analysis (%)	C 75.60 H none	C 69.11 H none	C 86.60 H 0.02	C 86.60 H 0.01	C 86.45 H 0.01	C 86.15 H 0.8
	Analysis (%)	C 0.01 H 10.80	C 0.07 H 10.08	None H 11.88	C 0.01 H 11.98	C 0.01 H 12.06	C 0.02 H 11.86
		C 13.79	C 20.74	C 1.86	C 1.40	C 1.38	C 1.17

Table II(B)15  
THERMAL TREATMENT OF PALE CREPE RUBBER WITHOUT CATALYST

No. of Exp.	Conditions of Reaction		Properties of Product			Remarks
	Temp. (°C)	Time (hr)	Mol. wt	Iodine Value	$\bar{M}_n$	
1	150	20	72,000	368.9	$3.8 \times 10^{-4}$	used 4 volumes of solvent
2	200	20	26,700	324.6	$3.8 \times 10^{-4}$	used 4 volumes of solvent
3	200	30	18,100	315.2	$3.8 \times 10^{-4}$	used 4 volumes of solvent
4	300	10	3,000	310	$3.8 \times 10^{-4}$	used autoclave
5	300	4	816	146.9	$3.6 \times 10^{-4}$	used autoclave without solvent

Table III(B)15  
THERMAL TREATMENT OF PALE CREPE RUBBER WITH ACID CLAY

No. of Exp.	Conditions of Reaction		Properties of Products			Remarks	
	Temp. (°C)	Time (hr)	Amount of Acid Clay	Mol. wt	Iodine Value		$\bar{M}_n$
1	150	20	25	9,700	240	$3.8 \times 10^{-4}$	used 4 volumes of solvent
2	200	20	25	6,400	278	$3.6 \times 10^{-4}$	used 4 volumes of solvent
3	200	20	5	9,800	186	$0.4 \times 10^{-4}$	used 4 volumes of solvent
4	200	10	10	9,000	101.2	$0.4 \times 10^{-4}$	used 4 volumes of solvent
5	200	10	20	7,000	70.9	$0.4 \times 10^{-4}$	used 4 volumes of solvent
6	300	2	10	1,020	135.8	$0.4 \times 10^{-4}$	non solvent used autoclave
7	300	4	10	578	133.7	$0.4 \times 10^{-4}$	non solvent used autoclave

\*The reason for using the Standing's value of cyclic rubber molecule, i.e.  $0.4 \times 10^{-4}$ , for the constant  $\bar{M}_n$ , was because considerable cyclization of the rubber molecule caused by the action of acid clay, made it unfeasible to use the constant of rubber hydrocarbon as it is.

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Table IV(B)15  
POLYMERIZATION OF SIMPLE CRACKED DISTILLATES OF PALE CREPE RUBBER

No. of Exp.		1	2	3	4	5	6	7
Conditions of Reaction	Range of Fraction (°C)	100~150	150~200	200~250	250~300	100~250	150~200	150~250
	Catalyst	AlCl <sub>3</sub>	AlCl <sub>3</sub>	AlCl <sub>3</sub>	AlCl <sub>3</sub>	Acid Clay	Acid Clay	Synthetic Acid Clay
	Type Amount (%)	5	3	3	5	20	20	20
	Temp. (°C)	100	120	120	100	150	300	300
	Time (hr)	8	8	8	8	8	6	6
	Time of Product (%)	22	38	64	57.7	6.3	15.4	14.6
Properties of Product	Iodine Value	72.4	139.9	38.7	68.4	108.9	48.2	37.6
	Density (20/4°C)	0.9628	0.9693	0.9612	0.9542	0.9438	0.9607	0.9830
	Viscosity (S.U.S.)	2,269	9,303	2,745	2,884	7,123	3,095	610
		72.0	117.6	69.1	90.2	54.8	65.3	47.3
	Viscosity Index	-159.6	-269.2	-28.9	-66.8	-211.1	-406.6	-226.2
	Flash Point	135	102	145	171	185	176	174

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Table V(B)15  
POLYMERIZATION OF ISOPRENE FRACTION OF THE CRACKED DISTILLATE OF PALE CREPE RUBBER

No. of Exp.	25		26		24		20		23		29		31		32		34	
	Solvent (parts)*	1	1	1	1	1	none	none	none	none	none	none	none	none	none	none	none	none
Conditions of Reaction	Temp. (°C)	80	80	80	60	60	35	35	30	35	30	30	30	30	0	0	-10	-10
	Time (hr)	8	8	8	8	8	8	8	8	8	8	8	8	8	8	8	8	8
Catalyst	Type	AlCl <sub>3</sub>	AlCl <sub>3</sub> + Acid Clay	AlCl <sub>3</sub>	AlCl <sub>3</sub>	AlCl <sub>3</sub>	AlCl <sub>3</sub>	AlCl <sub>3</sub>	AlCl <sub>3</sub>	AlCl <sub>3</sub>	AlCl <sub>3</sub>	AlCl <sub>3</sub>	SnCl <sub>4</sub>	AlCl <sub>3</sub>	AlCl <sub>3</sub>	AlCl <sub>3</sub>	AlCl <sub>3</sub>	AlCl <sub>3</sub>
	Amount (g)	3	2+5	5	5	5	3	5	1	5	1	5	10	5	5	5	5	5
Yield of Product (g)		5.5	22.0	30.7	20.0	20.0	20.5	20.5	12.0	12.0	12.0	31.3	11.0	12.5	12.5	12.5	12.5	12.5
Mixing Ratio of Methyl Oil		77	47	42	none	none	20	20	none	none	none	none	30	80	80	80	80	80
Incline Value		30.1	28.4	32.0	40.6	41.0	42.3	42.3	56.5	58.4	58.4	58.4	58.4	58.0	58.0	58.0	58.0	58.0
Density (20/4°C)		0.9234	0.9274	0.9218	0.9216	0.9216	0.9226	0.9211	0.9216	0.9210	0.9210	0.9210	0.9210	0.9231	0.9231	0.9231	0.9231	0.9231
Viscosity (S.U.S.)		1,260	2,180	1,501	2,726	2,726	3,026	4,212	1,608	1,608	1,608	1,608	1,608	1,608	1,608	1,608	1,608	1,608
at 210°F		60.6	77.7	70.1	92.3	92.3	106.7	178.4	70.1	70.1	70.1	70.1	70.1	70.1	70.1	70.1	70.1	70.1
Viscosity Index		-110.6	-85.1	-54.3	-4.1	-4.1	0	77.6	72.7	72.7	72.7	72.7	72.7	72.7	72.7	72.7	72.7	72.7

\*Solvent is a South Borneo's light oil boiling from 190°C to 250°C.

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Table VI(B)15  
CO-POLYMERIZATION OF THE CRACKED DISTILLATE OF PALE CREPE  
RUBBER WITH THE CRACKED DISTILLATE OF PARAFFIN WAX

No. of Exp.		7	8	9	19	
Conditions of Reaction	Cracked Distillate of Pale Crepe Rubber (150~250°C) (%)	50	40	30	20	
	Cracked Distillate of Paraf- fin wax (150~250 C) (%)	50	60	70	80	
	Temperature (°C)	100	100	100	100	
	Time (hr)	8	8	8	8	
Yield of Product (%)		30	24	30	14.1	
Properties of Product	Iodine Value	19.2	16.4	15.2	14.1	
	Density (20/4°C)	0.9124	0.9063	0.8726	0.8789	
Product	Viscosity (S.U.S.)	at 100°F	888	1,971	1,072	1,206
		at 210°F	68.8	107.6	99.0	104.1
	Viscosity Index	38.4	64.1	106.7	104.3	
	Viscosity Ratio	2.94	2.42	1.77	1.20	

\*In all cases the catalyst was 5% AlCl<sub>3</sub> (wt)



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Table VIII(B)15  
HYDROCRACKING OF PALE CREEPY RUBBER WITH 2 PARTS OF SOLVENT

Quantity of Reaction	No. of Exp.		Temp. (°C)	20	21	22	23	24	25	26	27	28	29
	16	17											
Reaction	300	300	300	300	300	300	300	200	300	300	300	350	300
	5	15	20	20	20	30	4	20	6	4	7	4	12
Solvent (2 parts total)	Light oil from rubber	Light oil from rubber	Light oil from rubber	Light oil from rubber	Light oil from rubber	Light oil from rubber	Light oil from rubber	Gas oil (170-220°C)	Light oil from rubber	Light oil from rubber	Light oil from rubber	Light oil from rubber	Light oil from rubber
	2700 G.P.R. 55.3	2700 G.P.R. 86.0	2500 G.P.R. 60.1	2500 G.P.R. 83.3	2500 G.P.R. 83.3	2500 G.P.R. 75.0	2400 G.P.R. 72.0	2400 G.P.R. 72.0	3450 G.P.R. 33.0	3200 G.P.R. 39.3	3000 G.P.R. 55.3	3000 G.P.R. 55.3	3000 G.P.R. 55.3
TABLE of Products (%)	1.054	1.074	1.004	1.197	1.081	904	1.292	1.664	1.142	1.142	1.142	1.142	1.142
	7.046	2.660	4.009	2.081	3.256	2.744	2.744	7.312	5.921	5.921	5.921	5.921	5.921
Properties of	346	346	318	23.1	148.0	181.0	149.7	181.0	149.7	149.7	149.7	149.7	149.7
	80.3	77.3	92.1	83.3	72.2	73.3	73.3	22.7	25.1	25.1	25.1	25.1	25.1
Products	105.6	10.1	16.8	5.4	21.9	63.9	49.8	10.1	113.9	113.9	113.9	113.9	113.9
	(4.5)	2.96	2.60	6.40	5.8	5.8	3.1	(3.1)	(2.9)	(2.9)	(2.9)	(2.9)	(2.9)
Products	0.9057	0.8996	0.8912	0.8854	0.8989	0.9066	0.9233	0.9149	0.9381	0.9381	0.9381	0.9381	0.9381
	192	151	168	140	136	138	200	180	166	166	166	166	166
Products	-8	-14	-13	-18	-16	-12	-12	3.5	15	15	15	15	15
	1.6970	1.6970	1.4895	1.4895	1.4920	1.4920	1.4960	1.4960	1.5147	1.5147	1.5147	1.5147	1.5147
Products	5	5	5	5	5	5	6	11	9	9	9	9	9
	5	5	5	5	5	5	6	11	9	9	9	9	9

The light oil from rubber was a fraction of hydrogenated oil of rubber. Mixed as the catalyst and two parts of solvent were used in all cases. All light oil fractions were 150-V25000 fractions. The aromatic content was determined from the refractive index and molecular weight by E.I. Matarman's method of ring-analysis.





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Table X(B)15  
 HYDROCRACKING OF PALE CREPE RUBBER WITH 5 PARTS OF VARIOUS SOLVENTS

Conditions of Reaction	No. of Exp.		Solvent	Type	Decalin (188°C)	46	47	47 <sup>1</sup>	39	31	49
	Temp. (°C)	Time (hr)									
Yield of Product (%)	0	12	Decalin (188°C)	0	100	100	73	70.2	50	93.3	46.3
Iodine Value	63.6	67.1	12.1	86.8	15.5	46.4					
							Density (20/4°C)	0.9017	0.8966	0.8916	0.9167
Viscosity (S.P.S.) at 100°F	1,709	1,184	1,602	1,709	1,414	758					
							Viscosity (S.P.S.) at 210°F	75.4	110.5	123.8	101.2
Viscosity Index	-41.0	129.0	106.4	67.0	115.9	59.5					
							Viscosity Ratio	3.36	15.4	1.71	5.25
Flash Point (°C)	172	95	172	135	134	172					
							Solvent	300	300	300	300
Type	15	15	15	15	15	15					
							Decalin (215°C)	Dodecane (215°C)	Dodecane (215°C)	South Smarale light oil (150-250°C)	Light oil from rubber (150-250°C)

In all cases, nickel was used as the catalyst and 5 parts of solvent were used. The components of the light oils were determined by Maternan's method.

ENCLOSURE (B)15

Table II(B)15  
 HYDROCRACKING OF RUBBER PASTE AND NON-PASTE RUBBER

No. of Exp.*		31	51	51'
Yield of Product (%)		93.3	57.5	
Properties of Product	Iodine Value	15.5	4.3	4.9
	Density (20/4°C)	0.8779	0.8724	0.8777
	Viscosity at 100°F	1.414	1.677	1.636
	(S.U.S.) at 210°F	129.2	125.9	119.3
	Viscosity Index	115.9	103.1	98.6
	Viscosity Ratio	2.23	2.03	1.33
	Flash Point (°C)	134	124	166
Remarks		non-paste	paste	16% of spindle oil was mixed to the product of No. 51

\*Reaction conditions were: Temp., 300°C;  
 Time, 15 hr; Solvent, 5 parts of light  
 oil from scrubber; catalyst, nickel

ENCLOSURE (B) 15

Table XII(B)15  
HYDROCRACKING OF RUBBER PASTE WITH FISCHER OIL AS SOLVENT

No. of Exp.	Raw Material	52	53	54	55	56	57	58
		pale craps	pale craps	smoke sheet	smoke sheet	smoke sheet	smoke sheet	smoke sheet
Conditions of Reaction		300	300 350	300	300 350	300 350	300 350	300 350
Temp. (°C)		15	15 3	15	15 4	8 7	8 7	8 10
Time (hr)			80.1	63.0	70.0	75.6	81.2	80.0
Yield of Product (%)		86.5	76.0	92.1	45.1	3.6	1.94	1.22
London Value		36.0	38.4 <sup>a</sup>	0.8903	0.8777	0.8521	0.8421	0.8101
Density (20/20°C)		0.8636	0.8658	1.071	1.252	1.520	1.746	1.759
Viscosity at 20°C		1.122	1.053 <sup>b</sup>	133	100.3	111.5	140.1	108.1
(S.S.A.) at 20°C		131.2	111.9 <sup>c</sup>	101.3	100.3	95.3	130.3	111.5
Viscosity Index		125.6	120.5 <sup>d</sup>	127.6	8.43	1.80	1.72	1.67
Viscosity Ratio		34.2	2.54 <sup>e</sup>	1.50	25.4			
Carbonyl Carbon			0.006	0.03				
Carbonyl Carbon After Oxidation Test			0.7					

<sup>a</sup>Based on the characteristics of the mixture of the product from rubber (75%) and Fisher catalysts (43.53%).

<sup>b</sup>20°C values reported; Fisher oil solvent and nickel catalyst were used.

ENCLOSURE (B)

Table XIII(B)15  
CONTINUOUS HYDROCRACKING OF NATURAL RUBBER PASTE IN THE PILOT PLANT

	1	2	3	4	5	6	7	8	9
No. of Run									
Condition of Reaction	375	375	375	375	375	400	400	400	400
Amount of Charge (lb/hr)	1.0	1.0	1.5	1.0	1.5	1.0	1.5	1.0	1.5
Amount of H <sub>2</sub> (cu ft/hr)	1.0	1.0	1.5	1.5	1.5	1.0	1.5	1.5	1.5
Yield of Product from Rubber (S)	11.7	39.0	11.9	58.7	52.6	26.9	11.8	40.0	21.7
Yield of Product from Rubber (S)	0.857	0.879	0.857	0.864	0.897	0.876	0.8472	0.8519	0.9238
Properties of Feedstock									
Flash Point (°C)	210	211	205	228	221	213	177	218	200
Viscosity at 200°F (cP)	47,995	7,935		3,463	7,246	1,898	2,236	1,712	3,927
Viscosity at 210°F (cP)	2319	543	264	268	428	171.7	377.0	341.1	288
Viscosity at 210°F (cP)				122.5		120.5	120.9	108.1	118.4
Proportion of Feedstock									
Feed Point (%)	5	9	11	9	4	15	12	13	8
Proportion of Product									
Feed Point (%)	0.49	0.98	1.29	1.30	1.35	1.47	1.20	1.25	1.23
Composition of Carbon (S)	0.13	0.15	0.16	0.15	0.11	0.15	0.17	0.16	0.15

More figures in line.

ENCLOSURE (B) 35

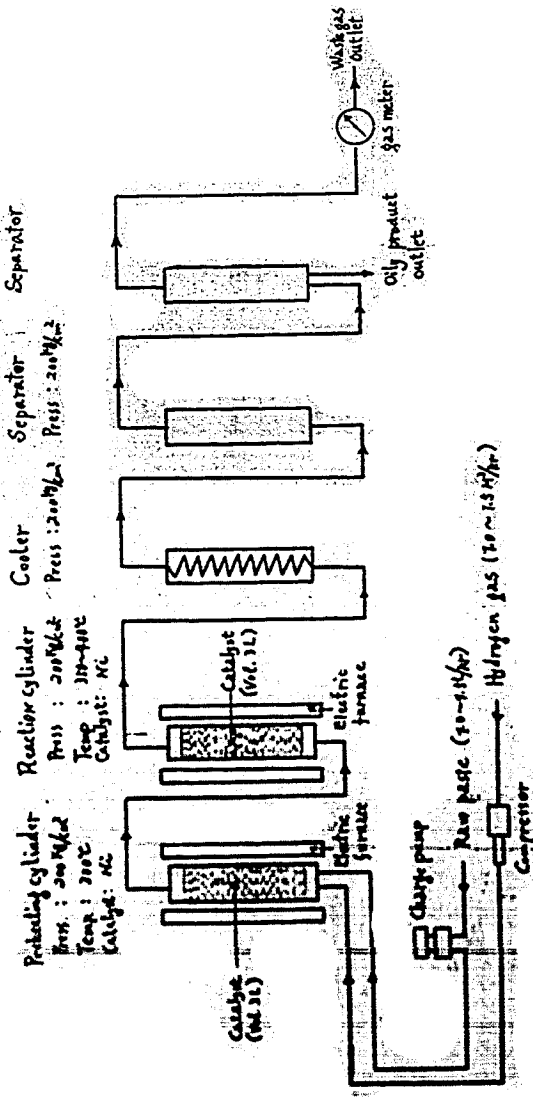


Figure 1(B) 15  
CONTINUOUS HYDROGENATING APPARATUS FOR NATURAL RUBBER PASTE