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ENCLOSURE (B) 36

R E S E A R C H O N L U B R I C A T I N G
S P E C I A L G R E A S E S
(In Three Parts)

by

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Research Period: 1938-1945

Prepared for and Reviewed with Authors by
the U. S. Naval Technical Mission to Japan

December 1945

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PART I
SPECIAL GREASES

by

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SUMMARY

The results of researches on manufacturing imported special greases and other greases for special uses led to the conclusion that these special greases could be made by using the proper metallic soaps and heating them together with suitable refined mineral lubricating oils.

I. INTRODUCTION

Special high class lubricating greases for aviation engines and its auxiliary parts, i.e. magneto grease, controllable-pitch propeller grease and rocker arm grease, were chiefly imported from U.S.A., and in Japan, there was practically no research on these greases. The authors, therefore, carried on researches on the components and methods of manufacture for these greases from 1938 to 1943 and obtained various ones.

In 1943 a special rust-proof grease was needed for the framework of the aero-torpedo engine, and another grease was needed as a better anticorrosive material for the interior of the compressed air chambers of the aero-torpedo because of the lack of satisfactory ones at that time. Research led to success in establishing a suitable method of manufacture.

In 1944 a sea water-proof grease for submarines was investigated and a superior one was developed and found very satisfactory by practical test, particularly in regard to resistance to removal by water washing.

In 1944 the authors carried these researches to the pilot plant stage and manufactured these greases which had been studied in the laboratory.

II. DETAILED DESCRIPTION

A. Analytical Methods and Tests

Chemical laboratory tests are classified under two headings and were carried out in the following manner.

1. Control Test.

   a. Percent water..................A. S. T. M. D95 - 30

b. Corrosion test. Place a clean strip of polished pure sheet copper about 1/2 inch wide and 3 inch long in a clean test tube. Add enough of the sample to be tested to cover the strip completely. Maintain for 3 hours at 100°C. Rinse the copper strip with sulfur-free benzene and compare it with a similar strip of freshly polished copper. Discoloration or pitting indicates corrosion.

c. Saponification value........A. S. T. M. D94 -28

d. Percent free alkali and free acid...............A. S. T. M. D128-27

2. Quality Test

a. Grease analysis.............A. S. T. M. D128-27

b. Percent fillers.............A. S. T. M. D128-27

c. Percent mineral oil.........A. S. T. M. D128-27

d. Mineral oil tests...........A. S. T. M. D128-27

e. Percent soap................A. S. T. M. D128-27

f. Kind of soap................A. S. T. M. D128-27

g. Total mixed fatty acids.....A. S. T. M. D128-27

Physical laboratory tests may be similarly classified.

1. Control Test

a. Consistency test at 25°C...A. S. T. M. D217-33T

b. Dropping point.................Ubbelohde method

c. Saybolt viscosity

2. Quality Test

Stability to heat: 20 to 25 gm of grease were heated to 100 to 105°C and held at that temperature for 3 hours. The specification is that after 24 hours standing no oil should separate.

B. Experimental Results

1. Magneto Grease. For an aero-motor magneto grease, "Bosch Magneto grease" imported from U.S.A. was generally used. Analysis of this grease showed the main composition was 26.8% of stearic and oleic acid sodium soap and 68.9% of low viscosity refined lubricating oil having a vis. of 43.2 S. U. S. at 210°F, vis. index, 109.

As a result of the experiments for manufacturing grease of the same property as the "Bosch Magneto grease", two trial samples were made.

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One was composed of 20% of the sodium soap of castor oil (whole oil and not ricinoleic acid alone) heated together with 80% of turbine oil (sample No. 1, Table II(B)36); the other was heated with phenol refined oil (sample No. 2 Table II(B)36) at the temperature of 170°C to 180°C. After milling, with a roller type mill, these greases were comparable in general properties to "Bosch grease" and they were found very suitable for magnetos by engine tests at the First Naval Aeronautical Depot.

In this study, it was recognized that greases made from sodium stearate and sodium oleate soaps heated together with turbine oil are of fibrous texture. Therefore, they are not suitable for high speed ball-bearing greases.

The use of the sodium soap of castor oil plus milling led to success in obtaining a suitable smooth texture as possessed by "Bosch grease".

Next, sample No. 3, Table II(B)36 was made from 22.3% of the soda soap of castor oil with an excess of soda (1.33 times the theoretical saponification value) heated together with 68.7% of refrigerating machine oil at the temperature of 230°C. This grease was quickly cooled to prevent the grease from crystallizing out soap.

This sample possessed a higher dropping point and a better heat stability than any other sample. The data for "Bosch grease" and three samples are shown in the Table II(B)36.

2. <u>Controllable-Pitch Propeller Grease</u>. Up to 1940 "Mobile Grease" No. 2, produced by Standard Vacuum Oil Co., was used for controllable-pitch propeller. Analysis of this grease indicated a composition of 7.57% of aluminum soaps of palmitic, stearic and oleic acid, and 89.73% of high viscosity refined mineral oil (vis. of 131.5 S. U. S. at 210°F, vis. index 69). As the result of experiments on manufacturing a grease equal to this "Mobile Grease" No. 2, the grease sample was made by mixing 6.48% of aluminum stearate, 0.72% of aluminum oleate, 0.80% of lead oleate, 0.40% of glycerine and 91.6% of mineral lubricating oil (vis. S. U. S. at 210°F 133.4, vis. index 87.5) at the temperature of 150°C. It was confirmed by tests on coefficient of friction, and engine tests at the First Naval Aeronautical Depot that this sample of grease was as applicable to controllable-pitch propellers as "Mobile Grease" No 2. The data for Standard Vacuum Oil Co. Mobile Grease No. 2 and experimental grease are shown in Table III(B)36. Determination of Coefficient of Friction.

The Kinetic coefficients of friction of sample greases were determinted at various temperatures by means of a Bearing Type Testing apparatus described in detail in the report. "Studies on the Additives of the Submarine Diesel Engine Lubricant" by Eng. Capt. Dr. I. KAGEHIRA and Chem. Eng. Lt. Comdr. N. HIRATA.

The schematic view of the test apparatus is shown in Figure 1(B)36. The diameter of the test shaft was 3cm, and the length of the line of contact was 2cm.

The test piece and the test shaft were polished with 04 emery paper and washed with well refined gasoline.

The sample grease is charged in the oil cup (C) and then maintained at constant temperature by electric heating. A constant weight (F) is loaded on the test piece (A) and then the test shaft (B) is rotated under various rubbing speeds. Frictional resistance at various rubbing speeds is determined by the weight of balance (E) and frictional resistance divided by load (F) gives a value from which the coefficient of friction of motion may be calculated. The plots of the data are shown in Figure 2(B)36-a to Figure 2(B)36-b.

From these data it was shown that experimental grease No. 1 containing lead oleate matched Standard Vacuum Mobile grease No. 2 in lubricating value.

Experimental greases tested for determination of coefficient of friction had compositions and properties shown in Table I(B)36.

3. Rocker Arm Grease. Up to 1941 "Super Gear Lubricant" made in U.S.A. by the Vacuum Oil Co. was used as aviation rocker arm greases. Analysis of this grease showed the following composition: 4.4% of calcium soap of stearic and oleic acid, 95.7% of heavy lubricating oil (vis. S. U. S. at 210°F, 108, vis. index 89.2)

As the result of experiments, sample No. 1, Table IV(B)36, was prepared by the following treatment. Sodium oleate (4.55%), 2.45% of sodium stearate, 0.70% of glycerine and 92.30% of heavy refined lubricating oil (vis. S. U. S. at 210°F, 100.8, vis. index 77) were heated together at the temperature of 150°C. This experimental grease was analogous in general properties and was indicated to be as serviceable as "Super Gear Lubricant" by the engine tests at the First Naval Aeronautical Depot.

Sample No. 2, Table IV(B)36, consisted of 3.0% of sodium stearate, 3.0% of sodium oleate, 1.0% of calcium stearate, 1.0% of calcium oleate, 0.4% of glycerine and 91.2% of heavy refined lubricating oil (vis. S. U. S. at 210°F, 95, vis. index 67), was made to possess a good water-proof character and by engine test it was proved to give good service.

Compositions and properties of "Super Gear Lubricant" and two experimental greases are shown in Table IV(B)36.

4. Special Grease for the Framework of the Aero-Torpedo. For the framework grease of the main engine of the aero-torpedo, the experimental grease was prepared as follows: 7% of aluminum stearate, 0.21% of calcium stearate and 92.7% of cylinder oil for aero-torpedo (vis. S. U. S. at 210°F, 61.9, vis. index 85.3, Setting point (-)36°C) were heated together at a temperature of 150°C. The role of calcium stearate in the oil was to liquify and semi-plastify the aluminum stearate.

By engine test this experimental grease was proved to be excellent for the prevention of the corrosion of engine parts. Its composition and general properties are shown in Table V(B)36.

5. Special Grease for Preventing Corrosion of the Interior of Compressed Air Chamber of Aero-Torpedoes. Up to 1943, as an anti-corrosive for the interior of the compressed air chamber of the aero-torpedo, a heavy cylinder oil was used. However, it was unsatisfactory as an anticorrosive compound from the standpoint of adhesiveness and antifreezing character.

To improve these points the following grease was prepared, applied to practical tests and proved to be excellent, having very good adhesive and anticorrosive properties.

Aluminum stearate (12%), 0.36% of calcium stearate, and 87.64% of cylinder lubricating oil were heated together at a temperature of 150°C.

In each case the grease was milled three to four times in a roll type mill. Composition and general properties of this grease are shown in Table VI(B)36.

6. Sea Water Proof Greases (Anti-Corrosive and Anti-Wash). Sea Water Proof Greases to be used for the machine-gun mounted on submarines were studied and the following experimental greases No. 1 to No. 6 were prepared and subjected to practical tests at the Yokosuka Naval Arsenal.

Their compositions are shown in Table VII(B)36, sample No. 5, which consisted of 13% of aluminum stearate and 87% of heavy lubricating oil, was most suitable by the laboratory and practical tests. The composition and general properties of the grease are shown in Table VIII(B)36.

7. Summary of Laboratory Data. The results of researches are summarized in Table IX(B)36 and X(B)36, which show the best conditions for the preparation of each grease.

C. Detailed Description of Pilot Plant

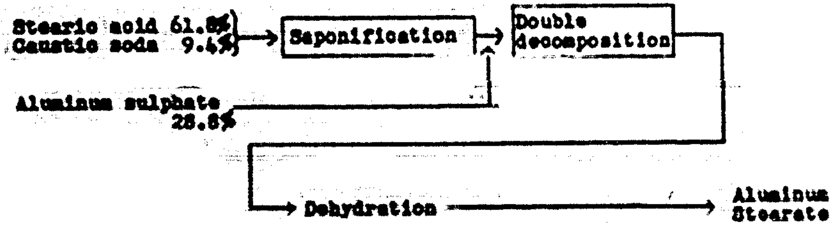
1. Description of Pilot Plant (erected February 1944)

a. Main equipment. This is shown in Table XI(B)36.

b. Flow sheets of pilot plant. These are shown in Plate I(B)36.

c. Process flow sheets of pilot units

Preparation of Metallic Soaps



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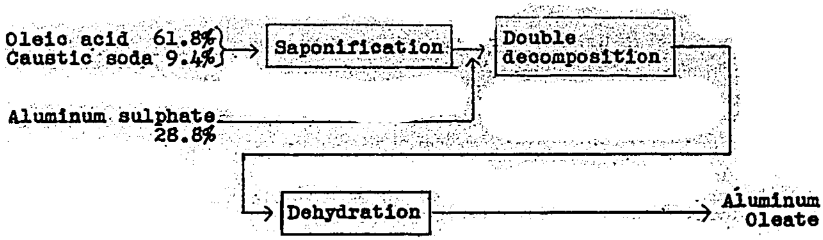
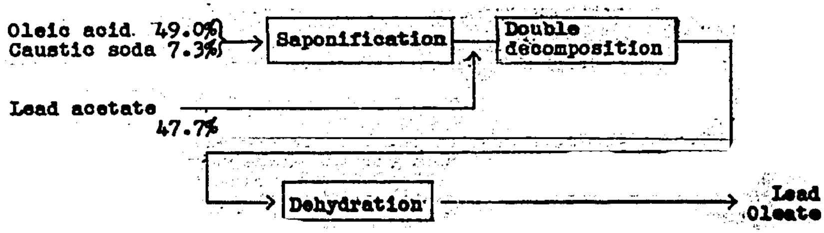
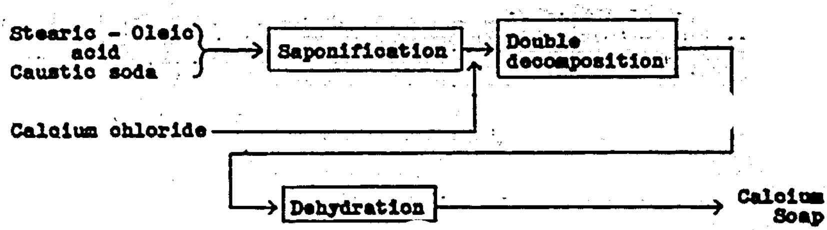
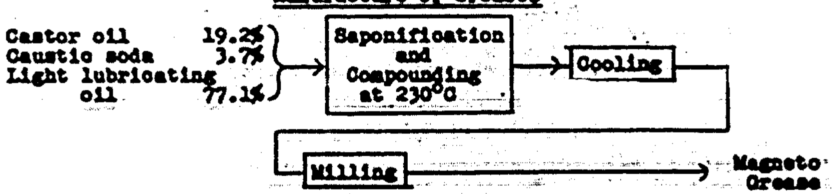






Manufacture of Greases



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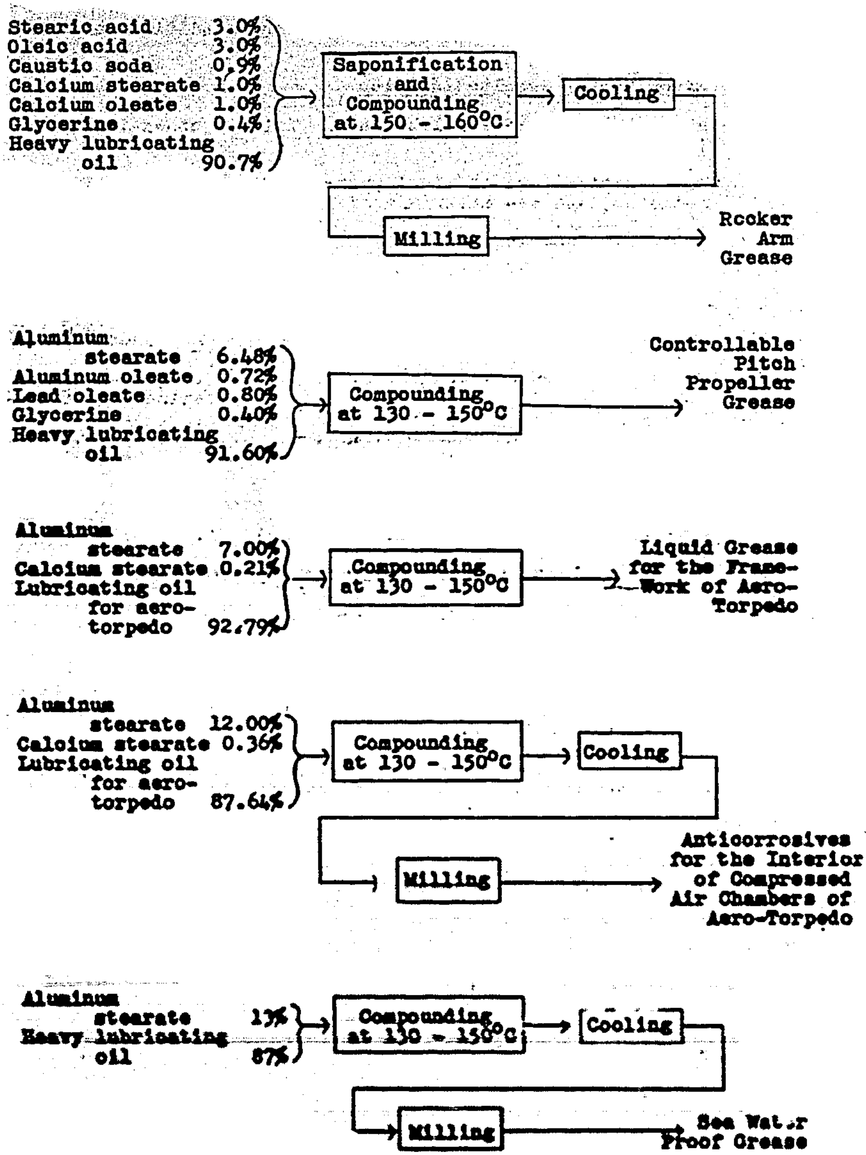

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2. Procedures. Details of procedures and conditions are shown in Table XII(B)36.

3. Experimental Results in Pilot

a. Yield and material balance. This is shown in Table XIII(B)36.

b. Physical and chemical properties of products, intermediates, and raw materials of feed stocks.

(1) Chemicals

Caustic soda.................................solid

Aluminum sulphate................$Al_2(SO_4)_3 \cdot 24H_2O$, Chemically pure

Lead acetate.....................$Pb(C_2H_3O_2)_2 \cdot H_2O$, Chemically extra pure

Calcium chloride.....................$CaCl_2 \cdot 2H_2O$, Chemically pure

Glycerine.............................98% pure

(2) Lubricating oils: see Table XIV(B)36.

(3) Soap stocks: see Table XV(B)36.

(4) Intermediates: see Table XVI(B)36.

(5) Products: see Table XVII(B)36.

D. Summary of Data in Pilot Plant. Summary of data in pilot plant and comparison of general properties of greases obtained in pilot plant and laboratory are shown in Table XVIII(B)36.

These data proved that the greases manufactured in the pilot plant have very nearly the same properties as those prepared in laboratory.

III. CONCLUSIONS

The results of research on the manufacture of special greases, i.e. Magneto grease, Controllable-Pitch Propeller grease, Rocker Arm grease and other special grease for the Framework of Aero-Torpedo, Anticorrosives for the Interior of Compressed Air Chamber of Aero-Torpedo and Sea-Water-Proof grease, were successful in establishing a suitable method for the preparation of each grease.

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Table I(B)36
COMPOSITIONS AND PROPERTIES OF GREASES
APPLIED TO THE FRICTION TEST

		Experimental Greases	
		No. 1	No. 2
Composition	Aluminum Stearate	6.1	6.56
	Aluminum Oleate	0.7	0.94
	Lead Oleate	0.7	absence
	Glycerine	0.38	0.38
	Mineral Oil	92.12	92.12
Properties of mineral oil	Flash Point 9C	268	
	Viscosity S. U. S. (at 210°F)	133.4	
	Viscosity Index	87.5	
	Carbon Residue (%)	0.4	
General properties	Appearance	Viscous, stringy	Viscous, stringy
	Dropping Point (°C)	86	84
	Consistency at 25°C	385	390
	Free Fatty Acid (%)	0.5	0.2
	Free Fatty Oil (%)	none	none
	Ash (%)	0.44	0.50
	Water (%)	trace	trace
	Corrosion	O. K.	O. K.
	Stability (100°C 3 hrs)	no oil bleeding	no oil bleeding

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Table II(B)36
COMPARISON OF SAMPLE GREASES
WITH BOSCH MAGNETO GREASE

Compositions

	Mineral Oil (%)	Soap (%)	Water (%)	Ash (%)	Free Alkali (%)	Free Fatty Acid (%)	Free Fatty Oil (%)
Bosch magneto grease	68.9	26.8	0.5	5.76	0	5.5	
Sample grease No. 1	79.4	20.5	trace		0.05	0	0.11
Sample grease No. 2	79.4	20.5	trace	2.5	0.095	0	0.34
Sample grease No. 3	68.7	22.3	0.5	3.44	0.12	0	

Properties of Mineral Oil

	F.P.(°C) **	Viscosity 210°F	Viscosity 100°F	Vis. Index	Viscosity# (Redwood Sec.)	Setting Pt. (°C)	C.R.(%) ##
Bosch magneto grease		43.2	144	109.2	176	-39	0.75
Sample grease No. 1	199	51.8	407.8	38	578	- 6	0.015
Sample grease No. 2	173	50.5	270.0	96	350	-14	0.010
Sample grease No. 3	155				130.8	-30	0.020

General Properties

	Appearance	Consistency* (-) 20°C	Consistency* (+) 25°C	Dropping Pt.	Corrosion	Stability (100°C 3 hrs.)
Bosch magneto grease	Smooth Texture		256	165	O.K.	no oil bleeding
Sample grease No. 1	Smooth Texture		200	165	O.K.	no oil bleeding
Sample grease No. 2	Smooth Texture		255	165	O.K.	no oil bleeding
Sample grease No. 3	Smooth Texture	180	254	175	O.K.	no oil bleeding

Soap Fatty Acid

	Acid Value	Sap. Value	Iodine Value
Bosch magneto grease	188.9	187.0	59.6
Sample grease No. 1		184.4	63.2
Sample grease No. 2		184.4	63.2
Sample grease No. 3	182.6	192.9	84.6

*Worked Penetration
**Flash Point
#Conradson Carbon Residue
##At 30°C

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Table III(B)36
COMPARISON OF EXPERIMENTAL GREASE
WITH STANDARD OIL CO. GREASE

	Compositions						
	Mineral Oil %	Soap %	Water %	Ash %	Free Alkali %	Free Fatty Acid %	Free Fatty Oil %
Standard Vacuum Oil Co. Mobile Grease No. 2	89.73	7.57	0.13	0.92	0	1.03	0.10
Experimental Grease	91.60	8.00	trace	1.08	0	0.67	0

	Properties				
	Appearance	Consistency* at 10°C	Dropping Pt. °C	Corrosion	Stability (100°C 3 hrs.)
Standard Vacuum Oil Co. Mobile Grease No. 2	Stringy Viscous Mobile	360	63.	O.K.	no oil bleeding
Experimental Grease	Stringy Viscous Mobile	347	40	O.K.	no oil bleeding

	Mineral Oil				
	F.P.(°C)	Viscosity (S.U.S.)	V. I.	S. P. (°C)**	C. R. (%)***
Standard Vacuum Oil Co. Mobile Grease No. 2		131.5	69.	2.5	1.2
Experimental Grease	268	133.4	87.5	(-)15.5	0.4

	Soap Fatty Acid			
	M. P. (°C)	Acid Value	Sap. Value	Iod. Value
Standard Vacuum Oil Co. Mobile Grease No. 2	52.5	175.2	206.9	10.4
Experimental Grease	Mixture of Stearic and Oleic Acid			

*Worked Penetration A.S.T.M.
**Solidification Point
***Conradson Carbon Residue

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Table IV(B)36
COMPARISON OF EXPERIMENTAL GREASES
WITH SUPER GEAR LUBRICANT

Compositions

	Mineral Oil(%)	Soap (%)	Water (%)	Ash (%)	Free Alkali (%)	Free Fatty Acid(%)	Free Fatty Oil(%)
Super Gear Lubricant	95.7	4.4	0.30	0.60	none	0.25	0.37
Experimental grease No. 1	92.3	7.0	0.02	1.24	0.006	none	none
Experimental grease No. 2	91.2	7.49	0.20	1.50	none	0.31	none

General Properties

	Appearance	Consistency* at 25°C	Dropping Pt.(°C)	Corrosion	Stability (100°C, 3 hrs)
Super Gear Lubricant	Semi Fluid Smooth	363	85	O. K.	Oil Bleeding, Soap-Separate
Experimental grease No. 1	Semi Fluid Smooth	370	65	O. K.	No Oil Bleeding
Experimental grease No. 2	Semi Fluid Smooth	360	119	O. K.	No Oil Bleeding

	Mineral Oil					Soap Fatty Acid			
	F. P.(°C)	Viscosity** S.U.S.	V. I.	C. R. (%)	S. P. (°C)	M. Pt. (°C)	Acid Value	Sap. Value	Iod. Value
Super Gear Lubricant		108	89.2	1.7	2.5	40-42.5	203.5	208	46.4
Experimental grease No. 1	230	100.8	77.1	0.86	(-)15	49-49.5	193.6	193.6	50.7
Experimental grease No. 2	218	95.0	67.3	2.01	(-)18	55	198	202.6	44

*Worked Penetration A.S.T.M. **At 210°F.

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Table V(B)36
AERO-TORPEDO FRAMEWORK GREASE

	Compositions						
	Mineral Oil (%)	Soap (%)	Water (%)	Ash (%)	Free Alkali (%)	Free Fatty Acid(%)	Free Fatty Oil(%)
Experimental grease for framework of aero-torpedo	92.79	7.21	0.05	0.50	none	0.50	none

	General Properties				
	Appearance	Consistency* (-) 40°C	Viscosity (S.U.S.)	Corrosion	Stability (100°C 3 hrs)
Experimental grease for framework of aero-torpedo	Liquid	284	1036	O. K.	no oil bleeding

	Mineral Oil (Base)					Soap Fatty Acid			
	F.Pt.(°C)	Viscosity** (S.U.S.)	V. I.	S. Pt. (°C)	C. R. (%)	M. Pt. (°C)	Acid Value	Sap. Value	Iod. Value
Experimental grease for framework of aero-torpedo	203	61.9	85.3	(-)36	0.36	65-67	202	206	0

*Worked Penetration A.S.T.M. **At 210°F

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Table VI(B)36
COMPRESSED AIR CHAMBER GREASE

	Compositions						
	Mineral Oil (%)	Soap (%)	Water (%)	Ash (%)	Free Alkali (%)	Free Fatty Acid (%)	Free Fatty Oil (%)
Experimental Grease for Interior of Compressed Air Chamber of Aero-Torpedo	87.64	12.36	0.04	0.80	none	0.50	none

	General Properties				
	Appearance	Consistency* at 25°C	Dropping Point (°C)	Corrosion	Stability (100°C, 3 hrs)
Experimental Grease for Interior of Compressed Air Chamber of Aero-Torpedo	Smooth	326	90	O. K.	No oil bleeding

	Mineral Oil					Soap Fatty Acid			
	F. P.(°C)	Viscosity** (S.U.S.)	V. I.	S. Pt. (°C)	C. R. (%)	M. Pt. (°C)	Acid Value	Sap. Value	Iod. Value
Experimental Grease for Interior of Compressed Air Chamber of Aero-Torpedo	203	61.9	85.3	(-)36	0.36	65-67	202	206	0

*Worked Penetration A.S.T.M. **At 210°F

Table VII(B)36
COMPOSITION OF EXPERIMENTAL GREASES

No. 1	No. 2	No. 3	No. 4	No. 5	No. 6
Calcium Stearate 10%	Calcium Stearate 9.7%	Calcium Stearate 9.7%	Calcium Stearate 11%	Aluminum Stearate 13%	Zinc Stearate 20%
Calcium Oleate 10%	Calcium Oleate 9.7%	Calcium Oleate 9.7%	Calcium Oleate 11%	Heavy Lubricating Oil 87%	Turbine Oil 80%
	Zinc Powder 2.0%	Zinc Stearate 1.0%	Heavy Lubricating Oil 28%		
Turbine Oil 80%	Turbine Oil 78.6%	Zinc Oleate 1.0%			
		Turbine Oil 78.6%			

Table VIII(B)36
SEA WATER-PROOF GREASE

	Compositions						
	Mineral Oil (%)	Soap (%)	Water (%)	Ash (%)	Free Alkali (%)	Free Fatty Acid (%)	Free Fatty Oil (%)
Experimental Grease No. 5 for Sea Water-Proof	87	13	trace	0.94	none	0.5	none

	General Properties				
	Appearance	Consistency* at 25°C	Dropping Pt. (°C)	Corrosion	Stability (100°C 3 hrs.)
Experimental Grease No. 5 for Sea Water-Proof	Adhesive, Smooth	310	88.3	O.K.	no oil bleeding

	Mineral Oil				Soap Fatty Acid		
	F. P. (°C)	Viscosity 210°F (S.U.S)	V. I.	C. R. (%)	M. P. (°C)	Sap. Value	Iod. Value
Experimental Grease No. 5 for Sea Water-Proof	270	141	15	5.0		193	3.1

*Worked Penetration, A.S.T.M.

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Table IX(B)36
VARIOUS SPECIAL GREASES

Name of Greases	Components		Conditions of Mixing or Compounding	Milling	Uses
Experimental grease for magneto	Castor oil sodium soap Light lubricating oil	22.3% 64.7%	Saponification and Compounding at 230°C	Milling	(1) For magnetos, generators and motors etc. (2) For back sliding gear box of V.D.M. type Controllable-pitch propeller.
Experimental grease for controllable-pitch propeller	Aluminum stearate Aluminum oleate Lead oleate Glycerine Heavy lubricating oil	6.18% 0.72% 0.80% 0.40% 91.60%	Compounding at 150°C	No	(1) For controllable pitch propeller of Hamilton-Type.
Experimental grease for rocker arms	Sodium stearate Sodium oleate Glycerine Calcium stearate Calcium oleate Heavy lubricating oil	3.0 % 3.0 % 0.4 % 1.0 % 1.0 % 91.2 %	Saponification and Compounding at 150-160°C	Milling	(1) For rocker box of aero-engine. (2) Will be useful for the lubrication of gears and valves which operate at high temperature.
Experimental grease for framework of aero-torpedo	Aluminum stearate Calcium stearate Cylinder lubricating oil for aero-torpedo	7.0 % 0.21% 92.79%	Compounding at 150°C	No	(1) For the anti-corrosives of engine parts.
Experimental anti-corrosive grease for interior of compressed air chamber of aero-torpedo	Aluminum stearate Calcium stearate Cylinder lubricating oil for aero-torpedo	12.0 % 0.36% 87.64%	Compounding at 150°C	Milling	(1) For the anti-corrosives of the interior of compressed air chamber of aero-torpedo.*
Experimental water-proof grease resistant sea water	Aluminum stearate Heavy lubricating oil	13.0 % 87.0 %	Compounding at 150°C	Milling	(1) For the lubrication and anti-corrosive of sliding parts of the machine gun mounted on submarines.

*For the lubricating of sliding parts of the periscope mounted on submarines.

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Table X(B)36
COMPOSITIONS AND PROPERTIES
OF SPECIAL GREASES

	Compositions								General Properties**			
	Mineral Oil(%)	Soap (%)	Water (%)	Ash (%)	Free Alkali (%)	Free Fatty Acid(%)	Free Fatty Oil(%)	Appearance	Consistency*	At 25°C	Viscosity 210°F (S.U.S.)	Dropping Pt. (°C)
Experimental Grease for magneto	68.7	22.3	5.5	3.44	0.12	0.0		Smooth texture	(At-20°C) 180	254		175
For controllable-pitch propeller	91.6	8.0	trace	1.08	0.00	0.67	none	Stringy viscous mobile		(At 10°C) 347		40
For rudder arms	91.2	7.49	0.20		0.00	0.31		Semi Fluid smooth		360		119
For framework of aero-torpedo	92.79	7.21	0.05	0.59	none		none	Liquid	(At-40°C) 284	Too fluid	1,036	
As anticorrosive for interior aero-torpedo	87.64	12.36	0.04	0.80	none		none	Smooth		326		90
For water-proofing against sea water	87.00	13.00	trace	0.94	none		none	Adhesive smooth		310		88

	Properties of Mineral Oil						Characteristics of Soap Fatty Acid			
	Flash Point(°C)	Vis(S.U.S.) 210°F	Vis. Index	Vis(Redwood sec.)	Setting Point(°C)	C. R. (%)	Melting Point(°C)	Acid Value	Sap. Value	Iod. Value
Experimental Grease for magneto	155			130.8	(-)30	0.02		182.6	192.9	84.6
For controllable-pitch propeller	268	133.4	87.5		(-)15.5	0.40				
For rudder arms	218	95.0	67.3		(-)18.0	2.01	55	198	202.6	44
For framework of aero-torpedo	203	61.9	85.3		(-)36	0.36	65-67	202	206	0
As anticorrosive for interior aero-torpedo	203	61.9	85.3		(-)36	0.36	65-67	202	206	0
For water proofing against sea water	270	141	15		(+)1	5.0		193	193	3.1

* Worked Penetration, A.S.T.M.
** All these greases were non-corrosive and in the stability test there was no oil bleeding (100°C, 3 hrs).

ENCLOSURE (B)36

Table XI(B)36
EQUIPMENT FOR GREASE MANUFACTURE

	Name of Equipment	Set	Remarks	Capacity
Part I				
For Metallic Soaps	Dissolving tank	2	Enameled ironware with stirring mechanism, steam coil heating	$1,000m^3$
	Saponification vessel	3	Enameled ironware with stirring mechanism, jacket steam heating	$1,000m^3$
	Double-decomposition vessel	4	Enameled ironware with stirring mechanism, steam jacket heating	$1,500m^3$
	Double-decomposition vessel	2	Enameled ironware with stirring mechanism, steam jacket heating	
	Centrifuge	5		
	Steam heated drier	4		
	Alkali tank	1		
	Oleic acid tank	1		
Part II				
	Name of Equipment	Set	Remarks	Capacity
Grease Kettles	Indirect-steam heated kettle	3	Steel with double-motion agitator	$1,500m^3$
	Indirect-electrically heated kettle	3	Steel with double-motion agitator	$500m^3$
	Measuring tank	3	Enameled ironware	$1,000m^3$
Part III				
	Name of Equipment	Set	Remarks	Capacity
Grease Milling	Three roller mixing machine	3		
Part IV				
	Name of Equipment	Set	Remarks	Capacity
Others	Transformer	3		
	Mineral oil storage tank	2		$10,000m^3$
	Mineral oil storage tank	1		$5,000m^3$
	Pump	6		

		Raw Materials	Procedures	Conditions	Used Plant
Greases	Controllable-pitch propeller grease	Aluminum stearate Aluminum Oleate Lead oleate Glycerine Mineral oil	Compounding: Aluminum stearate, aluminum oleate, lead oleate, glycerine and mineral oil is thoroughly mixed in the cold and then the mixture is compounded by heating. After the mixture is perfectly dissolved, the grease is then slowly cooled under slow agitation and then poured into product tank.	Temp. of compounding at 130~150°C Time of heating at comp. temp: 3~4 hrs. Cooling to room temp. by cooling water jacket. Time for cooling: 12 hrs. Total time from charge to products: 24 hrs.	Indirect steam heated steel kettle
	Special grease for framework of aero-torpedo	Aluminum stearate Calcium stearate Cylinder oil for aero-torpedo	Compounding: Aluminum stearate, calcium stearate and oil are thoroughly mixed in the cold and then the mixture is compounded by heating comp. & after treatments are same as case of controllable-pitch propeller grease.	Temp. of compounding at 130~150°C	Indirect steam heated steel kettle
	Special grease as the anticorrosives for the interior of compressed air chamber of aero-torpedo	Aluminum stearate Calcium stearate Cylinder oil for aero-torpedo	Compounding: Aluminum stearate, calcium stearate and oil are thoroughly mixed in the cold and then compounded by heating. After the mixture is perfectly dissolved, the grease is then poured into cooling pans & placed in ordinary temp. The grease is then milled.	Temp. of compounding at 130~150°C Time of heating at comp. temp: 3~4 hrs.	Indirect steam heated steel kettle Three roller mixer
	Sea water-proof grease	Aluminum stearate Heavy lubricating oil	Compounding: Same as anticorrosive grease. After the compounding and cooling, the grease is milled.	Temp. of compounding at 130~150°C Time of heating at comp. temp: 3~4 hrs.	Indirect steam heated steel kettle Three roller mixer

Table XII(B)36 (Cont)
GREASE MANUFACTURING PROCEDURES

ENCLOSURE (B)36

Table XII(B)36 (Cont'd)
GREASE MANUFACTURING PROCEDURES

		Raw Materials	Procedures	Conditions	Used Plant
Metallic Soaps	Aluminum stearate	Stearic acid Caustic soda Aluminum sulphate	Saponification: Stearic acid is saponified by heating with caustic soda and water. Double decomposition: 5% of sol'n. of sodium stearate is converted to metallic soap by treatment with 25% sol'n. of aluminum sulphate.	Temp. of saponification, at 95~98°C Temp. of double decomposition at 60°C	Saponification vessel Double decomposition vessel
	Aluminum oleate	Oleic acid Caustic soda Aluminum sulphate	Saponification: Oleic acid is saponified by heating with caustic soda and water. Double decomposition: Same as aluminum stearate.	Temp. of saponification, at 95~98°C Temp. of double decomposition at 30°C	Saponification vessel Double decomposition vessel
	Lead oleate	Oleic acid Caustic soda Lead acetate	Saponification: Same as aluminum oleate Double decomposition: 5% sol'n. of sodium oleate is converted to metallic soap by treatment with a 5% sol'n. of lead acetate.	Temp. of saponification, at 95~98°C Temp. of double decomposition at 30°C	Saponification vessel Double decomposition vessel
	Calcium stearate	Stearic acid Caustic soda Calcium chloride	Saponification: Same as aluminum stearate. Double decomposition: 5% sol'n. of sodium stearate is converted to metallic soap by treatment with a 5% sol'n. of calcium chloride	Temp. of double decomposition at 60°C	Saponification & Double decomposition vessel
	Calcium oleate	Oleic acid Caustic soda Calcium chloride	Saponification: Same as aluminum oleate Double decomposition: Same as calcium stearate.	Temp. of double decomposition at 30°C	Saponification & Double decomposition vessel
Greases	Magneto grease	Castor oil Caustic soda Lubricating oil	Saponification and Compounding: Castor oil and mineral oil are saponified by heating with 40% sol'n. of caustic soda & then compounded. The grease is then poured into cooling pans & may be cooled, is milled.	Temp. of saponification, at 100°C Temp. of compounding, at 230°C	Electrically heated steel kettle Three roller mixer
	Rocker arm grease	Stearic acid Oleic acid Caustic soda Glycerine Mineral oil Calcium stearate Calcium oleate	Saponification and Compounding: Fatty acids and mineral oil are saponified by heating with 30% sol'n. of caustic soda, and then calcium soaps are added to mixture and is compounded by heating. The grease is poured into cooling pans, after cooling, is milled.	Temp. of saponification, at 100°C Temp. of compounding at 150~160°C	Electrically heated steel kettle Three roller mixer

ENCLOSURE (B)36

Table XIII(B)36
FLOWSHEET OF GREASE MANUFACTURE

Raw Materials	Charge (kgs)	Balance	Process	Intermediates & Products
Stearic acid	40	100	Saponification → Double Decomposition Washing & Drying	Aluminum-stearate 34 kgs, 85% per stearic acid
Caustic soda	6	15		
Aluminum sulphate	18.8	47		
Oleic acid	25	100	Saponification → Double Decomposition Washing & Drying	Aluminum-oleate 22.5% kgs, 90% per oleic acid
Caustic soda	3.8	15		
Aluminum sulphate	12.0	47		
Oleic acid	25	100	Saponification → Double Decomposition Washing & Drying	Lead-oleate 22.5 kgs, 90% per oleic acid
Caustic soda	3.8	15		
Lead acetate	22.5	90		
Fatty acid	40	100	Saponification → Double Decomposition Washing & Drying	Calcium soap 32 kgs, 80% per fatty acid
Caustic soda	6	15		
Calcium chloride	14	35		
	(kgs)	(%)		(Product)
Castor oil	56	79.2	Saponification & Compounding → Cooling & Milling	Magneto grease 215 kgs Yield: 94%
Caustic soda	11	3.7		
Light lub. oil	250 lit	77.1		
Total	292	100		
	(kgs)	(%)		
Stearic acid	10.5	3.0	Saponification & Compounding → Cooling & Milling	Rocker arm grease 336 kgs Yield: 96%
Oleic acid	10.5	3.0		
Caustic soda	3.15	0.9		
Glycerine	1.40	0.4		
Calcium stearate	3.5	1.0		
Calcium oleate	3.5	1.0		
Heavy lub. oil	317.5	90.7		
Total	350	100		
	(kgs)	(%)		
Aluminum stearate	64.8	6.48	Compounding	Controllable pitch propeller grease 980 kgs Yield: 98%
Aluminum oleate	7.2	0.72		
Lead oleate	8.0	0.80		
Glycerine	4.0	0.40		
Lubricating oil	916.0	91.60		
Total	1000	100		
	(kgs)	(%)		
Aluminum stearate	70.0	7.00	Compounding	Anticorrosive liquid grease for aero-torpedo 990 kgs Yield: 99%
Calcium stearate	2.1	0.21		
Lub. oil for aero-torpedo	927.9	92.79		
Total	1000	100		
	(kgs)	(%)		
Aluminum stearate	120	12.00	Compounding → Cooling & Milling	Anticorrosive grease for the interior of compressed air chamber 960 kgs of aero-torpedo Yield: 96%
Calcium stearate	3.6	0.36		
Lub. oil for aero-torpedo	876.4	87.64		
Total	1000	100		
	(kgs)	(%)		
Aluminum stearate	130	13.0	Compounding → Cooling & Milling	Sea water proof grease 960 kgs, Yield: 96%
Heavy lub. oil	870	87.0		
Total	1000	100		

Table XIV(B)36
LUBRICATING OILS

Base oils for	Flash point (°C)	Viscosity (S.U.S.) (210°F)	Viscosity (Redwood) (sec.) at 30°C	Vis. Index	Setting point (°C)	Carbon Residue (%)
Magneto grease	155		130.8		30	0.02
Rocker arm grease	218	95.0		67.3	18	2.01
Controllable-pitch propeller grease	268	133.4		87.5	15.5	0.40
Aero-torpedo grease	203	61.9		85.3	36	0.36
Sea water-proof grease	270	141.0		15.0	1	5.0

Table XV(B)36
SOAP STOCKS

	M.P. (°C)	Acid Value	Sap. Value	Iod. Value	Remarks
Castor oil		0.5	192.9	84.6	
Stearic acid	63.0	189	193	3.1	Hydrogenated tea-seed fatty acid
Oleic acid					Tubaki-seed fatty acid
Stearic acid	65-67	202	206	0	Market, pure

Table XVI(B)36
INTERMEDIATES

	Moisture (%)	Residue after burning (%) Total	Residue after burning (%) Water Soluble
Aluminum stearate	0.5	8.16	.8
Aluminum oleate	1.0	10.5	1.6
Lead oleate	1.0		
Calcium soap (stearic 50% / oleic 50%)	0.3	12.3	.5

ENCLOSURE (B)36

Table XVII(B)36
PRODUCTS*

	Appearance	Consistency (at 25°C)		Dropping Point (°C)	Free Alkali (%)	Free Fatty Acid (%)	Water (%)	Ash (%)
Magneto grease	Smooth texture	180 at -20°C	250	171	0.10	none	0.3	3.5
Rocker arm grease	Semi-fluid Smooth texture		350	130	none	0.4	0.5	1.5
Controllable-pitch propeller grease	Stringy viscous fluid		350 at 10°C	75	none	1.0	trace	1.2
Liquid grease for the framework of aero-torpedo	Fluid texture	290 at -40°C	Vis.(S.U.S.) 210°F 1,500 sal.		none		0.4	0.37
Anticorrosives for the interior of compressed air chamber	Smooth texture		310	92	none	0.5	0.1	0.90
Non water-proof grease	Adhesive smooth		330	86	none	0.5	0.1	1.0

*All these greases were non-corrosive and possessed satisfactory stability (100°C, 3 hr).

ENCLOSURE (B)36

Table XVIII(B)36
COMPOUNDING AND PROPERTIES OF SPECIAL GREASES

	Products (Remarks)	Yield (%) per charge	Appearance	General Properties ** Consistency* at °C	Consistency* at 25°C	Dropping Pt. (°C)	Mineral Oil (%)	Free Alkali (%)	Free Fatty Acid (%)	Water (%)	Ash (%)
Magnetic grease	In pilot plant	94	Smooth texture	(-)20 189	250	171		0.10	none	0.3	3.5
	In laboratory		Smooth texture	180	254	175	68.7	0.12	none	0.5	3.44
Rudder arm grease	In pilot plant	96	Semi-fluid Smooth		330	130	90.7	none	0.40	0.50	1.5
	In laboratory		Semi-fluid Smooth		360	119	91.2	0.0	0.31	0.20	1.5
Controllable-pitch propeller grease	In pilot plant	98	Stringy Viscous Fluid	(+)10 350		75	91.6	none	1.0	trace	1.2
	In laboratory		Stringy Viscous Fluid	350		40	91.6	none	0.67	trace	1.08
Anti-corrosive liquid grease for the framework of aero-torpedo	In pilot plant	99	Fluid	(-)40 290		Viscosity (S.U.S.) 210°F 1500	92.79			0.4	0.37
	In laboratory		Fluid	284		1036	92.79		0.5	0.05	0.50
Anti-corrosive grease for the interior of compressed air chamber of aero-torpedo	In pilot plant	96	Smooth texture		310	92	87.6		0.5	0.10	0.9
	In laboratory		Smooth texture		330	89	87.6		0.5	0.04	0.8
Sea Water-proof grease	In pilot plant	96	Adhesive smooth		330	86	87	none	0.5	0.1	1.0
	In laboratory		Adhesive smooth		310	88	87	none	0.5	trace	0.94

*Worked Penetration, A.S.T.M.
**All these greases were non-corrosive and in the stability test there was no oil bleeding (100°C 3 hrs).

ENCLOSURE (B)36

Table XVIII(B)36 (Cont'd)
COMPOUNDING AND PROPERTIES OF SPECIAL GREASES

Name of Grease	Formula		Charge	Type of Grease Kettle Used	Performance Treatments
Magneto grease	Caster oil Caustic soda Water Light lubricating oil	19.2% 3.7% 77.1%	56 kgs 11 kgs 250 lit Total 300 kgs	Electrically heated kettle 500 liter capacity	Cooking and compounding at 230°C Cooling without agitation in cooling pan Milling by three roller mill
Rocker arm grease	Stearic acid Oleic acid Caustic soda Calcium stearate Calcium oleate Glycerine Heavy lubricating oil	3.0% 3.0% 0.9% 1.0% 1.0% 0.4% 90.7%	Total charge 350 kgs	Electrically heated kettle 500 liter capacity	Cooking and compounding at 150-160°C Cooling without agitation in cooling pan Milling by three roller mill
Controllable-pitch propeller grease	Aluminum stearate Aluminum oleate Lead oleate Glycerine Lubricating oil	6.48% 0.72% 0.80% 0.40% 91.60	Total charge 1000 kgs	Indirect steam heated kettle 1500 liter capacity	Compounding at 130-150°C Slow cooling with agitation
Anti-corrosive liquid grease for the framework of aero-torpedo	Aluminum stearate Calcium stearate Lubricating oil for aero-torpedo	7.0% 0.21% 92.79%	Total charge 1000 kgs	Indirect steam heated kettle 1500 liters capacity	Compounding at 130-150°C Slow cooling with agitation
Anti-corrosive grease for the interior of compressed air chamber of aero-torpedo	Aluminum stearate Calcium stearate Lubricating oil for aero-torpedo	12.0% 0.36% 87.64%	Total charge 1000 kgs	Indirect steam heated kettle 1500 liter capacity	Compounding at 130-150°C Cooling without agitation in cooling pan Milling by three roller mill
Sea water-proof grease	Aluminum stearate Heavy lubricating oil	13.0% 87.0%	Total charge 1000 kgs	Indirect steam heated kettle 1500 liter capacity	Compounding at 130-150 C Cooling without agitation in cooling pan Milling by three roller mill

ENCLOSURE (B)36

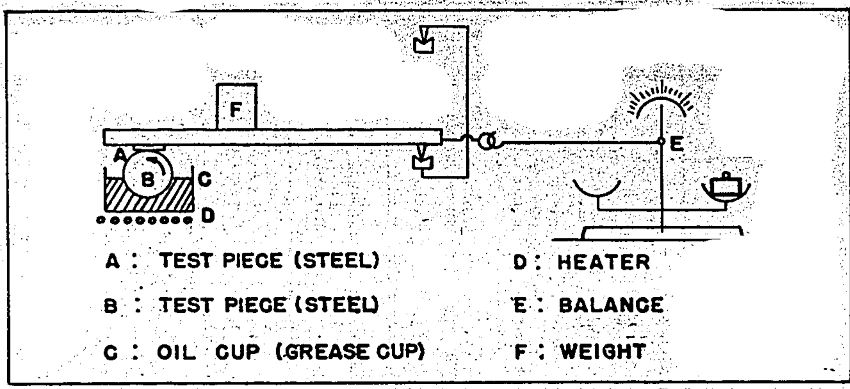


Figure 1(B)36

SKETCH OF MECHANISM OF BEARING TYPE TESTING APPARATUS

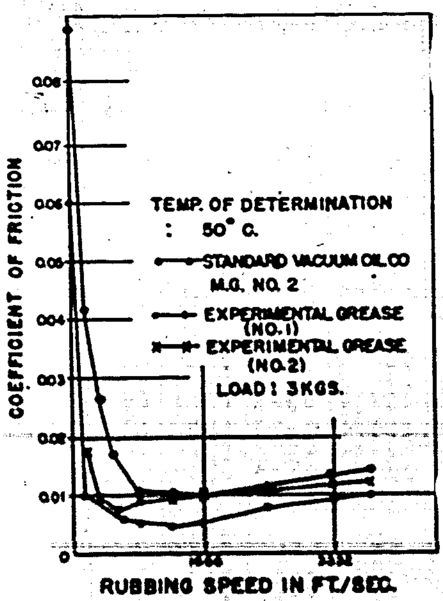


Figure 2(B)36-a

CHART OF COEFFICIENT OF FRICTION

BEARING TYPE TESTING MACHINE

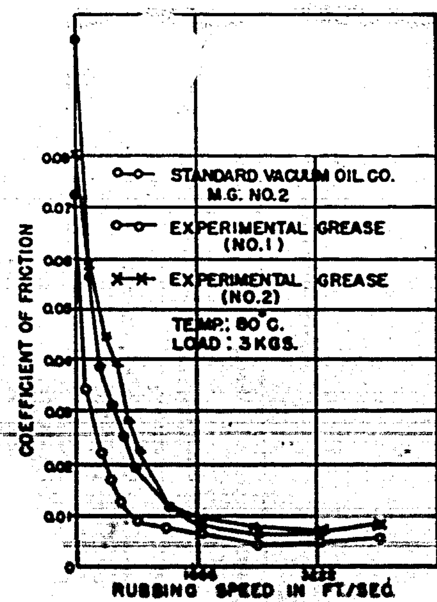


Figure 2(B)36-b

CHART OF COEFFICIENT OF FRICTION

BEARING TYPE TESTING MACHINE

ENCLOSURE (B)36

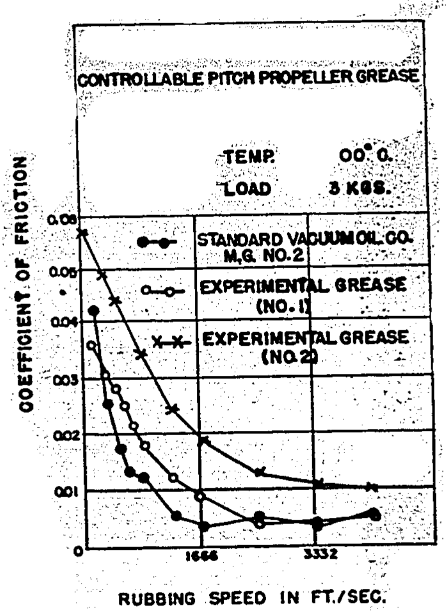


Figure 2(B)36-c
CHART OF COEFFICIENT OF FRICTION
BEARING TYPE TESTING MACHINE

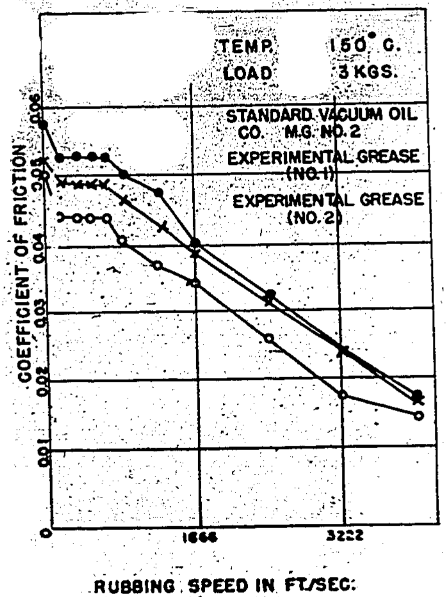


Figure 2(B)36-d
CHART OF COEFFICIENT OF FRICTION
CONTROLLABLE PITCH PROPELLER GREASE

Figure 3(B)36
PILOT PLANT FOR MANUFACTURING GREASES

ENCLOSURE (B)36

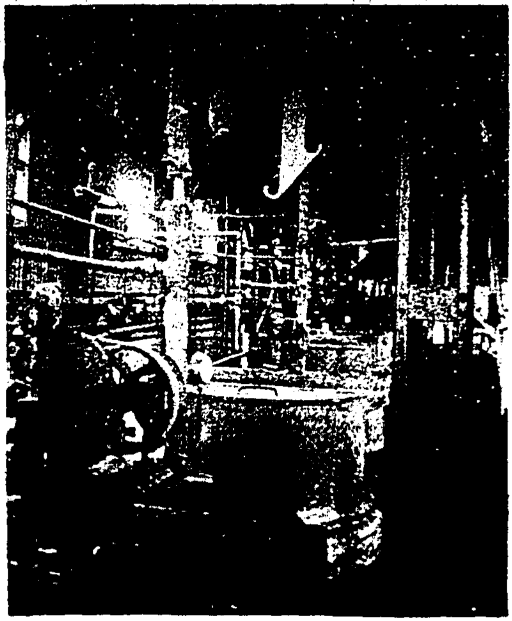


Figure 4(B)36
DOUBLE DECOMPOSITION VESSELS

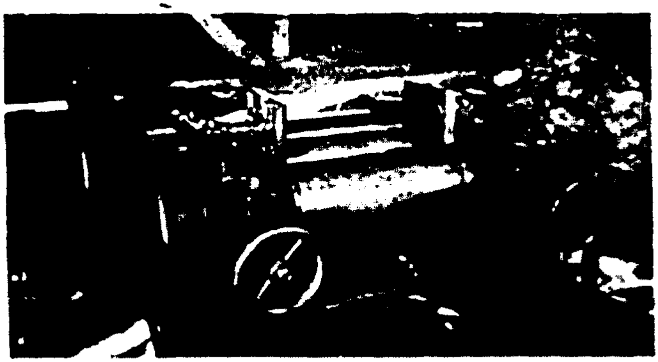


Figure 5(B)36
THREE ROLLER TYPE MILLING MACHINE

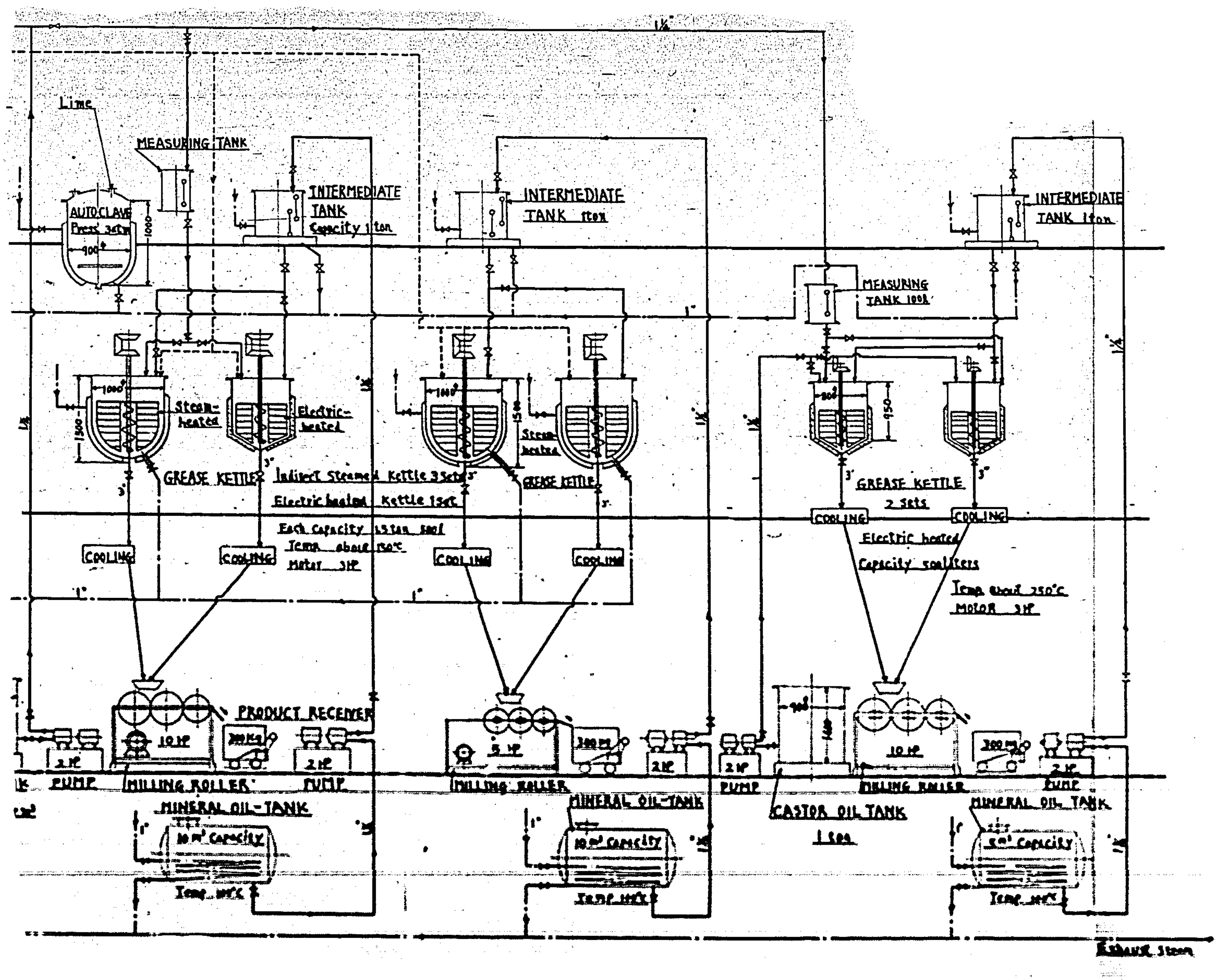
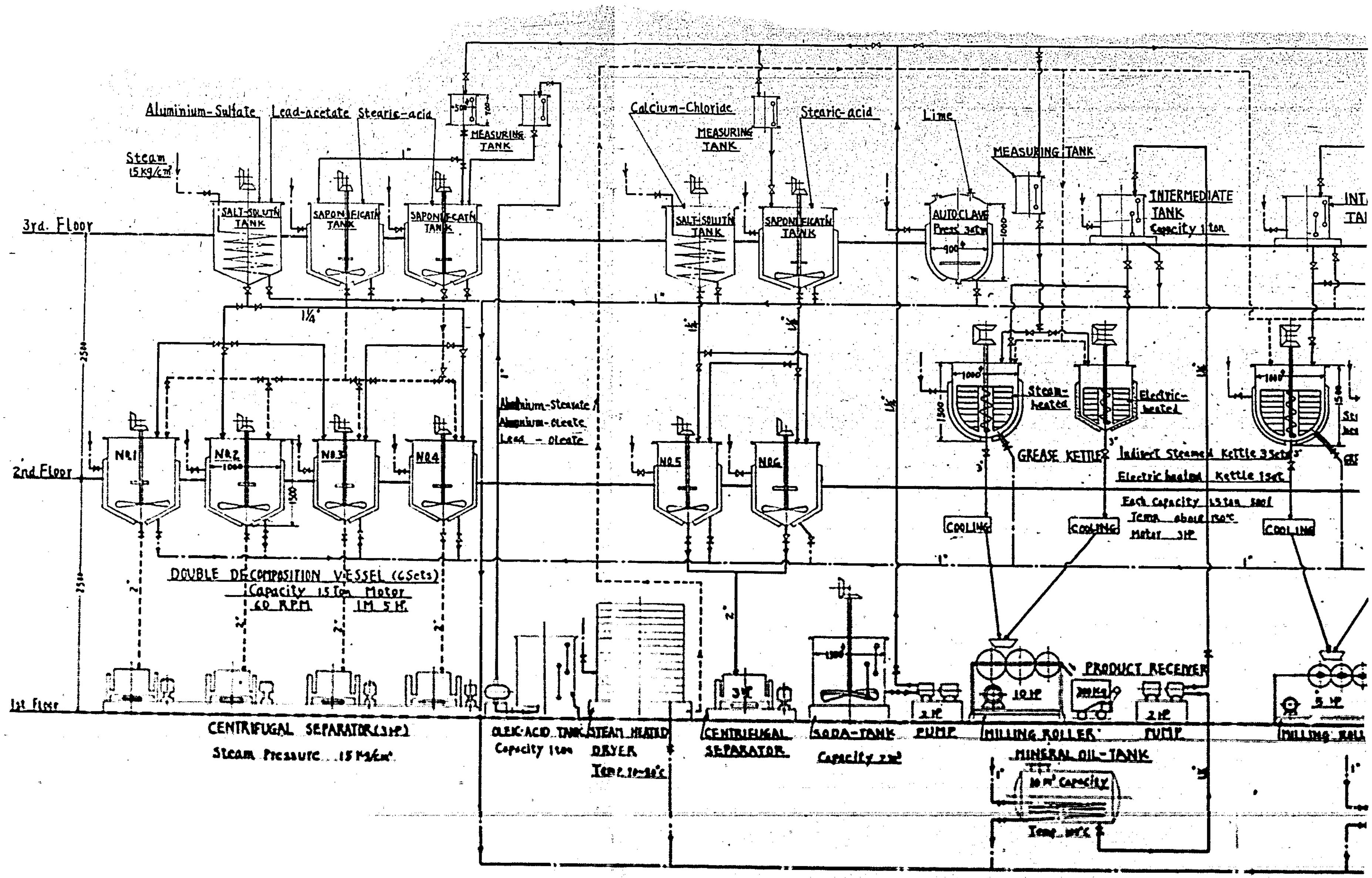


PLATE I(B)36
FLOW SHEET OF PILOT PLANT FOR GREASE MANUFACTURE



Aluminium-Sulfate
Lead-acetate
Stearic-acid
Steam 15 kg/cm²
MEASURING TANK
SALT-SOLUTN TANK
SAPONIFICATN TANK
3rd. Floor
2nd Floor
1st Floor
NO.1
NO.2
NO.3
NO.4
NO.5
NO.6
DOUBLE DECOMPOSITION VESSEL (6Sets)
Capacity 1.5Ton Motor
60 RPM 1M 5HP
CENTRIFUGAL SEPARATOR(3HP)
Steam Pressure 15 kg/cm²
Aluminium-Stearate
Aluminium-oleate
Lead-oleate
Calcium-Chloride
Stearic-acid
Lime
AUTOCLAVE
MEASURING TANK
INTERMEDIATE TANK Capacity 1 ton
Steam-heated
Electric-heated
GREASE KETTLE
Indirect Steamed Kettle 3 Sets
Electric heated Kettle 1 set
Each Capacity 1.5 ton
Motor 3HP
COOLING
OLEIC-ACID TANK Capacity 1 ton
STEAM HEATED DRYER
CENTRIFUGAL SEPARATOR
SODA-TANK
PUMP
MILLING ROLLER
PRODUCT RECEIVER
MINERAL OIL-TANK
Capacity