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Report on

Self-Sealing Materials for Lubricating Oil Tanks of Military Aircraft

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TABLE OF CONTENTS

Page No.

APPENDIX

AUTHORIZATION

1. This project was authorized by BuAero Letter AER-E-25-SGE, L11-1(3) of September 3, August 12, and September 29, 1940.

STATEMENT OF PROBLEM

2. The problem of protecting the lubricating systems of military aircraft from failure due to the damage of enemy fire is a matter of unquestioned importance. In view of the success of self-sealing tanks for gasoline, it has been hoped that a similar type of protection could be extended to lubricating oil tanks and lines. Protection of oil tanks, however, involves factors not encountered with gasoline, and of these not the least is the wide range of temperature, high enough at times to come within the curing range of rubber and rubber-like materials, under which oil systems sometimes operate Moreover, lubricating oil appears to attack self-sealing materials in a fashion somewhat different from gasoline, and it is understood that certain properties of the oil which make it useful as a lubricant tend to render sealing of the punctured tanks less effective than with gasoline.

3. In order to be really effective, self-sealing materials for oil tanks must be capable of withstanding high operating temperatures for reasonable periods of time, must suffer no more than small impairment of the original sealing characteristics during installed service life, and must not affect deleteriously the essential properties of the oil. This last factor is of prime importance to this Laboratory.

4. In order to determine the effect of self-sealing materials on aviation oils, and of oils on rubber and rubber-like synthetics, tests were set up as described below and run at varying temperatures simulating actual operating conditions. The self-sealing agents were the same as used in corresponding work with gasoline described in NRL Report No. P-1662.

Materials Investigated

The self-sealing materials were products of the U. S. Rubber Company. The number and the identity of these samples are given below:

Number	Identification
1	21 oz. duck and neoprene.
2	21 oz. duck and neoprene with 4 coats of cellulose acetate.
3	21 oz. duck and neoprene with 2 coats of thiokol cement.
4	0.035 inch neoprene.
5	0.035 inch neoprene with 2 coats of thiokol cement.

Number (cont.)	Identification (cont.)
6	0.25 inch Rubatex.
7	S.T. (single sandwich sealer).
8	Cured high shrink stock.
9	T.S.T. (double sandwich sealer).
10	1 inch cellular neoprene.

Though the above materials were intended for gasoline tanks and not for lubricating oil, they were the only ones available at the time that work on this phase of aircraft protection was begun. Nonetheless, it is thought that the behavior of these substances with oils is of interest to the Navy since, on the basis of that behavior methods can be evolved for evaluating the more highly oil resistant sealing agents, especially designed for lubricating systems, which are now becoming available. Samples 1 through 5 are intended for use as lining materials and are neoprene sheet, plain or surfaced with thiokol or supported on fabric. Numbers 6 through 10 represent compositions whose prime function is to act as sealing agents, and are considerably more sensitive to oil and gasoline than are the former.

5. These samples were tested by immersion in two oils, E and F, both of which are Navy Symbol 1080 oils. These oils had the following characteristics:

	Oil E	Oil F
S.U.V. at 100° F., sec.	912	715
S.U.V. at 210° F., sec.	84	75
Viscosity Index	95.4	97.4
Aniline Point, °C	115.4	120.0
Specific Gravity 60°/60° F.	0.892	0.881
Viscosity - Gravity Constant	0.816	0.804
Conradson Carbon Residue, %	0.83	0.25
Neutralization Number	0.03	0.01
Average Molecular Weight	542	550

6. These lubricants are representative of those supplied the Navy under Symbol 1080 for use in aircraft engines. They are examples of the so-called "paraffin base" oils as evidenced by their high viscosity indices and aniline points. The viscosity-gravity constant of approximately 0.8 further confirms this. These oils are considerably different in some characteristics, notably viscosity and carbon residue, even though their average molecular weights are approximately the same. Because they may be considered typical of Naval aviation oils, yet show considerable differences in some characteristics, they were selected for this study as it was thought possible that self-sealing materials might react differently with the two oils.

EXPERIMENTAL

7. Tests were run on the samples by immersing them in oils at the temperature of the test in the ratio of one square centimeter of rubber

surface to five cubic centimeters of oil. The oil was charged at room temperature. The samples were placed in 200 cc. Pyrex Berzelius type beakers, and their behavior investigated under the following conditions of time and temperature.

1. Room temperature - 7 days.
2. 75° C. - 24 hours.
3. 150° C. - 24 hours.

8. The samples run at room temperature were charged into the beakers, covered and left in a quiet dark place at 15 to 28° C. for the length of the test; those treated at 75° C. and 150° C. were placed in an electrically heated, thermostatically controlled, convection type oven. The vent in the top of the oven was opened so as to permit air circulation, but no attempt was made to measure the amount of air circulating through it. The temperature was controlled to a ±0.5° C. A blank determination was made under each of the above-mentioned conditions.

9. After the test was completed, the rubber samples were removed from the oil, washed with A.S.T.M. precipitation naphtha, and dried at room temperature, and then examined to determine any changes as compared with the original rubber sample. The oils from the tests were strained through a 100-mesh sieve and saved for further tests.

10. The following tests were made on the oil to determine the changes which had taken place, as compared to the blank, under the various test conditions.

1. Viscosity, Saybolt Universal
    (a) Viscosity, Kinematic, A.S.T.M. - D445-39T
    (b) Viscosity, Conversion of Kinematic to Saybolt Universal, A.S.T.M. - D446-39
2. Neutralization Number, A.S.T.M. - D188-27T; F.S.B.-510.31
3. Copper Strip Corrosion at 212° F.,F.S.B.-530.31
4. Conradson Carbon Residue, A.S.T.M.-D189-39; F.S.B.-500.13
5. Insolubles and Gums. These were determined by filtering 10 gram portions of the oils previously diluted with A.S.T.M. precipitation naphtha through diatomaceous earth in a weighted Gooch crucible as described in detail in the Appendix. The differences in weights of the insolubles and gums in the samples of oils exposed to the rubber materials above that given by the blank oil determination is a measure of the constituents dissolved from the rubber or the decomposition products of the oil which is catalyzed by the rubber, or both.

DATA OBTAINED.

11. The results of these determinations are shown in the following tables and plates.

TABLE I

7 Days at Room Temperature
Oil E

					Insolubles and gums, mgms. *	
Identification	S.S.U. 100° F.	Neut. No.	Corrosion	Conradson C.R. %	Total	Differ.
E, blank	912	0.03	Passes	—	8.4	
E1, liner	912	0.03	Passes	—	8.4	0.0
E2, liner	908	0.03	Passes	—	7.7	-0.7
E3, liner	910	0.03	Passes	—	8.3	-0.1
E4, liner	912	0.03	Passes	—	8.4	0.0
E5, liner	906	0.03	Passes	—	8.1	-0.3
E6, sealer	908	0.03	Fails, mottled	—	8.8	0.4
E7, sealer	902	0.04	Fails, black	—	8.7	0.3
E8, sealer	909	0.03	Passes	—	8.7	0.3
E9, sealer	903	0.03	Fails, black	—	8.6	0.2
E10, sealer	907	0.03	Passes	—	8.4	0.0
Oil F						
F, blank	715	0.01	Fails, very slight	—	8.4	
F1, liner	715	0.01	Fails, very slight	—	8.4	
F2, liner	714	0.01	Fails, slight	—	7.8	-0.6
F3, liner	714	0.01	Fails, very slight	—	8.1	-0.3
F4, liner	714	0.01	Fails, slight	—	8.4	0.0
F5, liner	712	0.01	Fails, mottled	—	8.5	0.1
F6, sealer	715	0.01	Fails, slight	—	8.5	0.1
F7, sealer	709	0.03	Fails, black	—	8.3	-0.1
F8, sealer	715	0.01	Fails, mottled	—	9.1	0.7
F9, sealer	710	0.01	Fails, black	—	8.6	0.2
F10, sealer	713	0.01	Fails, very slight	—	7.9	-0.5

* Basis, 10 gm. oil sample.

TABLE II

24 Hours at 75° C.

Oil E

Identification	S.S.U. 100° F.	Neut. No.	Corrosion	Conradson C.R.%	Insolubles and gums, mgms.*	
					Total	Differ.
E, blank	917	0.03	Fails, very slight	0.84	8.6	
E1, liner	914	0.03	Fails, slight	0.83	8.5	-0.1
E2, liner	917	0.03	Fails, purple	0.83	8.2	-0.4
E3, liner	914	0.03	Fails, very slight	0.84	8.6	0.0
E4, liner	914	0.03	Fails, slight	0.79	8.0	-0.6
E5, liner	915	0.03	Fails, purple	0.79	8.5	-0.1
E6, sealer	913	0.03	Fails, slight purple	0.82	8.8	0.2
E7, sealer	907	0.06	Fails, black	0.85	8.3	-0.3
E8, sealer	922	0.03	Fails, slight	0.79	8.3	-0.3
E9, sealer	907	0.04	Fails, black	0.83	8.6	0.0
E10, sealer	916	0.03	Fails, slight	0.83	8.4	-0.2

Oil F

Identification	S.S.U. 100° F.	Neut. No.	Corrosion	Conradson C.R.%	Total	Differ.
F, blank	716	0.01	Fails, very slight	0.23	8.0	
F1, liner	715	0.01	Fails, very slight	0.25	8.4	0.4
F2, liner	714	0.01	Fails, slight purple	0.23	8.4	0.4
F3, liner	714	0.01	Fails, very slight	0.25	8.0	0.0
F4, liner	716	0.01	Fails, slight	0.25	8.1	0.1
F5, liner	716	0.01	Fails, purple	0.24	8.3	0.3
F6, sealer	715	0.01	Fails, slight purple	0.25	8.6	0.6
F7, sealer	709	0.04	Fails, black	0.24	8.4	0.4
F8, sealer	722	0.03	Fails, slight purple	0.27	8.6	0.6
F9, sealer	711	0.03	Fails, black	0.25	8.4	0.4
F10, sealer	715	0.01	Fails, very slight	0.25	8.5	0.5

* Basis, 10 gm. oil sample.

TABLE III

24 Hours at 150° C.

Oil E

Identification	S.S.U. 100° F.	Neut. No.	Corrosion	Conradson C.R.%	Insolubles and gums, mgms.*	
					Total	Differ.
E, blank	927	0.06	Passes	0.96	11.4	
E1, liner	929	0.08	Passes	0.93	12.1	0.7
E2, liner	926	0.08	Passes	0.95	11.7	0.3
E3, liner	923	0.08	Passes	0.93	12.1	0.7
E4, liner	926	0.07	Passes	0.89	11.4	0.0
E5, liner	925	0.07	Passes	0.93	12.2	0.8
E6, sealer	969	0.06	Passes	0.95	44.5	33.1
E7, sealer	1002	0.21	Passes	0.93	45.0	33.6
E8, sealer	995	0.14	Passes	0.93	35.7	23.3
E9, sealer	986	0.15	Passes	0.93	40.2	28.8
E10, sealer	922	0.06	Passes	0.93	11.2	- 0.2

Oil F

Identification	S.S.U. 100° F.	Neut. No.	Corrosion	Conradson C.R.%	Insolubles and gums, mgms.*	
					Total	Differ.
F, blank	723	0.04	Passes	0.29	10.0	
F1, liner	722	0.05	Passes	0.27	10.5	0.5
F2, liner	724	0.07	Passes	0.30	11.1	1.1
F3, liner	721	0.07	Passes	0.28	10.7	0.7
F4, liner	723	0.05	Passes	0.30	9.8	-0.2
F5, liner	722	0.05	Passes	0.28	10.3	0.3
F6, sealer	765	0.03	Passes	0.37	51.3	41.3
F7, sealer	793	0.22	Passes	0.39	59.6	49.6
F8, sealer	818	0.10	Passes	0.34	54.5	44.5
F9, sealer	789	0.17	Passes	0.33	49.2	39.2
F10, sealer	721	0.05	Passes	0.28	11.0	1.0

* Basis, 10 gm. oil sample.

DISCUSSION OF RESULTS

12. Room Temperature - 7 Days. Oil E, when in contact with the rubber samples for 7 days at room temperature, showed no detectable differences from the blank except in the cases of samples E6, E7, and E9. Samples E7 and E9 had decreased in viscosity. This decrease is slightly greater than the permissible experimental error, and is evidence that some less viscous material was dissolved from the rubber and diluted the oil. This slight decrease in viscosity is of no significance in itself as this change is too small to be noticed in the operation of the engine. However, it is possible that this diluent from the rubber may have objectionable properties, such as corrosiveness, acidity, and tendencies to form gums and sludges under operating conditions. Sample E7 also increased in acidity from 0.03 to 0.04. This increase is approximately within the limits of experimental error. All of the above-mentioned samples failed the copper strip corrosion test at 212° F. This shows that some corrosive material was extracted from the rubber. Sulfur is the material most commonly used in vulcanizing rubber, and it is believed that the extracted sulfur or sulfur compounds were the cause of the failure of samples E6, E7, and E9 to pass the corrosion test.

13. The Conradson carbon residue was not run on these samples as the experimental error in this test is so great that small changes cannot be detected.

14. The insolubles and gums found were the same as the blank within the limits of experimental error.

15. As in the case of Oil E, samples F7 and F9 showed viscosity decreases. The per cent decrease was approximately the same for both oils. Sample F7 similarly to E7 showed an increase in acidity from 0.01 to 0.03.

16. The corrosion test on the blank, original oil, showed a very slight discoloration. It is doubtful whether this oil itself passes the test. Samples F1, F3, and F10 had about the same amount of discoloration as the blank. Samples F2, F4, and F6 were slightly more discolored. Samples F5, F7, F8, and F9 gave a definite discoloration to the copper strips ranging from mottled to black in color. As Oil F is on the border line of passing the corrosion test, it is not surprising that more samples failed this test in the F series than the E, as it would require less of the extracted corrosive material to push the discoloration of the copper strip over the border line of passing. There is also the possibility that the rubber samples are more soluble in Oil F than Oil E; thus more of the corrosive material would be extracted by Oil F.

17. The Conradson carbon residue was not run on the F series for the same reason as that given for the E series.

18. The insolubles and gums found were the same as the blank within the limits of experimental error, as was the case with the E series.

19. As this Laboratory is not equipped with apparatus for the testing of rubber, the changes which took place in the rubber test specimens had to be detected by visual inspection. Photographs of the various samples are shown on Plates 1 to 6, by which changes may be followed. There was a slight change in rubber samples, 6, 7, 8 and 9, when immersed in the oils. Sample 6 was altered most; it was swollen slightly and more wrinkled than the original. Samples 5, 8, and 9 showed slight swelling around the edges and corners. No differences could be detected between the action of the two oils.

20. <u>75° C. - 24 hours.</u> As was the case at room temperature, E7 and E9 decreased in viscosity, thus confirming the evidence that some lighter material was extracted from these samples. Rubber samples 7 and 9 are very similar, both being sandwich type sealers. Sample 7 is a single sandwich construction, and sample 9 is a double sandwich construction. E8 increased slightly in viscosity, evidencing the extraction of a more viscous material from this sample or the oxidation or polymerization of the extracted material to form this viscous material.

21. Samples E7 and E9 increased in acidity from 0.03 to 0.06 and 0.03 to 0.04, respectively. This increase in acidity will not be harmful unless it is due to strong acids, such as mineral acids or the lower homologs of organic acids.

22. All samples of the E series fail the corrosion test; even the blank discolored the copper strip slightly. Sample E2 gave about the same discoloration to the copper strip as did the blank, and E1, E4, E8, and E10 gave slightly more discoloration. The other samples discolored the copper strips to a greater extent ranging from purple to black in color. Thus, it is evident that more corrosive material is extracted in 24 hours at 75° C. than at room temperature in 7 days.

23. The Conradson carbon residues of all oils were the same as the blank within the limits of experimental error.

24. The insolubles and gums of the treated oils were the same as the blank within the limits of experimental error and were of the same magnitude of those at room temperature.

25. The viscosity changes of the F series were of the same order and magnitude as the E series. F7 and F9 decreased and F8 increased in viscosity.

26. F7 and F9 increased in acidity as did the corresponding E samples. In addition, F8 also increased in acidity.

27. All samples of the F series failed the corrosion tests, as did the E series. The samples were also placed in approximately the same order. F1, F3, and F10 were about the same as the blank, F6 slightly worse, and the other samples gave purple to black discolorations.

28. The Conradson carbon residues were the same as the blank within the limits of experimental error, as were the gums and insolubles.

29. The rubber samples 6, 7, 8, and 9 showed some changes after being immersed in the oil for 24 hours at 75° C. No differences could be detected in the other samples. Sample 6 was swollen and wrinkled, slightly more so that it was at the room temperature test. Samples 7, 8 and 9 also showed somewhat greater swelling around the edges and corners than they did at room temperature. No difference could be detected between the action of the two oils on the rubber samples under this test condition.

30. <u>150° C. - 24 hours.</u> The viscosity of samples E6, E9, E8, and E7 all increased in the order named. This increase in viscosity was due to the extraction of a more viscous material from the rubber, the oxidation of the extracted material, or the catalytic action of the rubber on the oxidation and decomposition of the oil. A combination of the first two hypotheses is more probable. This increase in viscosity is considerable in the abovementioned cases and shows that the oil is badly contaminated by these rubber samples.

31. There is very little increase in the acidity of the samples over the blank except samples E8, E9, and E7 in the order named. Oil from these samples is more acid than allowed by current specifications. If this acidity is due to strong acids, there is the possibility of the etching and corrosion of metal parts, especially the bearings. Sample E6 decreased slightly in acidity. This may be due to the use of ZnO or a similar material as a filler, which would give a basic reaction in aqueous solution, thus reducing the total acidity of the sample.

32. All samples of the E series passed the corrosion tests. However, it is believed that the corrosive material was extracted from the oil, as was the case at room temperature, and 75° C., but this material was volatilized from the oil at the temperature of the test or underwent some reaction to form a compound which was not corrosive. If either were the case, in actual engine use the corrosive material would be liberated in the crankcase, where it would come immediately in contact with metal surfaces and thus cause corrosion to such parts.

33. The Conradson carbon residues of all samples are the same as the blank within the limits of experimental error.

34. Samples E8, E9, E6, and E7 in the order named gave considerable increases in insolubles and gums. These samples were also the ones which increased in viscosity. However, they were not in this order. It would not necessarily follow that the increases in these two properties would be in the same order; in fact, it would be rather surprising if they were. Even if the same weights of materials were extracted from the different rubber samples so as to give the same weights of insolubles and gums, the properties of these extracted materials would probably differ, so they would change the viscosities of the oils by different amounts, depending upon their properties.

35. Samples E3 and E5 were contaminated by flakes of thiokol. As these flakes were so large that they could not be dispersed evenly throughout the oil, no concordant results could be obtained. The oil was strained through a 100-mesh sieve, which removed all these flakes, leaving the clear oil. The resulting oils had an insoluble and gum content of approximately the same value as the blank. Nonetheless it should be emphasized that such scaling and flaking of tank liners is undesirable because of the danger of plugged filters, pump parts, and oil lines.

36. Samples F6, F7, F8 and F9 increased in viscosity in the order named. This is the same order as the E series, except for the transposition of samples 7 and 8. As in the case of the E series, samples F8, F9, and F7 became considerably more acid. Likewise, F6 showed a decrease in acidity.

37. All F series samples pass the corrosion tests, as did the E series.

38. Samples F9, F8, F6 and F7 gave increased Conradson carbon residues over the blanks. These four samples also increased in viscosity. The corresponding samples in the E series showed no detectable increase. This seeming discrepancy may be due to one or more of the following reasons. (1) Experimental errors: small differences in the carbon residues are more difficult to detect in oils with high carbon residues. (2) The temperatures at which the two oils are volatilized under the test conditions may be different; thus the residue left from the extracted material would differ at different temperatures. (3) There is the possibility of different or no reactions of the extracted materials with the experimental oils. (4) The solvent effects of the two oils on the different constituents of the rubber samples may not be the same.

39. The insolubles and gums of samples F9, F6, F8 and F7 were considerably higher than the blank, as was the case of the corresponding E series samples, although the order was different. The gums and insolubles for these samples of the F series were greater than those of the E series. This leads to the conclusion that oil F attacks the rubber samples more readily than oil E, or that the extracted material is less soluble in oil F than oil E under the conditions of the insoluble and gum test.

40. As rubber samples 6, 7, 8 and 9 caused considerable increases in several properties of both oils, E and F, over that of the blanks it is apparent that these samples contaminate the oils badly under this test condition. Samples 7 and 9 in each series also left a black deposit which settled out on standing. This is probably lamp black or a similar substance used as a filler. Samples 6 and 8 in both series left a gummy deposit which separated when the oil cooled. As was the case with the E series, F3 and F5 were contaminated with flakes of thiokol.

41. The rubber samples when exposed to the oils for 24 hours at 150° C. exhibited considerable changes in some instances. Samples 1 and 4 showed no apparent swelling, but the neoprene cracked when bent sharply, indicating that the neoprene had lost its elasticity and resilience. Sample 2 was stiff and the cellulose acetate backing was rather birttle, cracking more easily when bent. Samples 3 and 5 flaked badly, the thiokol coat being practically removed by this treatment; that which remained could be wiped off

easily. Sample 6 was practically disintegrated, only a gummy mass remaining. Samples 7, 8 and 9 were swollen badly and were sticky to the touch. Sample 10 had charred somewhat and shrunk in size. Since this sample floated on the oil, about 1/3 immersed, it is believed that the heat was the principal cause of the change. There could be detected no difference in the action of the two oils on the rubber specimens.

CONCLUSIONS

42. I. Oil is contaminated by rubber and rubber-like materials in varying degrees, depending upon the rubber sample.

43. II. The degree of contamination increases with the temperature.

(a) After 7 days at room temperature, the contamination is due primarily to corrosive constituents, probably sulfides.

(b) After 24 hours at 75° C. the contamination of the oil is primarily reflected in increase of corrosiveness to a degree somewhat greater than that for 7 days at room temperature.

(c) After 24 hours at 150° C. the contamination of the oil by rubber samples 6, 7, 8 and 9 was considerable. This contamination is due principally to the extraction of gums and resins and their oxidation products increased the viscosity and carbon-forming tendencies of the oils. Also there was some insoluble material in the oils; this was filler from the rubber in the case of samples 7 and 9 in both the E and F series. Samples 3 and 5 in both series contained flakes of thiokol. Oil F showed greater contamination than E.

44. III. No one test is capable of showing the degree of contamination of the oil as one property may be affected by the rubber under one condition and another under a different condition.

45. IV. Samples 6, 7, 8 and 9 are affected by the oils only slightly at room temperature and 75° C. but at 150° C. swell and become sticky to the touch. These materials are self-sealing agents and normally would be protected from the action of oil by the inner liner of the tank. Samples 1, 2, 3, 4 and 5 are least affected by oil under different conditions of temperature. These samples also had little or no effect on the essential properties of the oil. They represent inner lining materials and should be the only portions of the sealing construction in direct contact with oil.

46. V. The thiokol coating on the neoprene sheet, samples 3 and 7, shows no effect from the oil at room temperature and 75° C., but at 150° C. the thiokol coating flakes off. The neoprene samples 1, 2, 4 and 10 appeared

to be unaffected by the oil at room temperature and 75° C. At 150° C. the samples 1, 2 and 6 break when bent sharply and have lost some of their resilience and elasticity. Sample 10 at 150° C. was shrunken and charred.

47. VI. No difference in the effect of the two oils could be detected by the somewhat crude visual method of evaluating changes in the rubber samples.

RECOMMENDATIONS

48. From the data and the discussion herein, it seems that only changes affecting the characteristics of the oils can be expressed on a quantitative basis. Though some attack of the tank sealing and lining materials does occur under some conditions, no attempt at precise evaluation of these changes has been made for reasons outlined elsewhere. As criteria for detecting alteration in the oil, it would appear that estimations of viscosity, neutralization number, and insolubles and gums, are the most useful. The method for Conradson carbon appears to be too insensitive, and copper strip corrosion is too sensitive and too erratic. Therefore, if specifications be written to safeguard the quality of engine lubricating oil which is contacted directly with self-sealing materials in an internally protected tank, they should be drawn around changes in the three former properties. It is the opinion of this Laboratory, with the qualifications listed below, that an accelerated test at 150° C. for 24 hours using a test specimen immersed in a volume of oil such that for each square centimeter of effective surface, there are present 5 cc. of oil, is sufficiently rigorous to insure against undue oil contamination. Such a test, on the basis of the experimental evidence presented herein, will exclude oil-sensitive rubbers but will not exclude materials as oil resistant as neoprene or better. Therefore, it is recommended that no lining material be used for internally protected, self-sealing oil tanks, which, when contacted with 1080 aviation lubricating oil, alters the properties of that oil as determined on a blank similarly treated by more than the following values:

1. <u>Viscosity</u>. The Saybolf Universal Viscosity at 100° F. shall not change by more than ± 5%.

2. <u>Neutralization Number</u>. The neutralization number shall not exceed that of the blank by 0.04.

3. <u>Precipitation number as determined by method for Insolubles and Gums, Appendix of Report</u>. The amount of insoluble and gummy matter from a 10 gram sample of treated oil shall be not more than 2.5 milligrams above the value for a 10 gram sample of the blank.

50. These recommendations are submitted with the following qualifications:

1. Though the proposed tests are designed to furnish information on the degree of contamination of engine oils by self-sealing materials, they are by no means perfect. For example, attention will be drawn in the Appendix to the fact that the procedure for insolubles and gums gives a measure of the amount of material extracted from the rubber and sludging of the oil, but this measure is not entirely quantitative. Moreover, nothing is known of the actual engine behavior of contaminated oils. Hence, it is impossible to say that application of the above requirements will entirely safeguard engine performance, though it is reasonable to assume that they will assist toward that end.

2. The experimental results on which these recommendations are based are drawn from the study of but one type of lining material, namely, neoprene, together with several typical sealing agents. The products of only one company have been investigated. Furthermore, these materials are primarily designed for the protection of gasoline tanks and carry no claims of especial resistance to hot lubricating oil. In the light of these qualifications, the recommendations herein submitted must be considered as more or less tentative.

APPENDIX

Procedure for Determining Insolubles and Gums

As there is no method for the determination of gums in oils, some method had to be devised. The following procedure was used. Ten grams ±0.1 gram of the oil were weighed into a glass stoppered Erlenmeyer flask, 125 ml. capacity, and the oil diluted to a volume of 100 ml. with A.S.T.M. precipitation naphtha. The solution was thoroughly mixed by shaking, after which the flask was allowed to stand at a temperature of 25° C. ± 2° C. for a period of one hour before filtering through an especially prepared Gooch crucible.

The Gooch crucible, 25 ml. capacity, was prepared for filtration by filling with an asbestos suspension, 6 grams of asbestos per liter of solution, allowed to set for a few minutes to permit the formation of a mat and then sucked dry. The crucibles were dried in an air oven at 110° C. After cooling, 1.5 grams of Celite ± 0.05 gram were weighed into the crucible. Celite is a diatomaceous silica earth, a product of Johns-Manville Company. The Celite was washed with three 20 ml. portions of A.S.T.M. precipitation naphtha, sucked dry and dried in air oven for three hours at 110° C. The crucible is allowed to cool in a dessicator and weighed to 0.1 milligrams. The crucible is placed in the suction assembly and the Celite dampened with A.S.T.M. precipitation naphtha, then stirred up by a stream from the wash bottle, care being taken not to splash any of the Celite out of the crucible. Suction is then applied at a reduced pressure of approximately 10 cm. of mercury. Before the Celite mat is sucked dry, the oil solution is added, care being taken not to let the mat become dry at any time during the filtration as the Celite mat is liable to crack, and thus, the oil solution would channel through. Any precipitate in the flask is washed into the crucible by a stream from the A.S.T.M. precipitation naphtha wash bottle. The crucible is washed with several portions of A.S.T.M. precipitation naphtha, 125 to 150 ml. total volume, to remove any oil and then sucked dry. It should be emphasized that at no time during the filtration or washing should the Celite mat be allowed to become dry. The crucible was then dried for three hours at 110° C., cooled in a dessicator and weighed to 0.1 milligram. The gain in weight is reported as insolubles and gums. To check the efficiency of the filtration through Celite, 50 gram samples of E, E4, E5, F, F4, and F5, which had been run for 24 hours at 150° C. were diluted with A.S.T.M. precipitation naphtha and filtered through Celite Gooch crucibles in the manner described above. The filtrates from the various samples were collected and the naphtha evaporated from the samples on an air bath with a blast of hot air blowing on the samples. The temperature of evaporation was 110° C. After the naphtha was mostly evaporated, the temperature was raised to 145° C., which is above the end point of A.S.T.M. precipitation naphtha, and kept at this temperature for 30 minutes to remove the heavy ends. It was found that this treatment was sufficient to remove the naphtha completely as a subsequent heating caused no change in the viscosity of the oils.

The results are as follows:

Sample	S.S.U. 100° F.	Neutralization Number
E, new oil	912	0.03
E, 24 hrs., at 150° C.	913	0.04
E6, 24 hrs., at 150° C.	928	0.04
E7, 24 hrs., at 150° C.	944	0.15
F, new oil	715	0.01
F, 24 hrs., at 150° C.	713	0.03
F6, 24 hrs., at 150° C.	723	0.03
F7, 24 hrs., at 150° C.	748	0.15

From these results, it is seen that the viscosity of oils E and F were reduced to their original values, but there was only a slight decrease in the neutralization number. Oils E6, E7, F6, and F7 were reduced considerably in viscosity by this treatment, but not all of the material which increased the viscosity was removed. The oils after filtration were clear, showing no evidence of insolubles and were considerably lighter in color than before this treatment, but not as light as the new oil. The acidity of these samples was reduced only slightly. Thus, it seems that this treatment removed all the insolubles and some of the gums. It is believed that a more drastic treatment could be devised which would remove all the gums, but the difficulties in analytical procedure would be increased.

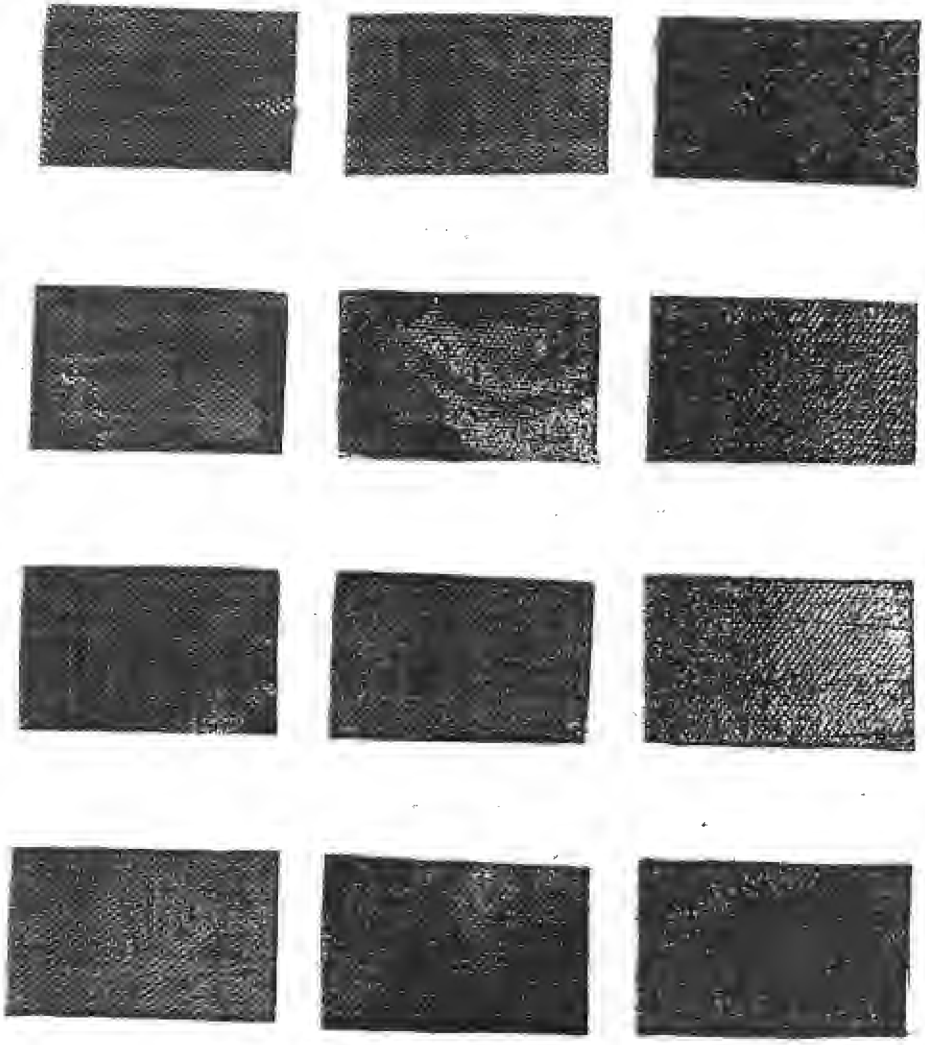


Plate 1

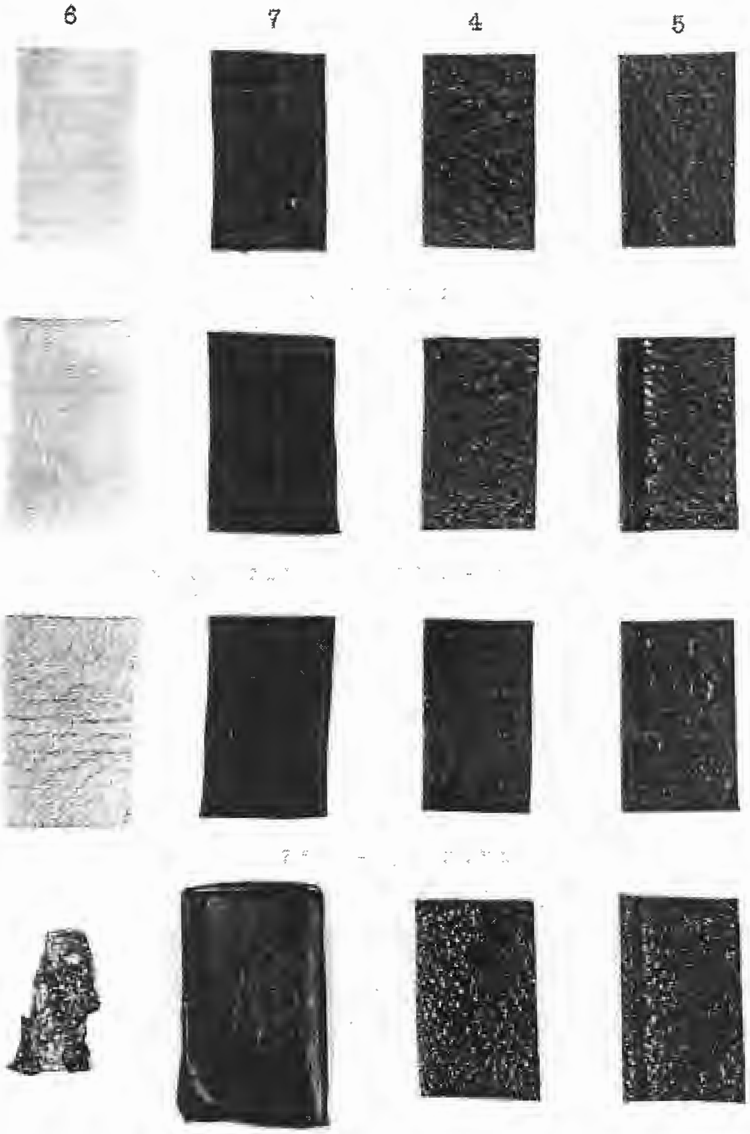


Plate 2

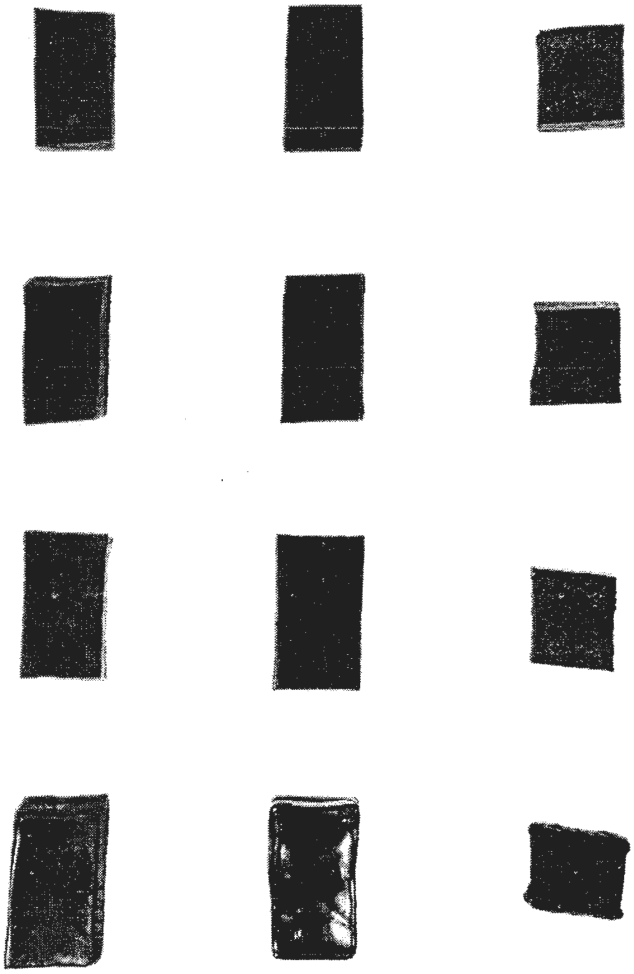
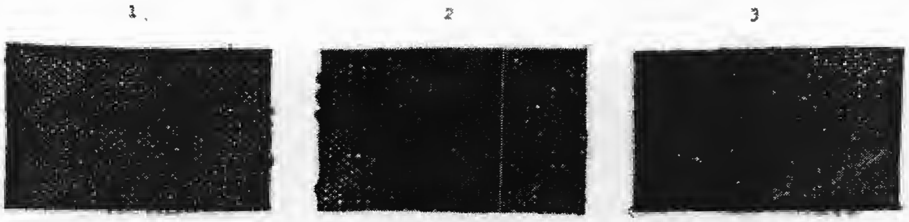


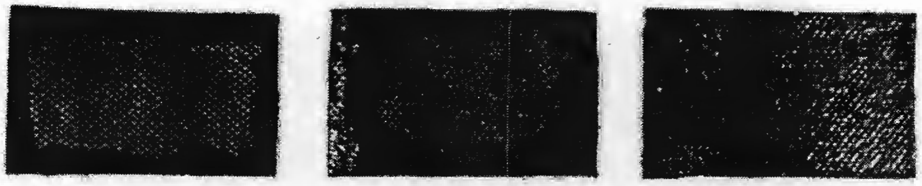
Plate 3

PLATE 4

OIL F



ORIGINAL



ROOM TEMPERATURE - 7 DAYS

75°C - 24 HOURS

150°C - 24 HOURS

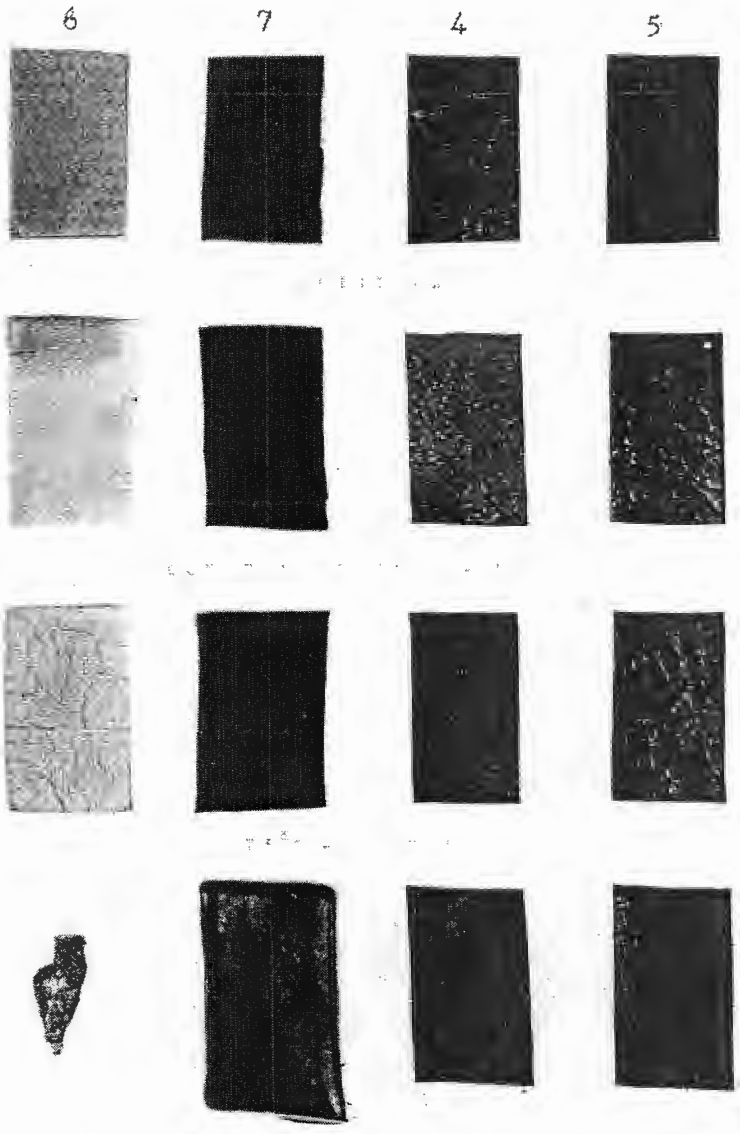


Plate 5