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THE MANUFACTURE, PROPERTIES AND TESTING OF NAPALM SOAPS

by
G. Broughton, Chemist, Eastman Kodak Company
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Supplement to
Report OSRD No. 2036
Copy No. 89
Date: March 7, 1944

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THE MANUFACTURE, PROPERTIES AND TESTING OF NAPALM SOAPS

Service Directives CWS 10 and 21

Endorsement (1) From Dr. H. C. Hottel, Chief, Section 11.3 to Dr. Irvin Stewart, Executive Secretary of the National Defense Research Committee.

Forwarding report and noting:

"This report supplements O.S.R.D. Report No. 2036, "The Manufacture, Properties and Testing of Napalm Soaps" issued November 17, 1943.

"In that earlier report recommendations were made concerning the advisability of investigation of the effect of raw material properties in the finished soap, further investigation of oxidation inhibitors, and investigation of the relationships among gasoline quality, moisture content, concentration and consistency. The present supplement reports progress on those recommendations.

"A study of the effect of raw materials leads to the following conclusions. Varying the composition of Napalm from the standard to 2:1:1 ratio of coconut to oleic to naphthenic acid indicates that the viscosity of the gel increases primarily with increased oleic acids and to a lesser extent with increased coconut acid above normal composition. The acid number of the coconut acid has been found important. Iron is an undesirable impurity when found in the alum but not in the acid.

"Since issue of the last report all Napalm has included an oxidation inhibitor. This is found to have no deleterious effect on consistency and to constitute a definite protection against oxidation.

"Although Napalm manufacture now appears to be under sufficient control to guarantee an acceptably small variation in quality of product from any one manufacturer, the viscositities of gels produced from equal concentrations of soap of different manufacturers vary considerably. For the 8 per cent gels the ratio of the strongest to the weakest is roughly 1.8; for the 4 per cent gels the variation is even greater. There is still need for a determination of how important this variation is in use of

the gel in flame throwers. Consistency varies also with aging, and the effect is most pronounced in gels of low concentration. The consistency of 8 per cent gels varies 1.5 fold with variations in gasoline quality; more concentrated gels less sensitive to gasoline variations, more dilute ones more sensitive.

"Quantitative data showing how little temperature affects the consistency of Napalm gels are presented in Fig. 5 following page 34. This temperature-insensitiveness makes Napalm the only available flame thrower fuel useful for low temperature operation."

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Introduction

This report constitutes a supplement to O.S.R.D. 2036, The Manufacture, Properties and Testing of Napalm Soaps" issued November 17, 1943. A considerable amount of new data has been obtained in the intervening months. Among these are the effect of (1) variations in raw materials, (2) the addition of oxidation inhibitors, (3) the addition of drying agents, (4) variation in gasoline and moisture content on consistency, and (5) temperature on dispersion and consistency. Also presented are data on the Columbia-CWS oxidation susceptibility test.

As before, we are indebted to C.W.S., and in particular Major deGray, for their cooperation in the preparation of this report.

The Manufacture of Napalm

I. The Effect of Variations in the Ratios and Specifications of the Raw Materials Employed.

A. Variations in Ratios of Acids

During the early days of Napalm manufacture, a considerable number of laboratory batches were made in an effort to decide what ratio of acids would give the best results. Many of the data are confused by the effects of oxidation and moisture (which were not appreciated at the time) and do not lend themselves to a reliable analysis.

Recently data have become available (1) which shed some light on the effect of variation of the three acids on the aluminum soap produced. Nineteen laboratory batches of Napalm were made by varying the percentage of each component acid ± 10% from the standard composition of 50% coconut, 25% oleic and 25% naphthenic. Fig. 1 shows on triangular coordinates the variations involved in these experimental batches. Fig. 2 shows the complete data on these batches.

Several interesting conclusions may be drawn from these data. Holding all variables as nearly constant as possible except the percentages of acids used, the strength appears to be a primary function of the amount of oleic acid, with the coconut and naphthenic acids contributing secondary effects. If the 24 hour 150°F. mobilometer values are plotted versus per cent oleic acid on semi-logarithmic coordinates, a straight line correlation is found having the following equation: (2)

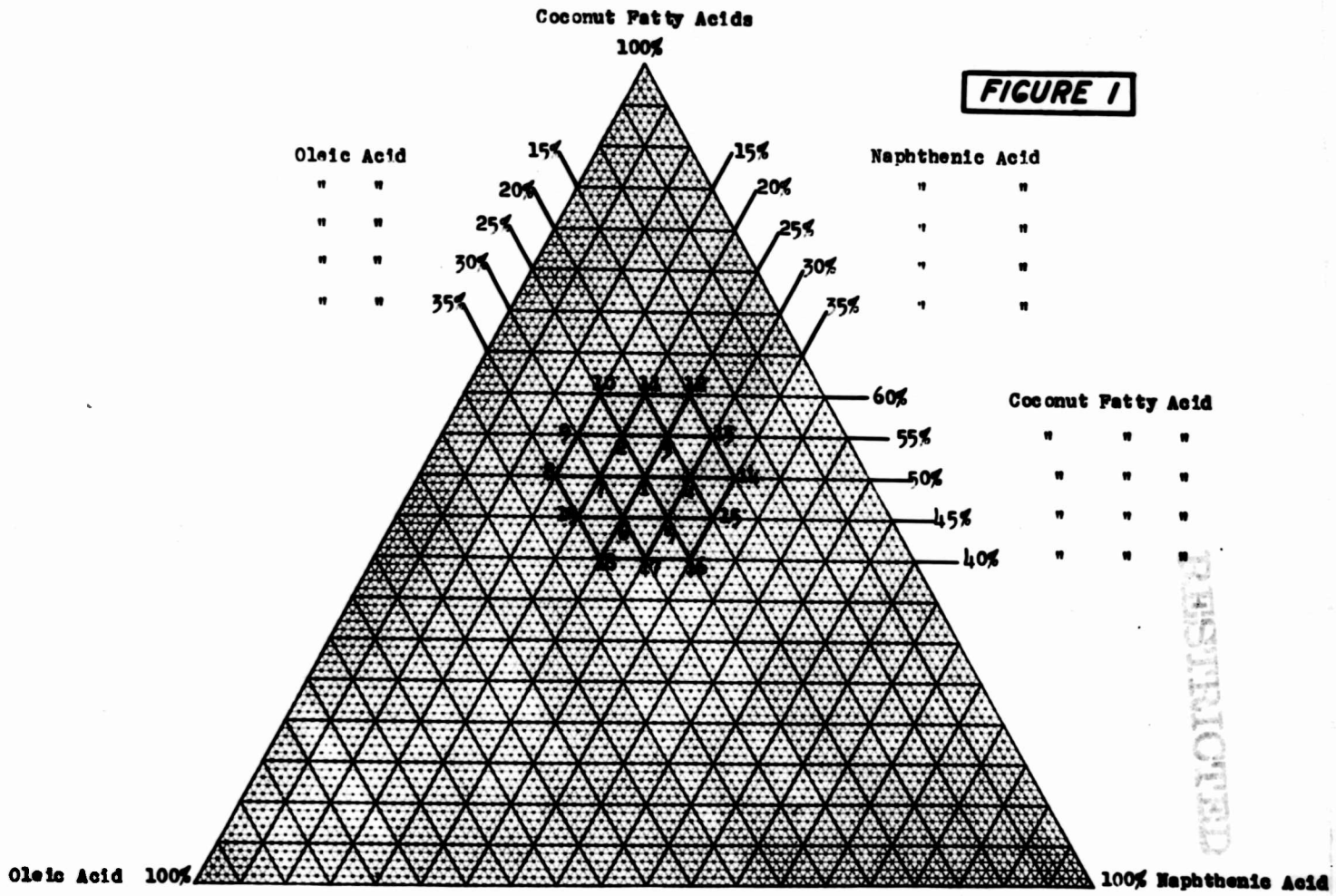
$M = 397 \log A + 110$ where M = 150°F, 24 hour mobilometer consistency
A = % oleic acid.

A more detailed analysis of the data (3) has been made by considering the percentage of each acid as an unknown in the following equation:

$m = aA + bB + cC$ where m = mobilometer consistency,
$A,B,C,$ = percentages of oleic, coconut and naphthenic acids respectively and a, b, c are constants.

Using the method of least squares to solve the nineteen simultaneous equations involved, the following values of the constants a, b and c are obtained:

FIGURE 1



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TRIANGULAR CO-ORDINATE
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NAPALM

Harmon Color Works, Inc.
Belleville, N. J.

FIGURE 2

A report indicating effects on Napalm when the proportions of the three fatty acids used are varied. These variations may be noted from the attached triangular coordinate diagram. Equivalent weights (calculated from acid number) and iodine numbers change with every variation.

Each variation was prepared three times on different days and these three were blended, marked and used for the test. They were stored for sixty days in a sealed glass jar and moisture determinations as well as gel consistencies were made on the material before and after drying to remove any moisture picked up during storage.

In preparing these the caustic soda was left constant and the amounts of fatty acids used were in proportion to the acid number calculated from that particular mixture.

The following method was used in the preparation of each sample:

49 grams NaOH 98% - dissolved in 2800 grams water at 80° C.
Add with agitation -
(See Column 7) " Mixed Fatty Acids
Stir 10 mins. Adjust to 30° C.
Precipitate in 30 mins. with -
135 grams $Al_2(SO_4)_3 \times H_2O$ (Al_2O_3 = 17.5%) H_2SO_4 = 47.7%; Fe=0.001%
dissolved in 470 grams water at 30°C.
After precipitation - stir 10 minutes - filter - wash sulphate free with 3000 cc water - presscake should be about 50% solids - pass presscake through an 8 mesh screen into tray to a thickness of 1 inch - Dry at 100° F for 16 hours - pass dry through an 8 mesh screen.

O B S E R V A T I O N S AND D A T A

Percent Coconut Fatty Acids g278 Iodine g 6.73	Percent Oleic Acid No. 198.6 Iodine g 85.6	Percent Naphthenic acid. Acid No. 227.8 Iodine No. 3.56	Calculated Iodine No. of Mix of Three Acids	Calculated Equivalent Wgt. No. Mix of Three Acids	Wgt. No. Three Acids Dry Mix	Parts Mix-ed Acids Used in Experiments	Napalm Dry Physical Appearance	Gel Value per In. Dry	Tests in Experiment Number (Number of Days Aged) 1 Hours	24 Hours	48 Hours	Moisture	Tests in Series 8 Hours at 160° F (Consistency Prior to Drying) 1 Hours	24 Hours	48 Hours	Moisture
50	25	25	26.7	228.0	10x1122A-1	182.4	Yellow White	295	390	340	410	1.2%	645	740	725	0.5%
55	25	20	26.6	229.5	10x1122B-2	181.2	White	375	320	310	350	1.2%	635	695	735	0.5%
55	20	25	22.9	224.5	10x1122C-3	179.6	White	320	330	320	340	1.3%	595	620	665	0.5%
50	20	30	23.0	227.0	10x1122D-4	181.6	Yellow Fused	260	400	350	410	1.3%	640	625	655	0.5%
45	25	30	26.9	231.0	10x1122E-5	184.8	Dark Fused	260	340	370	400	1.2%	645	660	680	0.5%
45	30	25	30.7	232.0	10x1122F-6	185.6	Yellow Fused	275	410	400	490	1.3%	650	720	740	0.4%
50	30	20	30.5	229.5	10x1122G-7	183.6	White Fluffy	325	390	350	370	1.25%	645	675	720	0.5%
50	35	15	34.2	235.0	10x1122H-8	188.0	White Fluffy	380	450	410	430	1.2%	740	755	780	0.6%
55	30	15	30.3	227.0	10x1122I-9	181.6	White Fluffy	400	370	350	370	1.5%	630	670	720	0.7%
60	25	15	29.4	223.5	10x1122J-10	178.8	White Fluffy	325	340	370	350	1.2%	680	700	720	0.6%
60	20	20	22.7	222.0	10x1122K-11	177.6	White Fluffy	330	340	340	370	1.3%	615	680	690	0.5%
60	15	25	18.6	221.0	10x1122L-12	176.8	White Fluffy	345	370	330	330	1.2%	580	570	635	0.5%
55	15	30	19.2	223.0	10x1122M-13	178.4	Yellow White	295	350	330	350	1.3%	495	585	605	0.4%
50	15	35	19.4	225.5	10x1122N-14	180.4	Yellow Fused	255	400	370	400	1.2%	575	575	595	0.5%
45	20	35	23.3	229.0	10x1122O-15	183.2	Dark Fused	250	380	380	430	1.1%	555	600	645	0.5%
40	25	35	27.0	232.0	10x1122P-16	186.4	Dark Fused	250	370	340	400	1.3%	570	630	695	0.5%
40	30	30	30.7	234.0	10x1122Q-17	187.2	Dark Fused	245	350	450	410	1.2%	615	680	720	0.4%
40	35	25	34.4	240.0	10x1122R-18	192.0	Dark Fused	255	400	400	510	1.1%	680	735	790	0.5%
45	35	20	34.4	237.0	10x1122S-19	189.6	Yellow White	300	400	320	460	1.1%	690	705	785	0.5%

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Acid	2 hrs. 150°F.	24 hrs., 77°F.	48 hrs., 77°F.
a (oleic)	13.1	13.8	14.0
b (coconut)	5.1	5.8	6.0
c (naphthenic)	1.1	1.8	2.0

When used in the above equation, these constants permit the calculation of mobilometer consistencies for these data showing good agreement with experimental values. This analysis makes clear the predominant effect of the oleic acid on the mobilometer consistency. It should be emphasized, however, that these constants apply only to the raw materials and preparation procedure given in Fig. 2, but the mathematical analysis should be generally applicable.

B. Variations in the Acid Value and Quality of Coconut, Naphthenic and Oleic Acids.

The effect of acid number and quality of the component acids has received considerable attention during the past few months, and data are now available as the result of a large number of laboratory preparations (4, 5, 6, 7). The laboratory technique has been held constant and the raw materials varied to as great a degree as possible with the samples available. A standard batch formulation and procedure, corresponding closely to general plant practice, were selected and after a sufficient number of batches had been made to assure reproducibility and satisfactory gel-forming characteristics, the three acids were replaced, one at a time, by other grades of the same acids. Tables I, II, and III show the results obtained with various grades of oleic, naphthenic and coconut acids. In each case all variables except the one being investigated were held constant.

From these data it seems reasonable to conclude that the quality and acid number of the oleic and naphthenic acids (within the limits of the spec. suggested on p. 34 of O.S.R.D. Report 2036) used have little effect on the consistency of the final gel. There is some indication that the acid value of the naphthenic acid may have some influence, but the effect is not clear from the amount of data available. With oleic acid poor results have been obtained with acids of titre lower than 8 and as high as 15. On the other hand, the acid value of the coconut acid is definitely a factor in determining the thickening power of a Napalm soap. Providing that the acid number of the coconut acid used is specified, no difficulty should be encountered in producing satisfactory Napalm from a variety of commericially available acids.

Table I: Oleic Acid

Grade	Source	Acid Value	I_2 No.	Titer °C.	44 hrs. 72°F.	24 hrs. 150°F.	Moisture
Crystolene, Double Dist.	Hardesty	198	93	11.8	815	770	0.55
Redolene-Reg.	"	191	89	10.6	800	825	0.37
" Med.	"	192	86	18.1	820	810	0.46
" Heavy	"	194	79	25.1	810	770	0.40
Sapolene Reg.	"	187	87	11.5	800	815	0.51
" Med.	"	188	85	17.9	780	780	0.40
" Heavy	"	190	79	25.5	810	810	0.45
Special Redolene Reg.	"	192	89	10	805	790	0.68
" " Med.	"	192	83	17.7	780	835	0.33
" " Heavy	"	193	78	25.2	815	705	0.30
" Crystolene	"	193	91	11.8	815	790	0.68
White Oleine	A. Gross & Co.	199	87-90	6-9	705	660	0.33
L.C.P. Red Oil	"	197	88-91	8-10	800	785	0.55
Sap. Red Oil	Wilson-Martin	185	88-90	10-12	825	780	0.55
Dist. " "	" "	190	90	10-12	730	710	0.25
Double Dist. Red Oil	" "	195	90	10-12	810	740	0.48
Sap. O-22	Emery Ind.	175	78-83	15-18	800	790	0.25
Extra Elaine O-18	" "	190	90-93	8-12	825	715	0.40
Olive " O-20	" "	199	90-93	8-12	805	705	0.38
Canary Brand	Proctor-Gamble	198	85-95	5-7	710	580	0.43
				mean	793	758	
				max.	825	835	
				min.	705	580	

Table II: Naphthenic Acid

Grade	Source	Acid Value	I_2 No.	Unsap.	44 hrs. 77°F.	24 hrs. 150°F.	Moisture%
#212 Refined	Gen. Petroleum Co.	272	9.7	<10%	815	840	0.55
200x	" " "	200	18.6	app. 20%	745	705	0.55
Refined	Pennotex Oil Co.	294	10.1	<10%	800	880	0.50
#9110	Texas Co.	190	10.4	30%	725	680	0.51
Special Cut	Stanco	196	10.0	25%	820	805	0.45
Rectified	"	228	15.2	—	750	700	0.43
Crude	St'd. of Cal.	231	14	7.6	800	825	0.50
Semi-ref.	" " "	240	12	4.0	810	810	0.60
Refined	" " "	241	6	3.6	790	815	0.67
				mean	785	785	

* * * * * * * * * *

Table III: Coconut Acid

Grade	Source	Acid Value	Sap. No.	I_2 Value	Titer	44 hrs. 77°F.	24 hrs. 150°F.	Moisture
CA	E.F. Drew Co.	266	267.4	7.7	23.6	750	700	0.43
A	"	279.5	281	8-9	23.2	770	785	0.30
AA	"	348	350	2-3	<10	1150	1050	0.31
AAAR	"	229	250-260	13-15	26.5	785	685	0.45
AAARH	"	AAR hydrolysed				835	805	0.52
A	"	274	274	14.8	23.2	820	780	0.44
AB	"	254	258	18.1	26-27	810	730	0.63
Refined	St'd.of Cal.	268	268	5	22.6	820	800	0.38
Oronite Refined	Griffin Chem. Co.	277	277	5	23.7	795	800	0.50

II. The Effect of Iron on Consistency

A. Presence of Iron in the Acids

Table IV A (5, 6) shows the results of standard batches of Napalm in which iron was added to the acids used, and B gives data on the addition of iron (as ferric sulfate) to the alum both with and without inhibitors (α-naphthol and alphanil). Up to 0.6% iron present in the acids used in making the batch appears to have little effect on the consistency of the resulting gel, and it is probable that the iron is precipitated as ferric hydroxide during the formation of the sodium soaps.

When iron is introduced with the alum used for precipitation, the harmful effect on consistency is apparent at final iron contents greater than 0.1% (approx. 0.15% based on the alum). The presence of 0.2% α-naphthol (based on weight of finished Napalm) does not appear to prevent gel deterioration. It is likely that the effect of iron noticed in these data is due to the dispersing action of the iron salt rather than oxidation since the iodine numbers given are normal. Maximum permissible iron contents suggested elsewhere (8, 9) have ranged from 0.01 to 0.03%; these figures were arrived at by oxidation experiments without taking account of the dispersing effect. Since oxidation is catalysed by amounts of iron considerably smaller than the quantities necessary to cause low gel consistencies by the dispersing effect, the former must be considered the more important and alum specifications should consider primarily this factor.

Table IV: Effect of Iron on Consistency

A. Iron in Acids

Formulation	Iron Coconut	Iron Naphthenic	Iron Oleic	Total Iron in Acids	44 hrs. 77°F.	24 hrs. 150°F.	Moisture
CPU (st'd)	0.0007	0.003	0.008	0.0033	750	700	0.43
CPU-CI6	0.06	0.003		0.033	810	775	0.30
CPU-C13	0.03	0.003		0.018	820	775	0.48
CPU-CI 1.5	0.015	0.003		0.010	770	795	0.43
CPU-I 12	0.0007	0.12		0.032	805	780	0.45
CPU-I 9		0.09		0.024	825	795	0.55
CPU-I 6		0.06		0.017	800	800	0.38
CPU-I 3		0.03		0.010	750	715	0.30
CPU-I 120		1.2		0.30	825	780	0.30
CPU-I 240		2.4		0.60	800	830	0.53
CPU-OI 48		0.003	0.48	0.121	835	850	0.58
CPU-OI 96		0.003	0.96	0.24	800	835	0.43
CPU-OI 128		0.003	1.28	0.32	825	825	0.45

Alum is General Chemical Company - War Grade Iron Free

* * * * * * * * * * * *

B. Iron in Alum

Formulation	Iron Content of Napalm %	Inhibitor % α-naphthol	Initial Iodine Value	44 hrs. 77°F.	24 hrs. 150°F.	% Moisture
CPU-A 11	0.01	0	27.5	815	805	0.73
A12	0.02	0	25.8	805	810	0.38
A 15	0.05	0	27.0	825	785	0.45
A 120	0.20	0	26.6	600	605	0.45
A 160	0.60	0	26.6	390	470	0.45
A 12N	0.02	0.2	27.1	795	785	0.60
A 15N	0.05	0.2	26.4	770	750	0.50
A 110N	0.10	0.2	26.8	800	760	0.65
A 180N	0.20	0.2	27.3	670	650	0.48
A 160N	0.60	0.2	27.1	420	425	0.40

III. The Effect of Oxidation Inhibitors on Gel Consistency

The following table gives results for several laboratory batches of inhibitor-containing Napalm (5, 6).

Table V

Inhibitor	44 hrs., 77°F.	24 hrs., 150°F.	Moisture
0.1% alphanil	855	750	0.47
0.3% "	810	765	0.30
None	795	755	0.45
0.05% α-naphthol	805	770	0.58
0.10 "	800	780	0.35
0.30 "	790	780	0.33
0.80 "	775	750	0.35
1.20 "	770	770	0.53

It appears that the presence of oxidation inhibitors in Napalm has no deleterious effect on the consistency of the resulting gels.

The Properties of Napalm

I. The Moisture Effect

No new findings of great importance have been made recently on the moisture effect. Nevertheless, the conclusions of the previous report have been confirmed and in some cases extended by new work. This may be briefly detailed.

Several times in the past (10), it has been questioned whether or not water taken up by Napalm can be redistributed within the molecule in some way on standing so that it becomes inactive with respect to its effect on the consistency of the gasoline gel. It was also thought that moisture determinations made by a variety of methods might change their relative values due to such rearrangement. To test this point, three soaps were exposed to two relative humidities, 120°F. - 20% R.H. and 85°F. - 65% R.H. for varying periods of time (13). Samples were withdrawn and kept in tightly closed bottles in the same relative humidity rooms so that any small leak would have no effect, and moisture determinations were made by both vacuum oven and benzene distillations (15). No differences whatever in moisture content (in any one series) could be observed. Similar results were obtained with consistency measurements. (Full results are given in Table XVI, Appendix I.) It must be concluded, therefore, that no detectable redistribution of moisture within the Napalm structure occurs on standing, at least with the present available water and consistency determination methods.

Data have been obtained on the equilibrium moisture contents at 20, 50 and 70% R.H. of typical samples of Napalm from the various C.W.S. contractors (16). These, together with consistency results on 4% and 8% gels in Standard Oil Development test gasoline #14, are shown in Table VI. It will be seen that there is considerable variation in moisture content and gel consistency, although in nearly all cases the values for the samples as received were very close to those of the samples conditioned at 20% R.H. This seems to indicate comparative uniformity in packaging conditions and containers.

Table VI: Moisture Content and Consistencies of Typical Napalms

Sample	Per Cent Rel. Hum. at which Conditioned	C.W.S. Moisture	Gardner Consistencies 8% Gels 2 days 77°F. Grams	8% Gels 24 hrs. 150°F. grams	4% Gels 2 days 77°F. grams
Nuodex	As rec'd.	.7%	690	745	175
19889	20	.95	650	690	160
	50	1.2	565	410	90
	70	1.7	355	280	40
Eakins	As rec'd.	.65	540	385	80
N-3-2981	20	.45	640	350	110
431	50	.7	530	320	70
	70	1.0	370	240	27
Imperial	As rec'd.	.7	620	585	90
NR 232	20	.7	620	540	78
	50	.95	410	460	53
	70	1.45	280	320	31
McGean	As rec'd.	.75	770	620	215
462	20	.8	750	700	215
	50	1.45	500	400	62
	70	2.2	250	130	11
Ferro	As rec'd.	.65	745	680	228
184	20	.55	740	640	235
	50	1.0	590		112
	70	1.45	345	540	37
Pfister	As rec'd.	.7	635	615	120
N-3-2432-	20	.7	645	770	135
94	50	1.05	550	505	70
	70	1.30	440	530	65
Harmon	As rec'd.	.7	660	630	140
R11285	20	.75	670	640	155
	50	1.0	530	455	73
	70	1.2	270	250	20
Oronite	As rec'd.	.5	980	645	290
J-33-C	20	.45	1110	645	330
	50	.7	860	715	195
	70	.95	570	400	92
Calif.Ink	As rec'd.	.7	595	465	141
98	20	.8	565	460	130
	50	1.1	375	355	59
	70	1.55	208	310	14
Colgate	As rec'd.	.4	740	570	140
N-3-2854-	20	.5	700	480	125
56	50	.75	610	320	80
	70	.95	450	260	58

Much of the variability in soap consistencies must be ascribed to the moisture effect, but it is thought that one of the methods previously suggested (8), namely, the addition of a small quantity of a dehydrating agent, may serve to overcome this. Of the available dehydrating agents, phosphorus pentoxide, magnesium perchlorate and calcium chloride immediately caused breakdown of Napalm gels. Calcium chloride, while initially satisfactory, causes breakdown of the gel after long time keeping, particularly at elevated temperature. Sodium sulphate, calcium sulphate and alumina are not sufficiently active leaving silica gel and magnesium sulphate as the most promising materials for use (11, 12, 13, 14). Table VII and Fig. 3 show results obtained with these materials. It will be noted that the addition of these reagents makes the drop in consistency on keeping at 150°F. practically zero, even after prolonged storage.

As a result of this work, experiments are now in progress to compare the relative efficiencies of magnesium sulphate and silica gel, the minimum percentage of each required and the reduction in concentration of Napalm which could be made in gels for any given purpose when a dehydrating agent is present.

The form in which the dehydrating agent should be shipped is as yet doubtful. If packed in a separate hermetically sealed container within the Napalm tin, the Napalm could suffer moisture deterioration yet the dehydrating agent when added to the gasoline directly before mixing would pull it up to strength. Mixing of the agent with the soap is also a possibility. Preliminary results (19) indicate that gels made up from Napalm containing silica gel were very satisfactory even after the Napalm-silica gel mixture had been in storage for two weeks.

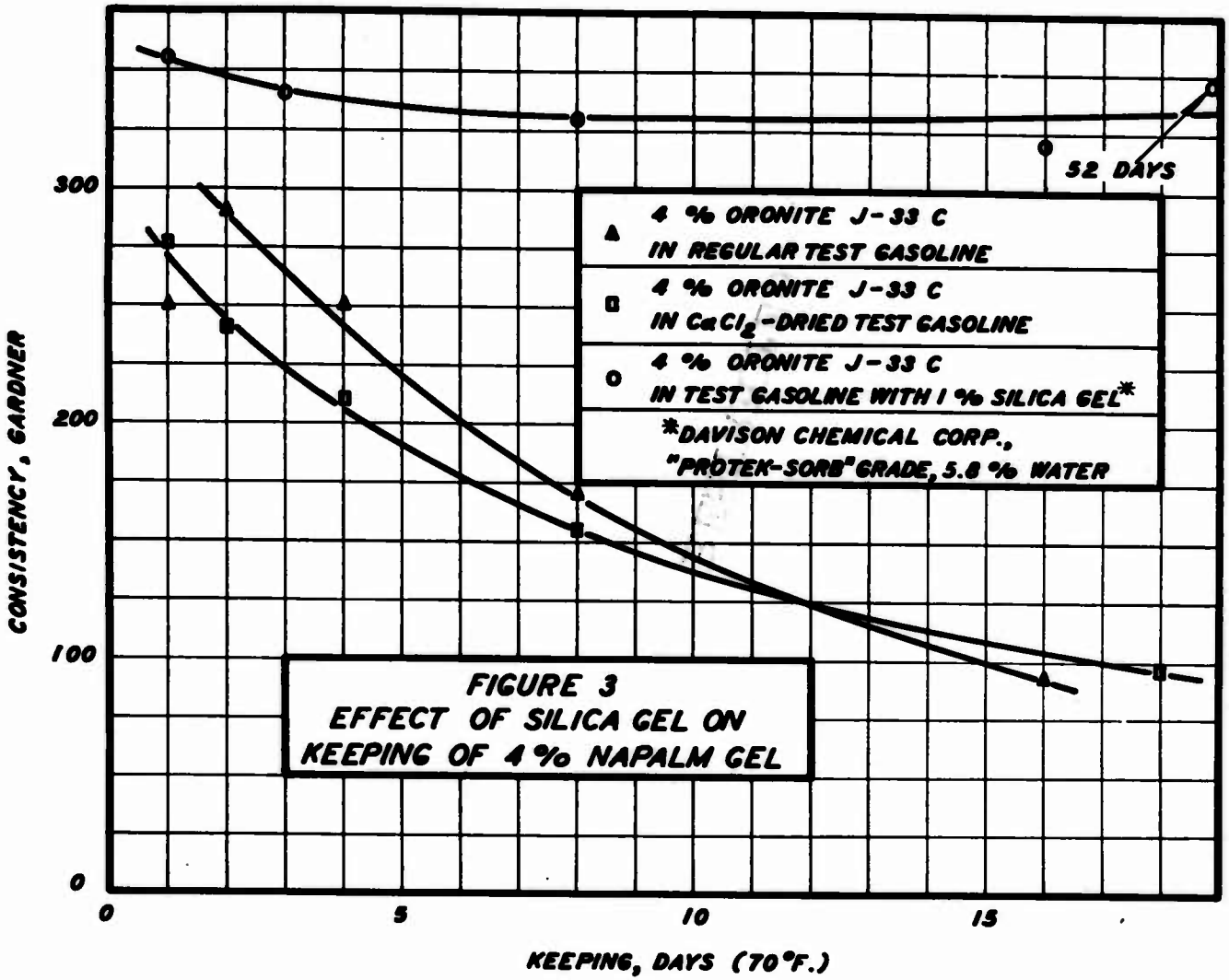

FIGURE 3
EFFECT OF SILICA GEL ON
KEEPING OF 4 % NAPALM GEL

RESTRICTED

Table VII (11, 12, 17): Effect of Drying Agents on 8% Napalm*

Drying Agent**	Days at 150°F.	Gardner Consistency grams
None	1	300
	7	150
1% $CaCl_2$ (anhyd)	1	700
	7	700***
	50	125
1% Na_2SO_4 (anhyd)	1	300
1% "Drierite" ($CaSO_4$)	1	450
1% Activated alumina	1	375
	5	260
3% Activated alumina	1	580
	5	360
1% Silica gel****	1	725
3% Silica gel	1	765
	7	780
	40	550
B. (Nuodex #89093, C.W.S. Moisture 1.1%.)		
None	1 hour	420
	1 day	450
	2 days	480
0.5% $CaSO_4$	1 hour	480
	1 day	480
	2 days	500
0.5% Activated alumina	1 hour	460
	1 day	510
	2 days	580
0.5% Silica gel*****	1 hour	610
	1 day	520
	2 days	570
0.5% $MgSO_4$	1 hour	530
	1 day	540
	2 days	650

*Nuodex #18032. Stored 90°F.-90% R.H. for 6 mos. in standard package. Vacuum oven moisture about 1.7%.
** Percentages based on weight of gasoline.
***Slight deterioration in top 10-20% of gel. Gardner viscosity in this region about 380.
****5.8% H_2O.
***** 9.1% H_2O.

II. The Oxidation of Napalm

Work on the oxidation of Napalm has continued, particularly with reference to causes and prevention of oxidation and the development of simple oxidizability tests.

Two further tests for susceptibility to oxidation have been suggested: (a) an adaptation of the Voorhees test for gasoline (20), and (b) a peroxide value test on the soap after heating for 24 hours at 80°C. in an atmosphere of oxygen (22).

The former has been tried on only a few samples of Napalm, but appears quite feasible. Results are given in Appendix I. The second has been given considerable study both at Columbia University and Eastman Kodak. In its final form, five grams (± 0.1 gram) of the soap to be tested are weighed into a Midvale absorption bulb (Stetser-Norton modification). The bulb is placed in an oven or thermostat regulated to 80°C. and connected with rubber tubing through a bubble counter to an oxygen tank. The bubble counter contains 50% sulfuric acid. A very slow stream of oxygen is passed through (about 50 ml. per minute) for 24 hours. The bulb is then disconnected, the soap transferred to a 250 ml. iodine flask and the peroxide number (mg. iodine liberated per gram of soap) is determined as follows: 50 ml. of glacial acetic acid - chloroform (60-40 by weight) are added and the flask shaken gently to disintegrate any lumps which may have formed. Three ml. of saturated potassium iodide solution are added, the mixture shaken vigorously for 1-1/2 minutes and then 100 ml. of water are added. The solution is titrated with .01 normal sodium thiosulphate solution and the peroxide number calculated according to the following equation.

$$\text{Peroxide No.} = \frac{127 \times \text{volume (ml.)} \times \text{normality}}{\text{weight of sample}}$$

Since an occasional erratic result is obtained, the test should be run in duplicate, although it is permissible to connect the two samples in series for the oxygen treatment. The peroxide number of the original untreated soap should also be determined according to the same method. Results on four typical soaps are shown in Table VIII below.

Table VIII

Oxidation Susceptibility Tests: (22)

Change in Peroxide Number After 24 Hrs. Oxygen Treatment at 80°C.

Soap	Peroxide Number	Peroxide Number After 24 Hrs. 80°C. under Oxygen
t Imperial #NR99	4.3, 4.3	28.1, 28.8
b Imperial #NR99	5.9, 5.4	29.5, 28.3
t Pfister #3-2432-78	1.6	20.9, 13.1
b Pfister #3-2432-78	1.4, 1.5	20.9, 12.8
t Eakins #N3-2981-182	0.3	1.8, 1.1
b Eakins #N3-2981-182	0.3	1.4, 1.0
t Harmon #R11242	0.2	0.9, 0.6
b Harmon #R11242	0.2	0.6, 0.5
t McGean #684	0.3	0.5, 0.4
b McGean #684	0.3	0.5, 0.5
Nuodex #89093	0.2	0.5, 0.5

t - Sample taken from top of drum and stored in a tightly covered jar. The jar had been opened a number of times to secure samples for experimental work.

b - Sample taken from bottom of drum especially for this test.

Surveillance Tests

Peroxide Numbers After Exposure to Air at 65°C.

Soap	Peroxide Number 0 days	7 days	9 days	12 days	14 days	17 days	19 days	21 days	23 days	104 days
Imperial NR99	1.8	17.7		7.4		6.2		3.1		
Pfister #3-2432-78	1.2	18.0	22.0		10.2		8.2		3.9	
Eakins #N3-2981-182	0.2		1.5		2.3		2.3		5.3	
Harmon #R11242	0.3		0.7		1.2		1.3		1.4	
McGean #684	0.4	0.8		1.8		2.5		14.6		
Nuodex #89093	0.2		0.3		0	0.6	0.7			3.0

A more complete study of peroxide number, iodine number and gelling properties of Napalm (Pfister #3-2432-78) is shown in Table IX.

Table IX

Correlation of Peroxide Number, Iodine Number and Gelling of Napalm* Exposed to O_2 at 70°C. (22)

Time Hrs.	Peroxide No.	% H_2O	I_2 No.	48 hrs. 25°C.	1 hr.65°C.	24 hrs.65°C.
0	1.1	0.8	35.7	590	570	570
4	1.2	0.8	35.7	630	610	540
6	1.3	0.8	35.7	---	---	---
24	2.1	0.8	35.2	630	570	540
30	2.4	0.8	---	---	---	---
36	3.5	0.7	34.3	670	610	590
60	6.4	---	33.6	---	---	---
76	7.4	1.0	32.2	700	620	540
82	11.9	0.8	32.2	680	630	640
94	14.9	1.0	30.9	710	580	670
102	17.2	---	29.3	Crumbly (710)	(650)	(640)
110	23.0	0.8	23.5	Too crumbly	---	---
116	22.2	---	21.8	" "	---	---
124	20.2	0.8	18.3	" "	---	---
132	22.1	---	16.1	" "	---	---
156	20.1	---	14.6	" "	---	---
180	17.2	0.8	---	" "	---	---

*Pfister Napalm #3-2432-78.

It will be seen that the soaps studied varied greatly in oxidizability and that the test appears to differentiate the soaps satisfactorily. Furthermore, the iodine value and peroxide value appear to change simultaneously, peroxide value increasing to a maximum and then decreasing. This last point is confirmed by results of McIntyre and Elliott (18, 19) (Figure 4). The properties of the gel appear to be little affected until the iodine number has fallen about ten points, after that the gels lose their string and become excessively crumbly.

As a result of these experiments, Birnbaum and Edmonds (22) recommended the following as a tentative specification for oxidation susceptibility of Napalm.

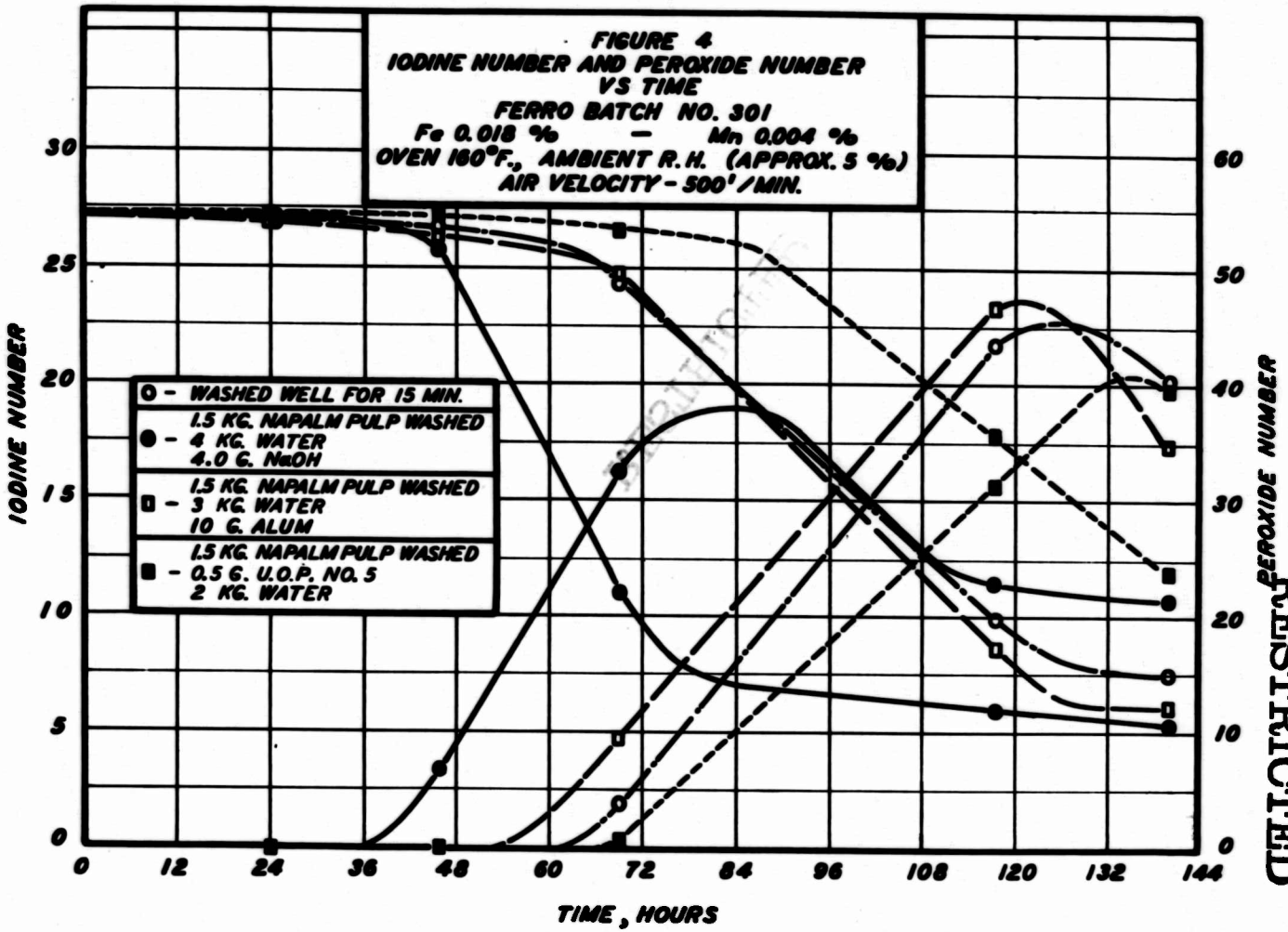
FIGURE 4
IODINE NUMBER AND PEROXIDE NUMBER
VS TIME
FERRO BATCH NO. 301
Fe 0.018 % — Mn 0.004 %
OVEN 160°F., AMBIENT R.H. (APPROX. 5 %)
AIR VELOCITY - 500'/MIN.
○ - WASHED WELL FOR 15 MIN.
● - 1.5 KG. NAPALM PULP WASHED 4 KG. WATER 4.0 G. NaOH
□ - 1.5 KG. NAPALM PULP WASHED 3 KG. WATER 10 G. ALUM
■ - 1.5 KG. NAPALM PULP WASHED 0.5 G. U.O.P. NO. 5 2 KG. WATER
IODINE NUMBER
PEROXIDE NUMBER
TIME, HOURS
0
5
10
15
20
25
30
0
10
20
30
40
50
60
0
12
24
36
48
60
72
84
96
108
120
132
144

RESTRICTED

(a) All soaps having an initial peroxide value above 5 shall be rejected.

(b) After the 24 hours at 80°C. oxygen treatment, all soaps with peroxide numbers
(1) below 1.2 shall be considered satisfactory,

(2) between 1.2 and 5 shall be marked for manufacture of the gels within two weeks, and in no instances be shipped for type A use, and

(3) over 5.0 shall be rejected.

At Eastman Kodak a large number of soaps received from the filling plants and other sources have been tested for oxidizability by three methods: namely, the Mackey (100°C.), the Modified Mackey(130°C.) and the peroxide value test just described (14). Simultaneously, long time keeping tests at 120°F. - 20% R.H. have been made following the iodine value of the soap. (Appendix I). In general, the relation between these tests and long term oxidizability appears to be good, particularly for the two month keeping times. The chief discrepancies have been noted for the Eakins soaps, several of which showed induction periods and high peroxide values after the Mackey test, but which failed to show oxidation after two months' keeping. The peroxide value test, possibly because it was run on fewer soaps, shows almost perfect correlation if 1.0 rather than 1.2 be taken as the value for rejection of the soap.

Since the writing of O.S.R.D. 2036 the addition of alpha-naphthol to all soaps during manufacture is required by C.W.S. Specification 196-131-107 A. The results of Tables V and XI indicate that this material has no deleterious effect on the consistency of gels made from soaps containing it and that it is beneficial as an oxidation inhibitor, as has been shown by experiments detailed in the previous Napalm report. Some results on a large scale batch containing alphanil as an inhibitor are summarized in Table X. It will be seen that to date, because of the excellent stability of the check, little distinction between the various batches with regard to oxidizability can be made.

Table X: Ferro Batches Containing Inhibitors

Batch No.	Ind. Period	at 100°C Heat Rise	Perox. Resi-due	Ind. Period	at 130°C Heat Rise	Perox. Resi-due	Columbia Test	Iodine No. Orig.	Iodine No. 1 mo.	Iodine No. 2 mos.
531	24	—		1.5	1°			30.2		
532	24	—	5.4	0	3°	20.2	.18,.20	28.7	28.7	
α-Naphthol .1%										
533	20	0.5	25.5	0	1°	14.9	0.25	29.0	28.1	
403	24	—	7.5	4	-	13	.3, .3	29.2	27.7	27.8
404	24	—	9.1	6	-	12	.7, .3	29.0	29.0	27.8
Alphanil .1%										
405	24	—	8.0	6	-	16	.2, .3	29.0	28.2	27.3

	As Received		Conditioned at 120°F.-20% RH		
	Moisture	24 hrs. 150°F.	Moisture	44 hrs. 77°F.	24 hrs. 150°F.
531	0.78	810,840			
532	0.66	720,740	0.48	840,880	770,730
533	0.54	680,700	0.49	820,850	750,730
403	0.73	---			
404	0.72	680,720			
405	0.70	650,680			

Table XI (23) shows the effect of α-naphthol on the oxidation susceptibility of four Napalms as determined by the Columbia Test (22). Iodine numbers are also shown for comparison. It is evident that the inhibitor has a beneficial effect on those soaps susceptible to oxidation by this test.

Table XI: Columbia CWS Lab. Oxidation Susceptibility Test (23)

	Ferro		Pfister		Eakins		Nuodex	
	(1)	(2)	(1)	(2)	(1)	(2)	(1)	(2)
Peroxide No. Before	0.4	0.3	0.7	0.4	3.4	0.9	0.2	0.4
" " After	0.5	0.4	1.1	0.4	6.1	0.5	1.0	0.5
Iodine No. Before	27.1	26.9	30.6	27.9	39.1	37.6	25.2	27.7
" " After	27.3	26.6	--	--	38.6	38.0	26.0	28.0

(1) Without α-naphthol
(2) With " "

* * * * * * * * * *

Table XII: The Effect of Metallic Ions on Napalm Oxidation

Metal Salt Added % Metal in Al Soap			Peroxide No.	
Alum Solution	Soda Soap Soln.	% H_2O	Original	24 hrs 80°C. in O_2
--	--	0.3	0.2	0.0
.004% Mn(ous)		0.3	0.1	0.2
.009% Mn (ous)		0.3	0.1	0.2
.018% Mn (ous)		0.4	0.3	32.5
.045% Mn (ous)		0.3	0.3	31.9
.02% Cu(ic)		0.6	0.2	0.3
.06% Cu(ic)		0.5	0.6	0.6
.10% Cu(ic)		0.4	0.8	0.9
.005% Cr(ic)		0.4	0.1	0.1
.01% Cr(ic)		0.6	0.1	0.1
	.05% Pb(ous)	0.2	0.1	0.2
	.10% Pb(ous)	0.3	0.1	0.2
	.15% Pb(ous)	0.4	0.1	0.1

According to Southern and Roth (9) alpha-naphthol does not protect completely against the presence of manganese and iron in the alum solution used for the precipitation. (Table XII) On the other hand, Dickenson and Long (7) report that 0.2% alpha-naphthol is effective in reducing Napalm oxidation in presence of iron (Table XIII). Nevertheless, it is recommended that any alum used, even though alpha-naphthol or other inhibitors be present, should contain as few metallic impurities as possible and in particular that the manganese content be kept below .01% and the iron content below .03%. This is confirmed by practical manufacturing difficulties which have been experienced by one C.W.S. contractor due to excessive manganese in the alum used.

TABLE XII Continued

% Metal in Al Soap			Peroxide		Fines Thru Stan. 40% Sieve, %	Specification		
In $Al_2(SO_4)_3$ Solution	In soda soap	% Water	Orig.	24 hrs. 80°C. in O_2		1 hr. 150°F.	24 hrs. 150°F.	48 hrs. 77°F.
—	--	0.3	0.2	0.0	—	—	—	—
.025% Fe(ous)	--	0.5	0.4	0.4	2.0	550	530	580
.075	--	0.4	0.8	1.0	—	—	—	—
.025% Fe(ic)	--	0.5	0.4	0.4	1.2	500	530	670
.075% Fe(ic)	--	0.4	1.1	13.5	—	—	—	—
	0.125%	0.5	0.2	0.1	1.5	670	650	730
	0.000* (added as iron stearate)	0.6	--	--	8.4	650	660	770
	0.125%+ Fe(ous)	0.7	0.2	0.1	6.8	600	600	650
	0.125%** Fe(ic)	0.7	0.2	0.1	5.6	620	570	650

*Iron stearate added but precipitate allowed to settle, soap made from supernatant liquid.

+ Iron dissolved in oleic acid under nitrogen.

**As under + but solution exposed to air overnight.

Table XIII (7)

Effect of α-Naphthol on Oxidation of Napalm in Presence of Iron

Iron Content of Napalm,%	Inhibitor Content,% Alpha-Naphthol	Initial Iodine Value	After Aging 8 days 120°F.	19 days 120°F.	36 days 120°E.
.01	0	27.5	28.4	26.9	25.2
.02	0	25.8	27.0	27.1	25.9
.05	0	27.0	27.5	27.4	26.9
.20	0	26.6	26.8	23.8	16.3
.60	0	26.6	26.4	22.7	12.2
.02	.2	27.1	27.3	28.9	27.0
.05	.2	26.4	27.4	28.5	26.9
.10	.2	26.8	27.4	26.6	26.6
.20	.2	27.3	28.6	29.6	26.5
.60	.2	27.1	27.7	29.9	25.8

Experiments at Ferro Enamel (Figure 4) indicate that poor washing or too high a pH of the pulp after washing may be conducive to easy oxidation.

The effect of temperature on induction period is also indicated by the Ferro (19) experiments, increase of temperature from 160° to 195°F. cutting the induction period to approximately one-quarter of its initial value.

It had been thought that the quality of the oleic acid used in making Napalm might play a role in its susceptibility to oxidation. This is probably true, although the effect is masked when alpha-naphthol is used as an inhibitor. In a series of experiments at C.W.S. Columbia Laboratory (24), a number of oleic acids were used for the preparation of Napalm. The acids had widely varied susceptibility to oxidation as shown by the Mackey test, as did their pure aluminum soaps; however, the Napalms prepared therefrom showed no significant difference in induction period. The reason for this undoubtedly lies in the fact that alpha-naphthol was used as inhibitor in all the experiments.

The inhibiting effect of the anti-oxidants present in naphthenic acid has again been shown by Shell (25). Mixtures of oleic with 4, 8, 16 and 24% of naphthenic acid had induction periods of 2.5, 3.9, 5.3 and 7.5 hours, respectively.

Fundamental Properties of Napalm

Some interesting work has been carried on at Stanford University (26,27). X-ray patterns of a number of pure soaps have been investigated and compared with that of Napalm. Napalm and aluminum dilaurate $Al(OH)L_2$ show practically identical diffraction patterns, suggesting that this soap may well be one of the main consitiuents of Napalm. An oxidized sample of Napalm shows a somewhat different pattern indicating a definite change has occurred.

The osmotic pressure of aluminum dilaurate in 1% solution in benzene has been studied. The results showed an unexpectedly high temperature coefficient for the osmotic pressure of such solutions. At 18°C. The osmotic pressure was slightly less than 1 mm., while at 25° it was about 75 mm. This indicates that the degree of association of the aluminum dilaurate in benzene varied from about 30 at 40°C. to over 6000 at 18°C. Such a continuous increase in particle size leading to the formation of particles with a molecular weight as high as one million, must correspond to a continuous transition from a liquid sol or solution to a jelly having an elastic structure.

III. The Dispersion of Napalm

Experiments are being continued at Standard Oil Development on the influence of gasoline quality on gel consistency. While not yet complete, the following preliminary statements may be made (28).

1. The consistency of Napalm gels varies with the soap and gasoline.

2. There is a change in consistency on aging which is most pronounced at lower concentration. This aging effect is shown by all gasolines but it is not so prominent with pure hyrocarbons.

3. All gels appear to attain a minimum consistency.

The precise effect of each of these variables has not yet been ascertained but for three gel consistencies the change due to variations in gasoline or hydrocarbon quality is shown in Table XIV.

Table XIV

Soap Conc. %	Variations in Consistency (Grams Gardner) due to Gasolines Min.	Gasolines Max.	Pure Hydrocarbons Min.	Pure Hydrocarbons Max.
4	40	250	110	390
8	600	900	550	1060
12	1250	1700	1300	2230

It is apparent that aging and gasoline quality cause a wide variation in viscosity. Undoubtedly, the third variable (variation in soap) will further increase this range. Some of the change due to moisture content may be lessened by use of dehydrating agents (Table VII, Fig. 3).

It is as yet uncertain how great an effect such a variation has upon performance.

The Testing of Napalm

There have been few developments of any importance in knowledge concerning Napalm testing and specifications. Compounding temperature apparently influences consistency but little (29) .

Further results have been obtained on the influence of temperature upon Gardner consistency. (Appendix II).

Birnbaum and Edmonds (10) have compared the Karl Fischer, C.W.S. Benzene Distillation, and vacuum drying on a number of soaps, reaching the conclusion that no method is absolute and can be related definitely to a certain definite type of water in the soap. The Karl Fischer method is reproducible for any given Napalm and will give satisfactory results, although they will be higher than those shown by benzol distillation.

A new specification, C. W. S. 196-131-206, has been issued covering gasoline for use in testing Napalm according to the methods described in C.W.S. 196-131-107A. This gasoline is designed to replace the standard test gasoline previously furnished by Standard Oil Development Company. While a somewhat higher boiling material than the previous test gasoline, it gives comparable consistency results (Table XV). It does not give the same dispersion times, being slower, than the old standard when a soap is tested for this factor according to CWS 196-131-107.

Table XV (23)

Comparison of Old and New Standard Gasolines

Nuodex Batch #88955R

Date	Gasoline	Gardner Consistencies 1 hr., 150°F.	24 hrs., 150°F.	48 hrs. 77°F
Dec. 6	Conoco (New)	690	590	730
Jan. 4	" "	680	650	700
Sept. 10	SOD (Old)	690	640	700
"	SOD (Old)	730	600	690
Jan. 17	Conoco (New)	660	640	680
"	" "	650	630	680

APPENDIX I.

Properties of Napalm

(a) Equilibrium of Moisture in Napalm

It has been questioned several times in the past whether or not water taken up by Napalm can be redistributed on standing in some way within the molecule so that it becomes inactive with respect to its effect on the consistency of the gel formed in gasoline. It has also been thought that such "unactivated" moisture might not show up in vacuum oven measurements, while it could be determined by the benzene distillation method. With this in mind, the experiment reported in Table XVI was devised. Three soaps were taken and exposed to two relative humidities, 120°F. - 20% R.H. and 85°F. - 65% R.H., for varying periods of time. Samples were withdrawn, kept in tightly closed bottles in the same relative humidity rooms so that very small leaks would have no effect, and moisture determinations were made by both vacuum oven and benzene distillation methods. It will be seen that no differences whatever in moisture content could be observed. Similar results were obtained with consistency measurements shown in the lower part of the table, but these were not continued beyond the third day because of the labor involved.

It can be concluded from this experiment that Napalm, after reaching equilibrium, shows no further changes which may become apparent through consistency or moisture content determinations even after long standing. An interesting side light from this experiment was the much lower susceptibility of the Oronite soap to atmospheres of high relative humidities. This may possibly be due to the rotating cylinder method of drying employed.

Table XVI: Moisture Determinations on Soaps Exposed to Different Relative Humidities

	Original			Kept 3 da. in Tightly Closed Bottle After Exposure			Kept 7 da. in Tightly Closed Bottle After Exposure		
	McGean #462	Harmon R11285	Oronite J-33-C	McGean #462	Harmon R11285	Oronite J-33-C	McGean #462	Harmon R11285	Oronite J-33-C
Original									
Vac. Oven*	.72,.71	.80,.77	.47,.48						
Original CWS	.8, .8	.8, .6	0.8						
Exposed 1 day 120°F. -20% R.H.									
Vac. Oven	.81,.79	.62,.61	.34,.36	.83,.83	.70,.69	.38,.36	.79	.70	.36
CWS	1.0,1.0	.8, .7	.6, .6				.9, 1.0	.7, .7	.6, .6
Exposed 3 days 120°F.-20% R.H.									
Vac. Oven	.79,.77	.57,.56	.33,.31	.80,.80	.63,.66	.37,.38	.73	.55	.32
Exposed 7 days 120°F.-20% R.H.									
Vac. Oven	.76,.72	.50,.46	.29,.32	.74	.56	.30	.82,.79	.55,.56	.32,.21
CWS	1.0,1.0	.8, .7	.6, .6				1.0,1.0	.7, .7	.5, .5
Exposed 1 day 85°F.-65% R.H.									
Vac. Oven	2.06,1.97	1.37,1.4	.73,.74	2.05,2.06	1.47,1.47	.73,.74	2.03	1.42	.74
CWS	2.0,2.3	1.3,1.3	1.0,1.0				2.0,2.1	1.5,1.5	1.0,1.2
Exposed 3 days 85°F.-65% R.H.									
Vac. Oven	1.90,1.86	1.45,1.43	.73,.69	2.06,2.0	1.65,1.62	.79,.79	2.02	1.58	0.75
Exposed 7 days 85°F.-65% R.H.									
Vac. Oven	1.95,1.88	1.58,1.56	.71,.72	1.93	1.34	.74	2.01,1.97	1.35,1.36	.75,.75
CWS	2.2,2.2	1.4,1.3	.9, .9				2.1,2.3	1.8,1.8	1.0,1.0

*24 hrs. 60° C. 10 mm.

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Table XVI Continued

				Kept 3 days in Tightly Closed Bottle After Exposure		
	McGean #462	Harmon R11285	Oronite J-33-C	McGean #462	Harmon R11285	Oronite J-33-C
Original						
24 hrs. 150°F.	620,620	620,640	650,670,620			
48 hrs. 77°F.	680,660	720,700	960,960			
Exposed 1 day 120°F.-20% R.H.						
24 hrs. 150°F.	600	600,680	815,720	600,630	630,580	720,710
48 hrs. 77°F.	930,940	720	1080			
Exposed 3 days 120°F.-20% R.H.						
24 hrs. 150°F.	620,570	630,590	710,670	600,620	640,700	800,810
48 hrs. 77°F.	830,830	760,740	920,910			
Exposed 1 day 85°F.-65% R.H.						
24 hrs. 150°F.	240,180	240,230	400,450	220,260	270,290	450,430
48 hrs. 77°F.	295,310	390,370	740,760			
Exposed 3 days 85°F.-65% R.H.						
24 hrs. 150°F.	200,210	200,210	410,430	230	210,260	450,275
48 hrs. 77°F.	325,315	300,290	710,730			

(b) Prevention of the Moisture Effect

The main results on the prevention of moisture deterioration of Napalm gels by the addition of dehydrating agents have been described in the body of the report. Nevertheless, Table XVII, showing the efficiency of magnesium sulphate, and Table XVIII, showing some further results with silica gel may be of interest. The latter table indicates that it may be possible to reduce significantly the percentage of Napalm required in gels for any given purpose. Thus, if 5% was formerly required for the flame thrower, 4% may be sufficient to give the same Gardner consistency if a dehydrating agent is present. Unfortunately, no direct comparison of silica gel and magnesium sulphate is as yet available, since the silica gel used in R. W. Little's (17) studies had too high a moisture content (0.1%) to be effective.

Table XVII: Effect of Addition of Magnesium Sulphate to Gasoline on Gel Consistency

Hours Soap* Exposed to Air	CWS Moisture Content	% $MgSO_4$	Gardner Consistencies grams 2 days	9 days	16 days	50 days
0	0.9	none	520	530	580	640
		0.8	690	680	800	740
4	1.4	none	370	400	430	570
		0.8	640	740	850	750

*McGean #684.

Table XVIII: Effect of Silica Gel* on 8% Napalm Gel Consistency (After 8 Days' Storage at 75°F.)

Batch No.	Soaps Conditioned at 90°F. - 70% R.H.			Soaps Conditioned at 90°F.-20% R.H.		% Increase in Consistency with Silica Gel	
	Moisture Content of Soap (CWS Benzene Dist.)	No Silica Gel	3% Silica Gel	Moisture Content of Soap (CWS Benzene Dist.)	No Silica Gel	over 70% R.H.	over 20% R.H.
Oronite J-33-C	0.9	760	1350	0.4	990	78%	36%
Pfister N-3-2432-94	1.3	490	810	0.7	675	65%	20%
Ferro 184	1.4	445	850	0.6	770	91%	10%
Imperial NR232	1.4	330	850	0.7	640	160%	33%
Harmon R11285	1.2	310	960	0.8	690	210%	39%
McGean 462	2.2	280	1000	0.8	720	257%	39%
Calif. Ink 98	1.6	235	720	0.9	560	207%	29%

*Davison Chemical Corp. Water content 5.8%

(c) Oxidation of Napalm

The Voorhees test for the oxidation stability of gasoline (30) can apparently be adapted for use with Napalm. Three samples of Napalm, one of which was known to oxidize badly on keeping, were examined with the results shown in Table XIX, below.

Table XIX (20): Oxidation of Napalm and the Voorhees Test

	Induction Period Hrs.	Iodine Number: Before Oxid.	After Oxid.	Orig.	After keeping at 120°F. for 1 mo.	2 mos.
Pfister #N-3-2432-94	5	34.1	31.0*	32.9	16.7	14.8
	5.5	--	21.1**			
Ferro No. 184	None	28.1	25.1**	30.5	28.2	29.2
	None	--	--	--	--	--
Nuodex No. 19869	14	--	--	--	--	--

*4 hrs. 35 min.
** 71 hrs.

The unstable soap showed a definite induction period of about five hours, while the stable sample continued to oxidize slowly for 71 hours. A third soap on which no long time keeping results are available gave an induction period of 14 hours. There was no apparent change in the appearance of the stable sample, while the unstable sample showed a slight change in color, and the Nuodex sample sintered somewhat. The results are tabulated above with iodine number before and after oxidation, determined by the Hanus method. They indicate that the test has possibilities for measuring oxidation stability of Napalm. Advantages compared with the metal bomb test are: more readily available glass equipment, simplified procedure, and more rapid temperature equilibrium.

Oxidizability tests have been run on a number of soaps received from the filling plants. Mackey tests at 100°F. and 130°C. were run on most of the soaps, together with a peroxide value determination on the soap residue after completion of the tests. Peroxide values were run on some of the samples according to the method suggested by Birnbaum and Edmonds (22). The results are summarized in Table XX. Also given in the Table are the iodine values of the soaps, initial

and after exposure for 1, and 2 months in a thin layer at 120° F. - 20% R.H. It appears that any of the oxidizability tests of this table might be used if necessary to predict long term behavior of the soap. However, the inclusion of alpha-naphthol as an inhibitor in all Napalm batches, probably renders further study and adoption of a test for specifications unnecessary.

Table XX

Soap	Fe	Mn	I.P. hrs.	Temp. Rise °C.	Per. No. Residue	I.P. hrs.	Temp. Rise °C.	Per. No. Residue	Columbia	I_2 Value Orig.	1 mo Aged at 120°F. 20% R.H.	2 mo.	Peroxide on Soap As. Rec'd.
McGean													
#462	.018	.0017	3	1°	36	0	3.4°	24	2.3,5.4	26.9	16.0	12.1	2.5
#760	.030		4	3					2.7, 4.4				
#1039	.018		1	3	33	0	29	21	20,14	27.7	10.0		
#1094	.024		6	2		0	5	23	3.9,3.1	28.8	7.5		
#1125	.027		5.5	4	45	0	1	12.5		27.5	12.8		4.0
#1160	.021		6	1	71					28.0	10.4		
#1169	.030		6	6		0	2			26.9	19.5	12.0	
#1220	.015		7.5	>2		0	1	16.4	1.2,1.1	27.9	10.5		
#1241	.025		2	6.5						28.1	16.0	9.0	1.8
#1264	.016		3	1.5	68					27.5	8.3		
#1418	.018		>24	–	61					27.4	12.5	8.2	
#1434	.022		2.5	2.8	40	0	9	17		27.5	14.1	9.5	
#1473	(.025,.022)		6	1	59	0	9	22		25.7	11.3		
#1577	.024		1.75	3	36	0	10	12		25.1	14.6	9.5	
#16939	&.022		1	1.5	60	0	13	14	19.6,21.6	25.9	10.1	7.6	
16843*	(see p. 33)									26.3	12.8		
Ferro													
#2	.021		6	2	70	0	13			27.9			
#34	.090		6	1	54					28.2			
#67	.019		5.5	5	61.2	.8	6	25.9	1.0,2.0	28.4	9.5	6.2	
									1.9,11				
#184	.016	<.0005	>24		6.6	>6	–	22	1.7,0.5				
#360			?	0.5?	10.5	none		11.2	0.9,0.9	30.5	28.2	29.2	0.9
#403			>24	–	7.5	>4	–	13	.18,.15	30.2			
#404			>24	–	9.1	>5.5	–	12	0.3,0.3	29.2	27.7		
#404			>24	–	3.7	1	0.5	11.9	0.7,0.3	29.0	29.0		
#405			>24	–	8.0	>6	–	15.9	0.7,0.5	29.7			
#478			>24	–	3.8	>5	–	11.6	0.2,0.3	29.0	28.2		
#479	.005		>24	–	1.0	>4	–	8.6	0.3	31.5	30.5		
#449			>24	–	4.3	6	–	15.4	.03,.23	28.8	27.7		
									.15,.15	30.2			

Table XX Continued

Soap	Fe	Mn	I.P. hrs.	Temp. Rise °C.	Per. No. Residue	I.P. hrs.	Temp. Rise °C.	Per. No. Residue	Columbia	I_2 Value Orig.	1 mo. Aged at 120°F. 20% R.H.	2 mo.	Peroxide on Soap As Rec'd.
Ferro Cont.													
#531			>24	–	8.4	1.5	1	13.1	.15,.15	30.2			
#532			none		5.4	0	3	20.2	.18,.20	28.7			
#533			20	0.5	25.5	0	1	14.9	.25	29.0			
#585	.020		>24	–	15	0	1	16.4		31.4	31.0		
Imperial													
#NR 69	.013	.0001	7	0.5	32					28.4	29.2	23.8	
#NR 91	.032	.0002	>36							32.9	31.2		
#NR 111	.025	.0058	2.5	5						30.9	8.7		
#NR 219	.015	.0071	0	6.7	32	0	32	8.9	16.9,17.0	23.9	7.8	6.4	28.4
#NR 232	.015	.0010	6	1	21	0	1	10	1.1,1.1 1.1,1.6	30.2	19.3	18.9	2.1
#NR 245	.013	.0007	>24	–	18					29.4	28.2		
#NR 246	.013		3	2	40	0	1	4.6		28.4	11.0		
#NR 362			>24	–	15	>6	–	1.6	.25,.25	29.7	28.9		
#NR 358	.013		>24	–	11	>4.5	–	3.0		29.0	28.9		
Hermon													
#10532	.017		>24	–	4.9	>5	–	15.1	.23,.25	30.3	28.4	28.9	
#R10543	.034		>8	–						30.5	29.0		
#R10567	.017, .019		>24	–	3.4					28.3	27.8	26.7	
#R10576	.016,.021		>24	–	3.4	>4	–	16		27.9,	28.1	27.4	0.6
#R11202	.016		>24	–	2.9	>6	–	10.7	.18,.25	29.8	27.7		
#R11251	.019		>24	–	4.4					29.4	29.2	27.2	
#R11254	.021		>24	–	5.9					30.2	--	29.0	
#R11285	.094	<.0005	>24	–	3.4	>6	–	13	.6,.6, .8,.6	32.0	29.5	29.5	1.8
#88836	.019		11	–	36	0	4.8	21	1.6,1.7	31.3	30.2		
Oronite													
J-33-A	.025,.021		4	1	76					23.8	22.0	10.2	
J-33-C	.020	<.0005	3	1	71	0	10	29	1.9,1.8 1.8,2.0	23.3	22.1	12.8	1.9
California Ink													
#68	.022		5.5	1	65	0	3	27		27.4	25.8	17.2	
#74	.014		>24	–	67				0.9,1.0 1.5,1.5	26.1	25.0	13.8	
#98	.019	<.0005	9	1	74	0	11	33	1.1,1.0	27.6	25.9	18.7	1.9,1.6

Table XX Continued

Soap	Fe	Mn	I.P. hrs.	Temp. Rise °C	Per. No. Residue	I.P. hrs.	Temp. Rise °C.	Per. No. Residue	Columbia	I_2 Value Orig.	1 mo. Aged at 120°F. 20% R.H.	2 mo.	Peroxide on Soap As Rec'd.
Pfister													
#98	.022		2.5	2						35.0	15.8	14.1	
#106	.018		>33							34.4	32.2	30.7	
#N-3-2432-94	.020	<.0005	2.5	2	29	0	8	20	8.5,7.1	32.9	16.7	14.8	3.2
Eakins													
#60	.032		>24	-	50					42.0	40.0		
#116	.014		7	0.5	51					40.7	40.2		
#216	.034		8	0.5	51	Fuse			1.1,1.7	42.5	38.0		
#260	.014		7	0.5	33	Fuse				42.6	40.9		
N-3-2981													
-287	.015		0	0.5	54					42.9	40.1		
-397			>24	-	30	Fuse		5.0	0.4				
-397			14	1	49.8			5.7	.50,.35	40.5			
Huntsville**													
#342 lot 821	.017		>24		3.3					30.9	29.7		
#158 lot 889	.016		>24	-	3.4					29.7 30.2	29.7		
#389 lot 972	.013		>24	-	16					32.9	31.2	28.2	
#401 lot 1000	.014 .022		1.5	4	15				15.6,20	20.5 20.7	8.3	5.1	24

*This cannot be the correct McGean batch number, but was the only information on the bottle as received.

**Maker not known, therefore listed under filling plant from which it was received.

Note: I.P. is induction period in hours
Temp. Rise is from modified Mackey-Test
Per. No. Residue is Columbia-CWS Test on Mackey Residue

APPENDIX II

Testing and Specifications

From time to time the question has been raised as to whether the temperature of dispersion has any significant effect upon gel consistency. In order to determine this, four samples of Napalm, covering a dispersion range of fifteen seconds to eight minutes at 70°F., were made up in standard test gasoline at temperatures of 50, 70 and 90°F. To avoid evaporation or condensation of moisture at the low temperatures all the gels were made up in individual sealed mason jars and shaken by hand before transferring to the iron pipes for specification measurements. The consistency of the gels (8%) was measured according to C.W.S. Specifications after 24 hrs. at 150°F. and 44 hrs. at 77°F. The data are shown in Table XXI.

Table XXI (29)

Gardner Consistencies

	24 hrs. 150°F.			44 hrs. 77°F.		
Sample	Made up 90°F.	Made up 70°F.	Made up 50°F.	Made up 90°F.	Made up 70°F.	Made up 50°F.
Imperial NR 232	620	620	520	600	625	590
Nuodex 19889	640	645	625	760	805	790
Eakins N-3-2981-431	355	385	375	480	525	475
Calif. Ink 98	415	465	395	575	570	575

All of the samples stored at 77°F. checked within 50 grams Gardner over the 50° to 90° F. range of makeup temperatures. With one exception, good checks were also obtained on the 150°F. test. The single low point may be due to tube leakage. It seems probable, therefore, that over the range of mixing temperatures normally encountered variation in dispersion temperature has but little effect.

Further results have been obtained on the variation of Gardner consistency with temperature (Fig. 5) (14). It appears that variation with temperature is not very pronounced, thus giving Napalm one of its outstanding advantages over other thickening agents.

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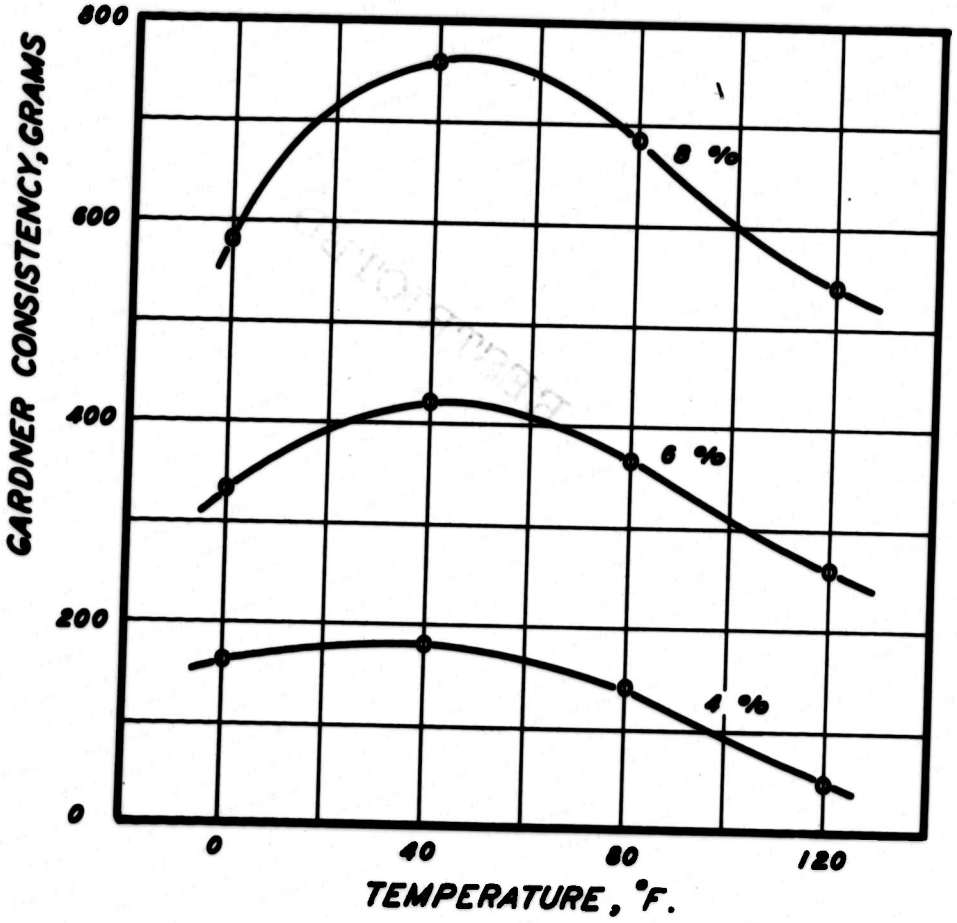
FIGURE 5
TEMPERATURE-CONSISTENCY
VARIATION OF NUODEX BATCH NO. 19889
(S.O. D. TEST GASOLINE)



APPENDIX III

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ABSTRACT:

A study of manufacture and properties of Napalm soaps is presented. The study is concerned with the effect of variation in the ratios and specifications of raw materials employed, and the effect of oxidation inhibitors of gel consistency. Relationships existing among gasoline quality, moisture content, concentration, and consistency, are investigated. It was found that varying the composition of Napalm from the standard to 2:1:1 ratio of coconut to oleic to napthenic acid, indicates that the viscosity of the gel increases primarily with increased oleic acids and to a lesser extent with increased coconut acid above normal composition. The acid number of the coconut acid has been found important.

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