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National Lefense Research Committee

of the

Office of Scientific Research and Levelopment

Report on "The Preparation and Properties of Aluminum Naphthenate Soaps."

by

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Date: January 3, 1944.

O. S. R. L. Contract No. OEMsr-862

INTRODUCTION:

An aluminum soap of oleic, naphthenic, and coconut oil fatty acids, has been developed as a thickener for gasoline. This material, known as Napalm, has shown wide variations in quality but at present production troubles have been corrected so that a number of manufacturers are producing satisfactory material.

Early in the research on the Napalm type of soaps it became apparent that sorption of moisture and the oxidation of the acid radicals were two factors which markedly affected the characteristics of the soaps. Because commercial naphthenic acid contains only small amounts of readily oxidizable unsaturated compounds and substantial amounts of natural antioxidants, unmodified aluminum naphthenate might be expected to show considerably greater resistance to aerial oxidation than Napalm containing no added antioxidants.

The relative merits of the two kinds of soap, now that an antioxidant has been included in regular Napalm, is not known.

Early in the Napalm program some work was conducted on the unmodified naphthenates by various organizations. This work was allowed to lapse because of the critical naphthenic acid situation and general lack of interest. So that there would be sufficient data to determine whether the unmodified aluminum naphthenates might serve usefully in the thickening program, the early work has been reviewed and some additional batches prepared and evaluated. Though the data presented is not complete because of the limited time available, a small amount of additional work should make possible production of satisfactory soap.

GENERAL METHODS OF PREPARATION:

There are three methods of precipitation of aluminum soaps which have been used to produce the Napalm type of soaps. Because the preparation of aluminum naphthenate so closely parallels Napalm production, the methods can be used unchanged.

Method 1.

Addition of Partially Neutralized Alum to Neutral Sodium Soap.

In this process stoichiometric quantities of naphthenic acid and caustic soda are reacted to form a neutral sodium soap. To this soap is added an alum solution previously reacted with soda ash so as to form some predetermined quantity of basic salts.

Method 2.

Two Stream Precipitation.

In this process a stream of alum and a stream of basic sodium soap are run simultaneously into a small volume, rapidly agitated starter bath. Usually the bath contains some controlled excess of alum at the start and this is maintained throughout the precipitation.

Method 3.

Addition of Alum to Basic Sodium Soap.

In this process the alum solution is added to the basic sodium soap while agitating. This method is the one most widely used in the commercial manufacture of Napalm.

Any of these methods are feasible, so the choice of method really resolves itself into determination of the procedure most readily used in the plant. Methods 1 and 2 for the preparation of the straight naphthenate have been tried at other laboratories but work at Ferro was concentrated on Method 3 for this is regarded as the most satisfactory for production. Before summarizing the work at Ferro, the data secured at Standard Oil of California and General Printing Ink will be considered. An anonymous method of preparation using Method 2 has been received and is recorded below. The data on General Printing Ink aluminum naphthenates is included in this same section for convenience although the method of preparation has not been revealed.

## EXPERIMENTAL METHODS OF PREPARATION.

Method 1.

Addition of Partially Neutralized Alum to Neutral Sodium Soap.

Standard Oil of California (Oronite) Procedure.

The exact operation followed by Oronite is not too clear but they describe their method as follows:

> "The method of preparation consisted of neutralizing the naphthenic acids with the caustic soda indicated, followed by a stepwise addition of the balance of the caustic soda and aluminum sulfate. The precipitate was filtered, washed and dried."

Chart I summarizes the data obtained by Oronite using the manufacturing method described. The formula used for 31457-R and 31456-R follows. Presumably the same general method was used to prepare 31462-R.

210.0 g naphthenic acid 267 A. V.

1620.0 cc 4° Be' caustic soda (for neutralization).

13.5 cc 50° Be' caustic soda (for basicity)

313.0 cc 3.59% Al Aluminum Sulfate Solution.

Method 2.

Two Stream Precipitation.

Anonymous Procedure (data received from N. L. R. C.)

A procedure for the preparation of unmodified aluminum naphthenates apparently found satisfactory by some one of the interested research groups has been received. The following materials were used for this work:

(a) 1170 cc 2 N aluminum sulfate solution (Sun Chemical and Color Co.) well filtered.

(b) 2250 cc sodium naphthenate solution containing 450 g. naphthenic acid (Harshaw Chemical Co., rectified grade, 239 A. V.) and 76.6 g. NaOH (Merck Reagent).

(c) 750 cc 1 N NaOH

(d) 10,000 cc water containing 50 cc 2 N aluminum sulfate solution.

Solutions b and c were well mixed. The mixture (b + c) and (a) were run into (d) at such a rate that precipitation was completed in 5 minutes. Propeller type agitation was used and agitation was continued 5 minutes after precipitation.

The product was filtered on a buchner and washed until the wash water gave no sulfate test. It was dried in thin layers for 24 - 48 hours at 60° C. To prevent caking during storage, 0.5 - 1.0% aluminum stearate was incorporated in the granular product.

General Printing Ink Soaps.

In Table II the chemical and physical characteristics of the General Printing Ink soaps are summarized. Table III data indicates the difference in moisture susceptibility and the change in the viscosity - concentration relationship between a Napalm and G. P. I. Sample No. 1.

Method 5.

Addition of Alum to Basic Sodium Soap.

The Napalm produced by the Ferro Enamel Corporation has all been produced using the method in which a stream or fine spray of aluminum sulfate solution is introduced at a controlled rate into the well agitated basic sodium soap. Though the technique has been criticized because a substantial amount of the precipitate forms over a short period near the end of precipitation, proper control of agitation minimizes any difficulties. The very satisfactory experience Ferro has had with the method has resultedin a concentration on this particular method of preparation.

There was no background to indicate the best composition to be used for an aluminum naphthenate gelling agent so a number of variables were investigated. The study included the following work:

1) Preparation of soaps more basic than ordinary Napalm.

2) Variation of the sodium soap or aluminum sulfate solution concentrations.

3) The use of particle coating agents to minimize particle adhesion during drying and storage.

4) The use of naphthenic acids of different acid values.

5) Evaluation of the characteristics of the aluminum naphthenate which looked most satisfactory.

Raw Materials. Unless otherwise noted the raw materials used met these specifications:

1) Naphthenic Acid 244 A. V.
Supplier - Std. of Cal.
Acid Value - 246
Sap. Value - 249
Iodine Value - 9.5
Color - Dark amber
% Fe - 0.02

2) Naphthenic acid 261 A. V. - Stanco Rectified.
Supplier - Stanco Distributors
Acid Value - 259
Sap. Value - 263
Iodine Value - 8.9
Color - Pale Yellow
% Fe - 0.002

3) Aluminum Sulfate.
Supplier - General Chemical
Fe - 0.015
Mn - 0.004

4) Caustic Soda
Supplier - Michigan Alkali

1. Preparation of Soaps of Variable Basicities.

A series of aluminum naphthenates were prepared in which the free caustic content of the soaps was gradually increased. The data on these batches are summarized in Table I. It is believed the difficulties met in the plant when handling an aluminum naphthenate prepared from a soda soap less basic than about 300 g. NaOH/1000 g acid would make such a product undesirable.

Both the naphthenic acids produced soaps which were satisfactory when the basicity was high enough. There was little apparent difference in their resistance to sintering during drying.

2. Variation of Concentrations.

Data in Table II pertains to soaps prepared by varying the concentration of reactants, the temperature of precipitation, and in one case, the time of addition. The conclusions reached follow:

a) The aluminum sulfate solution concentration is best maintained at 30 - 44% to secure a satisfactory particle size.

b) A 15% sodium soap seems preferable to lower concentrations if very fine particles are to be avoided.

c) The precipitation temperature must not be too low if fine particles are to be avoided.

5. Coating Agents.

Hydrated aluminum naphthenate which exhibited some tendency to fuse during the drying process was coated with 5% a) starch and b) 325 M talc. Starch made the sticking during drying more severe and talc helped only a small amount.

4. From the experience gained in preparing the other naphthenates, standard precipitation conditions were established and three batches of 310, 412 and 516 g NaOH per 1000 g. acid were prepared from each naphthenic acid. These batches were carefully dried, screened to pass 6 M, redried and bottled. These soaps were used for further testing and the consistencies of their 8% gels when dispersed in S. O. L. test gasoline are tabulated in Table III.

5. Moisture Sorption.

It was considered of interest to note whether the unmodified naphthenates sorbed moisture as rapidly and to the same degree as ordinary Napalm. The moisture absorption rates noted in Table IV were determined using 3/8" layers of material exposed under static conditions at 80° F. 25% R. H.

6. General Characteristics.

Characteristics of the unmodified naphthenates which have been investigated only briefly are as follows:

a) burning rates. Eight percent Napalm and straight aluminum gels were burned and the burning rates found to be comparable.

b) Extensibility. The extensibility of the aluminum naphthenate gels appears comparable to that of Napalm.

c) Cohesion. Cohesion in small size containers offers no great difficulties though it might cause trouble with large packages. A coating agent for the dried naphthenate would probably help considerably.

d) Oxidation. The induction periods of all of the unmodified aluminum naphthenates were not determined because of lack of time but there would seem to be little reason to suspect susceptibility to oxidation.

7. Research on the solvation rates of certain aluminum naphthenates (310 g NaOH/1000 g 244 A. V.) indicated that the set and solvation time in S. O. L. gasoline was relatively insensitive to temperature over the range 38° F to 90° F. though the magnitude of the set and solvation times was rather large. To check the characteristics at very low temperatures the naphthenate mentioned above was screened to pass 20 M and added to gasoline at - 10° F. Apparently there is an abrupt change in solvation rate for the soap had swelled to only half the total volume in 1 - 5 hours. Figs. 1 and 2 illustrate the change in set and solvation time and the spread between the two for the most satisfactory aluminum naphthenates.

Table I.

| G NaOH/ 1000 g. Acid | Precip. Temp. ° F | Soda Soap Conc. % | Naph. Acid A.V. | Alum Sulf. Conc. % | Time to Precip. min. | Precip. Characteristics. | Drying Characteristics. | Consistency 2 hr. | Consistency 24 hr. | Consistency 48 hr. | % $H_2O$ | % Al | % Na | Total $SO_4$ % | % $SO_4$ not combined with Na. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 165 | 72 | 15 | 246 | 44 | 20 | Very sticky | - - - - - | | | | | | | 1.55 | 2.18 |
| 206 | 72 | 15 | 248 | 44 | 20 | Sticky | Completely sintered. | | 616 | 578 | 0.55 | 5.55 | 0.64 | 5.51 | 2.18 |
| 248 | 73 | 15 | 246 | 44 | 20 | Sticky | Sintered badly. | 785 | 725 | 757 | 0.40 | 5.87 | 1.12 | 6.59 | 4.04 |
| 289 | 75 | 15 | 246 | 44 | 20 | Slightly sticky. | Sintered badly. | 770 | 605 | 626 | 0.75 | 6.34 | 1.09 | 6.45 | 4.14 |
| 330 | 75 | 15 | 246 | 44 | 20 | Fair part. size. Little stickiness | Sintered but could be broken easily. | 794 | 816 | 795 | 0.90 | 6.71 | 1.29 | 9.32 | 6.62 |
| 412 | 86 | 15 | 246 | 44 | 20 | Good part. size. Part soft. | Could be broken with difficulty. | | | | | | | | |
| 516 | 96 | 15 | 248 | 44 | 20 | Part. soft, mushy, hard to filter | Could be broken with difficulty. | | | | | | | | |
| 206 | 72 | 15 | 259 | 44 | 20 | Sticky | | | 300 | 900 | | 5.54 | | | |
| 248 | 72 | 15 | 259 | 44 | 20 | Sticky | | | 864 | 715 | | 601 | | | |
| 289 | 75 | 15 | 259 | 44 | 20 | Slightly sticky. | | | 856 | 950 | | 6.45 | | | |
| 330 | 75 | 15 | 259 | 44 | 20 | Little stickiness | | | 785 | 930 | | 6.82 | | | |

TABLE II

| G NaOH/ 1000 g Acid | Precip. Temp. ° F. | Soda Soap Conc. % | Naph Acid A.V. | Alum Sulf. Conc. % | Time to Precip. Min. | Precip. Charac-teris-tics. | Drying Character-istics. |
|---|---|---|---|---|---|---|---|
| 310 | 74 | 15 | 246 | 20 | 44 | Very soft and mushy | ----- |
| 310 | 80 | 15 | 246 | 20 | 45 | Very soft and mushy | ----- |
| 310 | 93 | 15 | 246 | 20 | 15 | Very fine unsatisfactory particle | ----- |
| 310 | 93 | 15 | 246 | 30 | 30 | Particle size good. | Sintered badly Broke up easily. |
| 310 | 78 | 9 | 246 | 44 | 20 | Very fine unsatisfactory particle. | Dried to a hard, brittle mass. |
| 412 | 86 | 15 | 246 | 44 | 20 | Good part-size Part soft | Could be broken with difficulty. |
| 412 | 90 | 15 | 246 | 44 | 20 | Part size Slightly better than 86° | Could be broken with difficulty. |
| 516 | 96 | 15 | 246 | 44 | 20 | Part. Soft, hard to filter. | Could be broken with great difficulty. |
| 516 | 120 | 15 | 246 | 44 | 20 | Part. fine but filtered easily | Could be broken with great difficulty. |

Table III

| G NaOH/ 1000 g Acid | Naph. Acid. | 2 hr. | 24 hr. | 48 hr. | % $H_2O$ | Extens. | Solv. | Set | Set. Solv. | % Al | Alum. Conc. %. | Soap. Conc. %. | Precip. Temp. ° F. | % Soluble Salts * |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 310 | 246 | 656 | 686 | 679 | 0.55 | Good | 8 | 15 | 7 | 6.33 | 44 | 15 | 80 | 8.51 |
| 412 | 246 | 490 | 605 | 665 | 0.55 | Good | 13 | 20 | 7 | 7.85 | 44 | 15 | 90 | 7.37 |
| 516 | 246 | 544 | 567 | 675 | 0.85 | O.K. | 22 | 32 | 10 | 8.69 | 44 | 15 | 120 | 8.19 |
| 310 | 259 | 672 | 550 | 720 | 0.60 | Good | 14 | 26 | 14 | 8.73 | 44 | 15 | 80 | 2.89 |
| 412 | 259 | 432 | 480 | 512 | 0.67 | Good (weak) | 20 | 35 | 15 | 8.10 | 44 | 15 | 90 | 8.85 |
| 516 | 259 | 385 | 382 | 537 | 0.80 | Low Visc. | 38 | 79 | 41 | 9.16 | 44 | 15 | 120 | 9.50 |

* Soluble salts were determined by ashing the soap, weighing, washing, igniting, and reweighing. Loss in weight was designated as soluble salts.

TABLE IV.

Moisture Sorption - Unmodified Aluminum Naphthenates.

| G. NaOH/ 1000 g Acid | % $H_2O$ Sorbed- % Weight Gain | | | | | |
|---|---|---|---|---|---|---|
| | 0.5 hrs | 1.0 hrs | 1.5 hrs. | 2.0 hrs. | 15.0 hrs. | Naph A. V. |
| 310 | 0.143 | 0.052 | 0.048 | 0.044 | .244 | 259 |
| 412 | 0.177 | 0.076 | 0.066 | 0.057 | .274 | 259 |
| 516 | 0.178 | 0.084 | 0.067 | 0.070 | .374 | 259 |
| 310 | 0.078 | 0.043 | 0.033 | 0.041 | .257 | 246 |
| 412 | 0.095 | 0.047 | 0.046 | 0.056 | .326 | 246 |
| 516 | 0.138 | 0.074 | 0.067 | 0.080 | .376 | 246 |

TABLE I.

| Sample No. | 31257-R | 31258-R | Harshaw X-104 | 31462-R |
|---|---|---|---|---|
| Drying Method | Tray | Air | --- | |
| Appearance | Granular | Powder | --- | Granular |
| % Al | 5.25 | 5.31 | --- | 5.67 |
| % $H_2O$ | 1.08 | 1.71 | --- | 0.95 |
| Ash | --- | --- | --- | 1.80 |
| Stability -40°C. | O. K. | O. K. | --- | |
| Stability-66°C. | No Thinning | Thins markedly | --- | |
| Solvation Time (Set Time to form) Stable 8, 9, 13.5% gels. | 24 hrs. | --- | | |
| 8% gel consistency (Oronite Method) | 10 sec. | | 18 sec. | |
| 9% gel consistency (Oronite Method) | 31 sec. | --- | | |
| Caking in package | Slightly sticky | --- | O. K. | |
| Consistency 48 hrs. @ 77° (May) | | | | 690 |
| Consistency 7 days @ 77° (May) | | | | 800 |
| Consistency 48 hrs. @ 150° (May) | | | | 820 |
| Consistency 2 hrs. @ 150° (Oct) | | | | 835 |
| 24 hrs. @ 150° (Oct) | | | | 850 |
| 24 hrs. @ 77° (Oct) | | | | 850 |
| % Through 6 Mesh | | | | 100 |
| % Through 40 Mesh | | | | 14 |

## TABLE II.

### The Chemical and Physical Characteristics of G. P. I. Soaps.

| | Nuodex #16,032 Average | G.P.I.#1 a. | G.P.I.#1 b. | GPI #3 | GPI #4 | GPI #5 | GPI #6 | GPI. #7 |
|---|---|---|---|---|---|---|---|---|
| 48 hrs. 77° F. Gardner in g. | 640 | 1160 | 1100 | 950 | 800 | 740 | 640 | 850 |
| 24 hrs. 150° F. Gardner in g. | 510 | 1150 | 1020 | 470 | 530 | 367 a.v. | 720 | 540 |
| 168 hrs. 77° F. Gardner in g. | 580 | 1220 | 1100 | — | — | — | — | — |
| 2 hrs. 150° F. Gardner in g. | — | — | — | 850 | 780 | 750 | 850 | 830 |
| Vacuum Oven Moisture | — | 0.28 | | 0.79 | 0.64 | 0.99 | 0.65 | 0.67 |
| Lean & Stark Moisture | — | 0.5 | | 0.80 | 1.00 | 1.50 | 1.00 | 0.90 |
| Iodine No. (Initial) | — | 11.0 | | 9.3 | 10.4 | 9.2 | 9.9 | 12.1 |
| Iodine No. (1 month-120° F | — | 5.2 | | — | — | — | — | — |
| Heat Stability (temp.rise°F.) | 0.0° | 3.0° | | — | — | — | — | — |
| Dispersion Time 55° F. | — | 20 | | — | — | — | — | — |
| Dispersion Time 70° F. | — | 15 | | — | — | — | — | — |
| Dispersion Time 90° F. | — | 11 | | — | — | — | — | — |
| Setting Time 55° F. | — | 28 | | — | — | — | — | — |
| Setting Time 70°F. | — | 21 | | — | — | — | — | — |
| Setting Time 90° F.(Viscosity Initial/Oscilation Method) in sec. | — | 17 | | — | — | — | — | — |
| 28 day 150° F. Viscosity (Oscilation Method) in sec. | 45 | 121 | | — | — | — | — | — |
| % Iron | 30 — | 36 0.015 | | — 0.10 | — 0.05 | — 0.04 | — 0.06 | — 0.04 |

TABLE III

Moisture Susceptibility and Viscosity -Concentration Characteristics of Aluminum Soaps.

| G. P. I. Soap No. 1. | % Vacuum Oven Moisture | Gardner 168 hrs. 77° F. | Gardner 48 hrs. 77°F. | Setting Time. | Dispersion Time. |
|---|---|---|---|---|---|
| Conditioned 24 hrs, 90°F. 20% R. H. | 0.38 | 1050 | 1000 | 20 | 15 |
| Conditioned 24 hrs, 85°F. 65% R. H. | 1.01 | 850 | 850 | 9 | 8 |
| Conditioned 24 hrs, 90° F. 90% R. H. | 1.50 | 590 | 440 | 9 | 6 |
| No Conditioning, 4% Concentration | --- | 310 | 325 | --- | --- |
| No Conditioning, 6% Concentration | --- | 705 | 740 | --- | --- |
| No Conditioning 8% Concentration | --- | 1100 | 1100 | --- | --- |
| No Conditioning, 10% Concentration | --- | 1450 | 1510 | --- | |
| No Conditioning 12% Concentration | --- | --- | --- | --- | --- |
| Napalm B. No. 87896 (Mfr.?) | | | | | |
| Conditioned 24 hrs, 90°F. 20% R. H. | 0.99 | 650 | 670 | --- | --- |
| Conditioned 24 hrs. 85°F. 65% R. H. | 1.75 | 255 | 300 | --- | --- |
| Conditioned 24 hrs. 90°F. 90% R. H. | 3.00 | 80 | 105 | --- | --- |
| No Conditioning, 4% Concentration | --- | --- | 97 | --- | --- |
| No conditioning, 6% Concentration | --- | --- | 240 | --- | --- |
| No Conditioning 8% Concentration | --- | --- | 680 | --- | --- |
| No Conditioning, 10% Concentration | --- | --- | 1000 | --- | --- |
| No Conditioning, 12% Concentration | --- | --- | 1410 | --- | --- |

SUMMARY:

Though additional work is yet to be done on the unmodified aluminum naphthenates, research has progressed far enough to indicate soaps of adequate physical characteristics and satisfactory gasol ine gelling ability can be prepared in the laboratory. Furthermore, the characteristics of the soaps are so close to those of the Napalm type there should be no unusual production problems.

Sintering of particles during the drying process might introduce more grinding difficulties than are met producing Napalm, but close control during precipitation will minimize such troubles.

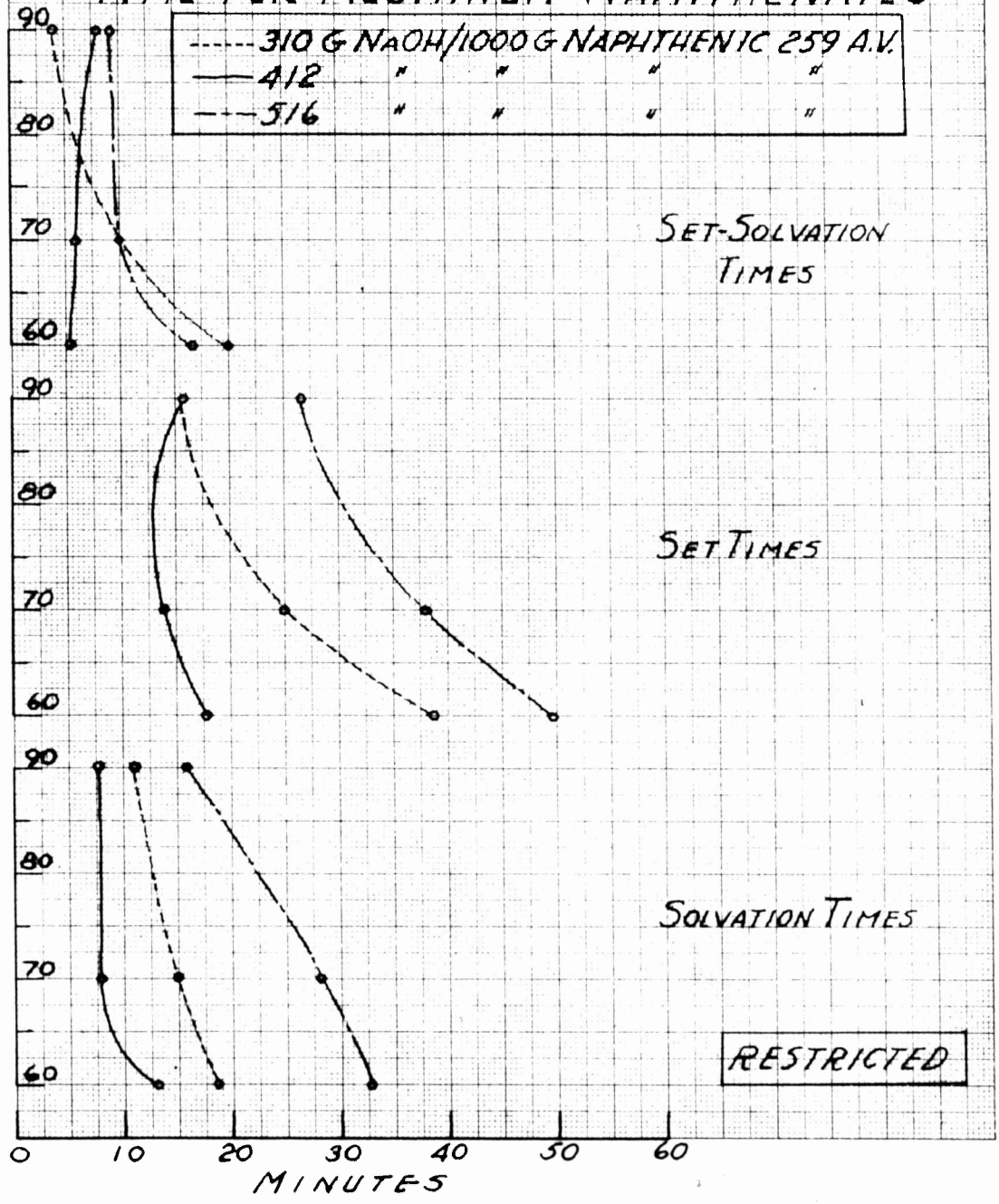
The naphthenates prepared at Ferro have shown no unusual gelling properties but when properly formulated appeared as satisfactory as Napalm. Thus, though the oxidation resistance of the two types of soaps have not been compared extensively, it would seem the greatest advantage of the unmodified naphthenates lay in their oxidation resistant structure.

Author S. B. Elliott

Supervisor G. H. McIntyre

FIG. 1

# SOLVATION, SET, AND SET-SOLVATION TIME FOR ALUMINUM NAPHTHENATES



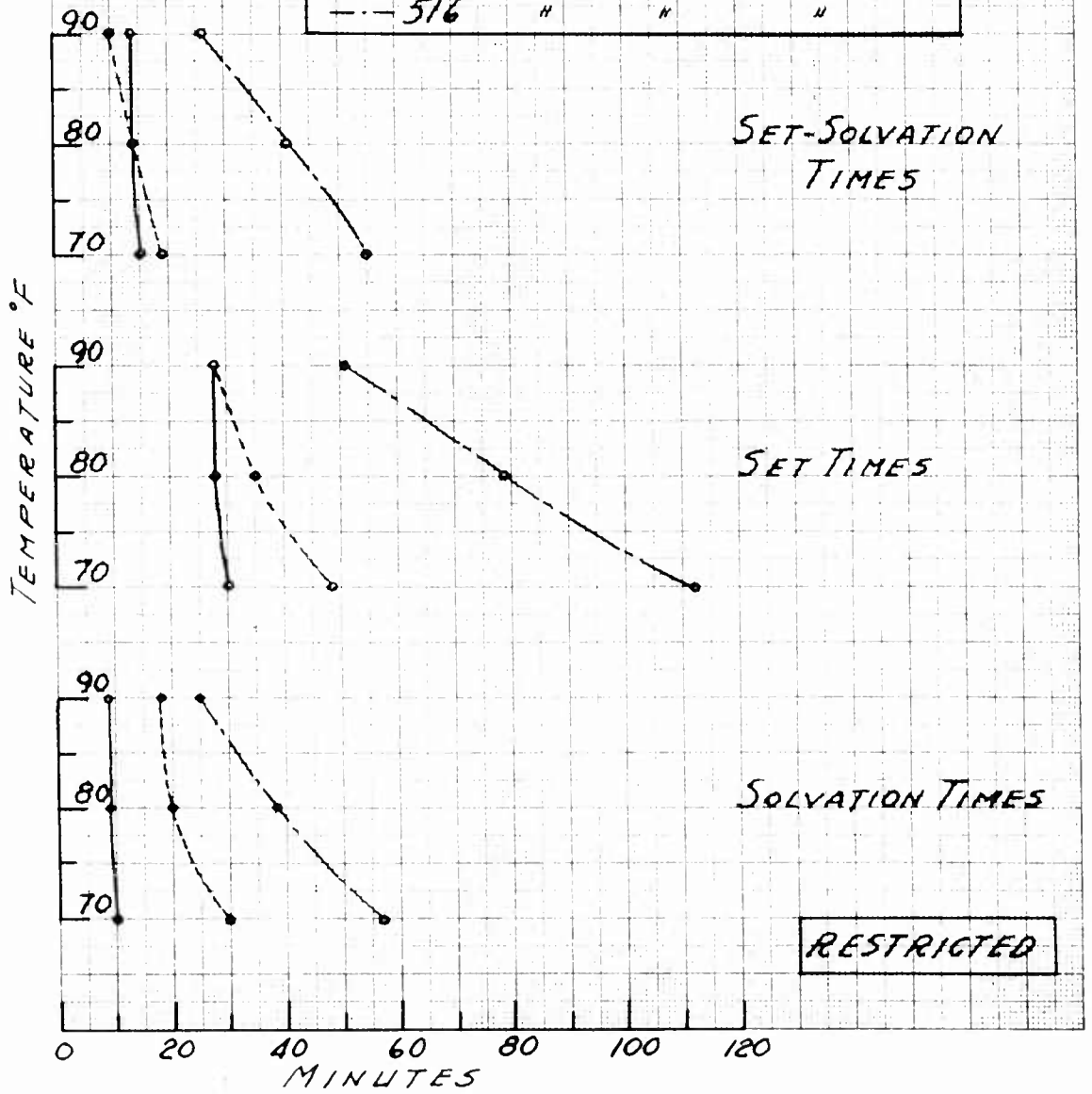

RESTRICTED

FIG 2

# SOLVATION, SET, AND SET-SOLVATION TIME FOR ALUMINUM NAPHTHENATES

## ORONITE NAPHTHENIC 246 A.V.

---- 310 G NaOH/1000 G NAPHTHENIC
—— 412 " " "
—·— 516 " " "

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ABSTRACT:

Though additional work is yet to be done on the unmodified aluminum napthenates, research has progressed far enough to indicate that soaps of adequate physical characteristics and satisfactory gasoline gelling ability can be prepared in the laboratory. Furthermore, the characteristics of the soaps are so close to those of the Napalm type that there should be no production problems. Sintering of the particles during the drying process might introduce more grinding difficulties than are met producing Napalm, but close control during percipitation will minimize such troubles. The napthenates prepared at Ferro have shown no unusual gelling properties, but when properly formulated they appear as satisfactory as Napalm. Thus, though the oxidation resistance of the two types of soaps have not been compared extensively, it would seem the greatest advantage of the unmodified napthenates lay in their oxidation resistance structure.



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