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Report on:

THE MANUFACTURE, PROPERTIES, AND TESTING OF NAPALM SOAPS

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By

by LUTHER A. WALKER, JR., 2nd Lt., Cml C  
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Report on "The Manufacture, Properties and Testing of Napalm Soaps"

by

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Report OSRD No. 2036

Copy No. 32

Date: November 17, 1943

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Division 11  
NATIONAL DEFENSE RESEARCH COMMITTEE  
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OFFICE OF SCIENTIFIC RESEARCH AND DEVELOPMENT  
Section 11.3

Report on "The Manufacture, Properties and Testing of Napalm Soaps"  
Service Directives CWS 10 and 21

Endorsement (1) From E. P. Stevenson, Chief, Division 11 to Dr.  
Irvin Stewart, Executive Secretary of the National Defense Research  
Committee.

Forwarding report and noting:

"This report presents the findings and conclusions of a reviewing committee appointed by Dr. H. C. Hottel, Chief, Section 11.3, to investigate the processes developed by the several N.D.R.C. contractors for the manufacture of the modified aluminum naphthenate soap commonly known as "Napalm." This product was originally developed under the Section's contract with Harvard University (Dr. Louis F. Fieser, Official Investigator, with Dr. E. B. Hershberg as Senior Assistant) to meet the emergency caused by the non-availability of natural rubber for thickening gasoline for incendiary uses. The present methods of manufacture are the result of extensive studies by Nuodex Products Company, Harshaw Chemical Company, Ferro Drier and Chemical Company, and of the companies now producing Napalm under contract with the Chemical Warfare Service.

"The primary purposes of this survey were to determine (1) whether with adequate technical supervision Napalm could be reliably produced in satisfactory quality, (2) the nature of the major troubles, and (3) how these might be remedied or minimized within safe limits. The conclusions on these points are that present data are sufficient to assure the production of Napalm of specification grade and that the troubles experienced in the past are due chiefly to moisture and oxidation.

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"As bearing upon the continuing research program of the Division it is recommended that,

- (1) Investigation of the effect of raw material properties on the finished soap be continued.
- (2) Investigation of oxidation inhibitors be continued.

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(3) Investigation of the relationships between gasoline quality, moisture content, concentration and consistency be prosecuted.

(4) A further study of the precipitation process be made.

"This report will be of further interest in that it reviews the work to date by N.D.R.C. contractors, by the Technical Division of the Chemical Warfare Service, and by some of the C.W.S. contractors now producing Napalm. In this review the original sources are cited, and much of the most pertinent data is tabulated for easy reference. The several processes now in use are critically presented with a detailed discussion of the important process variables and the chemical reactions involved.

"The report also contains a detailed description of a preferred manufacturing procedure. The reviewing committee recognizes that Napalm of satisfactory quality has been and can be reliably made by other processes, and that the availability of equipment must be a determining factor in the practices of the different C.W.S. contractors.

"Acknowledgement is made of the assistance of the several interested groups in the investigation and in the preparation of this report. Through the cooperation of the Technical Division of C.W.S., access was obtained to the different manufacturers, who in turn were entirely responsive to the requests for detailed information on manufacturing procedure and experience. The committee was further assisted by Major R. J. DeGray both during the investigation and in the preparation of the report."

\* \* \* \* \*

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\* \* \* \* \*

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## INTRODUCTION

Various war needs for a material to thicken gasoline led to the development of an aluminum soap of oleic, naphthenic and coconut oil acids now known as Napalm. The manufacturing process is one in which the water-insoluble aluminum hydroxy-soaps of oleic, naphthenic and coconut acids are coprecipitated from aqueous solution. When dried, the coprecipitated soap is granular, easily handled and rapidly dispersable in gasoline. Because of its many advantages, the coprecipitation method is now being used by all Napalm manufacturers and therefore discussion in this report will be limited to this process.

Two types\* of Napalm are made, differing only in setting time and in packaging; Type A, which should solvate slowly and be capable of long storage without impairment in properties, and Type B which normally has a shorter solvation time. Control of setting time is achieved by regulating the density and size of the finished soap particles, and is accomplished mainly during the dewatering operation. There appears to be no chemical difference between the two types of soap. The discussion in this report applies to the manufacture of both types of Napalm, operational variations for Type A being mentioned at the stage where they occur.

Little difficulty was experienced in preparing the first batches of Napalm soap. However, when attempts were made to produce Napalm on a larger scale, difficulties arose for reasons which will become clear in the following pages.

A survey of the Napalm problem, from both research and manufacturing aspects, has been made by an N.D.R.C. group with the help of the various C.W.S. divisions. It is believed that, given adequate technical supervision and satisfactory raw materials, no difficulty in making Napalm need be experienced. Accordingly, it was decided to issue an N.D.R.C. report concerning the manufacturing question, a certain amount of related research, and the properties of the product.

\* The new specification (Appendix VIII) calls for only one type of Napalm, the restriction on the setting time of Type A being removed. This, however, although contemplated, was not in effect at the time this report was written.

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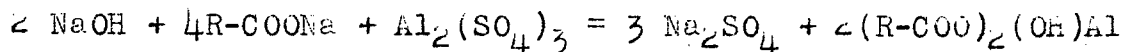
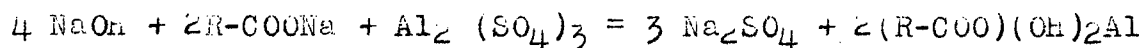
During the survey, excellent cooperation was shown by all the Napalm manufacturers and the various divisions of C.W.S. who made much information available. The collection of material for this report necessitated the free and open discussion by the manufacturers of their processes. It is a pleasure to record that the assembly of this material was possible only in consequence of the cooperation of all groups involved, including manufacturers, N.D.R.C. research contractors, and C.W.S. divisions.

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THE MANUFACTURE OF NAPALMA. The Mechanism of the Precipitation

The precipitation of the aluminum hydroxy-soaps of oleic, naphthenic and coconut acids may be approximately represented as follows:



The final product when gelled in gasoline contains long molecular chains built up by coordination between aluminum atoms and the carboxylic oxygen atoms, and possibly also by oxygen bridges similar to those found in silica gels. The final soap also contains free fatty acids and sodium salts occluded or possibly loosely held in the soap. (See Appendix I for a more detailed discussion of the precipitation chemistry.)

Analyses show that most satisfactory soaps contain about 5.4 to 5.3% aluminum, an amount which is in excess of the aluminum equivalent of the acids used. In order to metathesise the necessary additional alum, excess caustic soda ("free caustic") over and above that equivalent to the fatty acids used must be present. Experiment indicates that substantially all the aluminum which is supplied in the alum up to the coagulation point enters the final product. The resulting sodium sulfate leaves the precipitator in the clear liquor and in subsequent washing. Unreacted sodium soap in the product has been shown to be harmful, (1) but the presence of sodium sulfate in moderate amounts does not appear to be injurious. The amount of excess or "free" caustic used in precipitating a soap has been shown to affect the strength of a gasoline gel of the soap. Table I (2) illustrates this point.

Table I

The Effect of Excess Caustic Soda on Napalm Gel Strength (2)

Acid Ratio - 2 Coconut : 1 Oleic : 1 Naphthenic  
Acid No. of Mixed Fatty Acids - 240

| <u>% Free Caustic</u> | <u>Gardner Viscosities</u> |                      |
|-----------------------|----------------------------|----------------------|
|                       | <u>24 hrs. 150°F.</u>      | <u>48 hrs. 77°F.</u> |
| 26                    | <100                       | 240                  |
| 55                    | 300                        | 870                  |
| 65                    | 330                        | 350 <sup>20</sup>    |
| 125                   | <100                       | 710                  |
| <57                   | <100                       | 540                  |

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The maximum strength appears to occur at about 80% free caustic. Most manufacturers are using about this figure, and it is used, therefore, in the recommended batch procedure described in Appendix II.

The precipitation process for the manufacture of Napalm has been varied by the manufacturers and at the present time there are three basic methods in use; these are referred to as batch methods I and II and the two-stream precipitation process. A brier discussion of these methods follows.

1. Batch Method I.

In this process the total amount of caustic required is added to the mixed acids, the alum then being added until the precipitation is complete. This is the method used by the larger number of manufacturers and has been selected as the recommended procedure on the basis that more information is available on it than on any other process. A more detailed discussion of this method may be found in Appendix II.

2. Batch Method II.

This process differs from (1) in that only enough caustic is added to the mixed acids to produce the stoichiometric "neutral" soap solution, the balance being added as soda ash to the alum solution. The precipitation is begun at a lower pH than in (1) and a more gradual separation of aluminum soap than in Batch Method I is obtained throughout the alum addition. This method is discussed in more detail in Appendix III.

3. Two-Stream Precipitation.

This continuous method is the most desirable from the engineering standpoint, but information on plant operation is limited. In essence the procedure involves the addition, by controlled streams, of the alum and sodium soap solutions to a vessel supplied with vigorous agitation. The alum added in the first stage is insufficient to cause coagulation of the soap, and the resulting milky solution overflows into a second vessel along with another stream of alum solution to provide an excess of this reagent. The overflow from this second vessel passes to a third to allow time for completion of reaction, whence the suspended precipitate flows to a continuous washing and draining device.

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Space requirements for equipment used in this process are less than for (I) and (II), and it is believed that a satisfactory product may be obtained. However, few plant operating data are available and the method is not sufficiently understood at this writing to warrant a recommendation as a standard process.

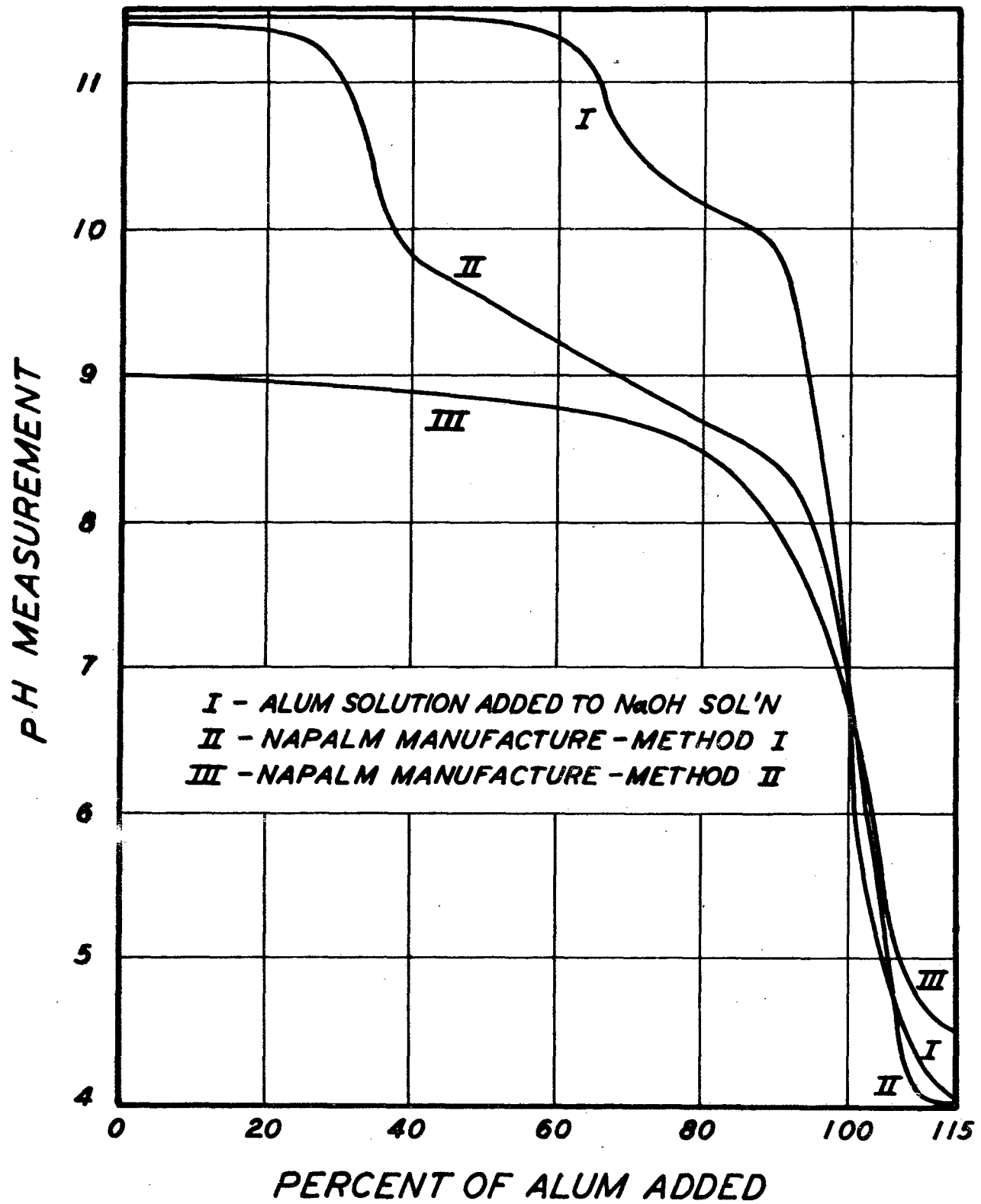
The actual precipitation may be viewed as a simple titration in which alum is added to a basic soap solution, the end point being judged by the coagulation of the precipitate. Figure 1 shows a typical plot of pH vs. amount of alum added for batch method I. The first drop in the curve, at about pH 10.5, occurs in the region where sodium aluminate decomposes and aluminum hydroxide begins to precipitate. The lower, more distinct drop occurs at about the neutral point and is caused by the coagulation of the basic aluminum soap. Laboratory results (2) show that about 75% of the soap is precipitated before the coagulation, but the particles are fine and it is possible that the pH measurements are a compromise between the basicity of the particle and that of the mother liquor. After coagulation, the mother liquor becomes quite clear and the pH drops as more alum is added.

Figure 1 also shows a typical pH vs. alum plot for batch method II. The initial pH is lower than for method I since less free caustic is present in the sodium soap solution. The pH drops more gradually in this method with precipitation proceeding throughout almost the entire operation. No sharp coagulation point is found and it is probable that less occlusion of sodium soap or sodium salts occurs in the precipitate. As described in Appendix III, the material formed by method II may be of such fine particle size that mechanical agglomeration by filter-pressing or roller treatment may be required prior to final comminution and sieving.

At the present time each manufacturer has devised a method for soap production which suits his particular equipment. Although all these methods fall basically into one of the three groups discussed above, each has slight variations which distinguish it from the others. The precipitation process involves a little-understood, highly complex equilibrium of sodium, acid, hydroxyl, aluminum and sulfate ions, and time does not permit a complete investigation of all the variables involved. It is, therefore, necessary to accept the results semi-empirically and attempt to control the known variables as closely as possible. The recommended procedure given in this report is, therefore, not necessarily the best, but is considered to be the safest and most foolproof at the present time.

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FIGURE I  
PH OF SOLUTION VS PERCENT  
OF ALUM ADDED



B. Dewatering and Drying of the Precipitate

The processing of the wet precipitate varies considerably among the various manufacturers. After the precipitation is complete the slurry may go to suction filters, filter presses or rotating screens, the type of equipment apparently having little effect on the product. If filter presses are used, it is necessary to break up the filter cake before and again after drying, in order to obtain an acceptable grain size in the product. In general, it appears that the type of dewatering used prior to drying is optional with the manufacturer and will depend on the facilities available.

If Type A Napalm is desired, it may be obtained at this stage by passing the wet filter cake between a pair of steel rolls. In this way, the precipitate is milled to a translucent, yellowish ribbon or sheet, and the individual small particles disappear. The ribbon is then comminuted to the desired particle size before drying. The finished particle produced in this way is dense, translucent and slow setting as compared with the granular, porous precipitate used for Type B.

The drying operation is one which requires considerable care. Dependent on particle size, free acid content and initial moisture content, Napalm soaps are temperature sensitive in several respects, the most important being aerial oxidation, moisture sensitivity, and fusion. As described in pp. 12-20 of this report, oxidation of Napalm affects the gelling properties to a considerable degree and may finally give a soap which is insoluble in gasoline. It is necessary, therefore, to avoid excessive drying times or temperatures which would use up too much of the induction period of the soap. Wet (50% water) Napalm fuses partially during the initial stages of drying, if the temperature during the first six hours exceeds about 165° F. The fusing of dry Napalm is less serious and can apparently be avoided if drying temperatures do not exceed 220° F. Laboratory data (3) show that the equilibrium moisture of Napalm at a given relative humidity decreases as the drying temperature increases, so that it is advantageous to use as high a drying temperature as possible without causing undue oxidation or fusion. Taking all the variables into account, the optimum drying temperature appears to be about 160° F., and this temperature is now being used by nearly all manufacturers. The final moisture content attained is usually between 0.4 and 0.3%, (C.W.S. benzol distn).

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There are three main types of driers in use at present, namely, tray, apron and continuous belt, the first being the most common. Drying times vary from one to twenty or more hours depending on the type of equipment used. The shortest drying time is used by one manufacturer who employs an apron drier, but the temperature used is about 200°F. Tray driers require the longer drying times with belt driers falling in between. Needless to say degree of oxidation depends both on time and on temperature, both of which must be kept to a minimum if a satisfactory product is to be obtained.

Unfortunately, few drier operating data exist and once again the empirical approach is necessary. Napalm appears to have a short constant-drying-rate period during which the surface is wet (or behaves as though wet) and evaporation takes place rapidly, followed by a falling-rate period during which evaporation takes place within the particle and the vapor must diffuse to the surface. During the falling-rate period it is advantageous, where possible, to agitate the Napalm in order to expose new surfaces and thereby hasten drying. It is believed that the continuous belt drier gives some particle disturbances as the belt passes over supporting rolls thereby shortening the total drying time.

### C. Packaging

Considerable difficulty has been experienced by some manufacturers due to a lack of appreciation of the extreme moisture susceptibility of Napalm. As discussed in pp. 3-12 of this report, most soaps having greater than 0.6-0.8% moisture give gasoline gels of low strength and stability. It has been found that Napalm soaps exhibit reproducible equilibrium moisture contents which depend on the relative humidity of the atmosphere; and that the time necessary for a sample substantially to equilibrate itself with the atmosphere may be a matter of minutes. It is necessary, therefore, that care be exercised in handling Napalm between the driers and the final packaging. Climatic conditions dictate to a considerable degree the amount of care necessary. For example, in cold dry weather it may be possible to allow Napalm to stand for a considerable period without serious damage. Conversely, packaging in hot humid weather necessitates keeping to a minimum the time between leaving the drier and final sealing of the package. The packaging of hot material is in general considered good practice, since in this way there is less chance for moisture absorption.

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THE PROPERTIES OF NAPALM

Since the purpose of this report has been to discuss the manufacture of Napalm with its related difficulties, no attempt will be made to cover all the properties of Napalm, e.g. rheology of the gels, for which other reports should be consulted. (5,6,7,52).

The Moisture Effect

Little difficulty was experienced with aluminum soaps in the early days when the evaluation depended largely upon visual examination. However, when more quantitative methods began to be employed, it soon became apparent that unaccountable variations were obtained in different laboratories using the same sample of solid Napalm. For some time the reason remained uncertain but it eventually became very clear that the absorption or desorption of moisture was the principal cause of the anomalies (4,2,8). Napalm is essentially a hygroscopic material, analogous to gelatin or paper in its affinity for moisture. Thus, if given sufficient time, it reaches a definite moisture content at any given relative humidity, the moisture-relative humidity curve resembling that of gelatin or paper in shape. Expressed in another manner, any soap will gain or lose moisture when exposed to the atmosphere, except at the one humidity with which it is in equilibrium (inert point).

Another difficulty, which at first tended to obscure the relationship between consistency and moisture, is the difference in values obtained by various methods of moisture determination (3,9,10). Undoubtedly, water is held in several different forms in the soap and according to the severity of the method of moisture determination used, different values will be obtained (Appendix VI). It can even be formed by reaction during determination. Best results thus far from the standpoint of correlation with consistency data, appear to have been obtained with the Dean and Stark method (benzene distillation) and vacuum oven drying. The former, a modification of which has been adopted as the official specification method (Appendix VIII), utilizes readily available apparatus but requires a large sample of soap and at low water-contents lacks precision, being good to + 0.1% (pp. 75-76). The latter is more suitable for use as a research tool, giving good reproducibility and requiring only small samples. Nevertheless, whatever method is adopted for moisture determination, the relationship between moisture content and surrounding psychrometric conditions is apparent from the data available.

Equilibrium at any given relative humidity appears to be attained fairly rapidly (Figure 2), being practically complete in eight hours in 1/8 or 1/4" layers under normal convection.

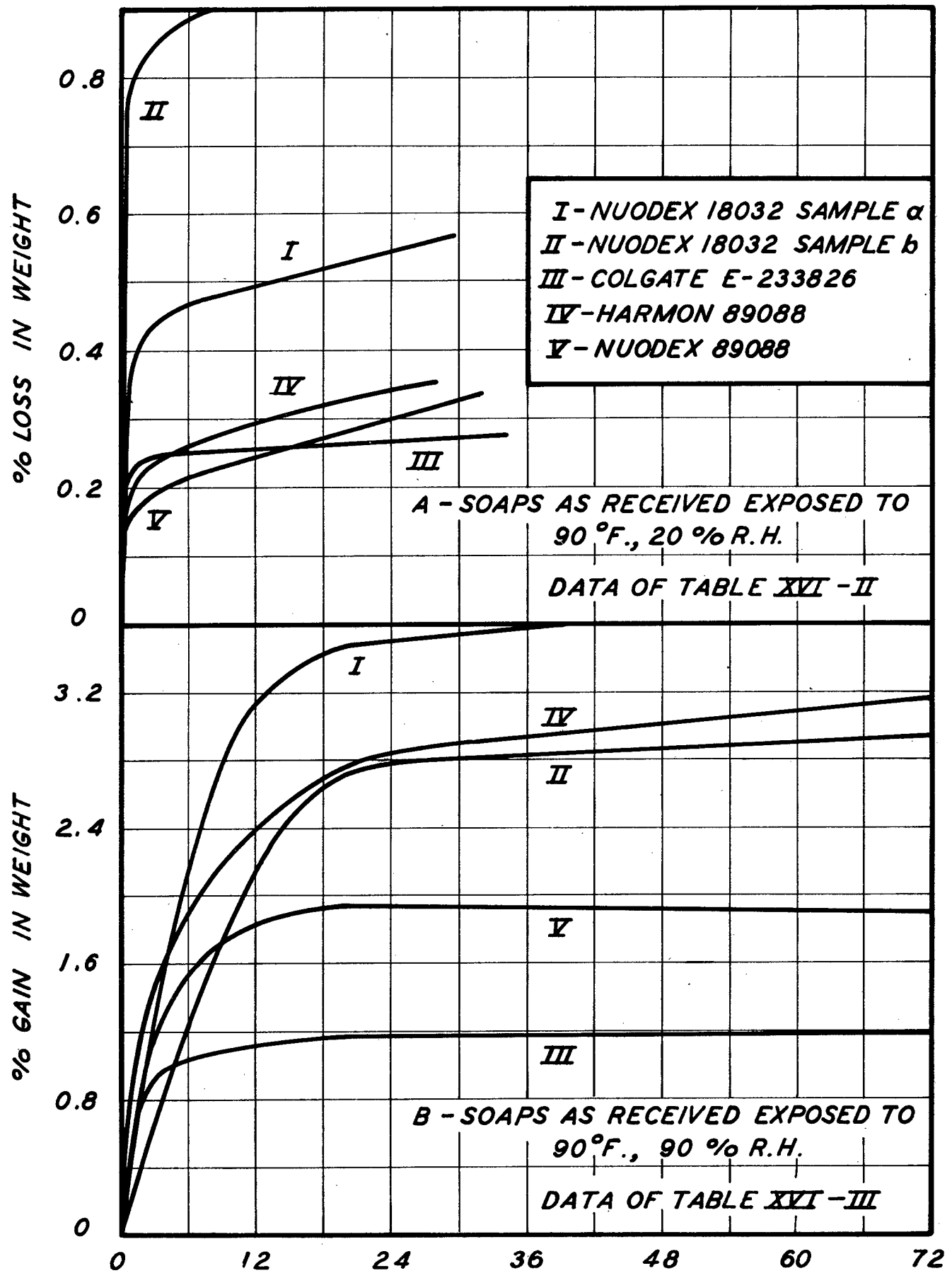
The water absorbed greatly affects the consistency of the gel formed when the solid soap is dissolved in gasoline. Table II shows representative results for two typical soaps, and gives the comparative Gardner mobilometer readings of gels made from soaps dried or equilibrated under different conditions. The average of a large number of tests made on many soaps at both Edgewood Arsenal and Eastman Kodak indicated that the absorption of 0.1% moisture measured by the vacuum oven method causes the consistency on the 24 hour 150° test (3% gel) to drop approximately 40-50 grams. The reasons for the discrepancy found in consistency by different laboratories now becomes very clear. According to the packaging and handling of the soaps between the two points at which they were examined they might either gain or lose moisture, thus radically changing their viscosity characteristics.

The cause of the moisture effect probably lies in the prevention of the formation or possibly the breaking of the soap chains by preferential coordination of water molecules at the points of attachment between the individual units. Osmotic pressure measurements (18) indicate that normal Napalm dispersed in gasoline has a molecular weight of 160,000 to 200,000. It seems probable that these large molecules are built up in large part by coordination of the aluminum atom of each soap unit with one of the carboxyl oxygens of the next unit. Such chains can obviously be broken if a molecule to which aluminum coordinates more strongly than the carboxyl oxygen is introduced. This may be the case when polar compounds such as water, amines (11), or free fatty acids of low molecular weight are added. The aluminum atoms coordinate preferentially with the amine or hydroxyl groups, the aluminum soap chains are broken, with a resultant drop in molecular weight and consistency.

In order to overcome the effect of moisture it has been found necessary to regulate the water content at the point of manufacture of the soap and also at times to redry Napalm Type B before use in the filling plant. Napalm Type A has been protected by hermetically sealing in metal cans, the only type of package which can be expected to withstand handling under atmospheric conditions prevailing in the tropics. Table III(a) shows data obtained on the tray drying of a typical batch of soap at the Harmon Color Works. It is apparent that unless drying is carried to a satisfactory moisture content, trouble will be experienced. Similar data are also shown in Table III(b) indicating that a soap can be redried after moisture pick-up without adverse effects other than possible partial oxidation.



**FIGURE 2 RESTRICTED**  
**RATE OF MOISTURE EQUILIBRATION**



TIME IN HOURS

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Table II

The Moisture Effect (3)

|  | <u>Vac. Oven Moisture</u>                           |                               | <u>Gardner Consistencies</u>    |                                  |                                  |
|--|---|-------------------------------|---------------------------------|----------------------------------|----------------------------------|
|  | <u>24 hrs.</u><br><u>60° C.</u><br><u>10 mm Hg.</u> | <u>Benzol</u><br><u>Dist.</u> | <u>48 hrs.</u><br><u>70° F.</u> | <u>24 hrs.</u><br><u>150° F.</u> | <u>163 hrs.</u><br><u>70° F.</u> |
| <u>Nuodex #87896 (8% Gel)</u>            |   |                               |                                 |                                  |                                  |
| Dried 1 mo. in Nitrogen                  | 0.10  | ---                           | 1220                            | 1350                             | 1250                             |
| Dried over P <sub>2</sub> O <sub>5</sub> | 0.46  | 0.65                          | 700                             | 650                              | 740                              |
| Dried in Vacuum                          | 0.61  | 0.8                           | 560                             | 520                              | 700                              |
| Equilibrated at 90°F-20% RH              | 0.99  | 1.1                           | 670                             | 390                              | 650                              |
| Equilibrated at 35°F-65% RH              | 1.75  | 1.5                           | 300                             | 190                              | 255                              |
| Equilibrated at 90°F-90% RH              | 3.06  | 2.7                           | 105                             | 100                              | 80                               |
| <u>Nuodex #15374 (6% Gel)</u>            |   |                               |                                 |                                  |                                  |
| Equilibrated at 90°F-20% RH              | 0.89  | 1.2                           | 600                             | 160                              | 530                              |
| Equilibrated at 35°F-65% RH              | 1.45  | 1.6                           | 340                             | 170                              | 175                              |
| Equilibrated at 90°F-90% RH              | 2.97  | 2.2                           | 47                              | 10                               | 17                               |
| <u>Nuodex #15374 (3% Gel)</u>            |   |                               |                                 |                                  |                                  |
| Equilibrated at 90°F-20% RH              | 0.89  | 1.2                           | 320                             | 760                              | 760                              |
| Equilibrated at 35°F-65% RH              | 1.45  | 1.6                           | 580                             | 400                              | 540                              |
| Equilibrated at 90°F-90% RH              | 2.97  | 2.2                           | 290                             | 43                               | 140                              |
| <u>Nuodex #15374 (10% Gel)</u>           |   |                               |                                 |                                  |                                  |
| Equilibrated at 90°F-20% RH              | 0.89  | 1.2                           | 1225                            | 1100                             | 930                              |
| Equilibrated at 35°F-65% RH              | 1.45  | 1.6                           | 1000                            | 770                              | 1050                             |
| Equilibrated at 90°F-90% RH              | 2.97  | 2.2                           | 600                             | 235                              | 520                              |

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Table III(a)(10)

Effect of Drying Time on Consistency and Moisture Content  
(Harmon Soap #11249)

| <u>Time</u> | <u>Temp.</u> | <u>Benzol Moisture</u> | <u>2 hours 150° F.</u> | <u>24 hours 150° F.</u> | <u>48 hours 77° F.</u> |
|-------------|--------------|------------------------|------------------------|-------------------------|------------------------|
| 12          | 160°F.       | 2.1                    | Fluid                  | Fluid                   | Fluid                  |
| 14          | "            | 2.6                    | "                      | "                       | "                      |
| 16          | "            | 1.3                    | 300                    | 220                     | 310                    |
| 18          | "            | 1.2                    | 290                    | 280                     | 330                    |
| 20          | "            | 0.8                    | 460                    | 340                     | 450                    |
| 22          | "            | 0.3                    | 670                    | 630                     | 700                    |
| 24          | "            | 0.4                    | 680                    | 670                     | 670                    |
| 29*         | "            | 0.4                    | 720                    | 670                     | 710                    |
| 48          | "            | 0.35                   | 730                    | 670                     | 700                    |
| 72          | "            | 0.45                   | 730                    | 680                     | 740                    |

\* Transferred at this point to laboratory oven.

Table III(b)(25)

| <u>Redrying Time at 150°F.</u>                                    | <u>% Water in Napalm*</u> | <u>24 hours 150° F.</u> | <u>48 hours 77° F.</u> |
|---|---------------------------|-------------------------|------------------------|
| Original  | ---                       | 810, 760                | 840, 820               |
| Original soap exposed to humid air (1) 18 hours at 80°F. 65% R.H. |                           |                         |                        |
| (2) 3 hours 86° F. 72% R.H.                                       |                           |                         |                        |
| None (1)  | 1.2                       | 475, 485                | 570, 530               |
| (2)   | 1.1                       | 535, 500                | 125, 660               |
| 10 mins. (2)  | 0.80                      | 635, 650                | 790, 790               |
| 20 mins. (1)  | 0.85                      | 695, 700                | 790, 750               |
| (2)   | 0.78                      | 710, 730                | 735, 795               |
| 40 mins. (1)  | 0.58                      | 815, 790                | 820, 785               |
| (2)   | 0.68                      | 680, 770                | 770, 795               |
| 60 mins. (1)  | 0.55                      | 865, 890                | 815, 815               |
| (2)   | ---                       | 770, 760                | 790, 785               |
| 120 mins. (1)   | 0.53                      | 330, 305                | 340, 330               |
| (2)   | ---                       | 775, 790                | 795, 805               |
| 240 mins. (1)   | 0.45                      | 330, 350                | 340, 345               |

\* Benzol distillation (C.W.S. method).

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The Oxidation of Napalm

Because Napalm contains oleic acid, with its double bond, it might be expected under suitable conditions to be susceptible to oxidation with consequent change in its properties. To a lesser extent certain fractions of the coconut acids used may contribute to their oxidation susceptibility. This was not at first noticed, but attention was drawn to the oxidation effect late in 1942, when one manufacturer was making Napalm Type A (then known as X-104-E) using 25% naphthenic, 40% coconut and 35% oleic acid. These were precipitated with a Victory grade aluminum sulfide from American Cyanamide which analyzed up to 0.4% iron. The soaps produced were very unstable, in some cases lacked solubility in gasoline and on at least one occasion took fire in the tray drier. Investigation showed these changes to be due to oxidation, strongly exothermic, and traceable by the drop in iodine number of the soap (12, 13). This was without doubt the most exaggerated form of the oxidation trouble yet encountered, although it has been found that, if kept long enough under unfavorable conditions, all samples of Napalm will oxidize (3, 14, 15). Shipments of Napalm have been received at a filling plant with the soap at the center of the barrel in a fused state owing to spontaneous oxidation. Figure 3 shows a photograph of a soap which oxidized spontaneously on keeping on the laboratory shelf under ordinary conditions for nine months. Complaints have also been received from the Pacific and from England of Napalm Type A which would not go into solution in gasoline (16). In the Pacific case, the iodine number of the soap had dropped to 3, the normal value being 25 to 30; this was undoubtedly due to high iron content, namely 0.23%. Napalm Type A may have to be kept in storage for many months before use. Type B is hot packed in large barrels so that the center cools off slowly and the exothermic oxidation reaction may be accelerated because the heat cannot be dissipated. It is clear, therefore, that if proper control is not exercised, oxidation can be a serious problem.

Table IV indicates the changes in iodine number occurring when typical soaps are spread out in 1/4 to 1/2" layers and exposed to the atmosphere. It will be seen (top section of table) that oxidation does not commence immediately, i.e. there is an induction period, which is greatly shortened by an increase in temperature or relative humidity. The bottom section indicates that a small drop in iodine number (compare lines 1 and 2) does not appreciably affect the properties of the soap either with respect to setting time or consistency if allowance is made for the tendency of "original" soaps to dry when exposed to 20% R.H., and therefore to produce more viscous solutions. A large drop in iodine number, however, causes very slow gelation with the formation of an inferior gel (compare lines 2 and 3, or 5 and 6).

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*FIGURE 3*



*SAMPLE OF NAPALM WHICH  
OXIDIZED DURING STORAGE AT  
ROOM TEMPERATURE*

*(HORIZONTAL LINE INDICATES  
ORIGINAL LEVEL OF SAMPLE IN BOTTLE)*

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Such tests as those reported in Table IV take considerable time to run (a matter of months) and hence are of little value for research work or for use in specifications. For this reason several accelerated tests for the oxidation susceptibility of Napalm have been suggested and used.

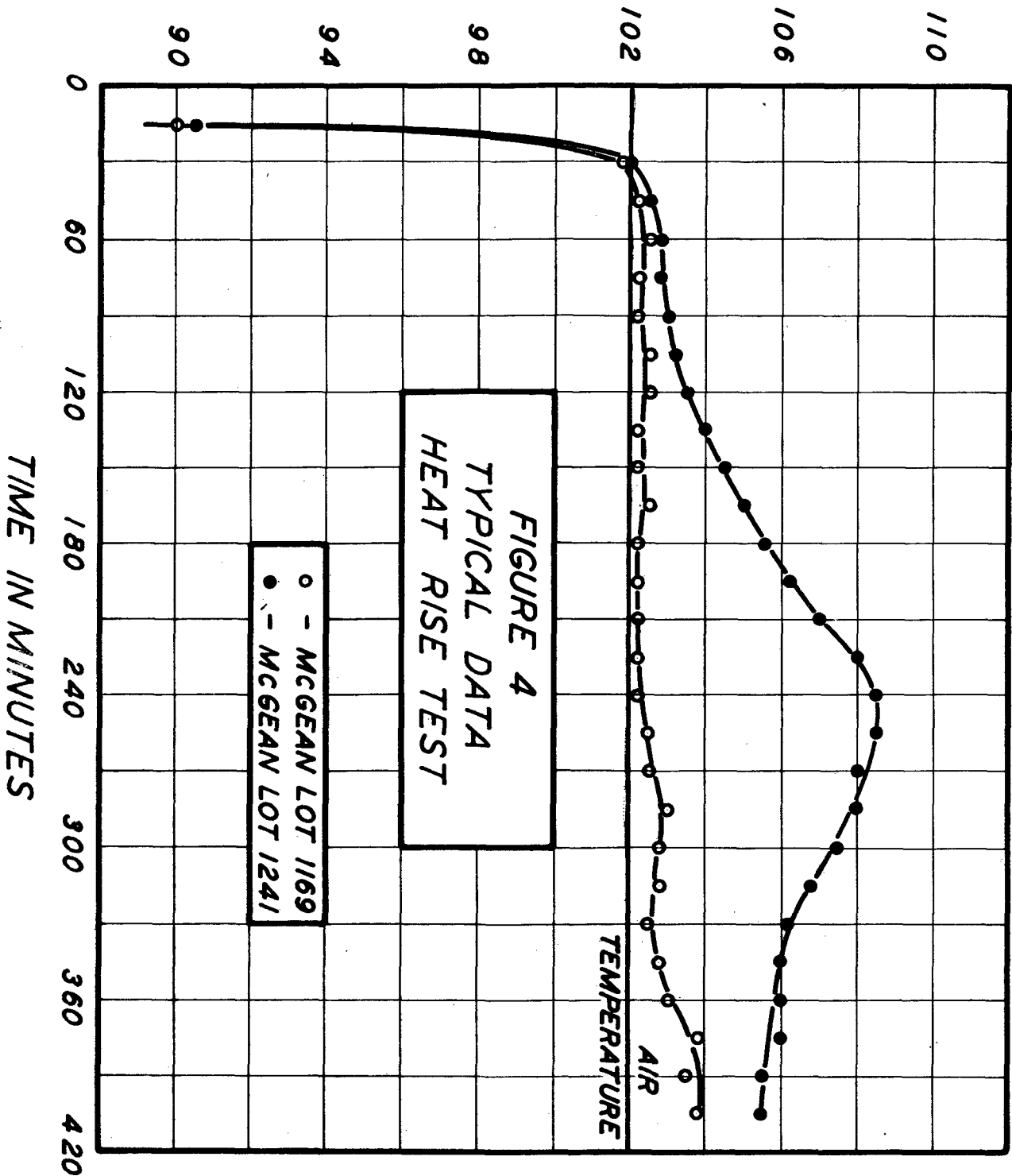
1. The U.O.P. test bomb method<sup>(15)</sup>. This procedure was adapted from that used in gasoline testing<sup>(17)</sup> and consists of subjecting the soap to the action of oxygen in a bomb under elevated pressure and temperature. The pressure in the bomb is recorded automatically on a chart, a sudden dip indicating oxygen absorption and the end of the induction period. Trouble is experienced when sintering occurs because no satisfactory induction period is obtained. For this reason, it has been necessary in most cases to extract soaps with acetone to remove free fatty acids and avoid the sintering. This acetone extraction renders the test of little practical value for ordinary Napalms (see below), although valuable orienting knowledge has been gained by its use.

2. The Mackey or modified Mackey test. In this test a sample of the soap is exposed at an elevated temperature to a current of air (either forced or caused by natural convection) and its temperature followed<sup>(13)</sup>. At first this remains constant but at the end of the induction period shows a sharp rise followed by a decline until the soap again takes the temperature of the surrounding batch (Figure 4). At 100°C. the induction periods of many soaps are too great (over 70 hours) to be followed conveniently by this method. Hence baths of varying temperature were used, 120 to 130°C. appearing to be the most convenient. Nevertheless, soaps which are known to be definitely unstable to oxidation do show a temperature rise at 100°C. and this may well serve, therefore, as a "go-no-go" test for control.

3. Peroxide value. When soaps, which have passed their induction period, are dissolved in chloroform and acetic acid and mixed with potassium iodide, iodine is liberated by reaction of the iodide with peroxides present in the soap, and can be titrated with standard sodium thiosulfate solution. Good soaps show a low peroxide value (mg. iodine/gram soap) over long periods of time while soaps which exhibit a temperature rise on test (2) give a high peroxide value. Figure 5 (49) shows results of iodine and peroxide values on a commercial batch of soap, which had been submitted to a variety of treatments. Curves I and III correspond to normal production batches, which had been washed with water and alum solution, respectively. It will be seen that the iodine and peroxide value curves are opposite in character, the latter going through a maximum and then decreasing. Treatment of the wet pulp with caustic soda

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TEMPERATURE, °C.



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FIGURE 5  
IODINE NUMBER AND PEROXIDE NUMBER  
VS  
TIME  
OVEN 160°F., AMBIENT R.H. APPROX. 5 %  
AIR VELOCITY - 500 FT./MINUTE

○, ● - I - WASHED WELL FOR  
15 MINUTES

△, ▲ - II - 1.5 KG. NAPALM PULP  
4 KG. WATER  
4.0 G. NaOH

□, ■ - III - 1.5 KG. NAPALM PULP  
WASHED  
3 KG. WATER

▽, ▼ - IV - 1.5 KG. NAPALM PULP  
0.5 G. U.O.P. NO. 5  
2 KG. WATER

— IODINE NUMBERS

--- PEROXIDE NUMBERS

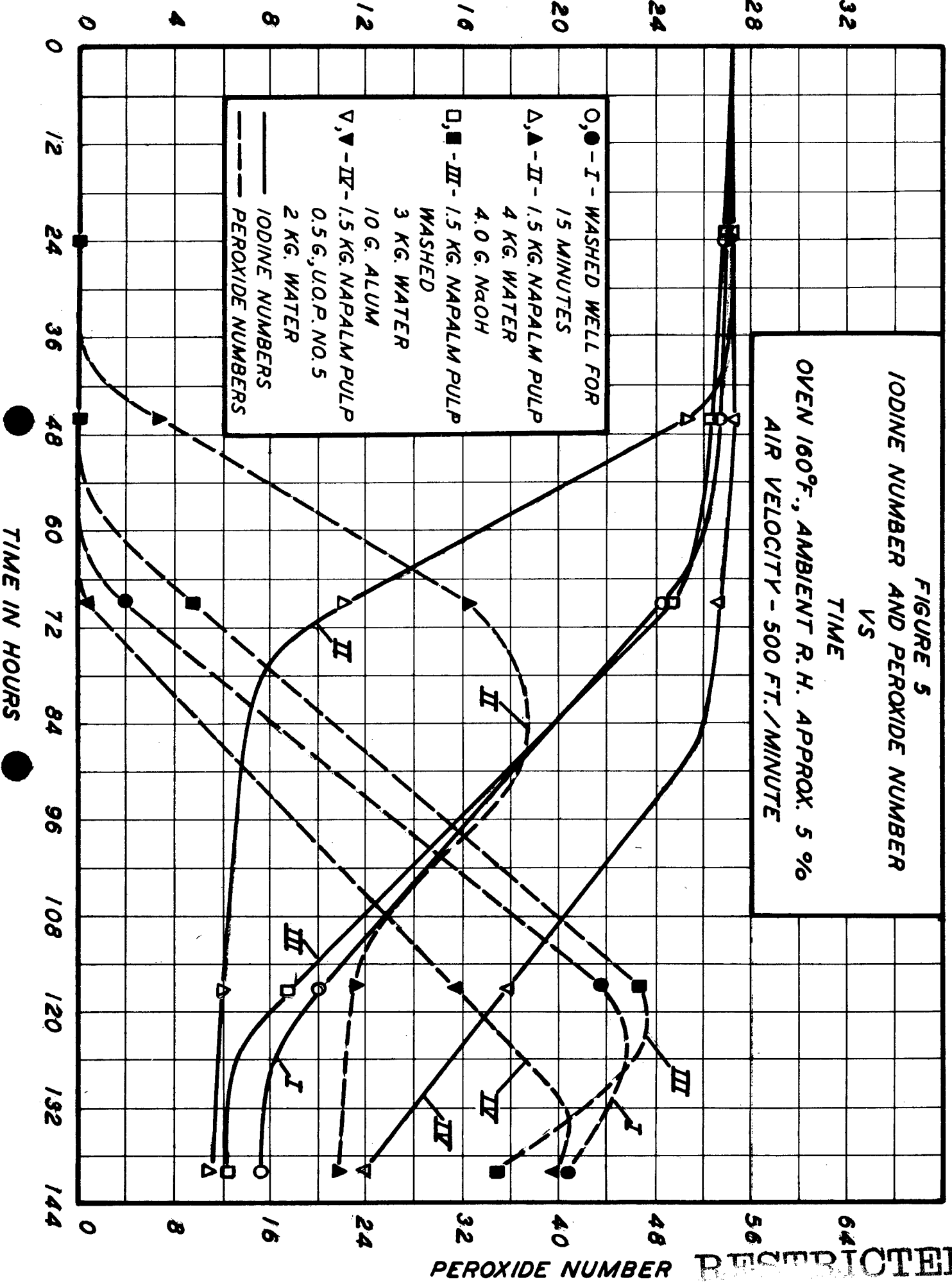




Table IV

Icebine Numbers of Seeps Exposed Under Different Psychrometric Conditions (48)

| Sample #                    | Condition         | Original | 36 Days  | 90 Days  | 120 Days | 165 Days | Gardner Mobilometer Reading | Extensibility |
|-----------------------------|-------------------|----------|----------|----------|----------|----------|-----------------------------|---------------|
| 87, 396                     | 90°F. - 20% R.H.  | 28.6     | --       | 27.3     | 27.6     | 24.6     | 48 hrs. 77°                 | 1"            |
| 87, 905                     |                   | 23.4     | --       | 27.0     | 26.1     | 24.4     | 48 hrs. 77°                 | 1 1/4"        |
| 87, 390                     |                   | 23.5     | --       | 27.5     | 25.1     | 24.7     | 48 hrs. 77°                 | 1 1/4"        |
| 87, 937                     | 120°F. - 20% R.H. | 31.5     | 30.8     | 26.2     | 20.5     | 16.0     | 48 hrs. 77°                 | 1 1/8"        |
| 87, 838                     |                   | 31.7     | 31.5     | 28.5     | 22.4     | 14.2     | 48 hrs. 77°                 | 1 1/4"        |
| 87, 834                     |                   | 27.3     | 27.8     | 23.3     | 17.4     | 12.4     | 48 hrs. 77°                 | 1 1/4"        |
| 87, 835                     |                   | 27.5     | 27.3     | 20.0     | 13.1     | 11.2     | 48 hrs. 77°                 | 1 1/4"        |
| 87, 392                     |                   | 23.7     | 27.4     | 17.3     | 11.5     | 8.5      | 48 hrs. 77°                 | 1 1/4"        |
| 87, 307                     |                   | 23.0     | 27.5     | 22.3     | 18.2     | 13.1     | 48 hrs. 77°                 | 1 1/4"        |
| 87, 891                     |                   | 27.3     | 27.4     | 19.3     | 15.2     | 11.2     | 48 hrs. 77°                 | 1 1/4"        |
| 87, 896                     |                   | 28.6     | 27.7     | 21.3     | 14.2     | 10.2     | 48 hrs. 77°                 | 1 1/4"        |
| 87, 905                     |                   | 28.5     | 27.0     | 21.6     | 15.2     | 11.3     | 48 hrs. 77°                 | 1 1/4"        |
| 87, 890                     |                   | 28.4     | 27.0     | 21.6     | 15.2     | 11.3     | 48 hrs. 77°                 | 1 1/4"        |
| 87, 396                     | 90°F. - 90% R.H.  | 28.6     | 27.2     | 22.9     | 21.0     | 14.8     | 48 hrs. 77°                 | 1 1/4"        |
| 87, 905                     |                   | 28.4     | 27.0     | 22.5     | 20.4     | 14.7     | 48 hrs. 77°                 | 1 1/4"        |
| 87, 390                     |                   | 23.5     | 26.6     | 25.6     | 24.5     | 18.7     | 48 hrs. 77°                 | 1 1/4"        |
| 87, 396 Original            |                   |          |          |          |          |          |                             |               |
| Exposed 90, 20% RH, 7 mos.  |                   | 28.6     | 20 secs. | 65 secs. | 43 hrs.  | 77°      | 48 hrs. 77°                 | 1 1/4"        |
| Exposed 120, 20% RH, 7 mos. |                   | 24.6     | 45       | 105      | 730      |          | 470                         | 1"            |
| 87, 905 Original            |                   | 10.3     |          | 65       | 420      |          | 730                         | 1 1/4"        |
| Exposed 90, 20% RH, 7 mos.  |                   | 23.4     | 25       | 65       | 350      |          | 440                         | 1 1/4"        |
| Exposed 120, 20% RH, 7 mos. |                   | 24.4     | 50       | 110      | 350      |          | 750                         | 1 1/2"        |
| 87, 390 Original            |                   | 10.1     |          | 55       | 330      |          | 500                         | 1 1/8"        |
| Exposed 90, 20% RH, 7 mos.  |                   | 28.5     | 25       | 55       | 780      |          | 420                         | 1"            |
| Exposed 120, 20% RH, 7 mos. |                   | 24.7     | 30       | 90       | 780      |          | 540                         | 1"            |
| Exposed 120, 20% RH, 7 mos. |                   | 12.2     | 44 hrs.  | <36 hrs. | 530      |          | 610                         | 1 1/8"        |

or an anti-oxidant is seen to change the induction period, curves II and IV, the relationship between iodine value and peroxide value persisting. Recently, a specification test based on peroxide value has been suggested (10). Obviously, in using the peroxide value test, care must be taken to ascertain on which side of the maximum the value is situated.

With the above tests it has been possible to trace some of the factors influencing the oxidation.

1. Using the same fatty acids, the length of the induction period appears to be dependent upon the soluble iron content of the alum used in the precipitation (Table V). Soaps with a high iron content usually, but not invariably, have a low induction period (15). Manganese, in quantities greater than 0.02%, has also been found to be a very powerful oxidation catalyst (50) and there is some evidence that copper may also serve in this capacity.

Table V

The Effect of Iron on the Induction Period of Napalm (3)

|   | <u>Iron Content</u> | <u>Induction Period on Modified Mackey Test</u> | <u>Peroxide Value After Drying</u> |
|---|---------------------|---|------------------------------------|
| Lab. batch made with pure alum                  | 0.04<br>0.005       | Negative<br>"                                   | 10<br>--                           |
| Lab. batch made with Victory grade alum         | 0.24<br>--          | 95 mins.<br>60 "                                | 29.6<br>9.5                        |
| Lab. batch made with alum, ferric sulfite added | 0.30<br>0.08        | 75<br>85  | 32.4<br>--                         |

2. The induction period can be lengthened by the addition of suitable antioxidants. The Shell Development Company (15), using the oxidation test #1 above as a guide, have done considerable work along these lines and suggest that UOP #5, n-dilauryl para-phenylene diamine and 4-ortho toluidino-stearic acid should all be satisfactory in preventing the oxidation of Napalm during storage for six months at 130° F. Limited data on gel strengths of Napalms to which these inhibitors have been added are shown in Table VI.

**RESTRICTED**Table VI

Oxidation Stability and Specification Consistency Tests  
on Napalm Batches Made at the California Ink Company, Inc. (51)

| <u>Sample</u>                                  | <u>Vac. Oven</u><br><u>Water, %</u> | <u>Viscosity</u><br><u>48 hrs. 77°F</u> | <u>Viscosity 150°</u><br><u>Heat Stab. Test</u> | <u>IP, min.</u><br><u>standard</u> |
|--|-------------------------------------|---|---|------------------------------------|
| California Ink<br>Batch 5<br>1% Naphthol       | 0.35                                | 760                                     | 560   | 195                                |
| California Ink<br>Batch 6<br>No inhibitor      | 0.6                                 | 830                                     | 780   | 720 ?<br>sintering                 |
| California Ink<br>Batch 8<br>1% Naphthol       | 0.68                                | 750,770                                 | 705,730   | sintered                           |
| California Ink<br>Batch 14<br>1% o-anyl phenol | 0.95                                | 725,735                                 | 705,680   | >77 hrs.*                          |
| California Ink<br>Batch 302<br>0.02% Lorene    | 1.05                                | 585                                     | 555   | >77 hrs. *                         |

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Table VII

Induction Periods of Various Napalms<sup>(15)</sup>

|   | <u>Temperature</u> | <u>Pressure<br/>Oxygen p.s.in.</u> | <u>Induction<br/>Period, mins.</u> |
|---|--------------------|------------------------------------|------------------------------------|
| <u>A. Experiments with Solid Napalm</u>         |                    |                                    |                                    |
| Napalm  | 100                | 100                                | 205                                |
| Acetone Extracted Napalm                        | 100                | 100                                | 50                                 |
| Aluminum Soap Talloil Acids                     | 80                 | 100                                | 120                                |
| Above + Napalm Extract                          | 80                 | 100                                | 360                                |
| Unextracted Napalm + 0.01%<br>UOP #5 sprayed on | 100                | 100                                | 1470                               |
| Extracted Napalm + 0.01%<br>UOP #5 sprayed on   | 100                | 100                                | 650                                |

B. Inhibitors Introduced During Napalm Preparation. Tests at 100°C. and 100 p.s.i.

|   | <u>% Inhibitor<br/>Added</u> | <u>Induction<br/>Period, mins.</u> |
|---|------------------------------|------------------------------------|
| None  | 0                            | 20                                 |
| Hydroquinone  | 1*                           | 25                                 |
| duPont #19  | 1                            | 150                                |
| Lecithin  | 1                            | 20                                 |
| SPC   | 1                            | 345                                |
| Rubber inhibitor composed<br>of phenyl $\gamma$ &<br>$\beta$ naphthylamines | 1                            | 97                                 |
| $\gamma$ -Naphthol  | 1*                           | 307                                |
| 2,4,6-Trimethylphenol   | 1                            | 95                                 |
| 2,4-Dimethylphenol  | 1                            | 63                                 |
| 2,4,5-Trimethylphenol   | 1                            | 49                                 |
| 2,4-Dimethyl-6-butylphenol  | 1                            | 70                                 |
| 3,3',5,5'-Tetramethyl-4,4'-<br>dihydroxy diphenyl                           | 1                            | 17                                 |
| Santolene A   | 1                            | 53                                 |
| UOP #5  | 1*                           | 519                                |
| Aerohib   | 1                            | 64                                 |
| Flöyd's base #28  | 2                            | 1                                  |
| Aerolube BU   | 1.49                         | 21                                 |

**RESTRICTED**Table VII Cont'd.

|  | <u>% Inhibitor<br/>Added</u> | <u>Induction<br/>Period, mins.</u> |
|--|------------------------------|------------------------------------|
| <u>C. Inhibitors Introduced by Varying Methods</u> |                              |                                    |
| None   | 0                            | 205                                |
| N-phenyl N'-dihydro-<br>isophoryl thiourea**       | 0.1                          | 195                                |
| Alphatol (o-ortho<br>toluidino stearic acid)**     | 1                            | 300                                |
| o-(para-amino anilino)<br>stearic acid**           | 1                            | 410                                |
| o-(para-oxyanilino)<br>stearic acid**              | 0.25                         | 1225                               |
| N-octadecyl paraamino-<br>phenol***                | 0.1                          | 830                                |
| N-dilauryl paraphenylene<br>diamine***             | 0.02                         | 730 (96.7°C)                       |

\* Considerable loss to mother liquor owing to solubility of inhibitor in same.

\*\* Coprecipitated.

\*\*\* Sprayed on soap granules.

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Of the materials readily available, UOP #5 and 2-naphthol appear most suitable.

3. Napalm itself contains natural antioxidants. Thus, (Table VIIA), a sample of Napalm had an induction period of 205 minutes (line 1) when tested by the UOP bomb method at 100°C. and 100 lb. oxygen pressure. After three extractions with five times its weight of acetone, the induction period fell to 50 minutes (line 2). The extract after removal of fatty acids was incorporated into an aluminum soap made from talloil acids in the same proportion at which it was removed from the Napalm, namely 1.6%. The induction period of the talloil sample was then found to be increased from 120 (line 3) to 660 minutes (line 4) at 80°C. and 100 lb. oxygen pressure. The acid-free acetone extract was found to contain about 25% of full range alkyl phenols, indicating that there are about 0.4% of these in the original Napalm. Their odor and physical properties indicate that they were introduced via the naphthenic acid, in which they were noted to be present as impurities. It is clear, therefore, that Napalm is stabilized to oxidation by the presence of impurities in the naphthenic acid and that we have here a known, but as yet uncontrolled, source of variation in the oxidation susceptibility of Napalm.

Oxidation would be expected to be favored by small particle size (3). There is some indication that the oxidation troubles in commercial production at one plant, and in trial batches made by an N.D.R.C. contractor were due to this cause.

At the present time the incomplete knowledge of the factors favoring oxidizability of Napalm is one of the most serious dangers of the Napalm program. It is known that many of the soaps prepared commercially show temperature rises within eight hours on the modified Mackey test at 100°C. while others show no rise under these conditions. Furthermore, sufficient long-time-exposure tests have now been run to show varying susceptibility to oxidation under ordinary keeping conditions. It is clear, therefore, that commercial samples of Napalm vary widely in their oxidation susceptibility and, therefore, not all will be suitable for storage for future use as Type A Napalm. Furthermore, while three possible causes, namely iron content of the aluminum sulfate, natural inhibitor content of the naphthenic acid and particle size, have been suggested for the variability, there is no guarantee that other factors may not also be at work. Preliminary work at the Columbia University Laboratories of C.W.S. (10) indicates that oleic acids vary widely in their susceptibility to oxidation. The Colgate-Palmolive-Peet Company also believe that the linolenic acid content of the oleic acid used in Napalm manufacture may have considerable influence upon the oxidizability of the Napalm.

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The above points require further elucidation. In the meantime, it seems advisable to be as cautious as possible in the selection of raw materials for Napalm manufacture. Although low iron content may not be essential in all cases, it would seem reasonable to use acids having as low iron analysis as possible, and to use iron-, manganese-, and copper-free alum. No method has yet been worked out for measuring the natural inhibitor content of the naphthenic acid, but it may be necessary to use naphthenic distilled at one source and adjusted to a definite inhibitor content. Alternatively, sufficient inhibitor such as  $\lambda$ -naphthol, UOP #5, etc., may have to be added to take care of all possible natural inhibitor variations. Particle size is relatively easily controlled in the manufacturing operations.

#### The Dispersion of Napalm

All Napalm for its final use must be dispersed in gasoline or other hydrocarbon solvent. It was apparent very early that the solvent played a part in determining the rate of solution of the soap, and the consistency and other characteristics of the gel formed. Cyclohexane and aromatic hydrocarbons in general were found to give stronger gels than aliphatic materials (21, 22). Furthermore, the setting and dispersion times of the soaps were markedly affected by the nature of the hydrocarbon used. For this reason, the standard test gasoline, prepared by Standard Oil Development, has always been used for specification work and comparative experiments on different soaps. Attempts are now being made to replace this by a standardized naphtha so that errors due to evaporation, difficulties in standardization of the gasoline, etc. can be minimized.

Investigations at Standard Oil Development and Edgewood Arsenal (22, 23), using a fairly wide range of motor gasolines, including those which have been used in the filling plants, indicate no comprehensive correlation between any of the physical properties of the hydrocarbons and those of the gels. In Table VIII, comprising results on a series of hydrocarbons ranging from cyclohexane to paraffins, the hydrocarbon had no appreciable effect on gel viscosity with the exception of cyclohexane, which yielded gels of higher viscosity, and a steam cracked gasoline which resulted in gels of very low consistency. Neither of these materials is likely to be met in practice. Nevertheless, difference in gasoline quality might be sufficient to give unstable gels with specification Napalm, and might be of extreme importance in the field of use of Napalm Type A.

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The deleterious effects of some commercial gasolines are illustrated in the following table. All of these gasolines represent commercial products which were submitted for use at Napalm gel compounding plants.

| Gasoline                                  | Mobilometer Readings<br>(3% Gel) |                    | Phenol*<br>Content<br>p.p.m. |
|---|----------------------------------|--------------------|------------------------------|
|   | 43 hrs.<br>77° F.                | 24 hrs.<br>150° F. |                              |
| Standard Oil Development<br>Test Gasoline | 770                              | 680                | --                           |
| A   | 760                              | 630                | 1.4                          |
| B   | 700                              | 580                | 1.4                          |
| C   | 630                              | 330                | 23                           |
| D   | 320                              | 650                | 46                           |
| E   | 270                              | 175                | 200                          |

\* Determined by the method of Wetlaufer, VanNatta and Quattlebaum (56)

Gasoline B had an Aniline Point of 137° F. Present specifications limit this test to 120° F. maximum. All of the other Aniline Points lay between 95 and 110° F. Some correlation between phenol content and gel performance may be noted, though other effects, such as the high Aniline Point of Gasoline B, also appear to be operating. This correlation is supported by the fact that Gasoline E when washed with dilute NaOH, rinsed with water, and dried gave a gel performance matching that of the Test Gasoline. Peroxides and acids, as well as the phenols, would be removed by this treatment, so it cannot be stated that only phenolic compounds caused the original gel instability.

Another gasoline has been encountered which was so corrosive that it discolored the brass Mobilometer disc. Actual Mobilometer readings are not available, but it can be stated qualitatively that the gels were unstable. Treatment with ammoniacal copper solution, to remove the active sulfur compounds, gave a gasoline showing proper gel stability. The phenol content of this gasoline was zero.

Certain inhibitors of the type usually added to gasoline were found to have little effect on gel consistency (Table IX).

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In contrast to consistency, the rate of solvation of the Napalm was changed by at least a factor of three in passing through the range of different gasolines (Table VIII). Limiting consideration to the regular motor gasolines, the solvation rate is predicted by the aniline point.

It was found that long time storage at  $-20^{\circ}\text{F}$ . was affected by the gasoline used (Table VIII). With gasolines of very high aniline point, synecresis occurred after several months. However, gasolines having aniline points below  $120^{\circ}$  did not show more than 4-5% synecresis over a period of three months storage at  $-20^{\circ}\text{F}$ .

Table VIII  
Effect of Gasoline on Properties of Magnal Thickened Fuels  
(All gels contained 3% by weight Type B soap compounded at 77°F.)

| Batch No. | Origin of Hydrocarbons | Grav. % API | Pt. of Anil. | Acid Heat | Bromine No. | Refr. Index | ASTM Octane Number Clear | ASTM Number + TEL | Gasoline Inspections        |                        |
|-----------|------------------------|-------------|--------------|-----------|-------------|-------------|--------------------------|-------------------|-----------------------------|------------------------|
|           |                        |             |              |           |             |             |                          |                   | Breakdown (d) in 2 lb. drop | Minutes for 5 lb. drop |
| 1481      | Cyclohexene            | 50.6        | 35           | 5         | --          | 1.4106      | --                       | --                | 1100                        | 1440+                  |
| 1432      | Toluene                | 31.2        | --           | 22        | --          | 1.4362      | --                       | --                | 1440+                       | --                     |
| 1433      | Disobutylene           | 34.3        | 111          | 236       | 139         | 1.4106      | --                       | --                | --                          | --                     |
| 1484      | Ref. Nap. M3(c)        | 61.2        | 140          | 0         | 0.2         | 1.4109      | 22.7                     | --                | 915                         | 1440+                  |
| 1485      | Coastal Gas.           | 54.1        | 127          | 2         | 0           | 1.4220      | 64.0                     | 75.9              | 1440+                       | --                     |
| 1436      | Trade Gasoline         | 59.7        | 121          | 59        | 6.0         | 1.4041      | 73.0 (F)                 | --                | 430                         | 325                    |
| 1487      | Trade                  | 61.2        | 115          | --        | --          | 1.4119      | 55.1                     | --                | 135                         | 160                    |
| 1479      | Trade                  | 63.6        | 111          | 89        | --          | 1.4073      | 57.4                     | 30.5              | 130                         | 135                    |
| 1500      | Trade                  | 50.0        | 101          | 33        | 37.3        | 1.4173      | 74.8 (e)                 | --                | 375                         | 420                    |
| 1501      | Blended                | 57.3        | 75           | 116       | 42.6        | 1.4211      | 74.2                     | --                | 45                          | 30                     |
| 1502      | " Test"                | 50.9        | 96           | 154       | 49.4        | --          | --                       | 35.0              | 70                          | 30                     |
| 1513      | Hydroformed            | 55.4        | 77           | 20        | 5.6         | 1.4270      | 71                       | 35.4              | 305                         | 435                    |
| 1515      | Gasoline-98% Benzol-2% | 61.2        | 107          | 85        | 29.8        | 1.4111      | --                       | --                | 50                          | 55                     |
| 1514      | Steam cracked          | 52.2        | 29           | 448       | 130         | 1.4396      | --                       | --                | --                          | --                     |

(a) Stored at room temperature except where noted. Each batch of gel was stored in two containers. The 2 and 7 day tests were run on one sample and the later tests on the second sample. Gardner viscosities are for S.O.D. machine on which correction factor is 0.91

(b) Test carried out in accordance with specific instructions.

(c) Aniline point by S.O.D. method which gives values essentially equal to ASTM procedure.

(d) Breakdown run by S.O.D. method, using smaller sample (25 ccs) and bomb than ASTM standard.

(e) From Michigan Crude.

(f) Contained 1 cc. TEL/gallon.

(g) Contained 0.56 cc. TEL/gallon.

(h) Contained 2 cc. TEL/gallon.

Table VIII Cont'd.

| Batch No. | Compounding Time in Minutes | Dispense | Viscosity of Gels (a)<br>Gardner cms./100 sec. |        |         |         |                              |                              | Liquids Separation, % |                  |
|-----------|-----------------------------|----------|--|--------|---------|---------|------------------------------|------------------------------|-----------------------|------------------|
|           |                             |          | 2 days   | 7 days | 55 days | 72 days | 55 days + 44 hrs. at 150° F. | 72 days + 44 hrs. at 150° F. | 2 days                | 4 hrs. at 40° F. |
| 1481      | 0.33                        | 1.65     | 835  | 1030   | 1010    | -       | 950                          | -                            | 0                     | 0                |
| 1482      | 0.75                        | 1.05     | 835  | 790    | 910     | -       | 1020                         | -                            | 0                     | 0                |
| 1483      | 7.5                         | <1.0     | 330  | 790    | 635     | -       | 790                          | -                            | 0                     | 0                |
| 1484      | 2.5                         | 7.0      | 650  | 695    | 675     | -       | 625                          | -                            | 0                     | 0                |
| 1485      | 0.12                        | 2.0      | 800  | 820    | 930     | -       | 940                          | -                            | 0                     | 0                |
| 1486      | 0.12                        | 0.33     | 800  | 820    | 790     | -       | 755                          | -                            | 0                     | 0                |
| 1487      | 0.17                        | 0.50     | 710  | 700    | 960     | -       | 975                          | -                            | 0                     | 0                |
| 1489      | 0.13                        | 0.40     | 905  | 920    | -       | 350     | -                            | 0                            | 0                     | 0                |
| 1500      | 0.07                        | 0.20     | 345  | 320    | -       | 750     | -                            | 0                            | 0                     | 0                |
| 1501      | 0.07                        | 0.13     | 300  | 735    | -       | 830     | -                            | 0                            | 0                     | 0                |
| 1502      | 0.07                        | 0.17     | 855  | 815    | -       | 690     | -                            | 0                            | 0                     | 0                |
| 1513      | 0.20                        | 0.70     | 720  | 750    | -       | 690     | -                            | 0                            | 0                     | 0                |
| 1515      | 0.07                        | 0.17     | 735  | 730    | -       | 820     | -                            | 0                            | 0                     | 0                |
| 1514      | 0.13                        | 0.33     | 185  | 175    | -       | 55      | -                            | 0                            | 0                     | 0                |

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Table IX

Effect of Gasoline Inhibitors on Properties of Naphalm Thickened Fuels (a)

| Batch No. | Inhibitor  | Conc. of Inhibitor<br>gms./100 ml. | Viscosity of Gels-Gardner gms./100 sec. (b) |      |      |                     |
|-----------|------------|------------------------------------|---|------|------|---------------------|
|           |            |                                    | Days  | Days | Days | 24 hours at 150° F. |
| 1541      | Dupont #5  | 10                                 | 715   | 700  | 730  | 350                 |
| 1542      | Dupont #6  | 10                                 | 705   | 530  | 730  | 330                 |
| 1543      | U.O.F. #1  | 20                                 | 710   | 710  | 375  | 355                 |
| 1544      | U.O.F. #4  | 10                                 | 705   | 710  | 940  | 330                 |
| 1545      | U.O.F. #5  | 10                                 | 770   | 720  | 765  | 930                 |
| 1546      | FX-441     | 10                                 | 770   | 765  | 720  | 900                 |
| 1547      | X Naphthol | 10                                 | 765   | 700  | 765  | 810                 |

(a) Inhibitors added to "Fust" gasoline; 3% Naphalm in gasoline.

(b) Stored at room temperature except as noted. Made as one batch stored in two containers; one container being used for 2 and 7 day tests and the other for later tests. Gardner viscosities are for S.O.D. machine and may be multiplied by 0.91 to yield values corresponding to standard C.W.S. machine.

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THE TESTING OF NAPALM

The object of the testing procedure for Napalm has been to make tests available which would insure a satisfactory end product for use in the field.

The ideal requirements for a satisfactory thickening agent for gasoline are:

1. The rheological behaviour of the gel must be non-Newtonian and pseudoplastic.
2. A sticky gel with appreciable string must be formed. It must not be "short" or crumbly.
3. The gel must have reproducible consistency at any given concentration.
4. The consistency must not be too greatly affected by variations in gasoline quality.
5. Mixing of the thickener with gasoline to give a uniform gel must be easy.

For Type B uses

1. The gel must be stable from  $-20$  to  $120^{\circ}$  F. over long periods of time to allow for variable storage conditions.
2. The gel must be stable and ignite\* at  $-40^{\circ}$  F.

For Type A uses

1. The solid soap must be stable over long periods at both low and elevated temperatures giving a satisfactory gel when mixed with Quartmaster pool gasoline.

In addition to the routine analytical tests on the solid soap, which would describe its chemical composition, it seemed very necessary to have some form of consistency test on the gel formed in gasoline. Early work at Eastman Kodak<sup>(52)</sup> and Standard Oil Development<sup>(5)</sup> had indicated that the apparent consistency of the gel was closely related to its performance in actual use. With this in mind, the specification C.S. 196-131-107 was issued 11/19/42 to describe as closely as possible the first satisfactory samples of Napalm (originally known as X-104). It was believed to be unnecessary to differ-

\* This is more a function of the gasoline than of the solid thickener employed.

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entiate between Napalm Types A and B except for the specification of a certain minimum setting time for Type A, designed to allow satisfactory field mixing (5). Since little was known of the effect of many variables in the manufacture, it was believed necessary to include limits for aluminum content, a test for free sulfate, etc. On the basis of satisfactory results, the Gardner Viscosimeter was adopted as the standard instrument for measuring gel consistency. It was expected from the scanty knowledge of the soap available at the time that trouble might be experienced with the specifications and frequent revision would be necessary.

By the time of the January meeting on Napalm at Bayway (3), it had become clear that some revisions of the specifications might be required, and in particular that the method for running gel consistency would require more investigation and very close specification. To do this, a joint CWS-NDRC committee was formed and met several times at Edgewood to formulate adequate testing methods. With respect to consistency determinations, considerable work on ten Napalms at a number of concentrations (7,18,28) led to the following conclusions:

1. The Gardner instrument, of the viscosimeters tried, yielded most consistently reproducible results.
2. At soap concentrations above 9%, reproducibility is greatly impaired. For this reason 8% was picked as a standard testing concentration.
3. Providing the gel is worked once, reproducible readings are obtained at 8% concentration. It is unnecessary to work the gel ten times.
4. Check readings within 30 grams can readily be obtained on the same gel if a rigidly standardized procedure is followed.

As a result of this work, Directive No. 201 was issued 4/24/43, followed by its revision No. 201A, 7/16/43. (Appendix VIII).

As knowledge of the properties of Napalm increased, it became evident that some of the specifications were of little utility in guaranteeing good material. Hence, certain requirements of the original specification, e.g. aluminum and sulfate contents, were removed. It also became clear that the most important danger to be guarded against was inadequate

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stability of the gasoline gel on keeping. In this respect, however, the drop in viscosity on keeping from two days to seven days was found to have little significance.<sup>(5)</sup> Furthermore, the waiting involved in the seven-day test was troublesome to the manufacturers, causing a large tie-up of material awaiting approval. For this reason, the seven-day, 77°F. test was dropped when revised specifications were issued 4/26/43. In these, the 48 hr. 77°F. + 24 hrs. 150°F. test in a steel tube was retained, the viscosity being found to drop to a constant level and decrease no further. This drop is believed to be equivalent to that which the gel would undergo on standing for several months at room temperature. This test, however, still involved a wait of two days at 77°F. before the one day at 150°. It was thought that one day at 150°F. applied immediately after making up the gel would give similar results and this was found to be the case (24,25). Table X indicates negligible change in consistency after 24 hrs. at 150°F. Table XI presents a comparison of the two types of specifications.

Table X

Time and Consistency after Storage at 150°F. (53)

(pairs of numbers are duplicate tests, Gardner reading)

|                         | Nuodex  |         | McGean  | Eakins  | Pfister | Nuodex |
|-------------------------|---------|---------|---------|---------|---------|--------|
|                         | 89080   | 89088   | 684     | 24      | 30A     | 83955R |
| 2 hrs. 150°F.           | 510,490 | 710,730 | 540,560 | 730,710 | 500,480 |        |
| 4 hrs. 150°F.           | 430,440 | 740,730 | 540,530 | 720,730 | 470,430 | 600    |
| 16 "                    | ---     | 560     | ---     | ---     | ---     | 420    |
| 24 "                    | 350,350 | 580,560 | 520,530 | 640,640 | 480,530 | 510    |
| 48 "                    | 380,350 | 590,590 | 520,500 | 580,600 | 450,520 | 560    |
| 96 "                    | 350,270 | 530,530 | 370,430 | 570,570 | 430,460 | 540    |
| 48 hrs. 77° F.          | 530,660 | 750,300 | 540,550 | 740,700 | 640,620 | 610    |
| 163 hrs. 77°F.          | ---     | 540     | ---     | ---     | ---     | 640    |
| % Moisture              |         |         |         |         |         |        |
| Vac. Oven 5 hrs. 55° C. | 1.2     | 0.40    | 0.65    | 0.50    | 0.24    | --     |
| CWS Benzol              | 1.7     | 0.9     | 1.1     | 0.6     | 0.6     | 0.8    |

Table XI

Comparison of 150° F. Keeping Tests

|  | Nuodex <sup>(54)</sup><br>15374 | Nuodex <sup>(54)</sup><br>18032 | Harmon <sup>(54)</sup><br>88956 | Nuodex <sup>(23)</sup><br>89080 | Nuodex <sup>(55)</sup><br>88955R |
|--|---------------------------------|---------------------------------|---------------------------------|---------------------------------|----------------------------------|
| % Vac. Oven Moisture<br>(24 hrs. 60° C.) | ---                             | 1.10                            | 1.50                            | ---                             | ---                              |
| % Vac. Oven Moisture<br>(5 hrs. 55° C.)  | ---                             |                                 |                                 | 1.2                             | ---                              |
| % Moisture CWS<br>Benzol                 | ---                             |                                 |                                 | 1.7                             | 0.8                              |
| 44 hrs. 77° F.                           | 640,690                         | 590,550                         | 630,610                         | ---                             | ---                              |
| 48 hrs. 77° F.                           | 840                             | 640,620                         | 610,620                         | 560,630                         | 610                              |
| 43 hrs. 77° F. +<br>24 hrs. 150° F.      | 640                             | 560                             | 380,380                         | 370,350                         | 570                              |
| 24 hrs. 150° F.                          | 510                             | 510,510                         | 340,390                         | 360,350                         | 510                              |
| 2 hrs. 150° F.                           | 665,710                         | 550,550                         | 500,500                         | 510,490                         | 660                              |
| 168 hrs. 77° F.                          | ---                             | 690,690                         | 530,530                         | 640                             | 640                              |

The new specifications now allow testing to be completed in about fifty hours after preparation of the soap. At the same time that this last change was made, a two-hour at 150° F. test was also introduced. It was thought that this would indicate the maximum consistency attainable by the particular gel, but subsequent studies do not confirm this.

As pointed out in the next section, the moisture content of the solid soap has a profound effect upon its properties in the gel state. Nevertheless, the determination of moisture in a material of this type is exceedingly troublesome. In the original specifications, the Dean and Stark method for determination was adopted, it being thought that this measured approximately the "free" moisture of the soap. An investigation clearly showed the amount of water collected depended upon the boiling point of the solvent used, the length of time of distillation, etc. (18,20). Moisture specification consequently was allowed to lapse for a time. However, with the onset of

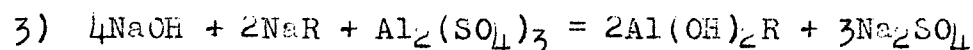
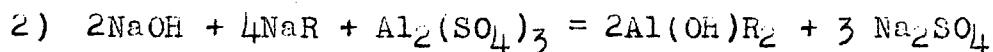
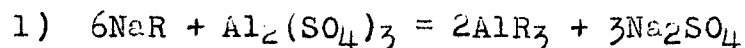


humid summer weather it became clear that many manufacturers were allowing soaps to pick up moisture before or during packaging, which subsequently caused failure in the 150° consistency test. For this reason the new moisture specification was introduced July 8, 1943, a limit of 0.8% by benzol distillation being set.

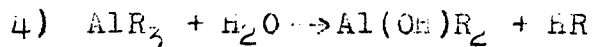
It will be evident from the present specifications (Appendix VIII) that principal reliance for securing good product is now placed upon (1) control of moisture content and (2) control of consistency after exposure to one day at 150° F. The introduction of a test for oxidation susceptibility into the specifications may be necessary, particularly since reports of oxidized soaps have been received from overseas and from compounding plants.

**RESTRICTED**Appendix IThe Manufacture of NapalmExpansion of Section A (p.3), The Mechanism of Precipitation.

A more complete representation of the Napalm precipitation process is given by the following equations:



Equation 1 is theoretical, since aluminum soaps hydrolyze --



In the above equations, HR denotes the mixture of coconut, naphthenic and oleic acids used in Napalm. In this case, the molecular weight of HR is approximately 233, and the corresponding aluminum contents of the three soaps shown above are:  $\text{AlR}_3$ , 3.74%,  $\text{Al}(\text{OH})\text{R}_2$ , 5.30%, and  $\text{Al}(\text{OH})_2\text{R}$ , 9.22.

Analyses show that most satisfactory Napalms contain between 5.4 and 5.8% Al. In terms of the above compounds, this indicates a mixture of  $\text{Al}(\text{OH})\text{R}_2$  and  $\text{Al}(\text{OH})_2\text{R}$  containing between 97.8 and 97.8%  $\text{Al}(\text{OH})\text{R}_2$ . Because of hydrolysis, all Napalms, contain some free acid, so the actual percentage of  $\text{Al}(\text{OH})_2\text{R}$  is probably higher than the 2.2 to 12.2% indicated.

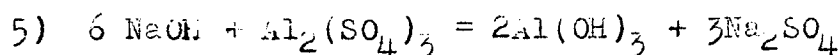
The NaR is formed by the reaction of NaOH and HR. It will be seen that Equations 1, 2, and 3 all involve a total of 6 mols of NaOH. In Equation 1, all 6NaOH have been converted to NaR. In Equation 2, 4NaOH are present as soap, and 2NaOH are free; thus, the ratio of free to combined NaOH is 50%. In Equation 3, the ratio is 200%. To give a mixture of 97.8% of the product of Equation 2, and 2.2% of Equation 3, the ratio of free to combined NaOH can be calculated to be 53.3%. For 87.8% of the produce of Equation 2 and 12.2% of the product of Equation 3, the ratio would be 69.3%. Hence, the range of aluminum contents can be controlled by the ratio of free to combined NaOH, over the range of 53 to 69%. Successful Napalm manufacture actually uses about 60% free caustic.

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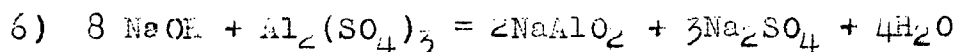
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$\text{Al}(\text{OH})\text{R}_2$  contains 91.3% of the gasoline-soluble R, while  $\text{Al}(\text{OH})_2\text{R}$  contains 79.2%. R. Expressed in another way,  $\text{Al}(\text{OH})\text{R}_2$  contains only 8.7% of the gasoline-insoluble Al and OH, while  $\text{Al}(\text{OH})_2\text{R}$  contains 20.8% of such groups. Hence  $\text{Al}(\text{OH})\text{R}_2$  would be expected to give a weaker, more extensible gel in gasoline, too weak for the intended purpose.  $\text{Al}(\text{OH})_2\text{R}$ , however, would lack solubility, would be extremely difficult, if not impossible to disperse at room temperature, and would give a low extensibility gel if dispersed. The mixture obtained by using from 53 to 69% free caustic is thus a balance of desirable and undesirable properties: sufficient extensibility and gel strength, with the ability to disperse at room temperature in gasoline. The ratio of free to combined NaOH controls the relative proportions of Equation 2 and Equation 3, and hence the gel strength of the product (Table I, p. 4). It is seen that high ratios of free NaOH give too much  $\text{Al}(\text{OH})_2\text{R}$ , which will not disperse, and hence the gel strength decreases if the ratio is too high. The maximum strength appears to occur at about 60% free caustic.

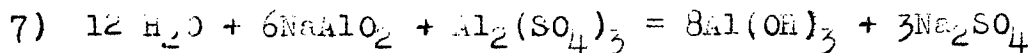
Equations 1, 2 and 3 indicate that 6 mols of NaOH are used in all cases, regardless of the ratio of free to combined NaOH. These 6 mols require, in all cases, one mol of  $\text{Al}_2(\text{SO}_4)_3$ . If no fatty acid were present, the reaction would be:



That is, one mol of alum reacts with 6 mols of NaOH, regardless of the amount of fatty acid present. The quality of napalm formed is controlled by the ratio of NaOH and HR, not by the ratio of NaOH and alum. This is further borne out by a secondary reaction which occurs when alum is added to NaOH:



Sodium aluminate forms when the pH is above 10.6. Aluminum hydroxide will not form until the pH drops below 10.6:



Similarly, Napalm will not form, if fatty acid is present, until the pH drops below 10.6. If HR is present, the R combines with the Al to form a soap. If no HR is present, the reaction is similar, except that the precipitate is a hydroxide.

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As stated in Section A, the actual precipitation may be viewed as a titration in which alum is added to NaOH and/or NaR. Figure I illustrates this. Curve I was obtained in the laboratory, using the plant solutions employed on a large scale in Curve II, but without the fatty acids. The break at pH 10.6 occurs with less alum in Curve II because only the free NaOH forms the aluminate (Equation 6). The second break occurs when both free and combined NaOH are neutralized. This is independent of the ratio of free to combined NaOH. Consequently, the coagulation point occurs at the same pH (about 7) regardless of the ratio of free to combined NaOH.

Curve III shows data for Method II. Here the free NaOH (as  $\text{Na}_2\text{CO}_3$ ) has been added to the alum. The absence of free NaOH in the soap precludes any aluminate formation. Napalm begins to precipitate as soon as alum is added, while in Method I, about 35% of the alum is added before the pH drops below 10.6, and Napalm precipitates. The presence of the free NaOH (as carbonate) in the alum explains the higher final pH of Curve III.

At the present time, there is evidence that the presence of  $\text{Al}(\text{OH})\text{SO}_4$  in the alum solution used for precipitation is an added source of hydroxyl ions, and may therefore effectively increase the ratio of free to combined caustic. Work is in progress to clarify this point.

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Appendix II

Batch Method I

As stated elsewhere in this report, this variation of the coprecipitation method is being recommended as a standard process. The basis for this choice is the greater quantity of information available from the manufacturers employing this method. The following discussion will, therefore, be expanded in considerable detail in order that it may serve as a plant procedure to any new manufacturers who might be asked to produce Napalm. Where specific quantities are given, they are averages of the actual quantities used by present manufacturers. Figure 6 shows a schematic flow sheet for a 2000# batch of Napalm manufactured by the recommended procedure. Table XII gives a summary of material quantities and tank sizes for any desired size of batch of Napalm with specific values for a 2000# batch.

1. Raw Materials

a. Acids

Variations in acid quality have been found to be partly responsible for differences in the character of the soap produced. Close adherence to specifications by regular laboratory testing of new shipments is a prerequisite of satisfactory Napalm production. It is also advisable to retain samples (about 1 gal.) of each new batch for future reference if necessary. The following specifications have been found suitable.

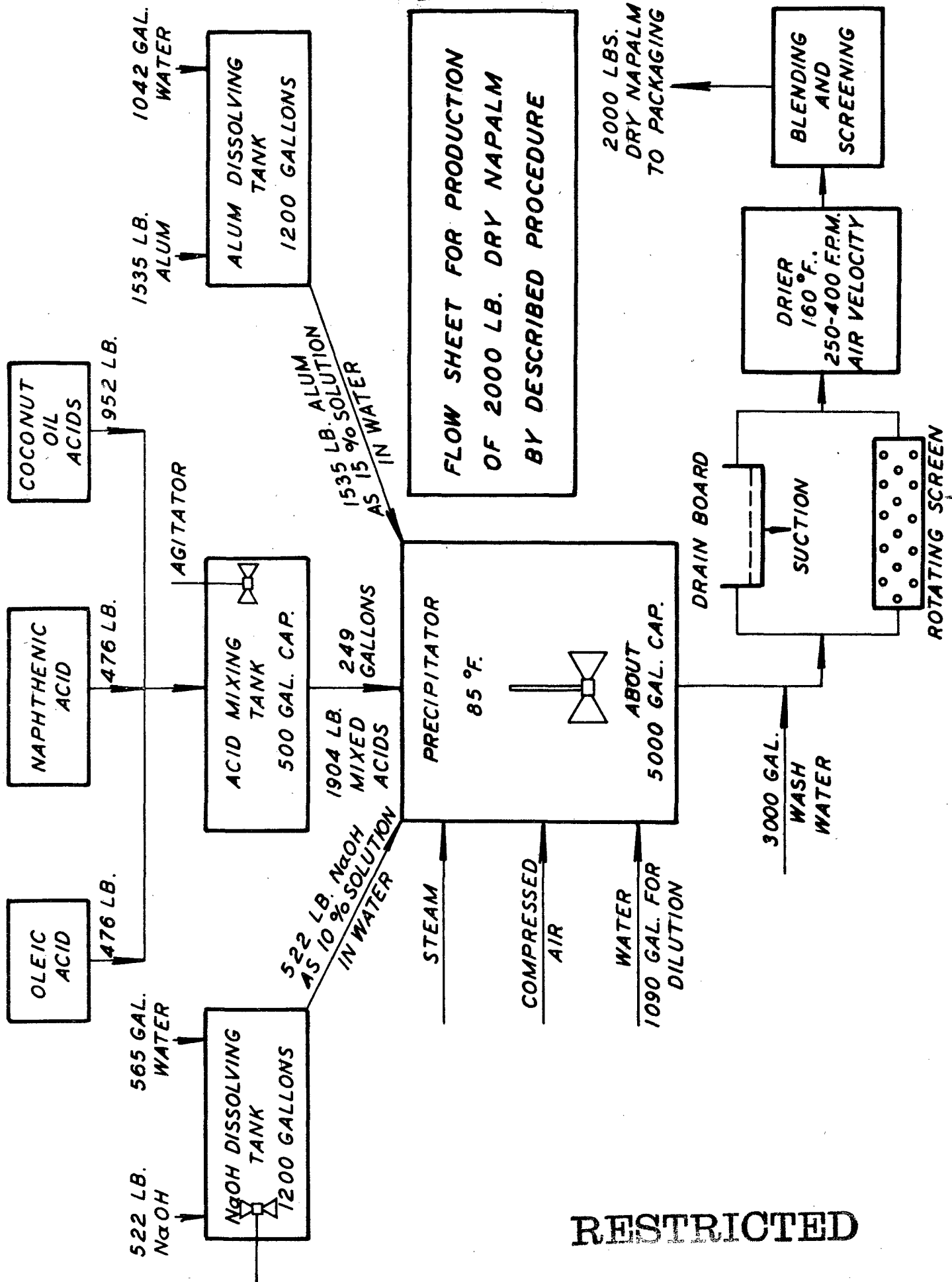
| <u>Specifications</u> | <u>Cocconut Fatty Acids</u> | <u>Naphtenic Acid</u> | <u>Oleic Acid</u> |
|-----------------------|-----------------------------|-----------------------|-------------------|
| Acid number           | 260-270                     | 230-245               | 190-200           |
| Iodine number         | below 15                    | below 10              | 85-90             |
| Iron                  | below 0.01%                 | below 0.01%           | below 0.01%       |
| Unsaponifiable        | 2% Maximum                  | below 3%              | below 2%          |
| Titre °F.             | 75-77                       | ---                   | 8°-12° C          |

b. Caustic

Flake or liquid caustic may be used, the quantities recommended for use in this report are based on 100% NaOH (77.5% Na<sub>2</sub>O). The iron content should be kept below 0.01%.

FIGURE 6

ACID STORAGE TANKS



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Table XII

Summary of Material Quantities and Tank Sizes

|                 | Amount #          | General Case             |                            | for 2000# batch |                     | Normal Tank Vol. Gals. |
|-----------------|-------------------|--------------------------|----------------------------|-----------------|---------------------|------------------------|
|                 |                   | Approx. Gals.            | Estimated Tank Vol.        | Approx. Gals.   | Estimated Tank Vol. |                        |
| Naphthenic acid | $\frac{x}{4.20}$  | $\frac{x}{33.6}$         | ---                        | 476             | 59.5                | ---                    |
| Oleic acid      | $\frac{x}{4.20}$  | $\frac{x}{31.3}$         | ---                        | 476             | 63.8                | ---                    |
| Coco acid       | $\frac{x}{2.10}$  | $\frac{x}{14.56}$        | ---                        | 952             | 137                 | ---                    |
| NaOH            | $\frac{x}{920}$   | $\frac{x}{352}$          | $\frac{x}{426}$            | 522             | 565                 | 1126                   |
| alum            | $\frac{x}{6.65z}$ | $\frac{x}{9.03}$         | $\frac{x}{6.9}$            | 1535            | 1137                | 1480                   |
| Mixed acids     | $\frac{x}{1.05}$  | $\frac{x}{3.04}$         | $\frac{x}{4.0}$            | 1904            | 249                 | 500                    |
| Precipitation   | ---               | $x(0.953 + \frac{y}{z})$ | $1.5(0.953 + \frac{y}{z})$ | 3041            | 4560                | 5000                   |

x = Pounds of dry Nepsalm to be produced per batch.

y = acid number of mixed acids (2-1-1 ratio).

z = per cent sulfate in alum.

c. Alum

War restrictions have been placed on the use of alum made from aluminum hydroxide - "trihydrate", and so-called "Victory" or "War Grade" alums made from kaolin clay have been offered as substitutes. There is no strong evidence that the "Victory Grade" material is not entirely satisfactory as long as the iron content is below 0.01% and other metallic impurities are kept very low. Nevertheless, it is highly desirable that the old pre-war grade, made from trihydrate, be used in order to avoid any possibility of trouble. Some "Victory Grade" alums contain a certain amount of insoluble siliceous residue, which settles during solution, but appears to have no effect upon gel properties (27). Acceptable alums contain 15.3-17.5% Al<sub>2</sub>O<sub>3</sub> and 43-47% SO<sub>4</sub>.

d. Water

No difficulties, with one possible exception, have been traced to water supplies, and it is believed that unless extremely hard waters are used, no attention need be given to this factor.

2. Preparation of Sodium Soap Solution.

The acids are mixed in the weight ratio of

|                 |     |
|-----------------|-----|
| Oleic acid      | 25% |
| Naphthenic acid | 25% |
| Cocoanut oil    |     |
| fatty acids     | 50% |

The mixing may be carried out in sufficient quantity for one batch, although where facilities are available, it has been found convenient to make up one or more days' supply at one time. The average yield of dry Napalm may be taken as 105 pounds per 100 pounds of mixed acids (57) and the average density of the mixed acids is about 7.66#/gallon. The quantities of acids required for one ton of dry Napalm are

|                          |            |
|--------------------------|------------|
| Naphthenic acid          | 476 pounds |
| Oleic acid               | 476        |
| Cocoanut oil fatty acids | <u>952</u> |

Total 1,904 pounds a volume of 249 gals.

Therefore, a tank of about 500 gallons capacity would be suitable. A sample of the mixed acids is withdrawn for determination of acid number.



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The required quantity of sodium hydroxide is calculated from the acid number of the mixed acids. This is indicated in the following example of production of 2000# of dry Napalm from mixed acids having an acid number of 240:

By definition, the acid number is the milligrams of KOH required to neutralize one gram of acid to an endpoint using phenolphthalein as an indicator.

NaOH required is 50% in excess of the amount necessary to neutralize the acids.

$$\frac{100}{105} \times 2000 \times .240 \times \frac{40}{56} \times 1.60 = 522 \text{ #NaOH/ton dry Napalm.}$$

The method of adding the caustic to the mixed acids will depend on the particular manufacturer's facilities. A convenient method is to dissolve the caustic in water to a concentration of about 10% by weight (565 gallons of water per ton of dry Napalm to be produced) in a separate small tank. This solution is then pumped or drained into the precipitator tank, the acids added through a cloth filter, and after 15 minutes agitation water is added until the proper total volume is reached. The recommended concentration of fatty acids in the precipitator tank prior to alum addition is one pound per gallon. Therefore, the sodium soap solution should be diluted to 1904 gallons before the alum is added.

This dilution drops the percent of sodium soaps and excess caustic in the solution to approximately 13% by weight, a figure agreed upon as about the optimum by most manufacturers. Lower concentrations of sodium soaps tend to produce Napalms which give weaker gasoline gels.

### 3. Preparation of the Alum Solution.

The amount of alum required is about 115% of that necessary to supply sulfate equivalent to the sodium added in the caustic. Actually the quantity of alum solution added may be controlled visually by the occurrence of coagulation (at about 100% of sulfate equivalent), after which about 15% excess is added.

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Assuming an alum containing 47%  $\text{SO}_4$ , the weight required is calculated as follows:

Basis: 1 ton dry Napalm  
from (2) the weight of NaOH to be used is 522

$$\therefore \frac{522}{40} \times \frac{1}{2} \times \frac{96}{.47} \times 1.15 = 1535\#$$

The required amount of alum is dissolved in water to a concentration of 15%\*, the temperature being held at about 105°F. The solution is cooled to about 90°F, and allowed to clear by settling. For one ton of dry Napalm, the volume of solution is then approximately:

$$\frac{1535 \times \frac{85}{15} + 1535}{9.0} = 1137 \text{ gallons}$$

The density of a 10% alum solution is about 9.0 #/gal.

#### 4. The Precipitation.

The precipitator tank should have a total volume about one and a half times that of the liquids it is to hold. This is equal to the volume of the sodium soap solution plus the volume of alum solution or, for one ton of dry Napalm,

$$1.5 (1904 + 1137) = 4560 \text{ gallons.}$$

The additional volume, while not absolutely essential, is desirable to allow for agitation and possible foaming. The tank itself may be of wood or steel and must be equipped with an agitator of sufficient power to give rapid mixing. Narrow radial baffles (usually four in number spaced 90° apart) around the inside of the tank have been found to aid in obtaining the degree of agitation desired. The alum feed lines should be placed in such a way that the alum solution is quickly diluted as it enters the tank. This may be accomplished by introducing the alum below the liquid surface by means of multiple pipes or by shower heads placed near the agitator blades. The precipitation tank should be equipped with steam and compressed air lines leading to the bottom of the tank, temperature and level indicators, and a dump valve large enough to avoid plugging when the precipitate is dropped to the filters. It is necessary to provide means for access to the top of the tank to enable the operator to observe the progress of the precipitation.

\* Up to 30% concentrations have been used successfully.

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The agitation requirements of the precipitating tank have not been correlated with batch size and information available is of a qualitative nature. Insufficient agitation, or too rapid alum addition results in local high concentrations of alum surrounded by an alkaline solution, and premature precipitation of large puffy agglomerates may take place. Furthermore, towards the end of the precipitation the coagulation may be so sudden that the soap cake cannot be broken up by the agitator and difficulty will be experienced in dropping the batch to the filters. Inclusion of unreacted sodium soaps may take place due to the sudden coagulation and subsequent washing with water may not remove these undesirable materials. Overagitation on the other hand, usually results in finer particle size, which, if densifying equipment is not available, will result in a higher percentage of fines during screening and may increase the oxidizability of the soap and decrease the time of setting.

Agitators may be either propeller or paddle types depending on availability. Power requirements are of the order of 15-20 H.P. for a 2000# batch of Napalm made in a tank of usual proportions. The actual power requirements and speed should be determined for the particular tank to be used.

The precipitation is carried out at 85°F. and this temperature should be held to within + 5° F. The alum is added slowly with agitation, the rate of flow being adjusted so that the total time of addition is about one hour. By means of an orifice in the alum line, the proper rate of addition may be insured without constant adjustment. Precipitation at first occurs slowly, final coagulation (the "strike") taking place after about 35% of the total alum is added. The precipitate floats to the surface of the liquid which gradually becomes clear as the alum addition is continued. When the required excess of alum has been added, the liquid is no longer turbid.

The precipitate should be washed until the extraneous salts are reduced at least as low as 1%. The amount of water necessary depends upon the method used for washing. Under poor conditions of contact between the precipitate and the wash water, adequate washing has been secured by the use of a volume of wash water equal to the volume of liquid present in the precipitating tank at the end of the precipitation. This water should be used in two or more portions. The extraneous ash may be estimated by determining the total ash and the aluminum content of the Napalm, correcting the total ash for the aluminum oxide present.

In some cases it has been found that a washing operation with dilute alum solution before the water wash has a beneficial effect (27,29). In cases where coagulation causes a stiff cake to form, the inclusion of sodium soap may be

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large and the alum washing will help by removing or reacting with these undesirable soaps. If an alum wash is used, the volume of solution should be about one half the volume of the finished batch and the concentration should be about 0.5-1%. The alum washing operation is carried out preliminary to and in the same manner as the water wash, except that a contact time of 15-30 minutes should be allowed.

#### 5. Dewatering of the Precipitate.

As discussed on page 6 of this report, the dewatering process varies considerably among the different manufacturers. Similar products are obtained using suction filters, rotary screens, and filter presses with equipment for breaking up the filter cake.

If equipment must be procured, a rotating screen is probably the most convenient and least expensive of the various methods. The screen is similar to those used for gravel washing and consists of a cylindrical open ended screen of about ten mesh. The cylinder is mounted with its axis at an angle of about 5° with the horizontal, and rotated at 20-30 rpm. The slurry is introduced at the upper end of the cylinder and travels to the lower end, the water leaving through the holes in the screen. Water sprays are placed along the axis of the cylinder and good washing action is obtained. A second similar screen may be placed in series with the first to provide additional washing. In this way the water content may be reduced to about 50% by weight.

As discussed on page 6 of this report, type A or slower setting Nopalms may be produced by a somewhat different treatment of the wet precipitate. The precipitate is partially dewatered in a plate and frame filter press and the resulting cake is fed to a pair of differentially rotating rolls. The rolls are of the general type used for clay crushing or paint pigment milling and may be about 15-20 inches in diameter geared to operate at a speed ratio of about  $2\frac{1}{2}:1$ . Clearance between the rolls should be about  $1/32$ ". The ribbon produced is removed from the fast roll by a steel doctor blade and passes to the comminuting machine where it is broken up to pass an 8 mesh screen. The material is then loaded into the drying trays and is processed in the same way as Type B.

#### 6. Drying.

The drying operation may be carried out in various types of equipment, and its choice depends on the relative availability of the equipment to the manufacturer. Since tray driers are most available their use will be discussed, but the drying principles are applicable to other types of driers.

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The dewatered precipitate is loaded into drier trays to a depth of about  $\frac{3}{4}$  of an inch. The trays have open, screened bottoms covered by muslin and normally are about 1" x 18" x 30" in size.

The drier temperature should be held at 160°F. with air velocities of 250 to 400 ft. per minute. Depending upon the equipment being used for size classification, the Napalm can be handled successfully on the screen when the moisture content has been reduced to some definite figure. It is recommended that the material be left in the drier only until this figure is reached and then handled in the screening and blending equipment. After this operation the material is replaced on the trays and the drying continued until the moisture content has been reduced to below 0.8%. Though this involves loading the trays twice, it has been found that the passage through the screen permits a more rapid drying rate so that the overall drying time is considerably shorter than it would be if accomplished in one operation. The screening and blending should have been accomplished at such a point that the redrying does not change the particle size distribution. In this case, re-screening at the completion of drying is unnecessary, and the material can be packed directly into the final container. This permits packing the material while it is still warm, reducing greatly the danger of moisture pickup after drying.

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Appendix III

Batch Method II

This method differs from batch method I in that the alum solution used for the precipitation has been partially neutralized with soda ash. By this procedure a uniform precipitation of soap is obtained throughout substantially the entire period of alum addition.

There seems to be no conclusive evidence that the products of the two batch methods differ in quality. Satisfactory soaps may be made by either method with equipment requirements about the same. At the present time operating data for this method are available from only one manufacturer, but it is planned to try the process on a plant scale using a different manufacturer's equipment.

Appendix IV

Materials of Construction

There is little evidence that the materials used in the equipment for manufacturing Nepalma have any great effect on the quality of the product, some manufacturers of satisfactory product using iron and others wooden or lead lined tanks. As stated elsewhere in this report, contamination by iron, copper, manganese and cobalt are undesirable and should be avoided.

Wood or steel tanks will in general be satisfactory if first carefully cleaned to remove traces of materials previously processed. Steel precipitator tanks become coated with soap after the first few batches and contamination with iron is thus prevented. Long periods of fatty acid storage in iron containers is undesirable since considerable contamination may take place. Similarly the alum solution will dissolve iron from the walls of a steel storage tank and result in contamination of the product.

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Appendix V

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Process Control

Adequate control of the manufacture of Napalm is necessary not only to direct plant operation, but to furnish a record which may be referred to in case an unsatisfactory batch is produced. Sampling and testing are advantageous at numerous points in the process and these will be discussed briefly. It is advisable that a log sheet be prepared and the operator be required to supply the necessary notations.

1. Raw Materials

All new batches of raw materials should be sampled and tested to be sure that they meet specifications (see page 34). A sample of at least two quarts should be taken from each new batch received and stored for future reference if necessary.

2. Mixed Acids

A sample of mixed acids should be taken from each batch made up and the acid number determined in the laboratory. If necessary the amount of caustic added to the batch should be adjusted to the acid number found (see page 37). Similarly, samples of the mixed sodium soaps should be titrated (27) with standard acid to phenolphthalein, and to methyl orange endpoints. The titration of the sodium soap solution may be carried out as follows. A 25 ml. sample of the soap solution is pipetted into a 500 ml. flask. One hundred ml. of a neutralized 50% alcohol and 100 ml. of petroleum ether are added. The soap solution is titrated with standardized one normal sulfuric acid, first to the phenol phthalein endpoint, then to the methyl orange endpoint. The latter endpoint may be judged by the clarity of the aqueous layer as well as by the color change. The amount of acid necessary to give the former is equivalent to the free caustic present and the difference in the amounts used to titrate to the two endpoints is equivalent to the sodium soaps. The ratio, of course, is a measure of the free caustic and may be used as a check on the operator making up the batch.

3. Preparation of Caustic and Alum Solutions

Temperature and quantities of material used should be noted on the operator's log-sheet.

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**RESTRICTED**4. Precipitation

The log-sheet should include a record of the temperature of the tank contents, dilution level before alum addition, time of addition and amount of alum used and the amount of water used during washing.

5. Drying

Notations should be made of drying temperature (a recorder is desirable), time of initial drying and redrying, if any, yield, and if possible, atmospheric temperature and relative humidity prevailing. Wet samples should be taken from the drums into which the Nepal is packed by means of a sampling thief. This sample should immediately be placed in a mason jar and closed tightly to prevent moisture absorption. A one-quart sample of each batch dried should be kept by the manufacturer for future reference. This sample should be kept in a tightly capped mason jar with a rubber sealing ring.

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Appendix VI

Properties of Napalm

The importance of moisture and oxidation have been demonstrated already in pages 8-20 . Here it is proposed to take up in more detail methods of water determination, to illustrate more fully the difference in behaviour of soaps on moisture absorption and to discuss more data available on the oxidation problem.

Determination of Water in Napalm

There can be no doubt that water exists in Napalm in two forms, namely absorbed water and water of hydration, the latter probably of several types. In addition under more or less drastic dehydration conditions there is potential water present, which can be evolved in varying degree by chemical reaction. No means has yet been found for analytically differentiating the three forms of water, because to a certain extent they are all interconvertable. Table XIII shows results by different methods on the same sample of Napalm. It is clear that, dependent upon the analytical conditions, any moisture content within limits can be obtained. In selecting a method for moisture determination, therefore, two considerations arise: (1) the moisture content indicated should correspond to the amount of gel breakdown found on the consistency test, (2) the method should be simple and require only common apparatus.

Table XIII

Moisture Determination by Different Methods (30)

| <u>Method</u>   | <u>Percent Water Found</u> | <u>Remarks</u>   |
|---|----------------------------|--|
| 1. Vacuum drying at 100°C. and 2 mm. pressure for 10 hours. | 1.0                        | Probably consists of most of adsorbed water and separation of doubled molecules of hydration.                  |
| 2. Dean and Stark with toluene or benzene (4 hours).        | 2.8                        | Above water plus that from a lesser degree of hydration and some water of reaction due to duration of process. |

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| <u>Method</u>   | <u>Percent<br/>water found</u> | <u>Remarks</u>  |
|---|--------------------------------|---|
| 3. Azeotropic distillation with dioxene.  | 2.5                            | Employed to avoid the inevitable water hold-up in apparatus and consequent lengthening of time of analysis. Distillation over in 30 mins. instead of 4 hrs. Fischer or acetyl chloride methods used to determine water in distillate. |
| 4. Fischer method as described in JACS <u>61</u> , 407, (1939).   | 2.1-3.0                        | Endpoints fade and procedure is time consuming due to lack of dispersion of sample in solvent and resultant slow reaction.  |
| 5. Fischer modified by swelling sample first in toluene and then breaking down to a liquid by addition of pyridine. | 3.7                            | Endpoint definite and fairly rapid. Water value probably includes some water of reaction due to extreme dehydrating conditions.   |
| 6. Fischer method on residue of dioxane distillation.   | 1.2                            | Total of 3. and 5. equals 5.  |
| 7. Acetyl chloride-pyridine method. JACS <u>57</u> , 841 (1935) Modified.   | 1.9-2.1                        | Acetyl chloride treatment at 60°C 5 to 40 mins. duration.   |
|   | 1.1-1.2                        | Acetyl chloride treatment at 5°C 5 to 40 mins. duration.  |
|   | 4.0-4.2                        | Acetyl chloride treatment at 30°C 5 mins. duration. Samples turned brown - obvious decomposition of soap.   |

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Notes: The vacuum oven method showed some fatty acids and neutral oil lost when the distillate was trapped by condensation in a tube surrounded by solid carbon dioxide. It may be, therefore, that samples containing large amounts of fatty acids would give fictitious results due to oil distillation of the latter. However, experiments at the CWS Columbia Laboratory (10) indicate the water insoluble matter amounts only to a few tenths of one per cent.

The water found by the benzol distillation method varies both with the time of distillation and with the boiling point of the solvent employed. With standardized conditions results are reproducible to  $\pm .1\%$  (Tables XXIV and XXV in Appendix VII).

The above Fischer method gives uncertain endpoints unless the sample is first gelled in toluene and then broken down to a limpid fluid by the addition of pyridine\*.

The acetyl chloride method gives quite arbitrary results depending upon the conditions imposed and exhibits a broad endpoint owing to precipitation of aluminum hydroxide during the caustic titration. Three titrations are required for each sample, a blank on the reagents and solvent, a fatty acid blank titre with caustic alone, and the sample. This method must, therefore, be considered very unsatisfactory.

-----

Consideration of the available methods from the above viewpoint selects vacuum drying and benzol distillation as the easiest and least complicated for use in practice\*\*. These are also the methods recommended for materials of the type of Napalm by Griffin(32). There is no doubt (pp. 55-58) that both can be made to correspond to the viscosity readings to a considerable degree.

\* Colgate-Palmolive-Peet report that they have had considerable success in correlating Karl Fischer moistures with gel viscosities. It appears that using a dead stop endpoint method (53) reproducible results can be obtained (10,3), but that different manufacturers' soaps have different base moistures perhaps due to different hydroxyl or "bound" moisture content.

\*\* For a fuller discussion of the various methods see (10).

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Consideration of many experiments had led both the Eastman Kodak group and the Technical Division group working at Edgewood Arsenal to the view that slightly better correlation of consistency results is obtained with vacuum oven measurements than with those made by benzol distillation. This may be due simply to the fact that reproducibility is better with the vacuum oven method and erratic results are less likely to be obtained. With the benzol distillation method results are probably good only to  $\pm 0.1$  and for research work this is somewhat too wide a margin. Comparison of vacuum oven and C.E.S. benzol distillation values is made in Tables XVII A and B.

Experiments in which a sample of Napalm is allowed to pick-up or lose a known amount of water (by direct weighing) and moisture content is determined before and after, have not proved too satisfactory (10,32,34). In general, neither the Karl Fischer, vacuum oven nor benzol distillation method check the results obtained by weighing. Table XIV compares in the two columns on the right, the change in moisture content found by direct weighing and by analysis. It will be noted that much closer agreement is found when water is being adsorbed by the soap than in the reverse case. It is believed that there is a possibility of internal rearrangement of water molecules within the Napalm, accounting for these peculiar results, which are not yet fully understood. Table XV-II also illustrates this point; the vacuum dried soaps show inert points considerably lower than those of the soaps as received.

Table XIV

Comparison of Moisture Determination Results with  
Actual Loss or Gain of Water by Weighing (10,34,57)

| <u>Soap</u>      | <u>Treatment</u>        | <u>Analytical Method</u> | <u>% Water by Analysis</u> | <u>% Water Change</u> | <u>% Water Change Found by Weighing</u> |
|------------------|-------------------------|--------------------------|----------------------------|-----------------------|---|
| McGeen<br>#22    | Original                | Karl Fischer             | 4.1                        |                       |   |
|                  | Vac. Dried at 60°C (1)  | " "                      | 2.9                        | 1.2                   | 2.2                                     |
|                  | Original                | Dean & Stark             | 1.1                        |                       |   |
|                  | Vac. Dried at 60°C (1)  | " "                      | 0.2                        | 0.9                   | 2.2                                     |
| Nuodex<br>Sample | Original                | Karl Fischer             | 3.2                        |                       |   |
|                  | Vac. Dried at 60°C (1)  | " "                      | 2.8                        | 0.4                   | 1.2                                     |
|                  | Original                | Dean & Stark             | 0.6                        |                       |   |
|                  | Vac. Dried at 60°C (1)  | " "                      | 0.2                        | 0.4                   | 1.2                                     |
| Nuodex<br>39053  | Original                | Karl Fischer             | 4.0                        |                       |   |
|                  | Dried at Room Temp. (<) | " "                      | 3.5                        | 0.5                   | 0.7                                     |
|                  | Original                | " "                      | 4.0                        |                       |   |
|                  | Dried at Room Temp. (<) | " "                      | 2.8                        | 1.2                   | 1.2                                     |

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Table XIV Contd.

| <u>Soap</u>          | <u>Treatment</u>                                 | <u>Analytical Method</u><br>10 mm. Hg. | <u>% Water by Analysis</u> | <u>% Water Change</u> | <u>% Water Change Found by Weighing</u> |
|----------------------|--|--|----------------------------|-----------------------|---|
| Nuodex<br>18032      | Original Vac.Oven<br>Cond. at 120°F.<br>20% R.H. | 60°C. 24 hrs.<br>" " "                 | 1.29<br>0.86               | 0.43                  | 0.54                                    |
| "                    | Original Vac.Oven<br>Cond. at 120°F.<br>20% R.H. | 10 mm. Hg.<br>60°C. 24 hrs.<br>" " "   | 1.74<br>0.93               | 0.81                  | 0.97                                    |
| Colgate<br>E-233826  | Original Vac.Oven<br>Cond. at 120°F.<br>20% R.H. | 10 mm. Hg.<br>60°C. 24 hrs.<br>" " "   | 0.46<br>0.34               | 0.12                  | 0.27                                    |
| Harmon<br>88955 R    | Original Vac.Oven<br>Cond. at 120°F.<br>20% R.H. | 10 mm. Hg.<br>60°C. 24 hrs.<br>" " "   | 0.76<br>0.63               | 0.13                  | 0.34                                    |
| Nuodex<br>89088      | Original Vac.Oven<br>Cond. at 120°F.<br>20% R.H. | 10 mm. Hg.<br>60°C. 24 hrs.<br>" " "   | 0.79<br>0.66               | 0.13                  | 0.30                                    |
| Nuodex<br>18032      | Original Vac. Oven<br>Cond. at 90°F.<br>90% R.H. | 10 mm. Hg.<br>60°C. 24 hrs.<br>" " "   | 1.17<br>4.85               | 3.68                  | 3.68                                    |
| "                    | Original Vac.Oven<br>Cond. at 90°F.<br>90% R.H.  | 10 mm. Hg.<br>60°C. 24 hrs.<br>" " "   | 1.66<br>4.45               | 2.79                  | 2.92                                    |
| Colgate<br>E-23382-B | Original Vac.Oven<br>Cond. at 90°F.<br>90% R.H.  | 10 mm. Hg.<br>60°C. 24 hrs.<br>" " "   | 0.54<br>1.62               | 1.08                  | 1.18                                    |
| Harmon<br>88955 R    | Original Vac.Oven<br>Cond. at<br>90°F. 90% R.H.  | 10 mm. Hg.<br>60°C. 24 hrs.<br>" " "   | 0.74<br>3.84               | 3.10                  | 3.14                                    |
| Nuodex<br>89088      | Original Vac.Oven<br>Cond. at 90°F.<br>90% R.H.  | 10 mm. Hg.<br>60°C. 24 hrs.<br>" " "   | 0.76<br>2.28               | 1.52                  | 1.90                                    |

(1) Pressure 15 mm. in N<sub>2</sub> stream(2) Vacuum desiccation over P<sub>2</sub>O<sub>5</sub>**RESTRICTED**

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The Effect of Moisture on Napalm

For investigation of the equilibrium between solid Napalm and atmospheres of different relative humidity, two methods, static or dynamic in character, may be employed. In the first, a sample of the solid soap is exposed to an atmosphere of known relative humidity and allowed to come to equilibrium. The moisture content may then be determined by one of the standard methods and the consistency of the gel formed in gasoline measured. This may be repeated at a number of different relative humidities and the variation in moisture content and gel consistency thus determined. In the dynamic method (10) the soap may be exposed in a stream of air or inert gas of known relative humidity and the rate of change in weight followed. By using atmospheres of several different relative humidities, it is possible to find by interpolation a humidity at which the soap neither increases nor decreases in weight. This may be called the inert point of the soap. Needless to say, the inert point will vary with the conditions under which the soap was dried. Results by both methods are summarized in Table XV:

Table XV

Moisture Content of Napalm and Relative Humidity

I - Static Equilibrium Method (3)

Water Content (Vacuum Oven 24 Hrs. 60°C. 10 mm. Hg.)

| <u>Soap</u>          | <u>90°F-20% RH</u> | <u>85°F-65% RH</u>   | <u>90°F-90% RH</u> |
|----------------------|--------------------|--|--------------------|
| Nuodex 15374         | 0.89               | 1.45   | 2.97               |
| " 87896              | 0.99               | 1.75   | 3.06               |
|                      | 0.94               | (Sample equilibrated at 90% RH, re-equilibrated at 20% RH) |                    |
| Nuodex 87905         | 1.04               | 1.75   | 5.23               |
|                      | 1.02               | (Sample equilibrated at 90% RH, re-equilibrated at 20% RH) |                    |
| Harshaw Experimental |                    |  |                    |
| Batch 27 JP          | 0.4                | 0.9  | 1.7                |
| 5 P                  | 0.6                | 1.1  | 1.6                |
| Ferro Batch #22      |                    |  |                    |
| Dried at 160° F.     | 0.9                | 2.1  | 4.36               |
| Dried at 185° F.     | 0.55               | 1.91   | 4.30               |

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Table XV Contd.

II - Dynamic Equilibrium Method (10)

| Soap         | Condition         | H <sub>2</sub> O <sup>(1)</sup> | Inert Points in Terms of % R.H. |     |              |       |
|--------------|-------------------|---------------------------------|---------------------------------|-----|--------------|-------|
|              |                   |                                 | Temperature                     |     |              |       |
|              |                   |                                 | 20°                             | 25° | 30°          | 40°   |
| Nuodex       | As Rec'd.         | 1.2                             | 39                              |     | 45           | 49    |
| Lot 89023    | Vac.Dried         | .4                              | ?                               |     | (14?)        | ?     |
| McGean       | As Rec'd.         | 1.0                             | 23                              |     | 25           |       |
| Lot 684      | Vac.Dried         | 0.2                             | (7?)                            |     | (14?)        | (14?) |
|              | <u>Time Dried</u> |                                 |                                 |     |              |       |
| McGean       | 18 hours          | 0.9                             |                                 | 26  | -            | -     |
| Lot 1100     | 20                | 0.9                             |                                 | 34  | -            | 36.5  |
|              | <u>Redried</u>    |                                 |                                 |     |              |       |
| (3)          | 2 hours           | 0.7                             |                                 | 17  |              | 26    |
|              | 4                 | 0.8                             |                                 | 19  |              | 27    |
|              | 5                 | 0.6                             |                                 |     | 28.5(2)      | 29    |
| Harmon       |                   | 0.4                             |                                 | 20  |              |       |
| R11249       |                   |                                 |                                 |     |              |       |
| 11242        |                   | 0.4                             |                                 | 19  |              |       |
| Imperial     |                   |                                 |                                 |     |              |       |
| NR99         |                   | 0.5                             |                                 | 19  |              |       |
| Eakins       |                   |                                 |                                 |     |              |       |
| N-3-2981-182 |                   | 0.4                             |                                 | 19  |              |       |
| Pfister      |                   |                                 |                                 |     |              |       |
| N3-243c-78   |                   | 0.5                             |                                 | 22  | At 70°C. 30% |       |

- (1) H<sub>2</sub>O determined according to C.I.S Specification.
- (2) This result is much too high.
- (3) All samples taken from plant drier. Plant practice is to dry for 20 hours, then grind and redry for 4 hours.

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There is considerable indication that all soaps do not have the same moisture content-relative humidity curve but that the relation is dependent upon the conditions of precipitation, drying, etc. This is readily understandable as it is known that soaps from different manufacturers have different alumina contents, different amounts of free sodium sulfate, contain different free fatty acids, etc.

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Moisture is picked up or lost by Napalm with considerable rapidity (Table XVI, Figure 2). In general, an exposure of a few minutes is sufficient to alter materially the water content of the soap unless it is at its inert point. It is of great importance, therefore, to keep samples of Napalm intended for test work in tightly closed glass bottles, canning jars with rubber rings having been found very suitable for this purpose.

Table XVIRate of Change of Moisture Content of Napalm

I - Soaps Exposed to 65% R.H. after Equilibrium at 20% R.H.  
1/8" Layers. Natural Convection (34).

| <u>Time</u>   | <u>Ferro Drier</u><br><u>Expt'l. Batch I</u> | <u>Ferro Drier</u><br><u>Expt'l. Batch II</u> | <u>Nuodex</u><br><u>#18032</u> |
|---|--|---|--------------------------------|
| Initial Moisture Content (Vacuum Oven 24 hrs. 60°C. 10 mm. Hg.) at 20% R.H. | 0.57%  | 1.11%   | 1.28%                          |
| 13 mins.  | 0.86   |   |                                |
| 18  |  | 1.43  |                                |
| 25  |  |   | 1.60                           |
| 30  | 0.93   |   |                                |
| 35  |  | 1.48  |                                |
| 37  |  |   | 1.64                           |
| 47  |  | 1.50  |                                |
| 49  | 0.99   |   |                                |
| 55  |  |   | 1.68                           |
| 74  | 1.04   |   |                                |
| 80  |  | 1.54  |                                |
| 87  |  |   | 1.73                           |
| 225   | 1.19   | 1.65  | 1.86                           |
| 340   | 1.22   | 1.66  | 1.91                           |
| 1020  | 1.27   | 1.71  | 2.01                           |
| 1450  | 1.28   | 1.71  | 2.03                           |
| 2500  | 1.28   | 1.69  | 2.03                           |
| Final Moisture Content (Vacuum Oven 24 hrs. 60°C. 10 mm. Hg.) 65% R.H.      | 1.08   | 1.39  | 1.73                           |

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Table XVI Contd.

II - Soaps as Received Exposed to 90° F., 20% R.H. 1/8" Layers.  
Natural Convection (57).

Loss in Weight, %

| <u>Time</u> | <u>Nuodex #18032</u> |                   | <u>Colgate</u> | <u>harmon</u> | <u>Nuodex</u> |
|-------------|----------------------|-------------------|----------------|---------------|---------------|
|             | <u>Sample (a)</u>    | <u>Sample (b)</u> | <u>E-23326</u> | <u>83455R</u> | <u>84088</u>  |
| 20 mins.    | 0.27                 | 0.49              | 0.12           | 0.39          | 0.09          |
| 75          | 0.44                 | 0.31              | 0.23           | 0.22          | 0.19          |
| 3-1/2 hrs.  | 0.44                 | 0.86              | 0.25           | 0.24          | 0.19          |
| 7-1/2 hrs.  | 0.45                 | 0.90              | 0.25           | 0.27          | 0.22          |
| 24 hrs.     | 0.54                 | 0.97              | 0.27           | 0.34          | 0.30          |

Initial Moisture Content (Vacuum Oven 60°C. 24 hrs. 10 mm. Hg.)  
1.29                      1.74                      0.46                      0.76                      0.79

Final Moisture Content (Vacuum Oven 60°C. 24 hrs. 10 mm. Hg.)  
1.36                      0.83                      0.34                      0.63                      0.66

III. - Soaps as Received Exposed to 90° F., 90% R.H. 1/8" Layers.  
Natural Convection (57).

Gain in Weight, %

|          |      |      |      |      |      |
|----------|------|------|------|------|------|
| 15 mins. | 0.21 | 0.10 | 0.26 | 0.28 | 0.37 |
| 1 hr.    | 0.63 | 0.32 | 0.56 | 0.78 | 0.61 |
| 3 hrs.   | 0.98 | 0.68 | 0.93 | 1.46 | 1.16 |
| 20 hrs.  | 3.44 | 2.71 | 1.16 | 2.78 | 1.74 |
| 68 hrs.  | 3.68 | 2.92 | 1.18 | 3.14 | 1.90 |

Initial Moisture Content (Vacuum Oven 60°C. 24 hrs. 10 mm. Hg.)  
1.17                      1.66                      0.54                      0.74                      0.76

Final Moisture Content (Vacuum Oven 60°C. 24 hrs. 10 mm. Hg.)  
4.85                      4.45                      1.62                      3.84                      2.28

IV - Soaps as Received Exposed to 50% R.H. Natural Convection (35).

Gain in Weight, %

|          | <u>Porous Napalm</u><br><u>1/8" Layer</u> | <u>Porous Napalm</u><br><u>1" Layer</u> | <u>Vitreous Napalm</u><br><u>1/8" Layer</u> |
|----------|---|---|---|
| 5 mins.  | 0.22%                                     | 0.11%                                   | 0.21%                                       |
| 15 mins. | 0.51                                      | 0.26                                    | 0.46  |
| 30       | 0.52                                      | 0.35                                    | 0.65  |
| 35       | 0.66                                      | 0.38                                    | 0.69  |

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Table XVII-A summarizes the results available on viscosity vs. moisture content for a large number of soaps. It was concluded from these that about 0.1% in vacuum oven moisture\* produces a difference of about 40 grams on the 24 hr. 150° F. consistency test.

Table XVII-B gives recent data (60) for three representative soaps dispersed in New York Chemical Warfare Service Procurement Division test gasoline. Compounding was done at approximately 70° F. and 40% relative humidity. Moisture was introduced into the samples by storage over water at 70° F. and removed by drying in a forced draft tray drier at 68° C. Moisture contents were determined by the C.W.S. benzol distillation, described in Specification #196-131-107 revised, and by vacuum oven drying for four hours at 55° C. In Table XVIII is shown the variation of consistency with moisture content for the three soaps determined by interpolation from smoothed curves. Over the low moisture ranges the change in consistency is again about 40-50 grams for 0.1% change in moisture content.

It will be appreciated from Table XVII that proper drying of Napalm in the factory is essential and also that adequate packaging must be provided for the finished soap.

Long-time drying (apart from the oxidation effect and shortening of the induction period which is discussed on pages 12-20) appears to do no damage as shown by experiments at the Columbia University C.W.S. Laboratory and at Ferro Drier and Chemical Company (1,10). The only effect appears to be a small decrease in the fatty acid content, presumably due to interaction with the free aluminum hydroxide in the Napalm. Nevertheless, the induction period is shortened rapidly with rise in temperature under oxidizing conditions, obeying the Arrhenius relationship (pp. 63). That is, for a rise in temperature of 10°C. the induction period would be halved.

\* The two methods in use at Kodak Park and Edgewood Arsenal Technical Division are somewhat different, making these figures only approximate.

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Table XVII-A

Variation of Viscosity with Moisture Content (3% Gels)

| Soap                            | Moisture Content                      |  | 48 hrs.<br>77° F. | 24 hrs.<br>150° F. |
|---------------------------------|---------------------------------------|--|-------------------|--------------------|
|                                 | Vacuum Oven<br>10 mm.<br>24 hr. 60°C. | Benzol<br>Distillation<br>(K.P. Method)* |                   |                    |
| <u>Eastman Kodak Laboratory</u> |                                       |  |                   |                    |
| Nuodex 18032                    | 1.1                                   | 1.65                                     | 570               | 510                |
| Imperial Paper                  | 0.36                                  | 0.9                                      | 770               | 550                |
| Harmon 88956                    | 0.38                                  | 1.25                                     | 850               | 600                |
| Harmon 83956                    | 1.10                                  | 1.50                                     | 620               | 365                |
| McGean Batch 22                 | 1.61                                  | 1.75                                     | 550               | 330                |
| McGean Batch 534                | 1.07                                  | 1.5                                      | 530               | 540                |
| Pfister E210D                   | 0.2                                   | 0.25                                     | 550               | 440                |
| Nuodex 33538                    | 1.03                                  | 0.9                                      | 600               | 390                |
| Nuodex 89023A                   | 1.33                                  | 1.1                                      | 400               | 190                |
| Nuodex 89033                    | 0.79                                  | 0.4                                      | 700               | 450                |
| Ferro Batch 22                  | 0.43                                  | 0.7                                      | 770               | 350                |
| Imperial X2105 NR<br>54         | 0.72                                  | 1.0                                      | 620               | 490                |
| McGean 1220                     | 0.72                                  | 1.3                                      | 700               | 560                |
| McGean 750                      | 0.60                                  | 1.1                                      | 800               | 620                |
| McGean 1094                     | 0.62                                  | 1.1                                      | 840               | 570                |
| McGean 1039                     | 0.59                                  | 1.15                                     | 730               | 620                |
| McGean 1125                     | 0.59                                  | 1.0                                      | 790               | 620                |
| Ferro 67                        | 0.90                                  | 0.35                                     | 330               | 480                |
| Nuodex 87396                    | 0.46                                  | 0.65                                     | 700               | 550                |
|                                 | 0.61                                  | 0.80                                     | 560               | 520                |
|                                 | 0.99                                  | 1.1                                      | 670               | 390                |
|                                 | 1.75                                  | 1.5                                      | 300               | 190                |
|                                 | 3.06                                  | 2.7                                      | 105               | 100                |
| Nuodex 87905                    | 1.04                                  | 1.0                                      | 650               | 460                |
|                                 | 1.75                                  | 1.9                                      | 280               | 230                |
| Nuodex 15374                    | 0.99                                  | 1.2                                      | 320               | 760                |
|                                 | 1.45                                  | 1.6                                      | 580               | 400                |
|                                 | 2.97                                  | 2.2                                      | 290               | 43                 |
| Harshaw Expt'l.<br>Batch 27 JP  | 0.4                                   |  | 630               | 770                |
|                                 | 0.9                                   |  | 470               | 630                |
|                                 | 1.7                                   |  | 36                | 25                 |

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Table XVII-A Contd.

| <u>Edgewood CWS Laboratory</u> | Vacuum Oven                |                    |
|--------------------------------|----------------------------|--------------------|
|                                | 10 mm. Hg.<br>5 hrs. 55°C. | 24 hrs.<br>150° F. |
| Nuodex 87835                   | 1.32                       | 350                |
|                                | 0.92                       | 450                |
|                                | 0.70                       | 520                |
|                                | 0.84                       | 540                |
|                                | 0.76                       | 610                |
| Nuodex 87937                   | 1.72                       | 200                |
|                                | 1.4                        | 350                |
|                                | 0.7                        | 380                |
|                                | 1.06                       | 390                |
|                                | 0.20                       | 620                |
|                                | 0.28                       | 660                |
| Nuodex 87396                   | 2.14                       | 130                |
|                                | 1.02                       | 470                |
|                                | 0.7                        | 700                |
|                                | 0.2                        | 750                |
| McGean 7026                    | 2.25                       | 250                |
|                                | 1.28                       | 400                |
|                                | 1.16                       | 630                |
|                                | 0.12                       | 710                |
|                                | 0.30                       | 720                |
|                                | 0.66                       | 720                |
| Nuodex 87391                   | 1.53                       | 220                |
|                                | 1.40                       | 330                |
|                                | 1.32                       | 390                |
|                                | 0.90                       | 510                |
|                                | 0.81                       | 540                |
|                                | 0.62                       | 600                |
|                                | 0.74                       | 660                |

\* 20 g. sample 200 ml. benzol 4 hrs. Oil bath 125-150° C.

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Table XVII-B

Harmon #88955-R Napalm Gels in Gasoline (A)

8% Gels

| <u>% Moisture By</u> |                  | <u>Gardner Consistency in Grams after Storage of</u> |                        |                       |
|----------------------|------------------|--|------------------------|-----------------------|
| <u>CWS Benzol</u>    | <u>Vac. Oven</u> | <u>1 hr. at 150°F</u>                                | <u>24 hr. at 150°F</u> | <u>48 hr. at 77°F</u> |
| 0.0                  | 0.09             | 900  | 750                    | 900                   |
| 0.1                  | 0.13             | 850  | 740                    | 850                   |
| 0.6                  | 0.59             | 650  | 550                    | 650                   |
| 1.4                  | 1.66             | 220  | 140                    | 180                   |
| 2.3                  | 2.48             | 140  | 80                     | 140                   |

7% Gels

|     |      |     |     |      |
|-----|------|-----|-----|------|
| 0.0 | 0.09 | 940 | 950 | 1050 |
| 0.1 | 0.13 | 930 | 930 | 1030 |
| 0.6 | 0.59 | 770 | 720 | 800  |
| 1.4 | 1.66 | 270 | 200 | 310  |
| 2.3 | 2.48 | 200 | 120 | 230  |

12% Gels

|     |      |     |      |      |
|-----|------|-----|------|------|
| 0.0 | 0.09 | --- | 1580 | 1500 |
| 0.1 | 0.13 | --- | 1430 | 1500 |
| 0.6 | 0.59 | --- | 1290 | 1290 |
| 1.4 | 1.66 | --- | 650  | 340  |
| 2.3 | 2.48 | --- | 350  | 660  |

California Ink #195-7-7-43 Napalm Gels in Gasoline (B)

8% Gels

|      |      |     |     |     |
|------|------|-----|-----|-----|
| 0.15 | 0.07 | 350 | 730 | 930 |
| 0.3  | 0.27 | 770 | 770 | 890 |
| 0.7  | 0.59 | 770 | 590 | 730 |
| 1.2  | 1.21 | 460 | 260 | 330 |
| 2.5  | 2.56 | 190 | 100 | 160 |

7% Gels

|      |      |     |     |      |
|------|------|-----|-----|------|
| 0.15 | 0.07 | 950 | 980 | 1050 |
| 0.3  | 0.27 | 960 | 950 | 970  |
| 0.7  | 0.59 | 330 | 810 | 970  |
| 1.2  | 1.21 | 580 | 450 | 560  |
| 2.5  | 2.56 | 240 | 170 | 250  |

12% Gels

|      |      |     |      |      |
|------|------|-----|------|------|
| 0.15 | 0.07 | --- | 1420 | 1500 |
| 0.3  | 0.27 | --- | 1470 | 1450 |
| 0.7  | 0.59 | --- | 1300 | 1430 |
| 1.2  | 1.21 | --- | 1160 | 1170 |
| 2.5  | 2.56 | --- | 530  | 700  |

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McGean #534 Napalm Gels in Gasoline (C)

8% Gels

| <u>% Moisture by</u> |                  | <u>Gardner Consistency in Grams After Storage of</u> |                        |                        |
|----------------------|------------------|--|------------------------|------------------------|
| <u>CWS Benzol</u>    | <u>Vac. Oven</u> | <u>1 hr. at 150°F</u>                                | <u>24 hr. at 150°F</u> | <u>48 hrs. at 77°F</u> |
| 0.3                  | 0.07             | 740  | 730                    | 840                    |
| 0.35                 | 0.10             | 730  | 700                    | 340                    |
| 0.5                  | 0.37             | 740  | 690                    | 810                    |
| 1.2                  | 0.94             | 530  | 430                    | 560                    |
| 2.0                  | 1.8              | 260  | 170                    | 260                    |

7% Gels

|      |      |     |     |      |
|------|------|-----|-----|------|
| 0.3  | 0.07 | 930 | 900 | 980  |
| 0.35 | 0.10 | 330 | 240 | 1030 |
| 0.5  | 0.37 | 330 | 390 | 1000 |
| 1.2  | 0.94 | 730 | 640 | 730  |
| 2.0  | 1.8  | 370 | 280 | 400  |

Table XVIII

Variation of Consistency with Moisture

Range of H<sub>2</sub>O%      Decrease in Consistency per 0.1% Increase in H<sub>2</sub>O in Soap

| <u>Range of H<sub>2</sub>O%</u>      | <u>A</u>                           |           |            | <u>B</u>  |           |            | <u>C</u>  |           |
|--------------------------------------|------------------------------------|-----------|------------|-----------|-----------|------------|-----------|-----------|
|                                      | <u>8%</u>                          | <u>9%</u> | <u>12%</u> | <u>8%</u> | <u>9%</u> | <u>12%</u> | <u>8%</u> | <u>9%</u> |
|                                      | <u>I - After 48 hrs. at 77° F.</u> |           |            |           |           |            |           |           |
| 0.4-0.8                              | 40                                 | 67        | 35         | 47        | 37        | 23         | 27        | 25        |
| 0.8-1.2                              | 45                                 | 30        | 57         | 57        | 75        | 77         | 43        | 45        |
| 1.2-1.6                              | 25                                 | 30        | 40         | 33        | 40        | 43         | 45        | 53        |
| 1.6-2.0                              | 5                                  | 2         | 17         | 40        | 25        | 30         | 30        | 30        |
| <u>II - After 24 hrs. at 150° F.</u> |                                    |           |            |           |           |            |           |           |
| 0.4-0.8                              | 53                                 | 65        | 57         | 50        | 43        | 27         | 35        | 30        |
| 0.8-1.2                              | 55                                 | 70        | 87         | 45        | 47        | 43         | 23        | 40        |
| 1.2-1.6                              | 23                                 | 30        | 63         | 30        | 40        | 50         | 43        | 47        |
| 1.6-2.0                              | 7                                  | 5         | 33         | 23        | 35        | 53         | 33        | 40        |

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Packaging

Because of the large influence of moisture on gel properties, packaging of the soaps must be carefully considered. The packaging problem is not so important for Type B soap as for Type A, since it is possible to redry the soap immediately before its use in the filling plant. It has been shown on many occasions that soap redrying has no deleterious effect on gel properties providing the soap has sufficient resistance to oxidation. Table XIX illustrates this point.

Table XIX  
Redrying of Napalm (3)

| <u>Soap</u>                  | <u>Gardner Viscosities 2 Days 77° F.</u> |                                     |                                       |
|------------------------------|--|-------------------------------------|---------------------------------------|
|                              | <u>Conditioned to 90° F.-20% RH</u>      | <u>Conditioned to 90° F.-20% RH</u> | <u>Reconditioned to 90° F.-20% RH</u> |
| Nuodex 87396                 | 570                                      | 105                                 | 500                                   |
| Nuodex 87905                 | 550                                      | 52                                  | 600                                   |
|                              | <u>Vacuum Oven</u>                       | <u>48 hrs.</u>                      | <u>24 hrs.</u>                        |
|                              | <u>Moisture</u>                          | <u>77° F.</u>                       | <u>150° F.</u>                        |
| <u>Nuodex 18032</u>          |  |                                     |                                       |
| Original Soap                | 1.20                                     | 540                                 | 515                                   |
| Conditioned at 90° F.-90% RH | 1.8                                      | 420                                 | 250                                   |
| Redried                      | 0.9                                      | ---                                 | 620                                   |

Nevertheless, one large packaging test has been run on the fiberboard drums commonly used to ship Napalm from the manufacturing plant to the filling plant. The results are shown in Table XX.

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**RESTRICTED**Table XXPackaging Test on Lot #18032 (38)

| <u>Initial Values</u>           | <u>Laboratory</u> | <u>Keeping Conditions</u>                     |                 |                  |
|---------------------------------|-------------------|---|-----------------|------------------|
|                                 |                   | <u>Tropical Room</u><br>110-90°F<br>75-72% RH | 90°F.<br>90% RH | 90° F.<br>20% RH |
| Iodine Number                   | 30.9              | 31.5  | 31.1            | 30.6             |
| Vacuum Oven Moisture            | 1.20              | 1.20  | 1.20            | 1.20             |
| Dean & Stark Moisture (E.K.)    | 1.5               | 1.35  | 1.5             | 1.5              |
| 2-Day 70°F. Gardner Vis.        | 540               | 530   | 540             | 530              |
| 7-Day 70°F. " "                 | 570               | 570   | 600             | 550              |
| 1-Day 150°F. " "                | 510               | 530   | 515             | 510              |
| <u>Three Weeks</u>              |                   |   |                 |                  |
| Outer Edge Iodine No.           | 31.9              | 31.4  | 31.9            | 32.0             |
| Center Iodine No.               | 31.6              | 31.0  | 31.8            | 31.9             |
| Outer Edge Vacuum Oven Moisture | 1.11              | 1.16  | 1.14            | 1.10             |
| Center " " "                    | 1.11              | 1.14  | 1.16            | 1.12             |
| Outer Edge Dean & Stark (E.K.)  | 1.75              | 1.75  | 1.65            | 1.20             |
| Center Dean & Stark (E.K.)      | 1.65              | 1.4   | 1.9             | 1.75             |
| Outer Edge 2-Day 70°F. Vis.     | 630               | 690   | 630             | 640              |
| Center 2-Day 70°F. Vis.         | 620               | 710   | 640             | 620              |
| Outer Edge 7-Day 70°F. Vis.     | 590               | 640   | 590             | 690              |
| Center 7-Day 70°F. Vis.         | 570               | 650   | 540             | 690              |
| Outer Edge 150°F. Vis.          | 570               | 660   | 540             | 550              |
| Center 150°F. Vis.              | 500               | 540   | 540             | 570              |
| <u>Twelve Weeks</u>             |                   |   |                 |                  |
| Outer Edge Iodine No.           | 31.6              | 31.2  | 31.0            | 31.5             |
| Center Iodine No.               | 31.5              | 31.1  | 31.5            | 31.2             |
| Outer Edge Vacuum Oven Moisture | 1.24              | 1.42  | 1.50            | 1.01             |
| Center " " "                    | 1.19              | 1.41  | 1.52            | 1.09             |
| Outer Edge Dean & Stark (E.K.)  | 2.25              | 1.9   | 2.25            | 1.7              |
| Center " " "                    | 2.25              | 1.0   | 2.25            | 1.4              |
| Outer Edge 2-Day 77°F. Vis.     | 750               | 740   | 690             | 850              |
| Center 2-Day 77°F. Vis.         | 800               | 830   | 690             | 800              |
| Outer Edge 1-Day 150°F. Vis.    | 370,370           | 350,380                                       | 270,310         | 390,320          |
| Center 1-Day 150°F. Vis.        | 350,420           | 360,280                                       | 330,400         | 350,410          |
| <u>Twenty Weeks</u>             |                   |   |                 |                  |
| Outer Edge Iodine No.           | 31.2              | 30.1  | 30.9            | 30.8             |
| Center Iodine No.               | 31.3              | 30.1  | 30.1            | 30.9             |
| Outer Edge Vac. Oven Moisture   | 1.3               | 1.6   | 1.8             | 1.0              |
| Center " " "                    | 1.34              | 1.6   | 1.7             | 1.0              |
| Outer Edge Dean & Stark (E.K.)  | 1.7               | 1.5   | 1.9             | 1.1              |
| Center " " "                    | 1.7               | 1.3   | 2.0             | 1.4              |
| Outer Edge 2-Day 77°F. Vis.     | 490               | 640   | 420             | 680              |
| Center 2-Day 77°F. Vis.         | 510               | 610   | 440             | 630              |
| Outer Edge 1-Day 150°F. Vis.    | 330               | 340   | 190             | 400              |
| Center 1-Day 150°F. Vis.        | 400               | 370   | 250             | 390              |

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Such fiberboard packages would obviously be unsatisfactory for Type A Napalm where the soap may be exposed for several months in atmospheres of widely varying humidity and where no facilities for redrying can be expected to be available. For this type the only completely satisfactory package is a hermetically-sealed metal can. This is now in use for Napalm Type A.

Prevention of the Moisture Effect

Until recently, little attention has been given to the possibility of preventing the troubles caused by moisture absorption. However, three distinct methods have been suggested so far for doing this:

1. Addition of a dehydrating agent to the gasoline in which the solid soap is to be dispersed. This dehydrating agent must naturally have no effect upon the gel formed even after prolonged standing. Phosphorus pentoxide, sodium\*, and calcium carbide, while satisfactory temporarily, have been found to cause breakdown of the gel on long keeping. Calcium chloride and calcium sulfate may be satisfactory. Surveillance tests with these materials are under way.

2. Coating the dried particles to slow down the rate of diffusion. It is conceivable that a thin layer of paraffin wax or other relatively moisture-impermeable, gasoline-soluble material distributed evenly over the exterior of the particles might slow down the rate of water absorption to such an extent that the soaps could be handled with no great difficulty. Such a layer might also assist in retarding oxidation of the solid soap. Preliminary experiments conducted at A.D. Little, Inc. (39) indicate that 10% of paraffin based on the weight of the soap is effective in reducing the rate of moisture pickup, while not unduly prolonging the solvation time of the gel. These experiments are being continued.

3. The addition, probably in the course of manufacture, of a material (capable of chain formation by coordination at two points in its molecule to the soap) which would coordinate preferentially with the carboxyl groups of the soap molecules, thus preventing water molecules from breaking the chains. This possibility has been considered by the Shell group (15) mainly in connection with the addition of oxidation inhibitors. Preliminary results with  $\alpha$ -ortho-toluidine stearic acid (Alphatol), 1% of which, based on the final soap yield, was added to the

\*Recent experiments (37) indicate that sodium may be satisfactory with certain soaps.

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neutral soap solution, reneutralized and precipitated with alum, gave a gel with excellent oxidation stability and increased stability toward water. This soap and a blank made and dried simultaneously to about 2% water content (C.W.S. benzol distillation method) were made into 8% gels in straight run gasoline. Mobilometer readings on the gels are shown below and the inhibitor action on the solid soap in Table VII C, p. 23. It will be seen that the Alphatol not only acted as an inhibitor but also stabilized the gel against the moisture effect. Further work is being done.

|             | <u>3 days 77°F.</u> | <u>6 days 77°F.</u> | <u>11 days 77°F.</u> |
|-------------|---------------------|---------------------|----------------------|
| 1% Alphatol | --                  | 605                 | 500                  |
| Check       | 560                 | 250                 | 39                   |

Oxidation

The main results of the oxidation of Napalm have been noted on pages 12-20. In this section it is proposed to go into greater detail on the methods for evaluating oxidation and other work which has been done on the oxidation problem.

1. The UOP Test Bomb Method (15). This method of testing aluminum soap products for oxidation stability has been adapted from that used in the testing of gasolines and consists of subjecting the soap to the action of oxygen in a bomb under elevated temperature and pressure. The UOP test bombs used (17) were modified to use a 250 lb. sheet safety disc to protect the pressure recorder.

15 gram soap samples are placed in 8 oz. polished oil sample bottles and inserted in the cold bombs. Air is displaced by admitting oxygen to 100 p.s.i. and releasing the pressure. This is done twice and the pressure brought to the desired value, usually 100 p.s.i. The bombs were then heated to the required temperature in a thermostatically controlled oil bath and allowed to remain until a rapid absorption of oxygen occurred as indicated on a chart (Figure 7), or by rupture of the pop-off disc. A correction of 45 minutes is subtracted from the total time to allow for the heating-up period.

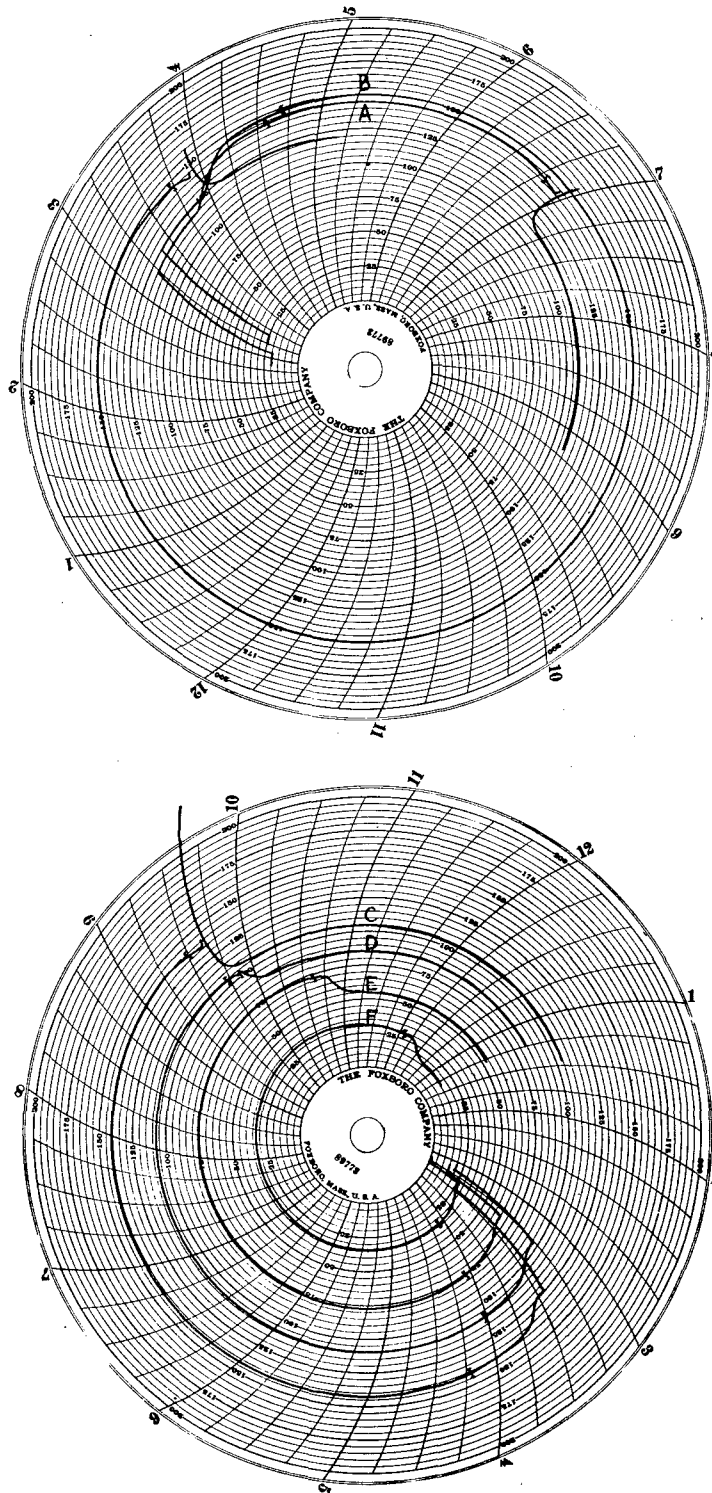
The samples used in the bomb tests are made in a special manner to prevent sintering, which when it occurs, causes no satisfactory induction period to be obtained. The procedure used is as follows:

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## Illustrative Charts from the Accelerated Oxidation Test Apparatus

Time vs. Oxygen Pressure (Psi. Gauge)

FIGURE 7



- Curve A - Straight Napalm 135 min. standard IP.  
" B - Sample A + 0.01% UOP#5, 662 min. standard IP.  
" C to F - Sample A at 80°C and 25, 50, 75, and 100 psi. O<sub>2</sub>.  
Variation of IP and violence of reaction with pressure.

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A one normal solution of basic aluminum sulfate is added slowly with very rapid stirring to an equal amount of neutral one normal soap solution. The very fine precipitate produced at first remains dispersed until just before the end point when the particles coagulate. After washing and drying the soaps still contain 3% to 10% free acids extractable with acetone. The soaps of the lower figure are usable directly in the bombs, but those containing above 6% free acid must be extracted with acetone before testing. The acetone not only removes free acid but also neutral oil and alkyl phenols. The samples are then screened through 50 mesh and tested. It was found for these extracted Napalms that a relationship between the induction period and the temperature,  $T$  in  $^{\circ}K$ , and pressure,  $P$  in p.s.i. absolute, could be deduced, namely:

$$\begin{aligned}\log IP (\text{mins.}) &= 5480/T - 13.0 \text{ (at 25 p.s.i.)} \\ \log IP (\text{mins.}) &= 3.021 - 0.56 \log P \text{ (at } 89^{\circ} \text{ C.)}\end{aligned}$$

2. The Heat-Rise Method (Mackey or Modified Mackey Test). It was early found that soaps susceptible to oxidation when heated in an oven at  $180^{\circ}$  or  $200^{\circ}$  F. finally showed a rapid rise in temperature followed by a drop (figure 8). This appeared to be caused by an autocatalytic oxidation. This procedure, while satisfactory from the research angle, is inconvenient for routine laboratory examination of soaps. For this purpose both the Mackey test (Allens Commercial Organic Analyses, 5th Edition Vol. 2 p. 759) and a modified Mackey test, the apparatus being shown in figure 9, were used. It was found that soaps which were known to oxidize readily gave a temperature rise in from 45 to 90 minutes by these methods while two Nuodex soaps, #15371 and #15374, which had been kept for over six months and were known to be stable, showed no temperature rise under these conditions even after exposure to the heat test for 10 hours. It seemed, therefore, that here was available a "go-no-go" test for detecting oxidation susceptibility in the soaps. For this purpose it seems to have been satisfactory although the temperature rise does not appear to be significant when soaps show sintering. However, an induction period can be measured if the soaps oxidize within a reasonable length of time. The test is influenced by air velocity and by the fineness of the soap particles.

3. Peroxide Value. It has been found (10,18,19) that those soaps which oxidize readily tend to show high peroxide values (mg. iodine/gm. soap) when treated with potassium iodide. The peroxide value can also be used as a guide to whether or no the induction period has been exceeded. The procedure is as follows:

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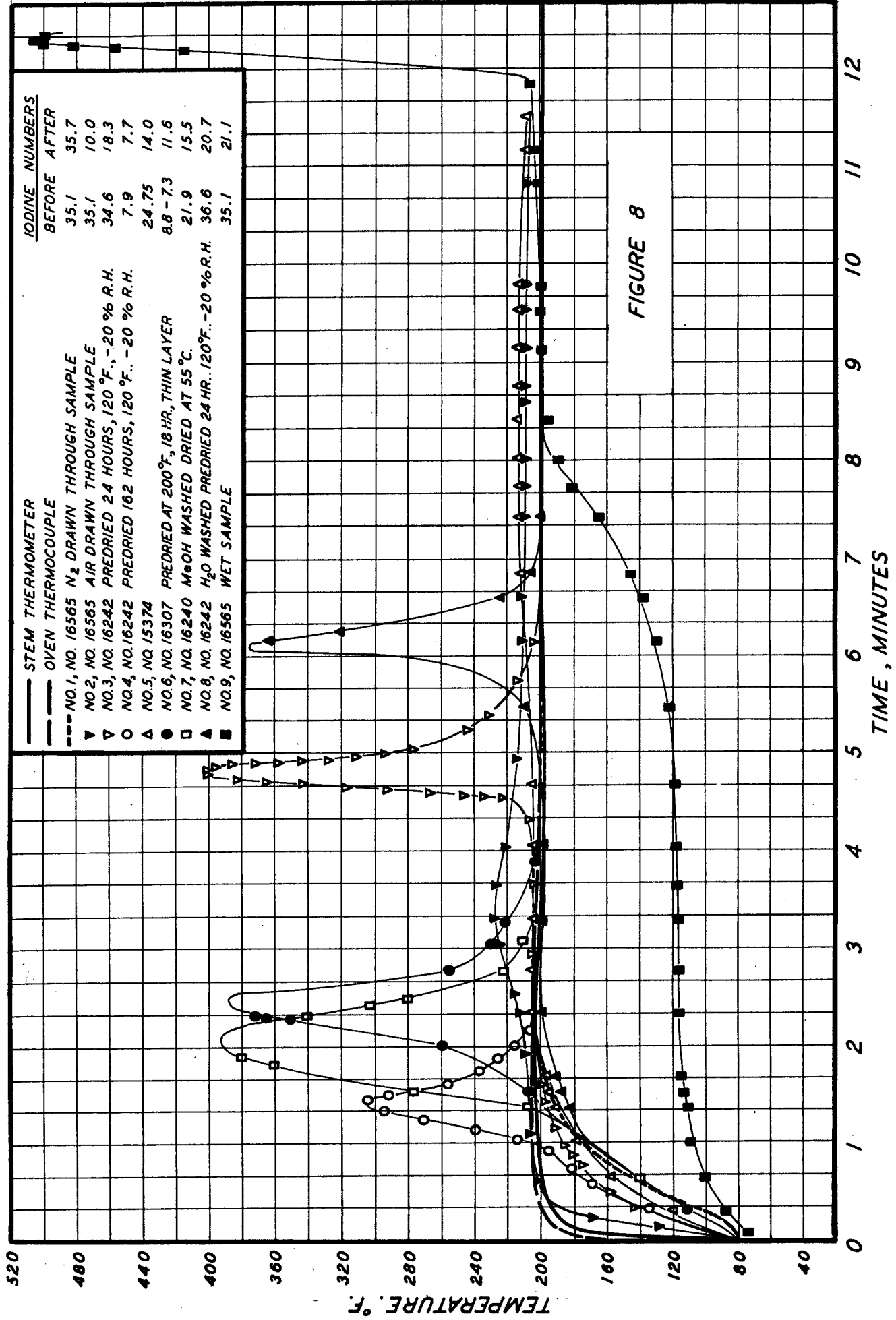
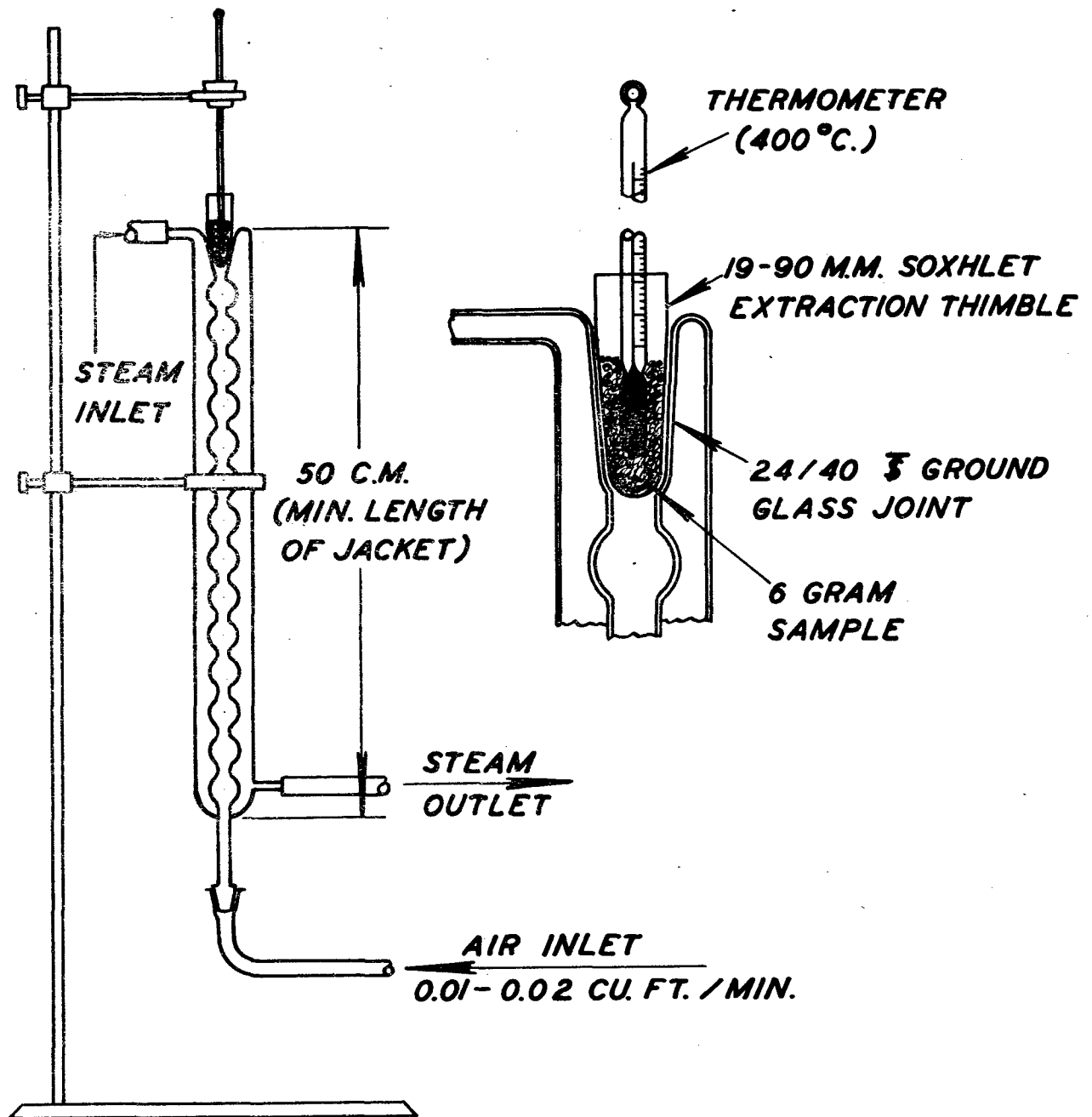


FIGURE 8

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FIGURE 9  
STABILITY TEST FOR X-104 TYPE  
THICKENERS AT 100°C.



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Five grams (+ 0.1 gm.) of the soap is weighed into an iodine flask and 50 ml. of a mixture of glacial acetic acid and chloroform (60/40 by weight) added. The flask is shaken gently to avoid formation of lumps and 3 ml. of a saturated aqueous potassium iodide solution is added. The mixture is shaken vigorously for 1.5 minutes (this must be carefully timed), 100 ml. of water is added, and then titration is carried out in the usual way with thio-sulfate solution using starch as indicator. The peroxide number is defined as the number of milligrams of iodine liberated from potassium iodide by one gram of the material and may be calculated from the expression:

$$\text{Peroxide number} = \frac{127 \times \text{volume (ml.)} \times \text{normality}}{\text{Wt. sample}}$$

It is convenient to use a standard thiosulfate solution of which the normality is 0.0394, in which case the peroxide number may be read directly from the burette if a five gram sample is used.

Unfortunately, the UOP bomb method does not lend itself to the routine investigation of Napalms because of the necessity for acetone extraction of the samples. Nevertheless, it has been used with great success to prove the presence of inhibitors in the Napalm and the effectiveness of adding inhibitors to prevent the Napalm oxidation (pages 15-20 ). A similar method<sup>(10)</sup> has been used in the C.W.S. Laboratories at Columbia where a 25 gram sample of the soap was sealed up in an atmosphere of oxygen in a 100 ml. flask attached to a mercury manometer (initial pressure 1 atmosphere). With this apparatus, it was demonstrated that Steolate (stearate-oleate) and Stearate - Staybelite soaps are considerably more susceptible to oxidation than the Napalms.

Table XXI

Oxidation of Aluminum Soaps in Sealed Flasks<sup>(10)</sup>

| Soap                | Induction<br>Period 25° C. | Induction<br>Period 45° C. |
|---------------------|----------------------------|----------------------------|
| Steolate            | 16 hours                   | 0.5 hours                  |
| Stearate-Staybelite | 72 "                       | 24 "                       |
| Napalm (Nuodex)     | --                         | 48 "                       |
| Napalm (McGean)     | --                         | 192 "                      |

It was further shown that Napalms differed in their induction period, confirming the Shell test.

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With the temperature-rise method considerable data have been accumulated and are shown in Table XXII. It will be seen that the oxidizability of Napalms varies widely and at the present time no complete explanation of this can be given.

There is no doubt that using the same fatty acids the introduction of iron either as a soluble soap (naphthenate) or in the alum is not deleterious to gel properties but greatly decreases the induction period of the soap (Table V, page 14). At the same time, Napalm samples made from different lots of naphthenic acids seem to possess variable susceptibility to the catalytic action of iron soaps. For example, one sample remained unoxidized for 300 minutes in the UOP bomb test in the presence of 0.05% iron without inhibitors. The addition of 0.01% UOP #5 raised this tolerance to 0.1%. Another sample broke down long before this time, although its iron content was only 0.004%.

It seems probable that an acetone soluble iron deactivator is present in Napalm since extraction of the soap with that solvent reduced the stability in the presence of 0.025% iron to practically zero if uninhibited, and to 150 minutes with 0.01% UOP #5 added. This deactivator may or may not be identical with the neutral alkyl phenols.

It is certain that iron has an adverse effect in many instances, although in isolated cases no trouble has been experienced with soaps of high iron content. Until the whole story is known, iron must be regarded with suspicion and should be kept as low as possible in Napalm by the use of substantially iron-free alum and acids containing the minimum amount of iron. Manganese, copper and cobalt are other alum impurities which should be avoided.

Another known factor which plays a considerable part in the oxidizability of Napalm is the natural inhibitor present in naphthenic acid. No progress has been made as yet in testing different samples of naphthenic acids for this variable so that a uniform product could be recommended for procurement. It has been suggested that control might be secured by testing a 50-50 mixture of the naphthenic acid in question and standard oleic acid with an accelerated oxidation test of the Mackey or UOP bomb type. Until some test of this type is devised, or alternately a single raw material source and a constant method of isolation is used, no certainty of this oxidation stability of Napalm without adding artificial inhibitors can be expected.

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Table XXII  
Induction Periods by heat-Rise Method (33)

| <u>Batch Number</u>           | <u>Induction<br/>Period*<br/>hours</u> | <u>Temperature<br/>Rise °C.</u> | <u>% Fe<br/>Content</u> |
|-------------------------------|--|---------------------------------|-------------------------|
| Nuodex 88538                  | 20                                     | None                            | 0.035                   |
| " 89023-A                     | 22                                     | None                            | 0.049                   |
| " 89033                       | 22                                     | None                            | 0.036                   |
| " 15374                       | 30                                     | None                            | 0.060                   |
| " 15923                       | 00                                     | 30°C.                           | 0.160                   |
| " 16242                       | 00                                     | 250°C.                          | 0.054                   |
| " 16307                       | --                                     | 10°C.                           | 0.053                   |
| " 16308                       | --                                     | 27°C.                           | 0.192                   |
| " 17016                       | --                                     | 6°C.                            | 0.210                   |
| " 16565                       | --                                     | 5°C.                            | 0.053                   |
| " 87937                       | --                                     | None                            | 0.0044                  |
| " 87938                       | --                                     | None                            | 0.0156                  |
| " 87884                       | --                                     | None                            | 0.0099                  |
| " 87885                       | --                                     | None                            | 0.0085                  |
| " 87896                       | --                                     | None                            | 0.0068                  |
| " 87892                       | --                                     | None                            | 0.0065                  |
| " 87907                       | --                                     | None                            | 0.0056                  |
| " 87905                       | --                                     | None                            | 0.0069                  |
| " 87890                       | --                                     | None                            | 0.0144                  |
| " 87891                       | --                                     | None                            | 0.0083                  |
| McGean 684                    | 20                                     | None                            | 0.031                   |
| " 1220                        | 7.5                                    | 2°C.                            | 0.015                   |
| " 760                         | 4                                      | 3°C.                            | 0.030                   |
| " 1094                        | 6                                      | 2°C.                            | 0.024                   |
| " 1039                        | 1                                      | 3°C.                            | 0.018                   |
| " 1125                        | 5.5                                    | 4°C.                            | 0.027                   |
| " 1169                        | 6                                      | 6°C.                            | 0.030                   |
| " 1241                        | 2                                      | 6.5°C.                          | 0.025                   |
| Harmon 88955-R                | 23                                     | None                            | 0.024                   |
| " R-10543                     | 8                                      | None                            | 0.034                   |
| " R-11202                     | 8                                      | None                            | 0.016                   |
| " 10X1186                     | 9                                      | None                            |                         |
| " 10X1183B                    | 9                                      | None                            |                         |
| Ferro 22 (dried at<br>160°F.) | 6.8                                    | 2°C.                            | 0.025                   |
| " 22 (dried at<br>185°F.)     | 3.7                                    | 2°C.                            | 0.025                   |
| " 67                          | 5.5                                    | 5°C.                            | 0.019                   |
| Imperial 6-4-43               | 6.5                                    | 7°C.                            | 0.049                   |
| " NR54                        | 28                                     | None                            | 0.023                   |
| " 91                          | 36                                     | None                            | 0.032                   |
| " NR111                       | 2.5                                    | 5°C.                            | 0.025                   |
| Pfister 30-A                  | 20                                     | None                            | 0.102                   |
| " 98                          | 2.5                                    | 2°C.                            | 0.022                   |
| " 106                         | 33                                     | None                            | 0.018                   |
| " N3-2432-94                  | 3                                      | 2°C.                            | 0.020                   |

\* Where no temperature rise is indicated, time under induction period is total time of heating. Where blanks appear data are missing.

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Another source of variable oxidation susceptibility lies in the oleic acid used. Investigation of a number of commercial oleic acids indicates that they have widely different induction periods (Table XXIII). Aluminum soaps were prepared from the different acids and dried in an inert atmosphere at 55°C. Mackey tests, columns 7 and 8, were made on both the soap and the acid. Peroxide values were obtained on the solid soaps at various times up to 22 days. The anti-oxidant effect of the naphthenic acid is clearly indicated by the peroxide values on soap 6-59B in the Table. It must be concluded from these results that oleic acid used in the manufacture may also be a factor in the oxidation of Napalm.

Certain details in the precipitation of the Napalm have been shown to affect the oxidation susceptibility of the finished Napalm. The addition of insufficient excess alum after the coagulation point produces Napalm which is more susceptible to oxidation than that made with the proper excess of alum. Even with the proper excess of alum, insufficient washing of the finished Napalm gives a product somewhat more susceptible to oxidation than is properly washed Napalm. Finishing of the product at too high a pH can also cause greater oxidation susceptibility (Figure 5).

#### The Free Fatty Acid Content of Napalm.

It appears to be impossible to prepare  $AlX_3$ , where X is a fatty acid radical by aqueous precipitation. No matter how great an excess of aluminum is employed free fatty acid always appears to be present in the final soap. This acid is extractable by a number of solvents most of which also appear to extract some aluminum soap, as shown by the ash of the fatty acid extract. The most satisfactory solvent from the standpoint of freedom from aluminum soaps of the free fatty acids appears to be cold acetone.<sup>(10,19)</sup> The amount of material extracted depends upon:

1. The number and length of time of the extracts,
2. The temperature of extraction,
3. The dryness of the acetone used.

It appears probable that continued extraction particularly with moisture present in the acetone tends to hydrolyze the soap, liberating more free acid which in turn is removed by the solvent. Table XXIV shows results obtained with a variety of treatments. On the basis of the above results, the C.W.S. Laboratory at Columbia has made the following suggestion for free fatty acid determination, results obtained on a number of soaps being shown in Table XXV.

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Table XXIII

Induction Periods of Commercial Oleic Acids and Their Aluminum Soaps (10)

| Acid                     | Source               | I <sub>2</sub> No. | % Fe   | Acid No. | Color              | Composition                                       | Mackey Induction            |           |
|--------------------------|----------------------|--------------------|--------|----------|--------------------|---|-----------------------------|-----------|
|                          |                      |                    |        |          |                    |   | Period of Acid              | Al Soap   |
| Elaine Oleic             | Emery                | 94.4               | 0.027  | 194      | reddish brown      | 67% oleic<br>18.9% linoleic<br>14.1% sat. acids   | 48 mins.                    | 30 mins.  |
| Ruby red oil             | Proctor & Gamble     | 98.5               | 0.034  | 193      | reddish brown      | 69.8% oleic<br>19.7% linoleic<br>10.5% sat. acids | 48, 39<br>37, 38            | 45        |
| Canary Brand red oil     | "                    | 91.0               | 0.0015 | 200      | light straw        | 74.3% oleic<br>13.5% linoleic<br>12.5% sat. acids | 73<br>71<br>78              | 50        |
| Arctic red oil           | Welch Holme Clark    | 87.6               | 0.53   | 194      | dark red brown     | 81.2% oleic<br>10.2% linoleic<br>8.6% sat. acids  | 22<br>21                    | 24        |
| Naphthenic oil acids     | obtained from Nuodex | --                 | --     | 238      | light amber        | ---   | a                           | a         |
| Cocconut oil acids       | "                    | --                 | --     | 261      | amber              | ---   | 225<br>(max. temp. 101° C.) | 18.5 hrs. |
| Rosin acids (Staybelite) | Hercules             | 135                | --     | 162      | U.S. Rosin Stan. X | ---   | a                           | a         |
| Stearic acid             | Proctor & Gamble     | 2.4                | --     | 208      | --                 | 49.7% palmitic<br>50.1% stearic acid              | a                           | a         |

(a) no rise in temperature in 48 hours.

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Table XXIII Contd.

| No.     | Soap                                  | Peroxide Values of Aluminum Soaps, Days After Drying |      |      |      |      |      |    |     |     |     |     |
|---------|---------------------------------------|--|------|------|------|------|------|----|-----|-----|-----|-----|
|         |                                       | 0  | 2    | 3    | 4    | 6    | 9    | 10 | 11  | 21  | 22  |     |
| 6-59 C  | Elaine Oleic                          | 4.4  | -    | -    | 22.4 | -    | -    | -  | -   | 97  | -   | 123 |
| 6-60 B  | Ruby red oil                          | 28.4   | -    | 55.7 | -    | -    | -    | -  | 96  | -   | 203 | -   |
| 6-60 C  | Canary Brand red oil                  | 3.85   | -    | 18   | -    | -    | -    | -  | 90  | -   | 193 | -   |
| 6-60 A  | Arctic red oil                        | 40.0   | -    | 70.5 | -    | -    | -    | -  | 121 | -   | 185 | -   |
| 6-59 B  | Naphtthenic -<br>Elain Oleic<br>50:50 | 0.3  | -    | -    | 0.4  | -    | -    | -  | -   | 0.4 | -   | 0.5 |
| 31-22 B | Cocoanut oil acids (1)                | 9.15   | 14.4 | -    | -    | 16.9 | 15.9 | -  | -   | -   | -   | -   |
| 31-22 A | Rosin acids (Staybelite) (1)          | 3.65   | 5.45 | -    | -    | 6.95 | 7.20 | -  | -   | -   | -   | -   |

(1) Test temperature 65° C.; in all other cases 25° C.

Table XXIV

Typical Results of Acetone Extractions of Napalm (10)

| Soap   | Temp. | Hrs. & Type of Treatment | Liters of Acetone | % Extract (A) | % Ash in Extract | Neut. Eq. of Extract | Size of Sample |
|--|-------|--------------------------|-------------------|---------------|------------------|----------------------|----------------|
| Nuodex orig. 1.0% H <sub>2</sub> O               | 40°   | 16 Sox.                  | ---               | 20.8          | 0.30 (1)         | 293 (1)              | 40 gms.        |
| " " " 0.5% H <sub>2</sub> O                      | 40°   | 16                       | ---               | 23.0          | ---              | ---                  | 15-20          |
| " " " vac. dried 1.0%                            | 40°   | 16                       | ---               | 26.0          | ---              | ---                  | 15-26          |
| " " " hydrated 2.0                               | 40°   | 16                       | ---               | 27.9          | ---              | ---                  | 15-20          |
| " " " hydrated 2.6                               | 40°   | 16                       | ---               | 33.5          | ---              | ---                  | 15-20          |
| " " " hydrated 2.6                               | 40°   | 16                       | ---               | 34.5          | ---              | ---                  | 15-20          |
| " " " orig. using 2% H <sub>2</sub> O in acetone | 40°   | 16                       | ---               | 21.5          | 0.32 (1)         | 286 (1)              | 40             |
| " " " orig. 1.0%                                 | 40°   | 8 1/2                    | 7.48              | 27.0          | ---              | ---                  | 15-20          |
| McGean, orig. % H <sub>2</sub> O                 | 40°   | 8 1/2                    | 6.78              | 26.3          | ---              | ---                  | 15-20          |
| " " " " dried acetone                            | 40°   | 6                        | 2.26              | 17.9          | ---              | ---                  | 15-20          |
| Nuodex, orig. after 5 extractions                | 45°   | 1 wash                   | 4.0               | 7.1           | .08              | 366 (5)              | 2000           |
| " " " orig.                                      | 45°   | 2                        | 4.0               | 0.7           | .73 (4)          | 238                  | 2000           |
| " " " orig.                                      | 45°   | 13                       | 10.0              | 11.7          | .27              | ---                  | 1000           |
| " " " orig.                                      | 25°   | 1 1/4                    | 4.0               | 7.23          | .03              | 393 (5)              | 2000           |
| McGean orig.                                     | 25°   | 1 1/4                    | 4.0 (3)           | 7.16          | .03              | 340 (5)              | 2000           |

(A) Method I. Difference in weights of sample and residue. Method II. Weight of extract.

1. Obtained on extract after 3 hours treatment.
2. In five steps of 2 liters acetone each, washed on filter with 1 liter after each step.
3. Plus 4 liters for washing on filter.
4. Extract probably contained suspended soap particles.
5. Not corrected for unsaponifiable matter.

**RESTRICTED**Table XXVSummary of Results Using Proposed MethodforPercentage Soap Extractable by Acetone (10)

| <u>Soap</u>          | <u>Date</u> | <u>Run No.</u> | <u>% Extracted</u> |
|----------------------|-------------|----------------|--------------------|
| McGean Lot 684       | 27/7/43     | 1              | 6.6                |
|                      | "           | 2              | 6.2                |
|                      | 4/8/43      | 1              | 6.7                |
|                      | "           | 2              | 6.3                |
|                      | 12/8/43     | 1              | 6.2                |
|                      | "           | 2              | 6.1                |
| Nuodex Lot 89093     | 23/7/43     | 1              | 7.0                |
|                      | "           | 2              | 6.9                |
|                      | 27/7/43     | 1              | 7.1                |
|                      | "           | 2              | 7.3                |
| McGean Experimental  |             |                |                    |
| normal               | ---         | -              | 4.9                |
| high excess caustic  | ---         | -              | 4.1                |
| low excess caustic   | ---         | -              | 7.7                |
| Steolates, 31-20-B-1 | ---         | -              | 15.8               |
| " 31-20-B-4          | ---         | -              | 15.1               |
| " 31-20-B-8          | ---         | -              | 14.5               |

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Method of Determination for Free Fatty Acid

1. Grind the soap in a mortar.
2. To a fifteen (15) gram sample in an iodine flask add fifty (50) ml. of dry acetone (dried over Drierite). Allow to stand fifteen (15) minutes with occasional swirling. At the end of that time decant through a filter into a weighed 250 ml. beaker. Repeat with a second fifty (50) ml. portion of acetone, decanting through the same filter into the first filtrate. Finally, give the soap a quick wash with a fifty (50) ml. portion of acetone, and decant the wash acetone through the filter.

3. Evaporate the combined filtrates on a steam bath until acetone vapor is no longer detectable, avoiding unnecessary overheating. Transfer the beaker to an oven and heat at 65°C. for forty-five (45) minutes. Cool and weigh. Tests have shown that the time of heating the extract at 65° is not too critical. An additional half hour of heating would decrease the weight of a 1.0 gm. extract by about 20 mg.

With Napalm no correlation has yet been made of free fatty acid content with physical properties, although it is known that the presence of a certain amount of free acid is essential for gelation. Thus, a soap thoroughly extracted with methyl alcohol does not disperse in gasoline unless a small quantity of a polar compound is added.

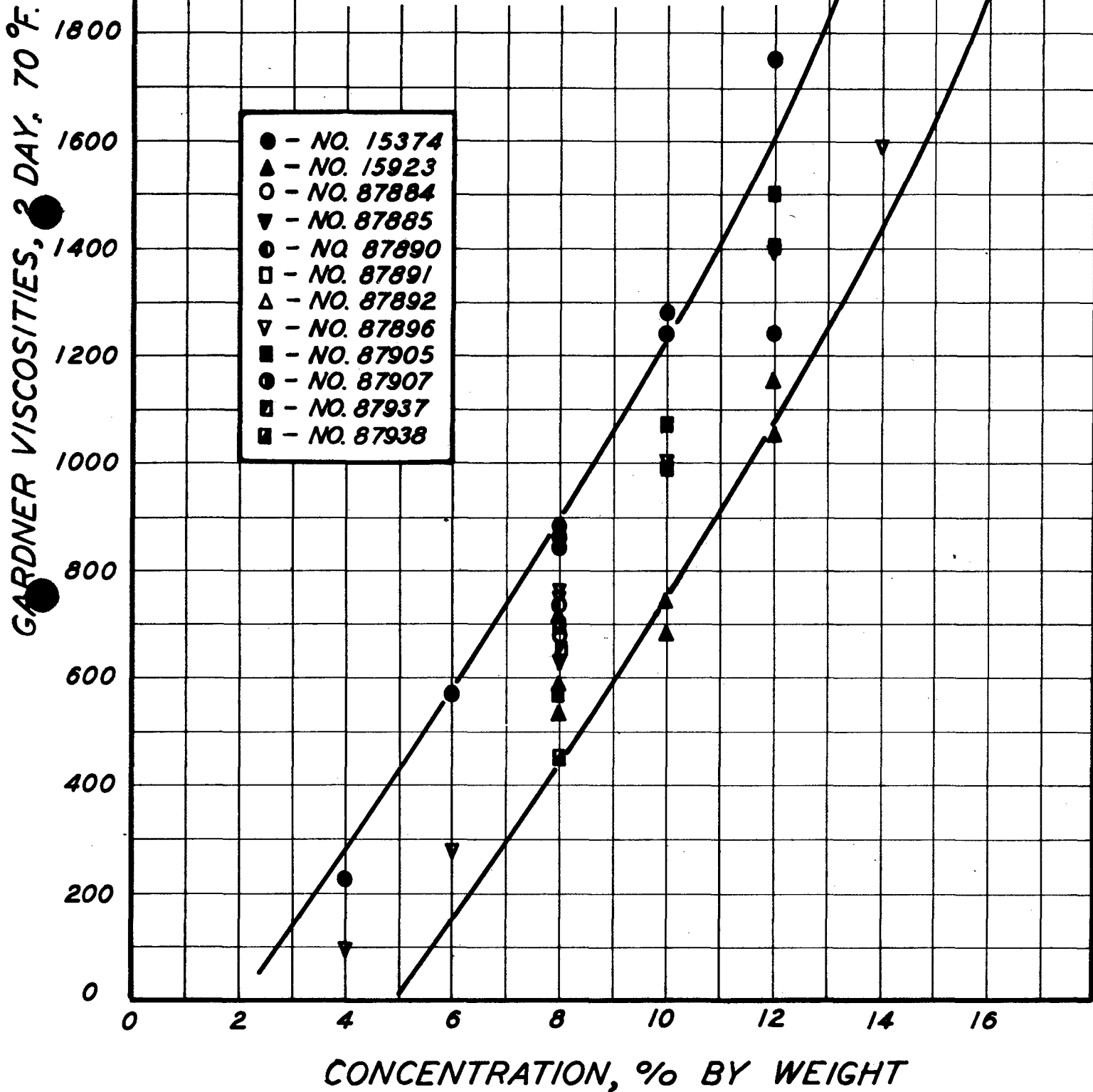
It is believed that under certain favorable conditions hydrolysis of the wet aluminum soap can occur during drying or even during washing. There is some evidence also to show that prolonged washing of the solid soap gives gels of somewhat lower consistency (43). The relationship of this to free acid content has, however, not been decisively proven.

Viscosity Concentration Relationship

Considerable work was done in the early days of the problem on the relationship between the consistency of the gels and their concentration (3,5). Much of this must be regarded as vitiated because no attention was paid to the moisture content of the gels used. Nevertheless, the general relationship is clearly shown in Figure 10, 1% in concentration over the range from 6% to 12% giving an increase of approximately 150-200 grams in consistency. The variability due to the use of different samples of Napalm (including their different water contents) is also clear. It will be seen that

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FIGURE 10  
VISCOSITY VS CONCENTRATION  
RELATIONSHIP  
ON SAMPLES AS RECEIVED



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without adequate control of the moisture and other variables, it is impossible to reproduce a solution to better than + 200 grams at the same concentration. This fact has been responsible for many of the disappointments and troubles previously reported with Napalm.

Recent data obtained at Edgewood (60) allows for this moisture effect and is summarized in Table XXVI. The soaps and procedure used were the same as those for Tables XVII-B and XVIII. It will be seen that the change in consistency for 1% concentration change is reasonably uniform over the ranges of moisture content and concentration investigated.

Further studies are planned on the viscosity-concentration relationship in which moisture content will be carefully controlled and the gasoline variable also investigated.

Table XXVI

Variation of Consistency with Concentration

△ Consistency per 1% Increase in Soap

| Moisture<br>CWS Benzol                     | A    |       | B    |       | C    |
|--|------|-------|------|-------|------|
|  | 8-9% | 9-12% | 8-9% | 9-12% | 8-9% |
| <u>A. Results after 48 hrs. at 77° F.</u>  |      |       |      |       |      |
| 0.4  | 210  | 160   | 160  | 120   | 170  |
| 0.8  | 100  | 190   | 200  | 170   | 180  |
| 1.2  | 60   | 200   | 170  | 170   | 170  |
| 1.6  | 40   | 190   | 140  | 170   | 140  |
| 2.0  | 50   | 230   | 100  | 160   | 160  |
| <u>B. Results after 24 hrs. at 150° F.</u> |      |       |      |       |      |
| 0.4  | 180  | 190   | 180  | 160   | 220  |
| 0.8  | 130  | 160   | 230  | 170   | 240  |
| 1.2  | 70   | 170   | 220  | 160   | 170  |
| 1.6  | 40   | 130   | 180  | 160   | 150  |
| 2.0  | 50   | 90    | 130  | 140   | 120  |

Testing and Specifications

In this section the background of the specifications (CWS 196-131-107 its revision 107A and Directive 201A, Appendix VIII) which has led to their formulation and the justification for the directions given will be considered.

Composition. This reads that the composition of the material shall approximate equal parts of basic aluminum naphthenate, aluminum oleate, and aluminum soap of cocoanut fatty acids. This was written at a time when it was still not certain that the best ratio of acids was two of cocoanut to one of naphthenic and one of oleic. Available evidence strongly indicates that the specification should now be changed to "The composition of the material shall approximate one part of basic aluminum naphthenate, one part of aluminum oleate and two parts of aluminum soap of cocoanut fatty acids".

Sieve Size. The requirements of this section safeguard against the presence of a large amount of fines, which solvate very rapidly causing premature gelation on mixing. Conversely, large lumps solvate too slowly. As long as excessive fines are avoided, there appears to be no reason why the limits should not be changed somewhat if desired. No difficulties in the actual determination using the apparatus suggested have been recorded.

Aluminum Content. This was originally written when it seemed that aluminum content might be some guide to gel behaviour. Sufficient evidence has now accumulated, that good gels can be obtained with soaps of widely varying metal content, that this no longer holds. The use of alum solutions containing water-insoluble aluminum compounds invalidates the test, because these are precipitated with the Napalm and must be included in the aluminum analysis.

Ash Content. Remarks similar to those made for aluminum content are also true here.

Moisture Content. Data have already been given in Tables II, XVII and XVIII to indicate that moisture is of primary importance in determining the viscosity of the gel, particularly in the 24 hour - 150° F. stability test. Soaps prepared by different methods undoubtedly differ in their susceptibility to moisture. Thus, three different soaps all showing 0.7% moisture by the benzol distillation method gave viscosities of 520, 380 and 700 in the 150° test. Nevertheless, most soaps studied seem to show satisfactory behaviour at moisture contents below 0.6%-0.8% by benzol distillation (CWS Method).

There has been considerable controversy as to the adequacy of the benzol distillation method for the determination of moisture. The method first used in which an amine was added to break the gel was shown to give erroneous results because of reaction between the amine added and free fatty acid of the soap. The method gives different results according to the boiling point of the solvent used (18). The specification issued July 8th, 1943, gives satisfactory results when operated carefully as shown by Table XXVII below where successive determinations were run on the same soap.

Table XXVII

Reproducibility of Benzol Moisture Determinations\*

| <u>Lot No.</u> | <u>No. of Drums</u> | <u>% Water From Opened Drum</u> | <u>Diff.</u> | <u>% Water From Unopened Drum</u> | <u>Diff.</u> |
|----------------|---------------------|---------------------------------|--------------|-----------------------------------|--------------|
| 54             | 12                  | .70                             |              | .75                               |              |
|                |                     | .88                             | .18          | .70                               | .05          |
| 55             | 12                  | .65                             |              | .50                               |              |
|                |                     | .87                             | .22          | .55                               | .05          |
| 56             | 12                  | .45                             |              | .50                               |              |
|                |                     | .50                             | .05          | .50                               | .00          |
| 57             | 12                  | .70                             |              | .45                               |              |
|                |                     | .63                             | .07          | .38                               | .07          |
| 58             | 11                  | .70                             |              | .70                               |              |
|                |                     | .72                             | .02          | .50                               | .20          |
| 59             | 9                   | .75                             |              | .75                               |              |
|                |                     | .70                             | .05          | .75                               | .00          |
| 60             | 12                  | .58                             |              | .50                               |              |
|                |                     | .53                             | .05          | .50                               | .00          |
| 61             | 12                  | .82                             |              | .63                               |              |
|                |                     | .73                             | .04          | .50                               | .13          |
| 62             | 12                  | .63                             |              | .75                               |              |
|                |                     | .63                             | .00          | .70                               | .05          |
| 63             | 12                  | .75                             |              | .75                               |              |
|                |                     | .80                             | .05          | .63                               | .12          |
| 64             | 12                  | .75                             |              | .53                               |              |
|                |                     | .75                             | .00          | .58                               | .05          |
| 65             | 12                  | .70                             |              | .63                               |              |
|                |                     | .68                             | .02          | .63                               | .00          |
| 66             | 12                  | .95                             |              | .63                               |              |
|                |                     | .75                             | .20          | .75                               | .12          |
| 67             | 12                  | .83                             |              | .70                               |              |
|                |                     | .78                             | .05          | .63                               | .07          |
| 68             | 11                  | .78                             |              | .70                               |              |
|                |                     | .75                             | .03          | .70                               | .00          |
| 69             | 11                  | .70                             |              | .80                               |              |
|                |                     | .90                             | .20          | .90                               | .10          |
| 70             | 5                   | .80                             |              | .78                               |              |
|                |                     | .75                             | .05          | .70                               | .08          |
| 71             | 5                   | .73                             |              | .83                               |              |
|                |                     | .70                             | .03          | .65                               | .18          |
| 72             | 1                   | 1.00                            |              |                                   |              |
|                |                     | .90                             | .10          |                                   |              |

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| <u>Lot No.</u> | <u>No. of Drums</u> | <u>% Water From Opened</u> |              | <u>% Water From Unopened</u> |              |
|----------------|---------------------|----------------------------|--------------|------------------------------|--------------|
|                |                     | <u>Drum</u>                | <u>Diff.</u> | <u>Drum</u>                  | <u>Diff.</u> |
| 73             | 1                   | .70                        |              |                              |              |
|                |                     | .75                        | .05          |                              |              |
|                | Average             | 0.72                       | .073         | 0.65                         | .076         |

\* Data from Rocky Mountain Inspection Laboratory through Captain W. Bauer.

Table XXVIII shows check results on the same soap between different laboratories.

Table XXVIII

Results with C.W.S. Benzol Distillation in Two Laboratories (10,33)

| <u>Soap</u>       | <u>Hours in Drying Ovens</u> | <u>% H<sub>2</sub>O</u>  |   |
|-------------------|------------------------------|--------------------------|---|
|                   |                              | <u>Harmon Color Wks.</u> | <u>C.W.S. Labs. Columbia University</u> |
| Harmon<br>R11249  | 12                           | 2.1                      | 2.2                                     |
|                   | 14                           | 2.6                      | 2.6                                     |
|                   | 16                           | 1.3                      | 1.1                                     |
|                   | 18                           | 1.2                      | 0.9                                     |
|                   | 20                           | 0.8                      | 0.8                                     |
|                   | 22                           | 0.3                      | 0.4                                     |
|                   | 24                           | 0.4                      | 0.3                                     |
|                   | 29                           | 0.4                      | 0.2                                     |
|                   |                              | <u>Eastman Kodak</u>     | <u>C.W.S. Lab., Edgewood</u>            |
| Harmon<br>83955 R | --                           | 0.55, 0.55               | 0.65                                    |

Setting. No difficulties have been reported with the official method for conducting this test.

Characteristics of the Gel. This section was originally written to insure that the gels formed should have the elasticity and stringiness characteristics of a good Napalm gel. Thus, a Napalm, if excessively oxidized, would fail to pass these tests.

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Nevertheless, as far as is known, no samples have ever been rejected on the basis of these tests and their abolition would seem desirable. If a stringiness or test for length is still desired then substitution of the extensiometer (52) for the spatula test would be indicated.

Stability at -40°F. This was originally inserted to insure that the Napalm gels would not show syneresis on standing for a time comparable to that experienced in a bomber flight at -40°F. No Napalm has ever been found to fail this test. Furthermore, it has been definitely shown (22) that this test does not correspond to the results obtained on long time low temperature storage such as might be experienced in actual use. It has been omitted in the final specifications.

Consistency. This is the most fundamental property of the soap since it governs behaviour in all its uses. Much study has been devoted to finding the right consistency for each different use (4, 5, 44). Although all concentrations are not yet fixed, it appears that it may be necessary to use Napalm at concentrations of (1) 4-5-1/2%, (2) 9%, and (3) 13%. Since it would obviously be unfair to ask the manufacturer to test his soap at each of these concentrations, 8% was chosen as a standard test concentration because it was the lowest concentration contemplated for use at the time the specification was written. This concentration has the advantage that it is about midway in the range of those used in practice and also allows reproducible readings to be obtained readily on the Gardner, which is difficult at higher soap concentrations.

From past experience it has become clear that there is no difficulty in the manufacture of a Napalm with a consistency of from 500 to 800 grams, if the moisture content is suitably controlled. At very low moisture contents, it is possible to obtain consistencies considerably in excess of 800 grams but such soaps would offer considerable difficulties in manufacture and handling.

Considerable trouble has been experienced in many places in running the consistency tests. The steps in the test, therefore, will be considered in detail.

1. Preparation of the Gel. The official method, which may be called the open vessel mixing technique, has received considerable criticism because of the possibilities of loss of solvent, condensation of moisture due to the refrigeration effect during vaporization and gel heterogeneity due to poor mixing. Perhaps the most thorough study of different mixing techniques was made by Allen and Levine (25). They prepared gels by the open vessel

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method, by tumbling the solid soap in gasoline in a quart jar, and by tumbling followed by stirring with a perforated piston or masher at the end of either two hours or 24 hours. Results are shown in Table XXIX. It will be seen that while there is little indication of either loss of gasoline or moisture condensation, there are signs that better mixing was obtained by methods other than the open vessel technique. Results on the 24 hour 150° test were not obtained, but it may be suspected that this would tend to make the results more uniform from top to bottom of the gels. Similar results with respect to evaporation were obtained by Elliott and McIntyre (46), who found that small gas losses in gel preparation affect gel strength very little. Thus, in two groups of 24 hour 150°F. tests:

|                |                  |     |       |
|----------------|------------------|-----|-------|
| 3% gas loss -- | average strength | 657 | grams |
| No gas loss -- | "                | 648 | "     |

This is well within the error of Gardner reading.

The possibility of moisture condensation has been tested at Eastman Kodak, gels being prepared in constant humidity rooms at 20% and 90% R. H. No consistent differences could be found.

It appears, therefore, that with reasonable care good results can be obtained with any method of gel preparation, although constant agitation and safeguards against evaporation are desirable.

2. Gel Transfer to Mobilometer. In the early days, because of the requirement that the 8% Napalm gels be poured, stored and evaluated in a capped mobilometer tube, the gel was not disturbed between completion of mixing and the first Gardner run. It soon became evident that storage would have to be made in tubes other than the mobilometer tubes and the question arose as to the difference in consistency caused by the transfer. Many inspection laboratories use a plunger or piston to transfer the gel from the storage tube to the mobilometer. Table XXX shows results obtained when this method of transference is used compared to values obtained with the static stored gel (25). This indicates that a slightly higher result is obtained when the gel is undisturbed between storage and measurement in the mobilometer by the method described in Directive No. 201.

Table XXX.

Effect of Gel Transfer on 48 Hour Viscosity (25).

(Directive No. 201 Method)

|                 | <u>Gardner Viscosities</u> |                 |                |
|-----------------|----------------------------|-----------------|----------------|
|                 | <u>Sample A</u>            | <u>Sample B</u> | <u>Average</u> |
| Static Gel      | 730 grams                  | 690 grams       | 710            |
| Transferred Gel | 660                        | 660             | 660            |

Table XXIX  
Uniformity of Gels (25)

| <u>Sample</u>               | <u>Open Vessel Mixing Technique</u> |               |                      |                              | <u>Tumbling Technique</u> |               |                      |                |
|-----------------------------|-------------------------------------|---------------|----------------------|------------------------------|---------------------------|---------------|----------------------|----------------|
|                             | <u>Gardner</u>                      |               | <u>Gardner</u>       |                              | <u>Gardner</u>            |               | <u>Gardner</u>       |                |
|                             | <u>Viscosity</u>                    |               | <u>Vis.Deviation</u> |                              | <u>Viscosity</u>          |               | <u>Vis.Deviation</u> |                |
|                             | <u>Top</u>                          | <u>Bottom</u> | <u>Test</u>          | <u>Average</u>               | <u>Top</u>                | <u>Bottom</u> | <u>Test</u>          | <u>Average</u> |
|                             | <u>grams</u>                        |               |                      |                              | <u>grams</u>              |               |                      |                |
| <u>Type B Napalm</u>        |                                     |               |                      |                              |                           |               |                      |                |
| Test 1                      | a.775                               | 770           | 5                    |                              | 810                       | 795           | 15                   |                |
|                             | b.775                               | 765           | 10                   | 7.5                          | 750                       | 795           | 45                   | 30             |
| Test 2                      | a.760                               | 720           | 40                   |                              | 740                       | 800           | 60                   |                |
|                             | b.715                               | 780           | 65                   | 52.5                         | 720                       | 730           | 10                   | 35             |
| Test 3                      | a.740                               | 800           | 60                   |                              | 735                       | 740           | 5                    |                |
|                             | b.775                               | 740           | 65                   | 62.5                         | 775                       | 755           | 20                   | 12.5           |
| Average                     | 757                                 | 763           |                      |                              | 755                       | 769           |                      |                |
| <u>Type A Napalm</u>        |                                     |               |                      |                              |                           |               |                      |                |
| Test 1                      | a.810                               | 655           | 155                  |                              | 730                       | 770           | 40                   |                |
|                             | b.820                               | 710           | 110                  | 132.5                        | 725                       | 760           | 35                   | 37.5           |
| Test 2                      | a.805                               | 715           | 90                   |                              | 750                       | 730           | 20                   |                |
|                             | b.820                               | 745           | 75                   | 82.5                         | ---                       | 730           | --                   | 20.0           |
| Test 3                      | a.775                               | 710           | 65                   |                              | 720                       | 775           | 55                   |                |
|                             | b.780                               | 730           | 50                   | 57.5                         | 730                       | 730           | 0                    | 27.5           |
| Average                     | 802                                 | 711           |                      |                              | 731                       | 749           |                      |                |
| <u>Tumbling 2 hour Mash</u> |                                     |               |                      | <u>Tumbling 24 Hour Mash</u> |                           |               |                      |                |
| <u>Type B Napalm</u>        |                                     |               |                      |                              |                           |               |                      |                |
| Test 1                      | a.775                               | 795           | 20                   |                              | 760                       | 785           | 25                   |                |
|                             | b.775                               | 775           | 0                    | 10                           | 810                       | 780           | 30                   | 27.5           |
| Test 2                      | a.735                               | 735           | 0                    |                              | 775                       | 755           | 20                   |                |
|                             | b.775                               | 775           | 0                    | 0                            | 730                       | 745           | 15                   | 17.5           |
| Test 3                      | a.775                               | 785           | 10                   |                              | 725                       | 765           | 40                   |                |
|                             | b.765                               | 760           | 5                    | 7.5                          | 765                       | 780           | 15                   | 27.5           |
| Average                     | 767                                 | 771           |                      |                              | 761                       | 768           |                      |                |
| <u>Type A Napalm</u>        |                                     |               |                      |                              |                           |               |                      |                |
| Test 1                      | a.760                               | 775           | 15                   |                              | 680                       | 755           | 75                   |                |
|                             | b.745                               | 745           | 0                    | 7.5                          | 750                       | 755           | 5                    | 40             |
| Test 2                      | a.740                               | 765           | 25                   |                              | 725                       | 725           | 0                    |                |
|                             | b.745                               | 750           | 5                    | 15.0                         | 695                       | 750           | 55                   | 27.5           |
| Test 3                      | a.730                               | 740           | 10                   |                              | 710                       | 730           | 20                   |                |
|                             | b.720                               | 740           | 20                   | 15.0                         | 740                       | 740           | 0                    | 10.0           |
| Average                     | 740                                 | 753           |                      |                              | 717                       | 743           |                      |                |

3. The Gardner Mobilometer and Directive No. 201. Owing to the unusual nature of the aluminum soap gels, discrepancies were early found when measurements on the Gardner Mobilometer were made by two different individuals. This raised considerable discussion as to the best method of running the instrument and a great amount of work has been done (28) It would seem unnecessary to detail this, but the main conclusions can be summarized. Providing the gel is worked once when it has been allowed to stand undisturbed for some time, further working offers no advantage and in some cases may lead to the introduction of air into the mobilometer. Among the many gels investigated, some appear to increase in viscosity and some to decrease on vigorous working. Short, or concentrated (10% or over) gels appear to increase in viscosity greatly on working. The latter become cut into small jelly-like particles, which do not coalesce again except on long standing.

Evaporation is not a serious error, but does occur to some extent during running of Gardner viscosities. Temperature of determination has a small effect as illustrated by the following table.

Table XXXI

Effect of Temperature of Determination on Gardner Viscosity (3)

(8% Napalm Gel)

| <u>Weight</u> | <u>Time of Fall (sec.)</u> |              |              |
|---------------|----------------------------|--------------|--------------|
|               | <u>58-60°F.</u>            | <u>70°F.</u> | <u>80°F.</u> |
| 850           | --                         | 4.2          | --           |
| 800           | 13.8                       | 9.6          | 10.6         |
| 750           | 19.4                       | 17.0         | 14.8         |
| 700           | 29.2                       | 21.6         | 22.0         |
| 650           | 67.0                       | 37.0         | 27.6         |
| 600           | 108                        | 57           | 44.2         |
| 550           | 187                        | 99           | 68           |
| 500           | --                         | 136          | 130          |
| 450           | --                         | --           | 191          |
| Consistency   | 600                        | 540          | 515          |

The points covered briefly above were considered at great length in preparing Directive No. 201 and its modification 201A. Consequently, it is believed that if the directions given in 201A are followed, no difficulty in securing reproducible results should be experienced.



4. Effect of Storage Containers on Consistency Results. In the early days of the Napalm program before the 150° F. accelerated test had been devised, storage of the gels after preparation was usually done in glass Mason jars. With good soaps, there was considerable indication that this storage in glass was equivalent to storage either in steel or glass jars containing steel. With the introduction of the 150° F. test, the glass jars, while usable, became of somewhat doubtful safety and many laboratories commenced to use steel or brass tubes. It was later shown that brass tubes were liable to give erratic results, particularly in presence of small amounts of moisture. Thus, a piece of polished brass placed in the gel will begin to show a green color, which slowly spreads out into the gel itself. Table XXXII below shows results on a number of different storage containers. It will be seen that brass gives lower values than either steel or glass and that a similar result can be obtained by adding 0.01% of copper as copper naphthanate. It must be concluded that (1) brass tubes are unsuitable either for the 48 hour or 150° storage test, (2) glass and iron are very similar in their action on the gel when used as storage containers.

Table XXXII

Effect of Storage Containers on Gel Viscosity<sup>(25)</sup>

| Storage                                    | Test | 48 Hr. Gardner Viscosity |          |     | 24 Hrs. 150° F. Gardner Viscosity |          |     |
|--|------|--------------------------|----------|-----|-----------------------------------|----------|-----|
|  |      | Sample A                 | Sample B | Av. | Sample A                          | Sample B | Av. |
| 48 hrs.<br>Glass                           | 1    | 800                      | 805      | 803 | 795                               | 780      | 788 |
|  | 2    | 820                      | 780      | 800 | 770                               | 820      | 795 |
| 48 hrs.<br>Brass (screw,<br>plug & gasket) | 1    | 900                      | 835      | 893 | 685                               | 695      | 690 |
|  | 2    | 820                      | 870      | 845 | 720                               | 740      | 730 |
| 48 hrs.<br>Brass (collodion<br>and cap)    | 1    | 865                      | 860      | 863 | 665                               | 685      | 675 |
|  | 2    | 880                      | 870      | 875 | 720                               | 710      | 715 |
| 48 hrs.<br>Steel (plug and<br>gasket)      | 1    | 845                      | 865      | 855 | 775                               | 795      | 785 |
|  | 2    | 840                      | 820      | 830 | 810                               | 760      | 785 |
| 48 hrs.<br>Glass ( plus<br>0.01% iron)     | 1    | 790                      | 780      | 785 | 800                               | 780      | 790 |
|  | 2    | 880                      | 830      | 880 | 790                               | 790      | 790 |
| 48 hrs.<br>Glass (plus<br>0.01% copper)    | 1    | 820                      | 840      | 830 | 710                               | 660      | 685 |
|  | 2    | 745                      | 755      | 750 | 620                               | 620      | 620 |
| 48 hrs.<br>Glass (plus<br>0.125% iron)     | 1    | 820                      | 820      | 820 | 610                               | 645      | 628 |
|  | 2    | 880                      | 830      | 830 | 690                               | 675      | 683 |
| 48 hrs.<br>Glass (plus<br>0.125% copper)   | 1    | 775                      | 810      | 793 | 625                               | 695      | 660 |
|  | 2    | 755                      | 760      | 758 | 670                               | 800      | 735 |

Table XXXIII

Effect of Storage Before Specification Tests (25).

| <u>Storage</u>    | <u>48 hrs. 77° F.<br/>Gardner Viscosity.</u> | <u>24 hrs. 150° F.<br/>Gardner Viscosity.</u> |
|-------------------|--|---|
| 48 hours in Glass | 800, 820<br>805, 780                         | 795, 770<br>780, 820                          |
| 24 hours in Glass | 800, 800<br>Lost, 860                        | 790, 300<br>Lost, 790                         |
| 2 hours in Glass  | 855, 810<br>Lost, 850                        | 760, 735<br>Lost, 810                         |

6. Reproducibility of Consistency Test. Results below (Table XXXIV) obtained from three different laboratories (46, 33, 45) indicate that with good technique no difficulty should be experienced in reproducing readings on the same soap. In studying the same soap in two different laboratories, perhaps the greatest difficulty, now that the different Gardner Mobilometers have been standardized at Edgewood, lies in the standard test gasoline employed. Although every effort is made to make this reproducible, it may be possible, due to this cause, to obtain differences of 50 to 100 grams in consistency measurements.

Table XXXIV

Reproducibility of Specification Consistency Tests

A. Consecutive Gels Prepared from Harmon #83955R using S.O.D. Test Gasoline (Pyrotechnics Division, Edgewood Arsenal). Moisture content 0.6% Benzol Distillation 0.55% Vacuum Oven. Stored in steel.

| Run No. | Compounding |      | Consistency After |                |               |
|---------|-------------|------|-------------------|----------------|---------------|
|         | Temp.       | R.H. | 1 hr. 150°F.      | 24 hrs. 150°F. | 48 hrs. 77°F. |
| 1       | 76° F.      | 71%  | 650, 680          | 600, 590       | 590, 610      |
| 2       | 75          | 77   | 640, 650          | 550, 560       | 600, 600      |
| 3       | 76          | 70   | 670, 660          | 580, 560       | 610, 590      |
| 4       | 75          | 78   | 650, 660          | 580, 580       | 610, 610      |
| 5       | 77          | 67   | 680, 680          | 570, 550       | 590, 630      |
| 6       | 77          | 67   | 660, 690          | 570, 600       | 620, 610      |
| 7       | 75          | 77   | 670, 680          | 590, 580       | 590, 610      |
| 8       | 75          | 78   | 680, 660          | 560, 550       | 570, 590      |
| 9       | 77          | 83   | 630, 630          | 550, 540       | 600, 560      |
| 10      | 77          | 81   | 670, 640          | 550, 550       | 600, 580      |

B. Consecutive Gels Prepared Using Standard Oil Development Test Gasoline (Eastman Kodak Co.). Stored in steel.

| <u>Harmon 83955-R</u> |                | <u>Ferro Batch #22</u> |                |
|-----------------------|----------------|------------------------|----------------|
| Run No.               | 24 hrs. 150°F. | Run No.                | 24 hrs. 150°F. |
| 1                     | 660            | 1                      | 450            |
| 2                     | 640            | 2                      | 480            |
| 3                     | 620            | 3                      | 490            |
| 4                     | 630            | 4                      | 500            |
| 5                     | 610            | 5                      | 480            |
| 6                     | 650            | 6                      | 460            |

C. Consecutive Gels Prepared by the Tumbling Technique. Stored in glass. (Nuodex).

| Sample | <u>Type A</u>     |                 | <u>Type B</u>     |                 |
|--------|-------------------|-----------------|-------------------|-----------------|
|        | 48 Hour Viscosity | 150°F Viscosity | 48 Hour Viscosity | 150°F Viscosity |
| 1      | 740               | 565             | 820               | 630             |
| 2      | 760               | 555             | 790               | 595             |
| 3      | 755               | 540             | 810               | 600             |
| 4      | 800               | 530             | 820               | 580             |
| 5      | 765               | 560             | 840               | 630             |
| 6      | 785               | 575             | 785               | 570             |

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TI No. 18215

C. W. S. Directive No. 201A

Chemical Warfare Center,  
Edgewood Arsenal,  
Maryland.

Superseding  
C. W. S. Directive No. 201  
24 April 1943

Air Documents Division, T-2  
AMC, Wright Field  
Microfilm No.

RC-6 F18215

DETERMINATION OF THE CONSISTENCY OF

INCENDIARY OILS

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DETERMINATION OF THE CONSISTENCY OF INCENDIARY OILS

I. GENERAL.

The purpose of this directive is to describe methods for the determination of the consistency of incendiary oils. Section II of this directive covers the Gardner mobilometer method which will be used for all incendiary oils of the Napalm type. Section III covers the parallel plate method which will be used for incendiary oils, containing isobutylmethacrylate polymers and sodium salts of fatty acids, such as Incendiary Oil, IM, Type III. (C.W.S. Specification No. 196-131-145).

II. GARDNER MOBILOMETER METHOD.

A. Description of the instrument.

1. The mobilometer is widely used in testing laboratories and can be purchased from most laboratory supply houses. (See Notes, Paragraph D, 1). The mobilometer consists of a tube, 4-hole disc, plunger rod, weight pan and bearing, mounted on a suitable base and support. The pertinent dimensions of the standard instrument are as follows:

Dimensions of Standard Gardner  
Mobilometer at Edgewood Arsenal, Maryland.

|                             |              |
|-----------------------------|--------------|
| Internal diameter of tube   | 1.535 inches |
| Diameter of disc            | 1.505 inches |
| Thickness of disc           | 3/32 inch    |
| Diameter of holes in disc   | 0.250 inch   |
| Diameter of plunger rod     | 1/4 inch     |
| Diameter of bearing opening | 17/64 inch   |

2. Cover. No tube cover is supplied with the instrument for the purpose of keeping the tube covered during the test. However, a suitable cover shall be made of cardboard, sheet metal, or thin wood and shall be large enough to cover the open end of the tube completely. The cover shall have a central opening large enough to permit easy passage of the collar on the disc.

3. Storage tubes. Tubes for storage of thickened fuel shall be made of steel. The internal diameter shall be approximately the same size or slightly larger than the external diameter of the mobilometer tubes used for testing. The length of the tube available for storage shall be approximately 10 inches and the tube shall be filled to give a void of  $10 \pm 2$  percent.

The storage tubes shall be capped at both ends with removable steel caps, capable of withstanding a pressure of 35 p.s.i. Gaskets of materials which do not affect the incendiary oil may be used (See Notes, Paragraph D, 2).

**B. Determination of consistency.**

1. In the determination of the consistency of Napalm incendiary oil, the following details, given in the order of their performance, should receive particular attention:

a. Fill the tube.

b. Assemble the mobilometer.

c. Make 5 working strokes when measuring gels containing less than 9 percent Napalm thickener. Take less than 1 minute to accomplish the 5 strokes. For gels containing 9 percent or more of the Napalm thickener, make only one working stroke.

d. Make the first run, using a total weight of 50 grams in excess of the specified upper limit of consistency.

e. Reduce the load by 50 grams on each succeeding run (except as in Paragraph B, 1, f).

f. Continue this process until the time of fall exceeds 100 seconds. If the time in Paragraph B, 1, d exceeds 100 seconds, increase the load by increments of 50 grams until a time of fall of less than 100 seconds is obtained.

2. A brief summary of the conditions standard for all runs made in determining consistency is as follows:

a. Start the complete stroke with the lower mark on the plunger  $1 \pm \frac{1}{4}$  inch above the bearing.

b. Allow the loaded plunger assembly to descend until the upper mark passes the upper surface of the bearing. Within 10 seconds thereafter, remove the weights, push the disc down to the bottom of the tube, and withdraw the disc until the lower mark on the plunger rod is  $1 \pm \frac{1}{4}$  inch above the bearing, wiping the rod on the upstroke. This procedure is to be used for gels containing less than 9 percent Napalm. For gels containing 9 percent or more Napalm, allow the loaded plunger assembly to descend until the upper mark passes the upper surface of the bearing. Within 10 seconds thereafter, remove the weights, push the disc down until it touches the bottom of the tube, then withdraw the disc until it strikes the tube cover, wiping the rod on the upstroke, and finally push the plunger down until the lower mark is  $1 \pm \frac{1}{4}$  inch above the bearing.

c. The time between runs shall be  $60 \pm 10$  seconds.

d. During the determination, and for at least 2 hours prior thereto, the temperature of the gel shall be  $77 \pm 2^\circ\text{F}$ .

3. Detailed instructions. The instructions outlined above are detailed as follows:

a. Filling the mobilometer tube. When the consistency of an incendiary oil is to be measured, the sample should be transferred from the storage tube to the mobilometer tube at the time of test. If the mobilometer tube will fit into the storage tube, transfer the oil as follows: Remove one cap of the storage tube and insert the mobilometer tube, without cap or base, into the storage tube, base end first. Push down until the base of the mobilometer tube has reached the lower cap of the storage tube. Remove the remaining cap of the storage tube and screw the base of the mobilometer tube into place. Withdraw the mobilometer tube containing the thickened fuel sample and cap until the consistency determination is made.

To return the sample to the storage tube, screw one cap of the tube loosely into place, so that the passage of air is freely permitted. Remove the base of the mobilometer tube containing the sample and insert the lower end of the mobilometer tube a short distance into the open end of the storage tube. By means of a solid disc on a rod, slowly push the sample from the mobilometer tube into the storage tube. After the transfer of the sample is effected, screw the remaining storage tube cap into place and securely tighten both caps.

If the diameter of the storage tube is approximately the same as that of the mobilometer tube, the sample may be transferred in a similar manner, except that the tube from which the gel is removed is held tightly over the receiving tube while the sample is extruded with a disc on a plunger. The base of the receiving tube should be loosely fastened until transfer is complete, to permit escape of air. Care must be taken not to entrap air in the base of the mobilometer tube.

Any convenient method of transfer of the sample from the storage to the mobilometer tube may be used, provided that the exposure of the sample to evaporation is no greater than that in the procedures described above.

The mobilometer tube shall be filled with the thickened fuel sample to a depth of not more than  $1/2$  inch below the top of the tube.

b. Assembly of the mobilometer. Upon receipt of the mobilometer, the plunger rod should be screwed firmly into place in the four-hole disc and thereafter left in place. To assemble the mobilometer for testing consistency, pass the plunger rod through the cover and the bearing, attach the weight pan, and fasten the bearing support to the mobilometer

tube. Clamp the bearing support to the mobilometer tube at such a height that the disc is 1 to 1-1/2 inches above the bottom of the tube when the upper mark on the plunger rod is at the bearing.

c. Working stroke. Force the plunger down manually until the disc touches the bottom of the tube, then withdraw until the lower mark on the rod is  $1 + 1/4$  inch above the bearing. This procedure shall require not more than 10 seconds. During the up stroke, hold a piece of absorbent cloth or paper around the plunger rod to prevent Napalm oil from being carried into the bearing. The presence of dried material on the rod, bearing, cover, disc or holes in disc will lead to error in measurement. The above procedure shall apply to the testing of gels containing less than 9 percent Napalm thickener. For gels containing 9 percent Napalm or more, use a similar procedure, but force the plunger down manually until the disc strikes the bottom of the tube, then draw it up until it strikes the tube cover, and finally force down until the lower mark on the rod is  $1 + 1/4$  inch above the bearing.

d. Making the determination. Determine the consistency of Napalm oil as follows:

(1) Each type of Napalm oil has a specified consistency range expressed in grams. In the first run in any determination, use a total weight, consisting of the sum of the weights of the disc, plunger, and weight pan plus added weights, of 50 grams in excess of the upper limit of consistency specified for the type of Napalm oil under test.

(2) After working the sample, grasp the plunger rod at the bearing and place the required weight on the pan. The interval between the end of the working stroke and the start of the first run shall be 60 + 10 seconds. The weights may be placed on the weight pan at any time during this interval. The placing of the disc so that the lower mark on the plunger is  $1 + 1/4$  inch above the bearing shall be considered as the end of the working stroke. Start the run by releasing the weighted plunger assembly, thus applying the load to the oil.

(3) When the run is started the plunger assembly moves downward. Start the stop watch when the lower mark on the rod passes the upper surface of the bearing. When the upper mark passes the upper surface of the bearing, stop the watch. Remove the weights from the pan, force the plunger down manually until the disc touches the bottom of the tube, then withdraw until the lower mark on the rod is  $1 + 1/4$  inch above the bearing, wiping the rod, as before, on the upstroke. In the case of oils containing 9 percent or more Napalm thickener, carry the disc upward until it strikes the tube cover, (wiping the rod) and finally down until the lower mark on the rod is  $1 + 1/4$  inch above the bearing. Not more than 10 seconds shall elapse between stopping the watch and placing the plunger assembly in the starting position ( $1 + 1/4$  inch above the bearing). Record the total weight used in the run and the elapsed time shown on the stop watch.



e. Completing the determination.

(1) Hold the rod at the bearing, and place a weight, 50 grams less than that used in the first run (exception: see Paragraph B, 1, f), on the weight pan. Release the weighted plunger assembly 60 ± 10 seconds after completion of the first run. Then finish the run by performing all the operations prescribed above (See Paragraph B, 1, f).

f. Reporting results. The two successive loads, in grams, giving times of fall less and more than 100 seconds respectively, shall be noted. The load required to give a time of fall of 100 seconds shall be obtained from the two loads so noted by linear interpolation. This figure, corrected by the proper correction factor for the tube and instrument used and reported to the next higher 10 grams, shall be considered the consistency of the material under test. (Thus, 803 grams shall be reported as 810 grams).

C. Standardisation of the instrument.

1. Each mobilometer which is to be used for testing Napalm oils must be standardized to correct for dimensional variations. Although some mobilometers are furnished with a standard liquid for standardisation purposes, all mobilometers intended for use on Napalm oils must be standardized using a Napalm oil.

2. For the purpose of official testing of the consistency of Napalm oils, a mobilometer, located at the Chemical Warfare Service Technical Command, has been designated as the primary standard instrument. Each laboratory engaged in the determination of the consistency of Napalm oils shall forward one complete mobilometer to "Commanding General, Chemical Warfare Service Technical Command, Edgewood Arsenal, Maryland; Attention: Chief, Chemical Research Division", for comparison with the primary standard mobilometer using Napalm oils. The instrument will be stamped and returned to the sender with a correction. This instrument will be used as a secondary standard against which all combinations of mobilometer tubes and disc-plunger-bearing assemblies shall be compared. Proper corrections shall be given to the several combinations of tube and instrument.

3. Calibration of mobilometers using secondary standard. Comparisons between a mobilometer to be calibrated and a secondary standard shall be made as follows: Prepare samples of incendiary oils, containing various percentages of Napalm thickener in gasoline, according to the procedure given in C.W.S. Specification No. 196-131-107. Store the sample oil in a steel storage tube for 24 hours at  $150 \pm 2^{\circ}\text{F}$ ., then place the storage tube in a water bath, at  $77 \pm 2^{\circ}\text{F}$ ., for at least 2 hours. Transfer the sample from the storage tube to the tube of the mobilometer to be calibrated and measure the consistency, at  $77 \pm 2^{\circ}\text{F}$ ., as prescribed in Paragraph B, 3. Then transfer the sample to the secondary standard instrument and measure the consistency. Ascertain the deviation between the consistency

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obtained in the mobilometer to be calibrated and the consistency obtained in the secondary standard (See Paragraph C, 4). This completes one set of measurements. Repeat the set of measurements, using a second sample of oil similarly prepared, but transfer the sample first to the secondary standard for measurement and finally to the tube of the instrument to be calibrated. Ascertain the deviation between the measurements of this set as before. Make not less than two sets of measurements on each of two samples of oils having consistencies of about 500 grams. Repeat using samples of oils having consistencies of about 650 grams and again with oils of about 800 grams consistency. A total of not less than 12 sets of two measurements each will be made.

4. Calculation of correction. In making measurements for calibration purposes, interpolate the consistency to the nearest gram rather than to the next higher 10 grams as specified in Paragraph B, 3, f. Calculate the mean of the deviations observed between the two measurements in each set. Reject any deviation which differs by more than 25 grams from the mean value. If more than two deviations are rejected, run additional determinations and recalculate the mean until at least 10 deviations are obtained, all of which are within 25 grams of their mean. Round off the mean to the nearer 10 grams. From the correction factor of the secondary standard and the mean deviation, calculate and record the correction factor of the mobilometer being tested. No mobilometer with a correction factor greater than  $\pm 30$  grams shall be used to test the consistency of incendiary oils.

D. Notes.

1. Mobilometers obtained from the Henry A. Gardner Laboratories, Inc., Bethesda, Maryland, have been found to be very satisfactory.

2. A non-swelling, non-extractable rubber substitute gasket material,  $3/32$  to  $5/32$  inch thick, procured from Hydro-Chemical Co., 355 S. Main St., Akron, Ohio, has been found to be satisfactory.

III. PARALLEL PLATE METHOD.

A. Description of the instrument.

1. Two pieces of plate glass are required, about 9 by 9 inches and  $1/4$  inch thick. It is convenient to place the plates in an open-top shallow wooden box or tray.

2. The top piece of glass, suitably marked, is weighed and a weight prepared such that the total weight of the glass and the weight is 2000 grams. It is convenient to make this weight with a circular base, 5.0 cm. in diameter.

3. A circle is marked on the under side of the bottom piece of glass, using a diameter of 5.0 cm. The center of the circle shall be at the center of the glass plate. Four straight lines are drawn through the center of the circle in such a manner as to divide the circle into 8 equal sectors of  $45^\circ$ .

4. For convenience, a plug syringe, similar to that shown on C.W.S. Drawing No. A18-60-21, is recommended.

B. Determination of consistency.

1. Clean both plates thoroughly, removing any incendiary oil from previous tests by rubbing with benzene and drying.

2. Set the bottom plate, with markings on the under side, in the wooden box and place on a table in a level position.

3. Place  $5.0 \pm 0.1$  ml. of the incendiary oil to be tested in the center of the circle marked on the bottom plate. This may be conveniently accomplished by means of the plug syringe (C.W.S. Drawing No. A18-60-21).

4. Place the rear edge of the top plate above the rear edge of the bottom plate. Lower the top plate on the incendiary oil smoothly, then place the prepared weight gently on the top plate, directly above the circle on the bottom plate.

5. When the gel has spread to a circle of 5 cm. diameter (up to the circle mark), start the stop watch.

6. Beginning 55 seconds after the stop watch is started, make 8 light marks, as with India ink, on the top plate, marking the edge of the circle of incendiary oil at points on the radial lines drawn on the bottom plate. These markings must be made in clockwise or counterclockwise order and must be completed 65 seconds after the stop watch was started. Remove the weight from the top plate.

7. Measure the distance, in centimeters, between the two marks located on each of the 4 lines drawn through the center of the circle.

C. Reporting results.

1. Average the 4 diameters measured in III, B, 7 above.

2. The consistency of the incendiary oil shall be reported as the average diameter, in centimeters, of the circle formed by the oil when tested as prescribed in III, B, above.

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CHEMICAL WARFARE SERVICE  
SPECIFICATION

No. 196-131-107A  
25 October 1943  
Superseding  
No. 196-131-107  
19 November 1942

**NAPALM THICKENER**

NO. 18216

**A. APPLICABLE SPECIFICATIONS.**

a-1. The latest issue of the following specifications and directive, in effect on date of invitation for bids, shall form a part of this specification:

a-1a. Governmental publications.

Sieves; Standard, Testing - Federal Specification No. RE-5-760.  
Gasoline, Test (For Napalm Thickener) - C.W.S. Specification No. 196-131-206.

Terne Plate (Long Termes) - Federal Specification No. QQ-T-191.  
Army-Navy General Specification for Packaging and Packing for Overseas Shipment - U. S. Army Specification No. 100-14.

Marking, Outside Shipping Containers - General Specification - C.W.S. Specification No. 197-54-329.

Stain, Water Soluble - Ordnance Department Specification No. AXS-843.

Stain, Oil Type - Ordnance Department Specification No. AXS-883.

General Specification, Protective Coating Materials; Synthetic Type - Quartermaster Corps Tentative Specification HQD ES-No. 680.

Determination of Consistency of Incendiary Oils - C.W.S. Directive No. 201.

a-1b. Non-Governmental publication.

Method of Test for Water in Petroleum Products - A.S.T.M. Designation: D95-40.

Air Documents Division, T-2  
AMC, Wright Field  
Microfilm No.  
RC-6 F/18216

**B. TYPE.**

a-3-1. This specification covers one type of thickening agent for use in the manufacture of flame thrower fuel and incendiary oil NP.

**C. MATERIAL AND WORKMANSHIP.**

a-3-1. Material. The material shall consist of a coprecipitated mixture of basic aluminum soaps of fatty acids and naphthenic acid (See Paragraph D-1 and Notes, Paragraph H-1). No Napalm thickener, once submitted for acceptance and rejected by the Chemical Warfare Service, shall be resubmitted for acceptance, nor shall it be used to admix with acceptable Napalm thickener to make a mixture or combination which will conform with the requirements of this specification.

## MATERIAL AND WORKMANSHIP (CCRT '0).

- C-2. Workmanship. This material shall be made in a manner consistent with the best commercial practice in the manufacture of metallic soaps.

## D. GENERAL REQUIREMENTS.

- \*D-1. Composition. The material shall be a basic aluminum soap of naphthenic, oleic and coconut fatty acids. The sodium soap used for the preparation of the aluminum soap shall contain 0.10 to 0.15 percent of alpha-naphthol. (See Notes, Paragraph H-1).

## E. DETAIL REQUIREMENTS.

- E-1. Physical and chemical requirements.

E-1a. Appearance. The material shall be granular.

\*E-1b. Sieve size. None of the material shall remain on a No. 6 U. S. Standard sieve and not more than 25 percent shall pass through a No. 40 U. S. Standard sieve.

\*E-1c. Moisture. The material shall contain not more than 0.6 percent nor less than 0.4 percent moisture when tested as prescribed in Paragraph F-6.

E-2. Performance.

E-2a. Gel characteristics. The material, when mixed with test gasoline in the proportions and in accordance with the method prescribed in F-7a, shall produce a gel which shall have the characteristics prescribed in Paragraph F-7b.

\*E-2b. Consistency. The consistency of the gel, formed as prescribed in Paragraph F-7c(1), and tested as prescribed in Paragraph F-7c, shall be not less than 500 nor more than 800 grams for each of the 3 tests.

## F. METHODS OF SAMPLING, INSPECTION AND TEST.

F-1. The material and work shall be subject at all times to inspection at the place of manufacture, and every facility shall be afforded authorized Government inspectors for the prosecution of their work.

F-2. Lot size. The size of a lot shall be as specified in the invitation for bids.

\*F-3. Batch size.

\*F-3a. Unit batch. A unit batch shall consist of the quantity of aluminum soap precipitated in one reactor at one time. The batch shall be processed as a unit.

**METHODS OF SAMPLING, INSPECTION AND TEST (CONT'D).**

- \*F-3b. **Blended batch.** If parts of more than one batch are combined to form a blended batch, the blended batch shall consist of the quantity of material mixed in the blender at one time.
- \*F-4. **Sampling.** Not less than 2 one-pound samples shall be taken from each batch of Napalm thickener for test (see Note). Each sample shall be placed individually in a clean, dry, air-tight container, sealed and labeled to assure the identity of the sample with the batch represented. One of the one-pound samples shall be reserved for all tests other than the performance tests prescribed in Paragraph F-7. The other one-pound sample or samples shall each be submitted to the performance tests. Note: The uniformity of a batch of Napalm thickener depends upon the processing used in its manufacture. Proper uniformity is indicated when all samples, representing a single batch, when tested for consistency, give consistencies which fall within a range of 150 grams. In each plant, the Government Inspector shall take as many one-pound samples from each batch as he deems necessary until proper uniformity of the material manufactured in the plant is established. The inspector shall then prescribe the number of samples (but never less than two) to be taken from each batch thereafter manufactured in the plant using the same processing methods. In the case of blended batches the inspector shall take not less than 5 samples from various points of each of the first two blended batches. If the 5 samples show proper uniformity (consistencies agreeing within 150 grams), all succeeding blended batches may be represented by 2 one-pound samples provided no change is made in the blending process.
- \*F-5. **Sieve size.** Test a No. 6 U. S. Standard sieve (Federal Specification TS-28-8-50) on a No. 40 U. S. Standard sieve equipped with a receiving pan. Place a 40-gram sample in the No. 6 sieve, cover, place the rest of sieves in a standard vibrating machine (See Notes, Paragraph H-2), and vibrate for 2 minutes with the tapper in operation. Weigh and calculate percent of sample remaining on the No. 6 sieve and the percent of sample passing the No. 40 sieve.
- \*F-6. **Moisture.** Weigh out rapidly about 40.0 grams of the sample and immediately disperse in 500 ± 10 ml. of benzene in a round-bottom liter flask. After the sample has been dispersed (about 5 minutes) determine the moisture content of the suspension in accordance with the Method of Test for Water in Petroleum Products - A.S.P.M. Designation: D95-40, with the following exceptions and additional details.

- (1) Use the entire 500 ml. of suspension instead of the 100 ml. specified.
- (2) Heat the flask by means of an oil bath. The temperature of the oil bath shall be 140 ± 5°C.

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**METHODS OF SAMPLING, INSPECTION AND TEST (CONT'D).**

- (3) Distill for 2 hours. Wash down the condenser tube with three 10-ml. portions of benzene, scrubbing the tube at the same time with a stiff brush to loosen water droplets. Resume the distillation for an additional 1/2 hour, then repeat the washing and scrubbing.
- (4) Protect the top of the condenser tube by a CaCl<sub>2</sub> tube or equivalent, except during the rinsing and scrubbing operations.
- (5) At all times during the distillation, maintain the oil in the bath at a level about 1/4 inch below the level of the suspension in the liter flask.
- (6) Make a blank determination by subjecting 500 ml. of benzene to the same treatment as the suspension and correct all determinations accordingly.
- (7) Determine the efficiency of recovery by treating similarly 500 ml. benzene to which 0.50 ml. water has been added. If after correction for the blank, the apparatus fails to recover  $0.50 \pm 0.05$  ml. of water, tighten all connections and repeat the test. No determinations shall be made with apparatus which has not demonstrated satisfactory recovery.

**F-7. Performance Tests.**

- \*F-7a. Preparation of gel for test purposes.** Uniform gels containing  $8.0 \pm 0.1$  percent Napalm thickener shall be prepared. Any suitable method may be used provided due consideration is given to proper protection against loss of gasoline by evaporation, absorption of moisture by the Napalm thickener or by the gel, and agitation for a sufficient length of time to insure uniformity of the finished gel. The following method is recommended:

Weigh the test gasoline (C.W.S. Spec. No. 196-131-206), to within one gram, into a glass fruit jar of suitable capacity, and cover immediately. Weigh the sample (to within 0.1 gram) into a beaker. Transfer quantitatively and as rapidly as possible to the jar containing the gasoline. Close the jar tightly and shake vigorously. Stop shaking from time to time to observe any settling of particles of Napalm thickener. When no more settling is observed continue to shake or roll the jar for a period of time at least equal to the previous shaking time.

- \*F-7b. Gel characteristics.** Prepare about 300 grams of gel in a one-pint glass fruit jar (See Paragraph F-7a) and store for 48 hours at room temperature (65°F. minimum). Examine the gel as prescribed in Paragraphs F-7b(1), F-7b(2), and F-7b(3). Failure of the gel to show the characteristics described in this section is an indication that the thickener is not suitable for the use intended.

**F-7b(1)** The gel shall be free from lumps and of uniform consistency.

**METHODS OF SAMPLING, INSPECTION AND TEST (CONT'D).**

F-7b(2) Insert an unpainted wooden stick,  $\frac{3}{8}$  inch in diameter, at right angles through the surface of the 48-hour old gel to a depth of one inch and draw once across a diameter. The surfaces of the gel, parted by the stick, shall adhere within 10 seconds after contact with each other, leaving only a line and not a torn surface.

F-7b(3) Gather a lump of the 48-hour old gel at the end of the wooden stick and draw smoothly away from the surface of the gel. The band or bands of gel which follow the lump shall not snap suddenly in a direction perpendicular to the line of motion but shall separate from the lump by stringing down to a thread.

F-7c. Consistency. Test the consistency of gels, prepared as prescribed in Paragraph F-7a, by the mobilometer method described in C.W.S. Directive No. 201, as follows:

F-7c(1) Preparation of gels for consistency tests. Prepare the necessary gels in accordance with Paragraph F-7a. For the 3 tests, prescribed in Paragraphs F-7c(2), F-7c(3), and F-7c(4), all of the gel required for one determination in each of the 3 tests may be prepared at one time (about 750 grams). The duplicate determinations, required in each of the 3 tests, must be run on another preparation of gel; in no case shall duplicate tests be run on 2 portions of the same preparation of gel.

F-7c(2) One hour test. Transfer a portion of the gel to a storage tube in accordance with C.W.S. Directive No. 201. Close the tube tightly and, within one hour after preparation of the gel, place the tube in a water bath at  $150^{\circ} \pm 5^{\circ}\text{F}$ . for  $60 \pm 5$  minutes. At the end of this time transfer the tube to a water bath at  $77^{\circ} \pm 2^{\circ}\text{F}$ . for an additional 2 to 4 hours. Remove the tube from the bath, dry the outside of the tube thoroughly, uncap the tube, transfer the contents to a mobilometer tube and determine the consistency of the gel in accordance with C.W.S. Directive No. 201. Run this test in duplicate (See Paragraph F-7c(1)).

F-7c(3) Twenty-four hour test. Transfer a portion of the gel to a storage tube in accordance with C.W.S. Directive No. 201. Close the tube tightly and, within one hour after preparation of the gel, place the tube in a water bath at  $150^{\circ} \pm 5^{\circ}\text{F}$ . for  $24 \pm 4$  hours. At the end of this time, transfer the tube to a water bath at  $77^{\circ} \pm 2^{\circ}\text{F}$ . for an additional 2 to 4 hour. Remove the tube from the bath, dry the outside of the tube thoroughly, uncap the tube, transfer the contents to a mobilometer tube and determine the consistency of the gel in accordance with C.W.S. Directive No. 201. Run this test in duplicate (See Paragraph F-7c(1)).



## METHODS OF SAMPLING, INSPECTION AND TEST (CONT'D).

- F-7c(4)** Forty-eight hour test. Transfer a portion of the gel to a storage tube in accordance with C.W.S. Directive No. 201. Close the tube tightly and allow to stand at room temperature (65°F., minimum) for  $44 \pm 4$  hours. At the end of this time transfer the tube to a water bath at  $77^\circ \pm 2^\circ\text{F}$ . for at least 2 hours. The total time of storage, including time in the water bath, shall not exceed 50 hours. Remove the tube from the bath, dry the outside of the tube thoroughly, uncap the tube, transfer the contents to a mobilometer tube and determine the consistency of the gel in accordance with C.W.S. Directive No. 201. Run this test in duplicate (See Paragraph F-7c(1)).
- F-7c(5)** Concordance of consistency test results. The results on the duplicate runs for each of the tests prescribed in Paragraphs F-7c(2), F-7c(3), and F-7c(4), shall agree within 50 grams. If this condition is not met, repeat the consistency tests completely.
- F-7c(6)** Reporting results. Average the two consistencies from each test. Report the average consistency for each test to the next highest multiple of 10 grams. The average consistency so reported must meet the requirements prescribed in Paragraph F-2b.

## G. PACKAGING, PACKING AND MARKING.

**G-1.** Requirements for packaging and packing will be specified in the invitation for bids. Unless otherwise specified, packing shall be as follows: (See Notes, Paragraph 3-5).

**eg-1a(1)** Type A packaging (for material to be used in flame thrower fuel). The material shall be packed in "square" 30-gauge, Class A terne plate cans (Federal Specification No. QQ-T-191), approximately  $8\text{-}1/8$  by  $5\text{-}1/2$  by 9 inches high, having a circular opening, approximately  $4\text{-}1/2$  inches in diameter, in the head. The cans shall be painted with lusterless olive drab enamel (Quartermaster Corp. Tentative Specification HQ) ES-No. 680). After filling with 5 lb. 4 oz.  $\pm$  1 oz. of Napalm thickener, net weight, the top opening shall be closed and hermetically sealed by soldering. Each can shall then be stenciled or labeled with a waterproof label as follows: "Mix the contents of this package with 10 U. S. gallons of gasoline."

**eg-1a(2)** Type A packing. Eight filled cans shall be placed in a nailed and strapped wood box with double face corrugated fiberboard, interlocking half-slotted partitions as interior packing. The box shall comply with the requirements of Section VI of U. S. Army Specification No. 100-24, and shall be approximately  $23\text{-}1/2$  by  $17\text{-}1/2$  by 9 inches high, inside dimensions. All wooden boxes shall be stained brown by a stain conforming to the requirements of Ordnance Department Specification No. AXS-843 or No. AXE-883.

**PACKING AND MARKING (CONT'D).**

- G-1b. Type H greasing (for material to be used in incendiary oil, EF). The material shall be packed in 100 ± 1 lb. units, in the following containers:
  - G-1b(1) Fullhead opening steel drums, complying with I.C.C. Specification No. 57D, finished on the inside with 2 coats of a high quality, iron-free, corrosion resistant enamel and lined with a creped sulfate kraft liner.
  - G-1b(2) Tied 50-lb. kraft paper bag placed in a 30-30-30 tied asphalt laminated duplex kraft bag, the whole being enclosed in a paraffin-waxed fiber drum complying with I.C.C. Specification No. 21A.
- G-2. Marking. All shipments shall be marked as prescribed in C.W.S. Specification No. 197-54-529. In addition, the top and bottom of the container shall be marked, in 2-inch yellow letters, "KERO DEX". In addition the batch number of the material shall be stenciled on the tops of all containers.

**B. NOTES.**

•H-1. The recommended formula for Napalm thickener is as follows:

a. Organic acids mixture:

|                     | <u>Parts by weight</u> |
|---------------------|------------------------|
| Coconut Fatty Acids | 50                     |
| Naphthenic Acid     | 25                     |
| Oleic Acid          | 25                     |

b. Alumina content: 5.4 to 5.8 percent

- H-2. By a standard vibrating machine as meant a W.S. Tyler Company, No-20 Testing Sieve Shaker, single eccentric type, directly connected to a motor having a speed of 1725 revolutions per minute.
- H-3. The following is furnished for the information of the contractor:

Impurities in Napalm thickener which may cause partial or complete breakdown of gels formed with gasoline, or oxidation of the thickener, include excess water, lime, caustic soda, scraps of sodium, copper, lead, iron, manganese, and cobalt, powdered or sheet zinc and lead, lead nitrate, rust preventatives containing amines, alcohols and all acids. Tetraethyl lead, on the other hand, has no injurious effects.

•H-4. A one-pound sample of Napalm thickener shall be taken at random from each week's production and shipped to the Chief, Technical Division, Edgewood Arsenal, Maryland, for surveillance tests. The sample shall be accompanied by the results of the inspection tests on the batch represented by the sample.

**NOTES (CONT'D).**

- H-5. Each shipment of Inpalm thickener shall be accompanied by copy of the inspection test results on each batch contained in the shipment.
- H-6. An asterisk preceding a paragraph number indicates that the corresponding paragraph in the previous issue was revised or a new paragraph added.
- H-7. The use of this specification, wherever applicable, is mandatory on all procuring agencies of the Army.
- H-8. **NOTICE:** This specification, together with specifications and drawings pertaining to it and bearing a 'Notice' of similar restrictions, is intended for use only in connection with procurement by the United States Government, and shall not be reproduced either wholly or in part except when authorized in connection with Government procurement, nor be used for any other purpose except when specifically authorized by the Chief of Chemical Warfare Service, Washington, D. C.
- H-9. **NOTICE:** This document contains information affecting the national defense of the United States within the meaning of the Espionage Act (U.S.C. 50:31,32). The transmission of this document or the revelation of its contents in any manner to any unauthorized person is prohibited.

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9-22-11-1

ATI- 18213

Broughton, G  
Byfield, A

DIVISION: Ordnance and Armament (22) 57  
SECTION: Chemicals and Incendiaries (11) 3  
CROSS REFERENCES: Thickness, Incendiary - Production (93930) 3

ORIG. AGENCY NUMBER  
O.S.R.D. - 2036

REVISION

AUTHOR(S)

AMER. TITLE: The manufacture, properties and testing of Napalm soaps

FORG'N. TITLE:

ORIGINATING AGENCY: O.S.R.D., N.D.R.C., Div. 11, Washington, D. C.

TRANSLATION:

| COUNTRY | LANGUAGE | FORG'N.CLASS | U. S.CLASS. | DATE   | PAGES | ILLUS. | FEATURES              |
|---------|----------|--------------|-------------|--------|-------|--------|-----------------------|
| U.S.    | Eng.     |              | Restr.      | Nov'43 | 119   | 53     | tables, diags, graphs |

**ABSTRACT**

Survey is submitted of the processes developed by various research agencies for the manufacture of Napalm, a modified aluminum naphthenate soap for thickening incendiary bomb gasoline. The process, selected for use on the basis of this review, is described in detail and shown to be adequate for the purpose for which it is intended.

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57 R-22-11-1  
3 OVER 3

ATI- 18214

Broughton, G  
Byfield, A

DIVISION: Ordnance and Armament (22)  
SECTION: Chemicals and Incendiaries (11)  
CROSS REFERENCES: Thickeners, Incendiary - Production  
(93930); Napalm (93930)

ORIG. AGENCY NUMBER  
O.S.R.D.-2036

REVISION

AUTHOR(S)

AMER. TITLE: The manufacture, properties and testing of Napalm soaps

FORG'N. TITLE:

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TRANSLATION:

| COUNTRY | LANGUAGE | FORG'N.CLASS | U. S.CLASS. | DATE   | PAGES | ILLUS. | FEATURES                      |
|---------|----------|--------------|-------------|--------|-------|--------|-------------------------------|
| U.S.    | Eng.     |              | Restr.      | Nov'43 | 99    | 46     | tables, diagrs, graphs, drwgs |

**ABSTRACT**

Napalm, a mixture of aluminum hydroxy soaps of oleic, naphthenic and coconut acid used for thickening incendiary bomb gasoline, is manufactured by a three step process. In the first step a mixture of the acids is reacted with aluminum sulfate solution in the presence of aqueous caustic soda. In the second step the precipitate of soaps, formed in the first step, is dewatered and dried, and in the third step the dried product is packaged in special containers. Details are given of control methods, of the various properties of Napalm, of the method of dispersing Napalm in gasoline, and of testing the finished gel.

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57 R-22-11-1

ATI- 18215

Chemical Warfare  
Center

DIVISION: Ordnance and Armament (22)  
SECTION: Chemicals and Incendiaries (11)  
CROSS REFERENCES: Oils, Incendiary - Testing (68165)

ORIG. AGENCY NUMBER

O.S.R.D. - 2036

REVISION

AUTHOR(S)

AMER. TITLE: Determination of the consistency of incendiary oils

FORG'N. TITLE:

ORIGINATING AGENCY: O.S.R.D., N.D.R.C., Div. 11, Washington, D. C.

TRANSLATION:

| COUNTRY | LANGUAGE | FORG'N.CLASS | U. S.CLASS. | DATE    | PAGES | ILLUS. | FEATURES |
|---------|----------|--------------|-------------|---------|-------|--------|----------|
| U.S.    | Eng.     |              | Restr.      | Jul '43 | 8     |        |          |

**ABSTRACT**

Directives are given on the use of the Gardner mobilometer method of determining the consistency of incendiary oils of the Napalm type, and of the parallel plate method for determining the consistency of incendiary oils containing isobutylmethacrylate polymers and sodium salts of fatty acids, such as incendiary oil IM, type III, CWS Spec.# 196-131-1145. Instructions include description of the two instruments and directions for their assembly and maintenance.

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Chemical Warfare  
Center

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57 R-3-11-1  
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DIVISION: Ordnance and Armament (22)  
SECTION: Chemicals and Incendiaries (11)  
CROSS REFERENCES: Thickeners, Incendiary - Specifica-  
tions (93935); Napalm (93935)

**ATI-** 18216

ORIG. AGENCY NUMBER

196-131-107A

REVISION

AUTHOR(S)

AMER. TITLE: Napalm thickener Applicable specifications

FORG'N. TITLE:

ORIGINATING AGENCY: O.S.R.D., N.D.R.C., Div. 11, Washington, D. C.

TRANSLATION:

| COUNTRY | LANGUAGE | FORG'N.CLASS | U. S.CLASS. | DATE   | PAGES | ILLUS. | FEATURES |
|---------|----------|--------------|-------------|--------|-------|--------|----------|
| U.S.    | Eng.     |              | Restr.      | Nov'43 | 12    |        |          |

**ABSTRACT**

Copy is submitted of CWS Specification No. 196-131-107A, dated 25 October 1943, to supersede No. 196-131-107, dated 19 November 1942. Specification includes Governmental and non-Governmental publications and pertains to Napalm, a thickening agent for use in the manufacture of flame thrower fuel and incendiary oil NP.

UNCLASSIFIED PER AUTHORITY

OSRD LIST 1, dated 1 Nov. - 14 Dec 45

*Yipik Lazar*, USCO

18 SEP 1950