UNCLASSIFIED

AD NUMBER	
ADA801489	
CLASSIFICATION CHANGES	
TO:	**UNCLASSIFIED**
FROM:	**CONFIDENTIAL**
LIMITATION CHANGES	
TO: **Approved for public release; distribution is unlimited.**	
FROM: **Distribution authorized to U.S. Gov't. agencies and their contractors; Administrative/Operational Use; 02 OCT 1944. Other requests shall be referred to Office of Scientific Research and Development, Washington, DC 20301.**	
AUTHORITY	
OSRD list no. 3 dtd 2-11 Jan 1946; OTS index dtd Jun 1947	

THIS PAGE IS UNCLASSIFIED


TECHNICAL
AMC
INTELLIGENCE

The U.S. GOVERNMENT

IS ABSOLVED

FROM ANY LITIGATION WHICH MAY ENSUE FROM THE CONTRACTORS INFRINGING ON THE FOREIGN PATENT RIGHTS WHICH MAY BE INVOLVED.

WRIGHT FIELD, DAYTON, OHIO

REEL – C

1344

A.T.I.

31510

CONFIDENTIAL

Division 11
NATIONAL DEFENSE RESEARCH COMMITTEE
of the
OFFICE OF SCIENTIFIC RESEARCH AND DEVELOPMENT

SYNTHETIC POLYMERS AS GASOLINE THICKENING AGENTS

by
E. C. Kirkpatrick
Chemist, E. I. duPont de Nemours and Company
Ammonia Department

Report OSRD No. 4202
Copy No. 39
Date: October 2, 1944

Copy Nos.	
1-28	Dr. Irvin Stewart
29	Office of the Chief of Chemical Warfare Service Attention: Technical Division, Liaison Branch
30-33	Chemical Warfare Center, Edgewood Arsenal Attention: Brig. Gen. W. C. Kabrich
34	Chief, Bureau of Ordnance Attention: Captain J. H. Sides
35	Air Corps Liaison Officer with NDRC - Colonel J. F. Phillips
36	Commanding Officer, Chemical Warfare Service Development Laboratory Massachusetts Institute of Technology Attention: Lt. Col. Willard Slagle
37	Division 11 Files
38-41	Dr. C. S. Keevil
42	Mr. E. P. Stevenson

Total Number of Copies - 45

This document contains information affecting the national defense of the United States within the meaning of the Espionage Act, U.S.C. 50; 31 and 32. Its transmission or the revelation of its contents in any manner to an unauthorized person is prohibited by law.

CONFIDENTIAL

CONFIDENTIAL

OSRD No. 4202

Division 11
NATIONAL DEFENSE RESEARCH COMMITTEE
of the
OFFICE OF SCIENTIFIC RESEARCH AND DEVELOPMENT

Section 11.3

SYNTHETIC POLYMERS AS GASOLINE THICKENING AGENTS

Service Directive: CWS-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 is a final summarizing report under Contract OEMsr-744 with E. I. duPont de Nemours and Company, Ammonia Department. The development of methacrylate polymer thickening formulas for use in the M-69, M-47, and E-9 incendiary bombs is reviewed. The formulas reviewed include IM-1, IM-2, and IM-3 which were accepted and utilized in the manufacture of incendiary bombs. Various possible substitutes for methacrylate are also reviewed."

This is a final report under Contract 11-364, OEMsr-744 with E. I. duPont de Nemours and Company.

CONFIDENTIAL

Ammonia Department
E. I. duPont de Nemours & Co., Inc.

****

Chemical Division
DuPont Experimental Station
Wilmington, Delaware

****

Synthetic Polymers as Gasoline Thickening Agents

Table of Contents

Table of Contents
(Cont'd.)

Table of Contents
(Cont'd.)

Table of Contents
(Cont'd.)

CLASSIFIED - CONFIDENTIAL

Ammonia Department
E. I. duPont de Nemours & Co., Inc.

****

Chemical Division
DuPont Experimental Station
Wilmington, Delaware

****

Synthetic Polymers as Gasoline Thickening Agents

ABSTRACT

A laboratory formulation study was undertaken to develop new gasoline incendiary mixtures containing polymeric viscolizing agents. Major emphasis was placed on combinations of isobutyl methacrylate polymers with diverse soap forming ingredients. The optimum type and amount of each of the basic gel ingredients was determined. Based on physical studies and surveillance tests, 80 gel formulas were chosen for evaluation in performance tests in one of five munitions. Three methacrylate gel formulas have been accepted and manufactured. Problems of commercial gel manufacture were investigated in a continuous pilot plant.

CLASSIFIED - CONFIDENTIAL

Ammonia Department
E. I. duPont de Nemours & Co., Inc.

****

Chemical Division
DuPont Experimental Station
Wilmington, Delaware

****

June 7, 1944

Synthetic Polymers as Gasoline Thickening Agents

I. Introduction

A study of the thickening of gasoline by synthetic polymers has been in progress in this laboratory since December 30, 1941. The major effort during the first six months was to assist the Technical Division of the Chemical Warfare Service* in the development of incendiary fillings for the M-47 bomb. From August 1, 1942 to February 29, 1944, the work was continued under an Office of Scientific Research and Development, National Defense Research Committee**, Contract, OEMsr-744, on incendiary fillings for M-47, M-69 and E-9 bombs and frangible grenades.

The first gel samples were submitted for inspection to CWS February 5, 1942 and were tested at Edgewood Arsenal as fillings for the M-47 incendiary bomb on March 3, 1942. The major effort during the next few months was to "high-spot" formulation, ingredient requirements, and methods of manufacture of gels containing isobutyl methacrylate polymer for use in the M-47 bomb. The operation of a continuous gel pilot plant was demonstrated to CWS on April 10, 1942. During the cooperative studies with CWS, an investigation of the manufacture of NR and AE grades isobutyl methacrylate polymer and the preparation of interpolymers of isobutyl methacrylate with small amounts of methacrylic acid were completed.

Prior to the start of the OSRD contract, several polymer samples were forwarded to groups working both on incendiaries and on flame throwers for the NDRC. At a request received April 30, 1942 from the NDRC, the F-241 formula gel (see Table 1, pg. 7) was prepared for inclusion in a fuel review for the M-69 (then termed M-56) bomb which was held May 3, 1942 at the Standard Oil Development Co. Laboratories at

---

* Abbreviated CWS throughout this report.
** Abbreviated OSRD, NDRC throughout this report.

Bayway, New Jersey. Following these tests, further formulation, firing, and surveillance tests on the F-241 gel were started in cooperation with the Standard Oil Development Company, and were continued under the OSRD contract after August 1, 1942. The program outlined under the OSRD contract included: (a) the formulation of methacrylate gels to develop superior gasoline incendiary mixtures and to reduce the need for critical ingredients, (b) the study of other polymers as gasoline viscolizing agents, (c) the development of specifications for gels and the individual components of the gels, (d) the development of physical methods of testing gels to control manufacture and to assist laboratory development of new formulas, and (e) the servicing of munitions with incendiary gels as requested either by NDRC or by CWS. During the course of this work requests were received for materials to be loaded into five munitions (M-69, M-47, and E-9 incendiary bombs, frangible grenades, and the portable flame thrower). Four methacrylate gel formulas (IM-1 to 4) have been adopted and manufactured by the CWS while other formulas are now under advisement.

This report is a complete review of the work done under OSRD Contract OEMsr-744 combined with such data from previous work as appear pertinent. Reports from this Department on specific phases of the work have been circulated to both NDRC and CWS and by reference constitute a part of this final report. These include:

1. Isobutyl Methacrylate-Methacrylic Acid Interpolymers as Gasoline Thickening Agents. Formal Report from E. I. duPont de Nemours &Co. to NDRC Section 11.3 by E. C. Kirkpatrick. April 26, 1944.

2. Chemical Ignition of Flame Throwers - A Critical Review of Proposed Techniques. Formal Report by E. C. Kirkpatrick to NDRC Section 11.3. February 22, 1944.

3. Proposed Surveillance Conditions based on Weather Information from the Air Force and the U. S. Weather Bureau. Letter H. R. Dittmar to Dr. R. H. Ewell, December 14, 1942.

4. Laboratory Examination of Experimental Incendiary Gels Produced at the Westerville, Ohio Plant of the Kilgore Manufacturing Company. Letter E. C. Kirkpatrick to H. R. Dittmar, January 18, 1943.

5. Report on Visits to the Kilgore Manufacturing Company. E. C. Kirkpatrick to H. R. Dittmar, December 22, 1943.
6. Inspection of IM-2 Gels Produced by the Kilgore Manufacturing Company. Letter E. C. Kirkpatrick to Brig. Gen. W. C. Kabrich, August 9, 1943.
7. Inspection of IM-3 Gels Produced by the Kilgore Manufacturing Company. Letters H. Peterson to Brig. Gen. W. C. Kabrich, December 1, 1943, and E. C. Kirkpatrick to Brig. Gen. W. C. Kabrich, August 9, 1943.
8. Properties and Examination of IM-3 Incendiary Fuel. Covering letter D. E. Strain to Brig. Gen. W. C. Kabrich, May 17, 1943.

II. Summary

A. General

In an extensive laboratory search for stable thickened gasoline mixtures of high strength and good incendiary characteristics approximately 2,500 different gels were prepared. Isobutyl methacrylate·polymers and other synthetic resins were combined with soap forming acids, strong bases, fillers, and gasoline with or without other fuels. Based on laboratory surveillance tests and physical measurements, 80 gels were chosen for field evaluation in one of 5 munitions. An additional 700 gels were then prepared to standardize the most effective compositions. Problems involved in plant scale manufacture of isobutyl methacrylate gels were investigated. A series of gel formulas of graded toughness, representing the compositions prepared during this study most suitable as incendiary fillings, are listed in Section F.

B. Gel Formulation with Methacrylate Polymers

The study of gasoline gels thickened by polymers of isobutyl methacrylate is reviewed by summarizing the effect on gel properties of varying in turn the nature and concentration of each of the basic gel components.

Gel preparation generally involved formation of a low viscosity gasoline solution containing isobutyl methacrylate polymer and soap forming acids which were gelled or thickened by the addition of a small amount of aqueous alkali. In general the polymer determines strength characteristics while the soap ingredients contribute body to these mixtures.

1. The range of strengths required of gels for the various incendiary munitions was covered by using NR or AE grade isobutyl methacrylate polymer for the weaker gels and one of a series of interpolymers of isobutyl methacrylate and methacrylic acid in the strongest gels. Polymer content of soap fortified gels was varied between 1 and 10%. The minimum polymer contents consistent with stability and the various desired gel strengths were determined.

2. A large number of soap forming acids were assessed as gel bodying agents. Formulation of the six most effective acids was intensively studied in gels containing various combinations of 2 or 3 acids. Of these six acids, stearic and oleic acids impart stiffness, body, and high temperature stability to all types of methacrylate gels. Naphthenic acid and dimerized soybean oil acids act as gel plasticizers, while wood rosin and Turkey red oil normally function as plasticizing agents but occasionally fulfill both of the above functions. The most effective acid combinations are stearic acid-naphthenic acid; stearic acid-naphthenic acid-wood rosin; stearic acid-dimerized soybean oil acid; and stearic or naphthenic acids alone.

3. To study the effect of the gelation agent strong and weak bases were tested at various ratios of acids to base to water. Only strong bases caused effective gelation. The use of aqueous sodium hydroxide, ground lime, and calcium hydroxide was studied in detail. Unsuccessful attempts were made to prepare stable gels with ammonia or amines.

4. Stiffness and a reduction in resilience were imparted to strong, fluid gels containing isobutyl methacrylate-methacrylic acid interpolymers by the addition of inert solid materials. Ground alpha-cellulose was the most effective filler tested.

5. Other NDRC research groups studied the gasoline requirements of methacrylate gels and concluded that an aniline point below 105°F. was required to obtain gel stability.

6. The addition of toluene improved the stability of soap-free isobutyl methacrylate-methacrylic acid interpolymer gels. Other solvents tested were less suitable.

C. Gel Formulation with Methacrylate Substitutes

In a search for isobutyl methacrylate substitutes a formulation study of other commercial resins was undertaken. A search for gasoline soluble polymers other than methacrylates revealed only the polyvinyl ethers, Vistanex (polyisobutylene) and the rubber substitutes derived from vegetable oils. Satisfactory strength in gels containing the latter two materials were obtained only when the polymer content exceeded 10%. The polyvinyl ether procured from the General Aniline & Film Corporation was tested as a direct substitute for polyisobutyl methacrylate and as a constituent of soap-free gels. The properties of polyvinyl ether gels are comparable to those prepared from methacrylate polymers. Evaluation of these mixtures as flame thrower fuels has been undertaken by other NDRC groups. Gel preparation was attempted with other commercial resins, especially ethyl cellulose, by adding an auxiliary solvent to the gasoline. It was concluded that without further modification such polymers do not impart sufficient strength to gasoline-soap gels and that the use of a water miscible auxiliary solvent results in poor high temperature gel stability.

Modification of existing commercial resins and the synthesis of new polymeric gasoline thickening agents was "high-spotted". While several gasoline soluble cellulose and vinyl resins were prepared, degradation occurred during the introduction of functional groups so that only low molecular weight materials were obtained.

Attempts to prepare polymer-free gels involved the use of asphalts, gums, factices, carbon black, and miscellaneous organic solids. When gels were obtained they were weak and had a high solids content.

D. Physical Measurements

The physical properties of various gels were compared with the results of field evaluations and the sensitivity of various tests to minor changes in composition of a given gel formula were determined. To obtain significant characterization of methacrylate gels it was necessary to modify existing methods and to develop new techniques. New physical measurements developed under this contract include the so-called Impact Strength, Parallel Plate, and Burning Rate tests. The Impact Strength, a measurement of consistency at a high shearing force, was useful in gel research to predict behavior of diverse gel formulas in static firing tests. The Parallel Plate test, a measurement of body under a low shearing force, was adapted to plant control on specific gel formulas where it showed excellent sensitivity to quality of ingredients and method of compounding. The burning rate test gave a comparative measure of the incendiary characteristics of diverse gels.

The stability to exposure to both high and low temperatures of gels prepared during the formulation study was determined and has been correlated with gel composition.

E. Pilot Plant Tests

A continuous method of preparing methacrylate gels which involved mixing the aqueous base with a gasoline stock solution (containing resin and soap-forming acids) in a centrifugal pump was developed and studied on a pilot plant scale. Operation of this unit was demonstrated to the CWS and to representatives of three industrial concerns under contract to load small incendiaries. The problems of continuous manufacture including method and rate of mixing, production capacity, the effect of temperature and the technique of pumping alpha-cellulose suspensions in gasoline were investigated both in our pilot plant and in cooperation with the CWS in their commercial loading plant at Westerville, Ohio. Performance tests were made on pilot plant product to confirm earlier conclusions drawn from gels prepared batchwise in the laboratory. Based on these results assistance was given to the CWS in drawing specifications on the ingredients and on the composite gel for each of three methacrylate gel formulas. Unsuccessful attempts were made to prepare lime gels in a continuous manner.

F. Applications in Munitions

The results of field tests in cooperation with other groups on methacrylate gels as fillings in four incendiary munitions are reviewed as they pertain to gel formulation studies. Based on such tests and on surveillance studies a preferred group of methacrylate gels of graded toughness has been listed in Table 1. For comparative purposes a few competitive gel formulas are included. These mixtures are discussed below in relation to the munition in which they were tested. The development of physical methods of evaluating gels and calibration of the test values against the results of field trials of incendiary munitions has yielded for each munition a specific range of physical properties which appears optimum.

Table. 1

CLASSIFIED - CONFIDENTIAL

CLASSIFIED - CONFIDENTIAL

Gasoline Incendrury Gels.

Gel Designation*	A-3942	A-3957	A-3989	8% Smoked Sheet Rubber	F-1440	IM-1***	IM-4 (F-1421)	F-107	Napalm 13.5%	Napalm 8%	IM-2 (F-241)	F-624	F-962	IM-3 (F-614)	F-1416	F-1951
Composition (weight percent)																
Methacrylate Polymer																
Grade NR											5					
AE					3	5	3	5						2	3	
0.1 Interpolymer												2	2			
0.3 "	6	4	4													
Napalm									13.5	8						
Smoked Sheet Rubber				8												
RE-302 Resin**																5
Stearic Acid					4	3	4	2.5			2.5	2	2	3	1	
Naphthenic Acid		1			1			2.5			2.5	2		3	3	
Dimerized Soybean Oil Acid													2			
Wood Rosin												2				
Alpha Cellulose												0.5				
40% NaOH soln.	1	1.5	1					3			3	3.7	2.1	4.5		
CaO (to pass 40 mesh)					3.5	2	4								3.1	
H_2O					2.2	1.25	2.5								2.3	
Toluene	20	20	20													
Gasoline.	73	73.5	75	92	86.3	88.75	86.5	87	86.5	92	87	87.8	91.9	87.5	82.6	95
Physical Properties																
Paralel Plate Consistency cm.	9	10.	9.5	9	8	8	8	8.	6.-	8.0	11.0	11.0	8.0	10	13	
Impact Strength																
B Scale units										5				5	2	
C " "			>15		15.	13	12.	12	8		14	10	6			
D " "	14	11.5	10.													
Munition in which gel has been used or for use in which gel is proposed.	Texas	E-9 40lb Bomb		M-47 "100"lb Incendiary bomb →						M-69 10lb Incendiary Bomb →					Frangible Grenades	Flame Throwers
Incendiary Characteristics																
Burning Time																
Method A min.	3.2		3.4			7.0	5.6						2.9	3.6	4.2	
Method B min.	3.9	2.7	2.8			6.4	6.5		5.8					5.5		

Notes: Tests Procedures are described in Section VI of this report.

* Gel formulas developed in this laboratory are designated by A or F numbers
The letters I.M. signify that the methacrylate gel formula has been accepted and standardized for production by the Chemical Warfare Service

** RE-302 Resin : A synthetic resin manufactured by the General Aniline and Film Corp.

*** IM-1 This gel formula was developed by the ~~Research Section~~ Technical Command Research Section of the Technical Division of the Chemical Warfare Service.

CLASSIFIED - CONFIDENTIAL

1. M-69 Incendiary Bomb

It is believed that a filling for the M-69 bomb should have an impact strength (test procedure described in Section IV-D) in the range of 4 to 9 C scale units. Somewhat weaker gels may prove satisfactory if they are fluid and adhesive.

Three gel formulas have been used during commercial production of M-69 incendiaries; two additional formulas are proposed for future consideration. The IM-2 formula was the original large scale M-69 filling. This was superseded by the IM-3 mixture which had equal if not superior firing characteristics, and which required only 40% as much isobutyl methacrylate polymer. Even with this polymer saving, the quantity allocated was insufficient for use in all M-69 bombs. The remainder of the M-69 manufacturing program used a gasoline gel containing 8% Napalm. In a joint CWS-NDRC field review of candidate M-69 fillings held November 19 to 21, 1942, it was found that the F-624 gel (Table 1) had incendiary characteristics superior to those of either the IM-2 or IM-3 gels. Acceptance of this formula was held in abeyance due to questions concerning stability and method of manufacture. These uncertainties have been successfully eliminated and its reconsideration is suggested. The F-962 formula which is also proposed for evaluation as an M-69 filling has a higher fuel content and a faster burning rate than any of the above mixtures. While its physical properties are in the range believed optimum for M-69 gels, its field performance has not as yet been determined.

2. E-9 Incendiary Bomb

The exact strength requirements of fillings for this bomb have not been clearly defined but are obviously high. For fluid resilient mixtures the optimum impact strength appears to be in the neighborhood of 12 D scale units.

Based on static firing tests by the Texas Company, gels A-3487 and A-3489, which are interpolymer gels of low soap content, have been chosen as fillings for this experimental bomb for use in field evaluations and acceptance tests. The latter has a slightly higher fuel content and a simpler composition but requires gasoline of low aniline point. The A-3487 gel is preferred by this group due to its greater ease of manufacture and its improved flow during combustion. The strength of the F-1948 formula is probably on the high side of that required. Its use is suggested only if the other formulas prove inadequate

3. M-47 Incendiary Bomb

The optimum incendiary fillings for the M-47 bomb have impact strengths in the range of approximately 11 to 16 B scale units. These limits are necessarily vague because of the possible variations in the type of burster well and case that come under this single classification.

Gels which have been employed in M-47 bombs in the field include 8% smoked sheet rubber, 13.5% Napalm, and the IM-1 and IM-4 formulas. The first mixture contains a critical material that could not be procured, while the high soap content of the second leads to friability. The two accepted IM formulas differ largely in polymer content. The F-1440 formula is also listed in Table 1 as an M-47 filling since it resembles the IM-4 gel but has improved adhesion to wood. The three methacrylate gels all contain lime as the gelling agent and thus have slow burning rates and do not adhere strongly to wood. Three methacrylate gels containing aqueous caustic rather than lime have proven acceptable in M-47 firing tests. The IM-1 formula was chosen over the first of these, the F-107 formula, due to manufacturing considerations. The other two, F-519 (4% of 0.3 interpolymer, 1.5% stearic acid, 1.5% naphthenic acid, 2% of 40% NaOH) and F-330 (5% of 0.1 interpolymer, 1% of 40% NaOH), in preliminary tests appeared superior to the lime gels. The later successful stabilization of soap-free interpolymer gels for use in the F-9 bomb by the addition of toluene suggests a re-evaluation of this type of filling in the M-47.

4. Frangible Grenades

Methacrylate gels with the proper consistency for use in frangible grenades had modified Stormer viscosities of 40 to 200 R.P.M./800 g. (see Section IV for test procedures) and Gardner Mobilometer values of 20 to 200 grams/1.5 min./10 cm. While the F-1416 gel meets these requirements, the use of existing methacrylate gels in frangible grenades is not recommended since other mixtures, notably gasoline solutions containing 2-5% of Napalm or Vistanex (polyisobutylene), have superior low temperature properties.

III. Discussion of Requirements on Incendiary Fillings

A discussion of the qualities which produce a superior incendiary filling involves the principles of incendiary attack. No incendiary bomb contains sufficient fuel to cause appreciable destruction per se. The purpose of the incendiary filling is to ignite combustible material present at the target and to initiate a destructive fire. An incendiary bomb should therefore deliver its active contents near combustible material in a concentration sufficient to ignite this material but not greatly in excess of this amount. Since fire raising is based on probability factors, the greater the number of units, each of effective size, into which the bombing load can be split the greater is the destruction obtained. It is therefore obvious that certain requirements on incendiary fillings will vary in different munitions These include the incendiary characteristics and the distribution in performance. Fundamental requirements which are independent of the munition include stability, ease of manufacture and availability. A general discussion of these requirements, as applied to gasoline gels, is followed by a description of the munitions in which these gels were tested.

A. Distribution

The complete analysis of the factors affecting gel distribution include not only the manner in which the gel is ejected from the munition but also its reaction on reaching the target and its properties during combustion. The most limiting requirement on all incendiary gels is that they have sufficient strength to resist disruption under the force designed to distribute the gel into favorable locations. This involves a high shearing force. It has been demonstrated in many tests that at least 2 pounds of gasoline gel must support a fire in a "normal" target if that fire is to spread rapidly. For small bombs with a gross weight of 10 pounds or less it is therefore essential that the major part of the fuel remain in contact. If all the fuel in large bombs, however, were to stay as a unit, then part of the gel would be wasted. In such munitions the gel should break into several pieces, each piece being of a size capable of initiating an individual fire. There is, however, an important psychological effect obtained from a large number of small fires It is therefore agreed that it is desirable that possibly 5 to 10% of the gel should scatter in small particles.

In an ejection type bomb, assuming gel strength to resist scatter on ejection, the gel is thrown from the bomb and its flight will be interrupted by an obstacle that may be combustible. The behavior of the gel on reaching such an obstacle is the second requirement that is readily studied. Elastic materials tend to bounce away from the obstacle to fall in a location that has little chance of being favorable. Weak gels will shatter on impact so that the incendiary effect is dissipated. It therefore appears desirable that on impact the gel spread enough to lose its momentum but remain as a single fire unit. Early wood combustion tests indicated that the optimum thickness of such a gel smear was 1/4 inch. For optimum action the gel should adhere to the obstacle. Once combustion of the target has been initiated the gel should then flow at the same rate as the fire travels to assist the spread of the fire. This means that the burning gel should have sufficient viscosity to adhere and stay in the fire area. A gel which neither scatters, adheres nor rebounds will spread along the base of the obstacle. This location is second in effectiveness only to complete adhesion. The extent to which the gel spreads on impact and its tendency to flow appear to be correlated with its viscosity under a low shearing force.

It is anticipated that when these munitions function in the field the gel temperature may be between room temperature and as low as -40°F. Since bombs are transported to the target in unheated bomb bays at high altitudes, it is believed that even in warm climates the bomb filling will be cooled to 70°F. or below before use. In high altitude, long range flights the bomb may cool to -50°F. resulting in a gel temperature of -40°F. For these reasons performance tests on gels were made at 70°F. and -40°F. while tests at higher temperatures appear irrelevant. This implies that the consistency of the gel, which in part determines the distributional properties described above, should be as nearly as possible independent of temperature in the range 70°F. to -40°F.

B. Incendiary Characteristics

The minimum conditions which will cause combustion of wood are a combination of temperature and time of exposure. Thus a good incendiary gel not only makes available a large quantity of thermal energy, but also is able to liberate this heat at a specified rate. The optimum rate of heat liberation is closely allied with both the particular munition and the type of target under consideration. A decision on the exact burning rate desired in a gel must be based on incendiary field tests which include an analysis of fire raising powers. The best estimates available indicate that the duration of combustion of the distributed gel should be in the range of 3 to 20 minutes with the shorter times for the most combustible targets. The shorter the time required for the fire to get beyond control, the greater the probability that enemy fire fighting will be ineffective. Comparison of the total heat available in different gels should be made on a volume not a weight basis since the bomb load of a plane in general is limited by the number and size of the bomb stations.

Some inferior gels exhibit partial instantaneous combustion when the bomb is fired. This flash fire is ineffective and is caused by the presence of free gasoline or by excessive breakup. Gel breakdown yielding free gasoline may occur either in gels unstable to temperature changes or in gels whose structure is broken by the impact forces.

While the burning rate of a gel is little affected by its initial temperature, the ignition of the gel is more difficult as this temperature decreases. The attainment and extent of ignition is, however, largely determined by the design of the munition.

C. Stability

Inferior gels exhibit phase separation during storage. The presence of an unthickened gasoline phase causes flash burn, alters the physical properties of the gel phase to change gel distribution on ejection, and in tail ejection bombs makes gel ignition difficult. The presence of two gel phases may decrease flight stability of a munition. It is therefore a requirement of an incendiary filling that it exhibit no breakdown and that it perform satisfactorily, after it has been held at such temperatures as it might experience in shipment to and in use in the field. These conditions are discussed in the section describing surveillance tests.

D. Ease of Manufacture

While the factors affecting performance are of major importance, a consideration of two equally effective fillings would also include the problems of procurement. An ideal gel would be made from available materials by a simple technique which would involve minimum safety hazards. The normal variations in composition of the individual ingredients and of the combined mixture experienced in large scale manufacture should not affect gel properties materially.

E. Description of Munitions

Polymer thickened gasoline gels have been prepared for appraisal as fillings in four incendiary munitions. These differ radically in the forces employed to disperse the gel. Since an increase in gel strength tends to decrease the incendiary quality of a gel it appears desirable to use in a given munition a filling which has only slightly more than the minimum required gel strength. For this reason the optimum gel formula varies for each of the following munitions listed in order of increasing ejection forces: frangible grenade, M-69 bomb, M-47 bomb, E-9 bomb.

1. M-69 Bomb

The 10 pound M-69 incendiary bomb (CWS Specification 196-131-99A) was developed by the Standard Oil Development Company under NDRC Contract OEMsr-354. It is a tail ejection bomb principally designed for area bombing of domestic targets. It has a terminal velocity when dropped from altitudes greater than 10,000 feet of 225 feet per second. This indicates that the bomb will penetrate and remain inside flimsy to moderately protected structures. Since the construction of such targets usually includes readily combustible material, a suitable filling should therefore be rapid burning.

The hexagonal shaped bomb contains a heavy steel powder chamber separated from a sheet metal enclosed fuel chamber by a diaphragm. The powder chamber contains the fuse, the delay element, and the ejection and ignition charge. The latter is a mixture of 7 2 grams of A-4 black powder and 4.8 grams of coated magnesium*. The fuel chamber is closed at the base by a cap which is seamed to withstand a pressure of 100 pounds gage but to disengage when the internal pressure reaches 150 pounds gage. The gel is supported in an open weave cheesecloth bag. The powder charge breaks the diaphragm when the pressure in the powder chamber reaches approximately 500 pounds per square inch This pressure wave then forces off the tail cup and ejects the gel in one piece mortar-wise. In flight from the bomb the gel is believed to reach an approximate speed of 100 feet/second. The cheesecloth sock is of such a strength that it prevents gel disintegration in ejection flight but breaks when it hits an obstacle, allowing the gel to spread. The bomb will hold 2.8 pounds of a gel having a density of 0.75.

2. E-9 Bomb

The 40 pound E-9 incendiary bomb now under development by the Texas Company under NDRC Contract OEMsr-898 is a tail ejection type bomb designed for precision bombing of industrial targets. It reaches its terminal velocity of about

---

*An alternate mixture of 10 g. of A-1 powder and 4 g. of coated magnesium has also been used.

900 feet per second when dropped from 30,000 feet. The present design of the bomb calls for the inclusion of 10 pounds of incendiary gasoline gel. Due to the relatively noninflammable nature of its intended target it is believed that this filling should act as a single fire unit on ejection and that it should burn over a slightly longer period than is optimum for the gels in the M-69 bombs.

The nose of the bomb contains an impact fuse, an anti-personnel element and the ejection charge. The ejection charge, 16 grams of black powder, is contained in a frangible dome which is surrounded by a second dome containing 1 pound of white phosphorous. The remainder of the body of the bomb and the cone supporting the tail are filled with gel. The weakest spot in this container is the joint connecting the body with the tail cone. The bomb functions when the explosion breaks both domes, breaks off the tail cone, and finally blows out the gel and the phosphorous. We believe this bomb requires a tougher gel than any other munition studied.

3. M-47 Bomb

The M-47 incendiary bomb differs from the M-69 and the E-9 bomb in that it is an internal burster well type and it is usually not clustered but rather hung as a unit in a bomb bay. The case was originally designed by the CWS to be filled with chemical agents and has the normal stream-lined shape of high explosive bombs. A 1 inch diameter hole at the point of the nose ojive is used to fill the bomb with incendiary gel and later after loading supports the end of the internal burster well and the impact fuse. The internal burster well extends the complete length of the axis of the bomb. The M-12 burster consists of a mixture of black powder and magnesium in a burster tube. The M-13 burster includes an outer shell of white phosphorous and an inner coaxial core of tetrytol pellets separated from the phosphorous by a steel wall. When the bomb functions the explosion breaks the burster well, forces the ignition agent (Mg or P) into the gel chamber, ruptures the case, and throws the gel in all directions over a circular area. In the tests described in this report the thin walled M-47A2 case was generally used. The 39 pounds of gasoline gel in the M-47 bomb transmits the explosive wave that ruptures the case. Under the shock of this explosive force the gel should break into a number of pieces whose approximate size should range between 1 and 5 pounds. This requires a stronger gel than is optimum for use in the M-69 bomb.

4. Frangible Grenades

Frangible grenades as produced by the Chemical Warfare Service consist of a squat 1 pint glass bottle filled with gasoline gel. Various ignition mechanisms have been tested with an external ignitor (M-3) favored. The grenade is thrown at a target such as a tank and should break and function on impact. The tactical effects of the grenade are achieved when the burning fuel (a) ignites lubricating oil on the tank treads or engine, (b) penetrates through appertures into the tank compartments with anti-personnel effect, or (c) obscures observation from the tank. All of these uses require a fluid, very rapidburning gel. The only limitation on minimum gel strength is that on impact the gel should not flow completely off the obstacle. A suitable grenade filling will adhere to metal, will flow readily and will burn with gusto.

IV. Procedures for Preparation and Evaluation of Gels

Specialized techniques developed to prepare and test methacrylate gels are outlined briefly in this section. The relative merits and the applicability of these tests are discussed. Specific detailed procedures are given in Appendix A.

The preparation of the IM-2 and IM-3 gel formulas for laboratory testing is described in CWS Specifications 196-131-120 and 196-131-145, respectively. A report on the properties and analysis of IM-Type III gels was prepared April 26, 1943, and was circulated (letter D. E. Strain to Brig. Gen. W. C. Kabrich) May 17, 1943. The above are supplemented by the following more general information.

A. Batchwise Preparation of Methacrylate Gels

The basic method of preparing methacrylate gels involves adding an aqueous solution of a base such as sodium hydroxide to a stock solution consisting of polymer and soap forming acids dissolved in gasoline. The stock solution is prepared by dissolving first the acids then the polymer with strong agitation in the gasoline. To insure solubility of stearic acid, the gasoline temperature must exceed 12°C. When mixtures of acids were used it was found convenient to weigh the acids into a container heated on a steam bath or when rosin was present on an electric heater, and to add the molten mixture to the gasoline while stirring. Solution of polymer was most readily obtained by adding the entire amount required all at one time. Stirring was continued until solution was complete. Fillers were sometimes added to this stock solution. Gelation was obtained by pouring the aqueous basic solution rapidly into the stock solution while stirring with an electrically driven stirrer. Agitation was continued for 1 minute or until the mixture had gelled sufficiently to climb the shaft of the stirrer. The usual size of a laboratory batch was 400 g. prepared in a wide mouth 1 quart bottle. The gels were allowed to set in the closed container at least 24 hours before examination.

When lime was used as the gelling agent the powdered dry lime was dispersed in the stock solution and water was then added to effect gelation. These mixtures were stirred at least two minutes after the addition of water. Since the particle size of the lime affected the rate of gelation and the final properties of the gel, a standard mixture was obtained by crushing USP lime, screening, and compositing the fractions to give a mixture with the following screen analysis:

35-60	mesh	- 22%
60-80	"	- 22%
80-100	"	- 22%
100-120	"	- 22%
past 200	"	- 12%
		100%

This synthetic mixture has a screen analysis which is the average of several analyses on limes ground to pass 40 mesh.

When aluminum or lead soaps were used as bodying agents, they were added to the polymer solution either as a dry powder or as a gasoline solution.

B. Pilot Plant Continuous Preparation of Methacrylate Gels

For larger than laboratory scale preparation of methacrylate gels it seemed desirable to develop a continuous rather than a batchwise process. Gelation involves rapid intimate mixing of a gasoline stock solution containing polymer and soap forming acids with the aqueous caustic solution. In preliminary tests the mixing obtained by injecting the two streams into a centrifugal pump seemed more controllable than the mixing obtained by passing the two solutions under pressure simultaneously through an orifice. The small scale unit shown diagrammatically in Figure 1 was therefore assembled. The stock solution is prepared batchwise in holdup tank T-1 while the 40% caustic solution is stored in tank T-2. These solutions are drawn from the tanks at calibrated rates by metering gear pumps. The two metered streams join in a tee

FIGURE 1

Continuous Unit for the Preparation of Methacrylate Gels

Flowsheet

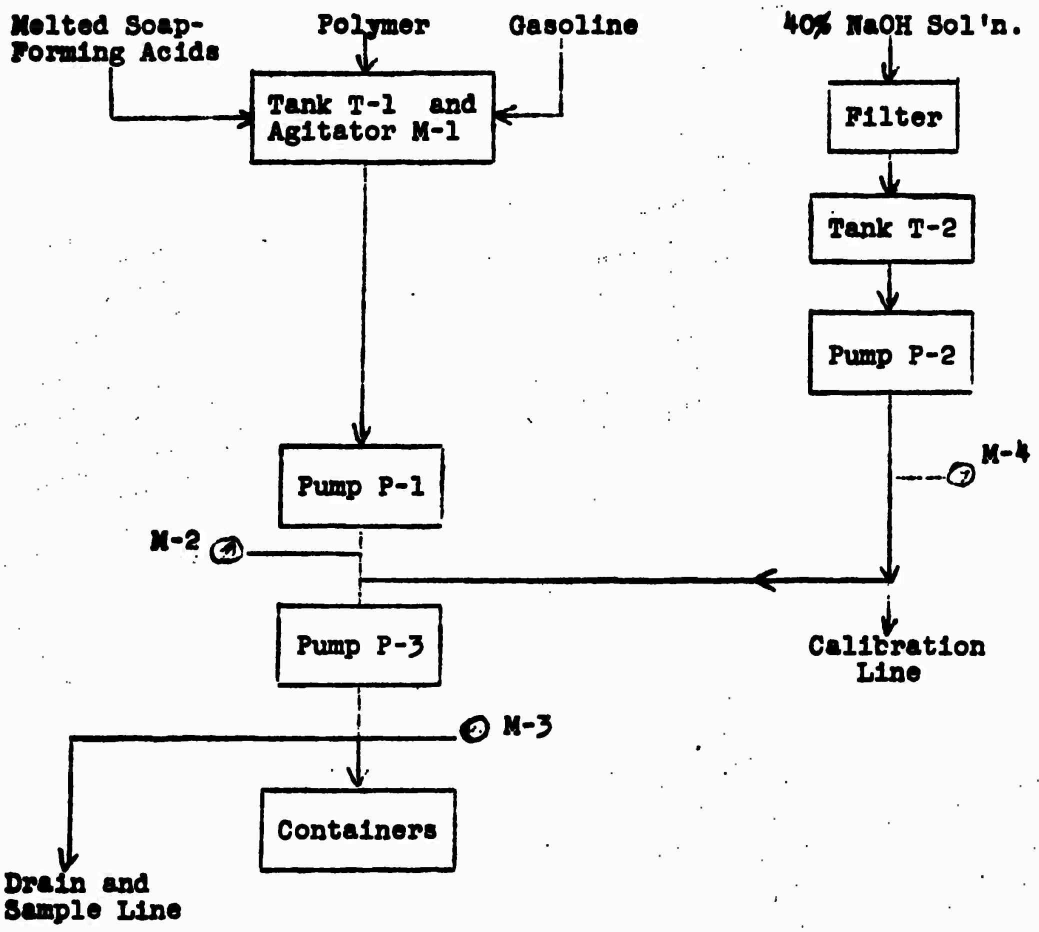


FIGURE 1
(Cont'd.)

Description of Equipment

1. Tanks

T-1 - 55 gallon open end steel drum.
T-2 - 5 gallon steel can.

2. Pumps

P-1 - Gould Figure 1676 steel gear pump driven by a 1/3 horsepower, single phase, 110 volt, 60 cycle motor.
P-2 - Zenith Products Company steel gear pump 1B-227 driven by a 1/4 horsepower, single phase, 110 volt, 60 cycle motor.
P-3 - Hills-McCanna Type A-219 steel centrifugal pump driven by a 1/4 horsepower, single phase, 110 volt, 60 cycle motor.

3. Variable Speed Drives

The drives on gear pumps P-1 and P-2 are made by Speed Master Company of Minneapolis, Minnesota.

The drive on P-4 was a Model A Serial 3067-UL All-Speed Drive made by Worthington Pump & Machinery Co., Harrison, N. J.

4. Piping and Valves

Standard steel or black iron pipe and all iron valves were used.

5. Miscellaneous

The caustic filter is a standard iron steam screen filter.

M-1 - 1/2 horsepower Lightning paddle type mixer.

M-2 to M-4 - pressure gauges range 0-200 lbs./sq.in.

or Y which immediately precedes the inlet to the centrifugal mixing pumps. Shut off and sampling valves are located so that the rate of flow of each stream can be calibrated separately. The gel produced in the mixing pump is forced through the discharge line and loaded directly into a bomb or container. The maximum production of the Ammonia Department unit, limited by pump capacity, was 10 lbs. of F-241 gel per minute. At this rate the back pressure developed in the discharge line which was 15 feet of standard 1" black iron pipe including two 90° bends was 5 p.s.i. gauge.

To obtain the flexibility required for experimental studies, all three pumps were operated by variable speed drives. In the unit producing IM-2 and IM-3 gel at the Kilgore Manufacturing Company it was found advantageous to operate both metering gear pumps from the same drive shaft and to predetermine the ratio of the two streams by the proper choice of gears coupling the pumps to the shaft.

All equipment was grounded and nonspark switches and explosion-proof motors were used throughout. Black iron but not galvanized pipe was completely resistant to the caustic and gel mixtures.

C. Qualitative Characterization of a Gel and Definition of Terms

The physical measurements to be described in the following sections are designed to quantitatively characterize specific properties of the gel. Qualitative observations may be made by manipulating the gel sample with a spatula.

To test for homogeneity the sample is cut in half and separated so as to expose a fresh surface. If there was insufficient agitation when the caustic was added to the stock solution, areas poor in caustic will be apparent by their lighter color. Such areas will also be much harder than the surrounding gel. This is confirmed by feel and yield under pressure. When the gel has been loaded into an M-69 case the areas of constant rigidity will tend to run lengthwise through the gel and the caustic content of the gel nearest the tail cup will exceed that of the gel near the diaphragm.

To determine friability and rate of heal the gel is cut with a spatula and a sample withdrawn. If the sample and the area from which it was withdrawn retain their shape and remain sharply outlined, the gel is hard and friable. In the most undesirable cases, shavings of gel can be produced by scraping the surface with the spatula. If a cut heals immediately or a smooth surface reforms as soon as a sample is withdrawn, the gel is soft and has insufficient body. The best gels are intermediate between these two extremes. The sharp edges of a cut or of a sample should become blurred almost immediately. The cut should be completely healed in approximately 5 minutes. The body of the gel can be evaluated by placing a sample on a plane surface. A soft gel will spread and lose its shape rapidly.

The strength and resilience of a gel are determined by forcing the flat surface of the spatula through the gel. The resistance offered to the passage of the spatula is dependent on the strength of the gel. A friable gel may offer considerable initial resistance and then break away. This is considered a rigid weak gel. When motion of the spatula is stopped, a resilient or elastic gel will tend to force the spatula back out of the "compressed" area.

When a sample of gel is withdrawn on the end of a spatula the length is qualitatively observed. The connection between the sample and the main body of the gel is maintained over a considerable distance with a long gel. If the gel is friable it will be very short. A long gel is either excessively fluid or is very elastic. This leads to inferior firing characteristics. The best F-614 gels are moderately short. The tendency to draw some of the remaining gel out with the sample is apparent.

A gel which is adhesive is difficult to remove from a spatula completely. A test of adhesion to wood may be conducted using a physician's tongue depressor as a standard wood strip. The strip in a vertical position is forced into the gel, the gel allowed to heal around it for a matter of 30 seconds and the strip is then drawn vertically out of the gel. With a good F-614 gel at least 50% of the submerged surface will be gel coated. A long elastic gel will be drawn part way out and will then snap back leaving the wood clean.

D. Determination of Physical Properties

Attempts to accurately characterize gels by measurement of specific physical properties were undertaken to correlate gel formulation work, to establish specification tests, and to obtain an insight into the factors influencing gel performance in incendiary munitions. Obviously no test will fulfill all these functions. In the light of the data discussed in Sections VI. and VII, the parallel plate test appears most suitable for specification work, that is, this test is sensitive to changes in composition and indicates when production of a specific formula is satisfactory. The performance of a gel of new or unknown formula, however, cannot be predicted from its parallel plate value. The impact strength of a gel on the other hand will fairly accurately determine the distribution of the gel on ejection from various munitions.

1. Consistency Measurements

The tendency of a material to flow under stress is inversely proportional to its consistency. True Newtonian liquids exhibit a constant ratio of shearing stress to rate of shear which is defined as the viscosity. Plastic materials such as gels, however, resist deformation by low shearing forces and tend to yield more easily to higher shearing forces. Since the overall performance of a gel is dependent on its behavior under both conditions, it is important to characterize a gel at both high and low shearing forces. A satisfactory method of accomplishing the former objective is the impact test while the latter measurement is most reproducibly observed by the parallel plate test.

a. The Parallel Plate Test

The parallel plate test is described in CWS Specification 196-131-108 and in CWS Directive 201. A more complete description of our technique may be found in Appendix A. Our procedure differs from CWS practice solely in the size of the plates (12 inches square in this laboratory, 6 inches square in CWS Directive 201). This test is a modification of a consistency test for putty (H. A. Gardner, Physical and Chemical Examination of Paints, Varnishes, Lacquers and Colors, 8th Edition, January 1, 1937, pages 599-601), and consists of measuring the diameter in centimeters to which 5 cc. of gel spreads in one minute between parallel plates of glass under a 2 kilogram load.

A study of various loads indicated that values in the most sensitive range were obtained with the recommended 2 kg. load. The majority of the gels will nearly reach an equilibrium spread in the 1 minute period of stress. If the load is applied for 5 min. to a typical F-241 gel, the parallel plate value would be increased from 10.4 to 11.6 cm. Straight lines are obtained by plotting the diameter against the logarithm of the time the load is applied. The original technique involved increasing the applied load by 2 kilogram increments every minute until a total load of 10 kilograms resulted. When the diameters observed by this method were plotted against the logarithm of the applied load, parallel straight lines were obtained for gels of quite diverse composition. The simpler technique involving a single measurement is therefore recommended. The temperature coefficient of the parallel plate values of F-241 gels was 0.15 cm./°C. To study the reproducibility of the test, 46 F-241 gels were prepared from various samples of the ingredients. At least three parallel plate measurements were made on each of these gels twenty-four hours after preparation. For a particular gel the maximum deviation from the mean of any single measurement was 0.7 cm., while for all of the gels three-quarters of the values were within 0.3 cm. of the average.

b. Impact Strength

To determine consistency at a high rate of shear a Schopper Dynstat plastics impact tester was modified to handle gels. A blade one-quarter inch wide was mounted on a pendulum with a total length of 15 inches. A trough to retain the gel sample was placed horizontally at the bottom of the swing of the pendulum. The horizontal length of the path of the blade through the gel was 26 cm. Various loads were placed on the pendulum arm. The pendulum was released from a horizontal position, allowed to swing down through the gel and the vertical height of the upward swing was determined. When the gel trough was empty the pendulum, being nearly frictionless, rose to the horizontal position. When the gel was present the energy consumed in forcing the blade through the gel caused the pendulum to swing through to a maximum height somewhat short of the horizontal or zero point. The decrease in the height of the maximum swing from the zero point is proportional to the work done and therefore to the strength of the gel. To cover all types of gels, it was necessary to use three different loadings on the pendulum which are referred to in increasing order of gel strength as B, C, and D scales. The B scale readings for the weakest gels ranged from 1 to 10 units. The units are empirical but in a very rough way correspond to kg. cm. The C scale covered a range of 1 to 20 units and in the lower half of this range B and C scale readings were nearly equivalent. A few tough gels had values exceeding the C scale. These were measured by placing a small additional weight on the impact arm and by raising the release point from 90° to 120° from the vertical. This increased angle of fall alters the zero point of the instrument. The D scale values are therefore not directly comparable with those of the B and C scale.

c. Modified Stormer Viscosimeter

The modified Stormer viscosimeter as described by Geddes and Dawson, J. Ind. Eng. Chem., 34, 163 (1942), consists of a sample cup containing a paddle which is driven by the gravitational pull of a falling weight. For a given load the speed with which the paddle rotates is inversely proportional to the viscosity of the material in the sample cup. A true determination requires that stirring be continued until a constant rate is obtained. Curves for R.P.M. versus applied load for F-288 and F-241 gels are shown in Figure 2. These curves show a pseudo yield point. Thus at low shearing forces, i.e., low applied load, the rate of rotation is extremely slow while under a somewhat higher load the increase in R.P.M. with an increase in the applied load is rapid. For these particular gels the most satisfactory but time-consuming criterion of consistency would be the load required to produce 10 R.P.M. With many gels, especially those containing interpolymers, the viscosity of the gel increased during the measurement, and results could be duplicated only when a fresh sample was used for each measurement. It was therefore not possible to obtain a constant rate. As a rapid generalized technique the procedure described in Appendix A-2 was therefore adopted. It consisted in measuring the average rate of rotation during 100 revolutions of the paddle after an acceleration period of 10 revolutions under a standard load (800 or 1400 g.) that appeared suitable for the particular type of gel.

d. Gardner Mobilometer

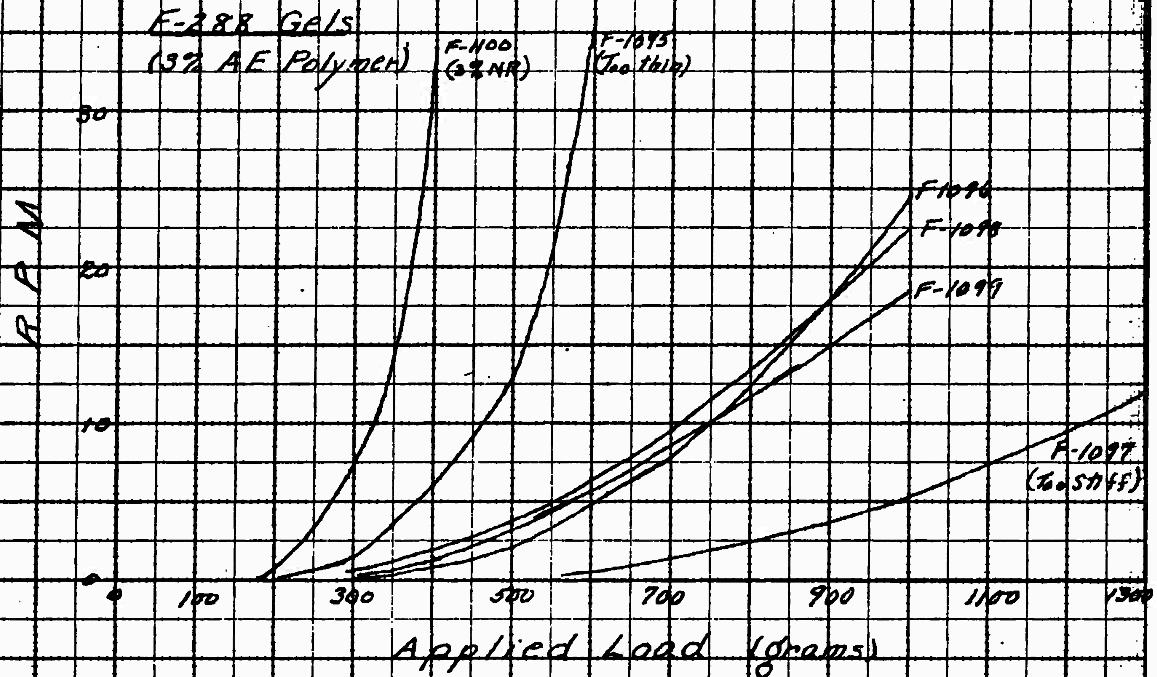
The Gardner mobilometer as described in CWS Directive 201 is an extrusion type viscosimeter. The rate of fall of a loaded piston through the sample contained in a tight-fitting cylinder is determined. Due to work hardening phenomena, the value observed with methacrylate gels was markedly dependent upon the treatment of the sample. Reproducible values were obtained only when freshly prepared gels were allowed to age 24 hours in the Gardner cylinder and subsequently a single measurement was made on the unworked sample. As shown in Figure 3, with F-107 type gels the logarithm of the time required for the piston to travel 10 cm. (rate of shear) plotted against the logarithm of the applied load (shearing force) gives parallel straight lines, and that the separation between these lines is proportional to the polymer content of the gel. This measurement was not used for routine characterization of laboratory gels due to the large number of mobilometer tubes that would have been required.

For extremely tough gels a special modification of the mobilometer measurement was devised. The gel sample was allowed to remain in the large container (1 quart or greater) in which it was prepared. The loaded piston was placed on the surface and allowed to fall through the gel. The time in minutes required for the piston face to penetrate from 1/2 inch to 1-1/2 inches below the surface is taken as the modified

Figure 2

Modified Stormer Viscosities

F-288 Gels: 3% AE Polymer 2.5% Stearic Acid 2.5% Naphthenic Acid 3% of 40% NaOH
F-241 Gels: 5% NR Polymer, remainder as in F-288.



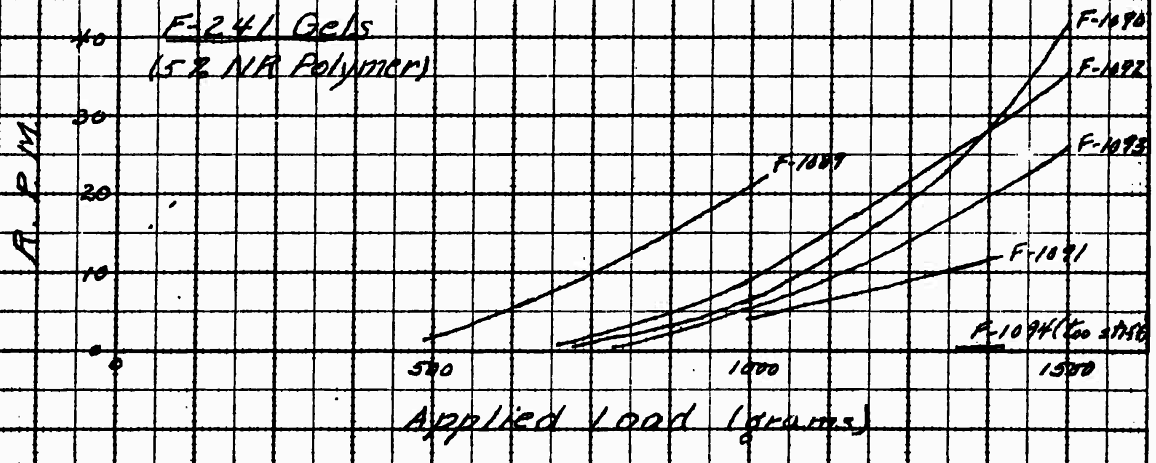
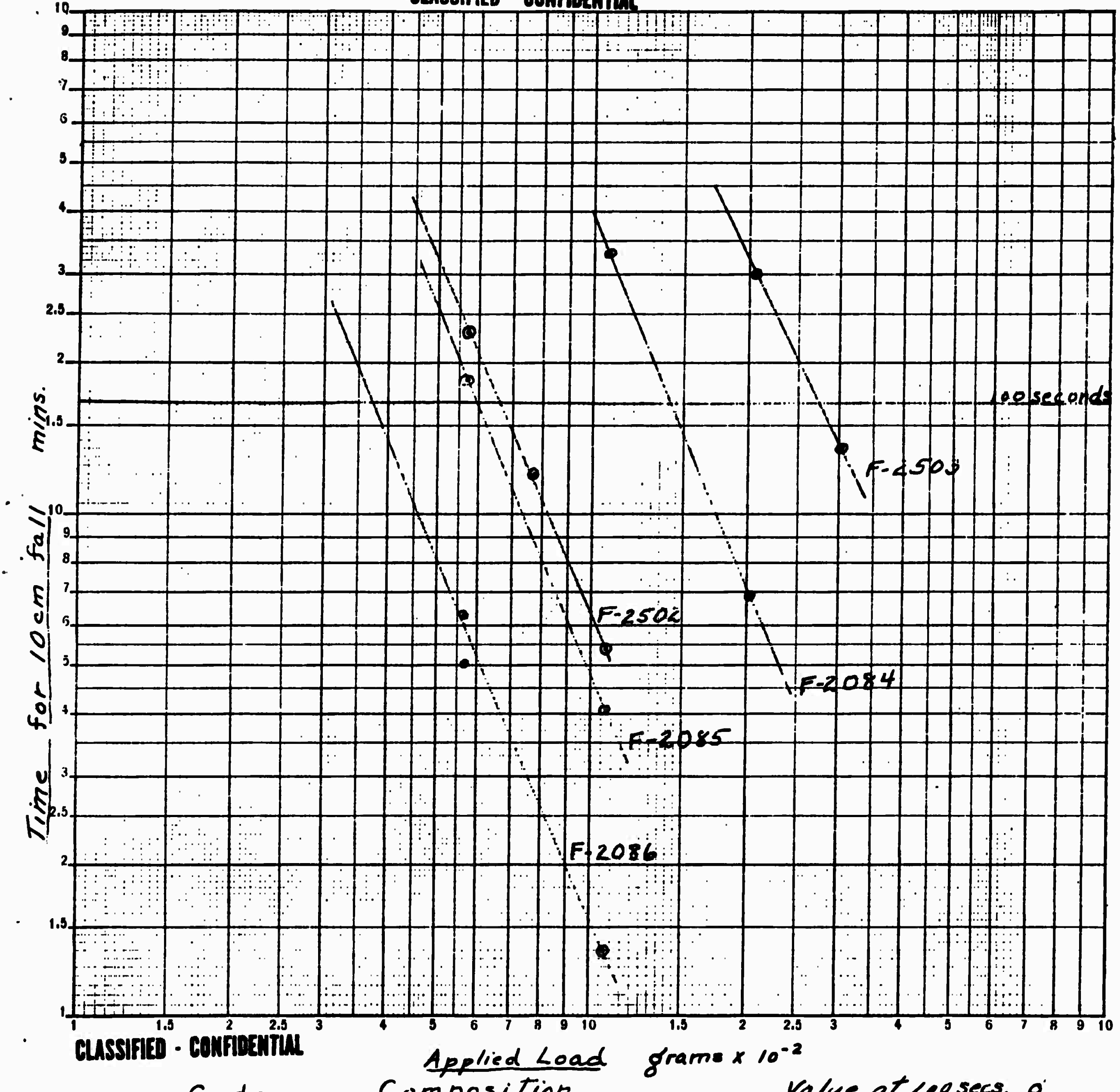


Figure 3
Gardner Mobilometer
4 hole Efflux Viscosimeter

CLASSIFIED - CONFIDENTIAL



CLASSIFIED - CONFIDENTIAL

Code	Composition	Value at 100 secs. g
2084	F-107 5% AE Polymer	1410
2085	F-107 3% AE "	600
2086	F-107 2% AE "	370
2502	F-241 5% NR Polymer	660
2503	F-107 5% AE-5 "	2700

mobilometer value (min./inch/567 g. load). This technique allowed the test to be run on unworked samples and eliminates the difficult step of filling the mobilometer tube.

e. A.S.T.M. Grease Penetrometer

The A.S.T.M. penetrometer determines the depth of penetration in hundredths of centimeters of a cone into the gel in 5 seconds. The standard A.S.T.M. procedure D-217-27T uses a load of 150 g. To obtain penetrometer values in the most sensitive region of the instrument, the load was reduced by a counter-balance to 50 and 25 g. Even under these conditions the correlation between penetrometer values and apparent stiffness was still not good. The roughness of the gel surfaces and the presence of air bubbles in the gels seemed to introduce errors which exceeded the normal differences between gels.

f. Clark-Hodsman Viscosimeter

In a fundamental study of the rheology of gels, the Eastman Kodak Company (Contract OEMsr-538, Monthly Report #1, June 18, 1942) studied the use of the Clark-Hodsman viscosimeter to determine the elasticity and viscosity of gels at very low shearing forces. An instrument built by the Eastman Kodak Company was loaned for test to the Ammonia Department of E. I. duPont de Nemours & Company. By trial and error it was necessary to determine the proper diameter wire and angle of torque for each gel in order to obtain a valid measurement. Many gels did not exhibit sufficient adhesion to the plumb to prevent slippage. The measurement did not therefore appear convenient as a method of characterizing new gels. The values observed on different preparations of the same gel formula did not show sufficient variation for this technique to serve as a control method. For a fundamental study of a particular gel, the device had the advantage over the parallel plate method that it gave the viscosity at low shearing forces directly in poises rather than in empirical units.

2. Miscellaneous Physical Measurements

While consistency of the gels at various shearing forces appeared to be the major factor determining their firing performance, other physical properties were determined in a search for critical tests.

a. Drop Test

As an empirical screening test to reduce the number of gels submitted for evaluation in firing tests, one pound samples were allowed to fall from a height of 60 feet onto plywood targets. The force of the impact in these drop tests seemed to duplicate that observed in static M-69 firing tests. When the samples were dropped onto a flat target the thickness of the resulting smears was close to that subsequently observed at Bayway. In the drop tests the samples which failed to adhere when the target was inclined at a 45 degree angle also fell off the target in firing tests both in the M-69 at Bayway and in the E-9 mortar tests at Beacon, N. Y. In this qualitative test the area of the smear and degree of adhesion for a series of gels was compared. The latter development of the more convenient impact strength test has outmoded the usefulness of the drop test.

b. Eastman Extensibility Test

The extensiometer developed by the Eastman Kodak Company (Contract OEMsr-538 Monthly Report #4, September 14, 1942) consists of two pieces of 4 mm. glass tubing which may be drawn apart at the rate of 0.5"/sec. The two tubes are brought into contact and filled with gel. The length of the gel is the distance the gel can be drawn without breaking. Some gel samples which do not adhere to glass will slip out of the tubes and give erroneous values. Gels with high soap content had average lengths of 1/8 inches with an occasional formulation giving values as high as 4 inches. Interpolymer gels normally had lengths in excess of 12 inches and thus beyond the limit of accurate measurement. The instrument was useful in special cases where the value observed fell in the narrow range where accurate determinations could be made.

3. Estimation of Incendiary Properties

The incendiary quality of a gel depends on the rate and quantity of heat liberation during combustion. The total thermal energy content can readily be calculated from the heat of combustion of the constituents. Since soap and polymer ingredients tend to burn slowly and incompletely, their heat of combustion can be ignored. The heat content of a gasoline gel is then directly proportional to the fuel content. To measure the rate of combustion three simple burning tests were devised.

a. Flash Point

The Tagliabue open cup method was used to determine flash points of various gels for shipping classification. With soap-free interpolymer gels the observed value varied from below 10° to +60°F. as the polymer content was increased from 2 to 6%. The dependence of the observed value upon the draft over the sample limited the accuracy of this technique. Apparently the determining factor was the rate at which the gel liberated gasoline vapor rather than the equilibrium vapor concentration.

b. Burning Times

To obtain a draft-free area for combustion tests a cubical box was constructed of "Transite" insulation boards 1 yard square. When this box was covered the air supply limited the combustion of samples of practical size. Under these conditions the temperature rise in the box during the combustion of 15 g. of gel did not exceed 20°C. The recommended practice is to use the box as a shield leaving the top uncovered. The length of time the gel remained burning was taken as an inverse measure of the burning rate. Two specific procedures differing in size and distribution of sample are described in Appendix A-3. Values obtained by the two test methods were approximately equivalent for stiff samples. Method B is recommended for strong gels. Burning times varied between 2.7 and 7.0 minutes. Comparative results taken on the same day appeared very accurate. Under varying weather conditions determinations on the same gel formula agreed within 0.3 minute.

E. Surveillance

Three sets of test conditions to determine the stability of gels during storage were standardized. As a control on manufacture of individual lots of a standardized gel the CWS specifications required that the gel structure be stable for 4 hours at both -40°F. and 125°F. For laboratory studies on gel formulation more reproducible data were obtained by extending the surveillance periods at these temperatures to one month. Results of such tests are summarized in appended Table 18. As a final acceptance test on new proposed gel formulas, cyclical surveillance tests were set up based on information assembled from U. S. Weather Department data. The conclusions of this study were reported by letter, H. R. Dittmar to Dr. R. H. Ewell, December 14, 1942. After exposure to low temperature the gel sample was inverted and the amount of unthickened gasoline which could be poured off was noted as per cent of the total sample. After exposure to high temperatures (125-150°F.) the presence or absence of separation into distinct phases was observed and the amount determined. The per cent syneresis after the sample returned to room temperature was also occasionally determined. The sample was worked with a spatula to initiate incipient syneresis. The best method of insuring proper temperature control during surveillance tests appears to be immersion of the sample in a sealed container into a liquid bath. The container must not make direct contact with the heating element in the bath. To test the reproducibility of the method a large preparation of IM-3 gel was split into four portions and each portion was held at 125°F. for one month. The syneresis observed on these duplicate samples varied between 1 and 3%. Upon cooling to room temperature the material gelled completely. A total of 12 different preparations of the IM-4 formula was placed in surveillance. Syneresis observed at 125°F. varied between 0.2 and 1.0%. One week's storage at 150°F. did not appear to be an accelerated test since there was no correlation between the breakdown observed under these conditions and that observed after one month at 125°F. or six months at ambient temperature.

F. Analytical Methods

Detailed procedures for the chemical analysis of methacrylate gels may be found in the report on IM-3 gels (D. E. Strain to Brig. Gen. W. C. Kabrich, 5/17/43). These techniques were useful in studying gel production problems but are not pertinent to gel formulation studies. Due to the incompleteness of the reaction of lime with acids in organic media, only the determination of total basic ingredients is applicable to IM-1 and IM-4 lime gels.

G. Performance Tests

Since all performance testing on incendiary gels prepared by this group was made by other organizations, only a general description of the tests will be given. Detailed procedures may be obtained from the reports of the indicated groups. For all standardized munitions CWS specifications include tests on performance of the fuse and powder charges and on gel ignition. The only tests discussed here are those designed particularly to study gel quality.

1. M-69 Static Firing Test

The static firing test studies gel distribution on ejection from an M-69. The bomb is fired in a horizontal position so as to deliver the gel to a 4' square vertical plywood target placed at a distance of 30 feet. If syneresis has occurred a flash burn will be observed near the mouth of the case. The fire is extinguished within one minute preferably by CO_2. All the gel is collected and placed in three piles and the amounts of each estimated. The first pile is the material scraped off the plywood target. The per cent of the total gel in this pile is the per cent adhesion. All gel on the ground within 2 feet of the base of the target would presumably support a fire on the target. This is reported as the per cent at the base. The remaining gel frequently estimated by difference represents scatter. The sum of the gel adhering to the target plus the gel at the base is the gel in the target area. Such tests are run on M-69's at room temperature and after cooling the bomb to -40°. In succeeding tests scatter is very reproducible, while a good evaluation of adhesion must depend on the average of several shots. At -40°F. a satisfactory F-614 gel will exhibit less than 20% scatter. At 70°F. the adhesion should exceed 50% and the scatter should be less than 10%.

2. E-9 Texas Mortar Test

At their Beacon, N. Y. Laboratories the Texas Company has constructed an outdoor mortar and target to similate the action of their E-9 bomb (Texas Company Contract OEMsr-898, Monthly Reports for periods ending August 15 and November 15, 1943). The mortar has a powder chamber and tail section identical with those of the E-9 bomb. In initial tests the target was a 1/4" plywood-covered steel frame containing approximately 80 square feet of surface (65 square feet in a vertical position). It has a 2 foot wide apron raised from the ground. In subsequent tests a plywood target of the same dimensions was supported against a cinder-block wall and cement floor. The incendiary filling was fired from the mortar point blank at the target 75 feet away. The resultant fire was extinguished after 10 minutes and the per cent of the target completely destroyed was estimated.

3. M-47 Static and Flight Tests

The Munitions Development Section at Edgewood Arsenal of the Technical Command of CWS tested M-47 incendiary fillings both by firing the loaded bombs statically or by releasing them from a plane.

In the static test the bomb was propped at a 45° angle from the ground and fired electrically. The number of fires still burning at 1 minute intervals after ejection was determined. Based on the average number of fires burning at 5 and 11 minutes a "fire index" was calculated. The extent of flash burn and the amount of unignited gel was also estimated.

4. Examination of Frangible Grenade Fillings

The Munitions Development Section of the CWS Technical Command tested frangible grenade fillings by throwing loaded grenades at a steel wall inclined at an angle 45° from the vertical. The grenade was thrown at the wall at an angle of 45° from the base of the target. The adhesion of the gel to the wall, the area covered and the duration of the fire were noted.

V. Laboratory Formulation of Methacrylate Gels

A. General Discussion of Composition Variables

The optimum methacrylate gels were mixtures containing the following basic components: viscolizing agent (methacrylate polymer), soap forming acids*, aqueous bases, and fuels principally gasoline. In a given gel formula the optimum concentrations and compositions of the various components are closely interrelated to an extent that may not be immediately apparent. For example, a change in the polymer content of a given formula requires a readjustment in the concentrations of all the other components in order that the resulting gel have the desirable properties specified in Section III. It also may not be feasible to maintain the ratios of all other ingredients constant and then to vary the fuel content. That each formula represents a balance dependent on all of the components present should be borne in mind during the following discussion on the effect of the individual components in gel formulation studies.

In a very general way the polymer imparts to the gel elasticity and strength (resistance to high shearing forces); fillers and soaps such as sodium or calcium stearate impart body (resistance to low shearing forces), while naphthenic acid soaps act as plasticizers.

1. Concentration and Nature of the Polymer

Early formulation experiments indicated that a satisfactory gasoline thickening agent should be gasoline soluble and of high molecular weight. The potentially available materials appearing to meet these requirements included polyisobutyl methacrylate grades NR and AE and a series of interpolymers of isobutyl methacrylate and methacrylic acid in which

---

*Note: The formulation of methacrylate gels free of soap-forming acids is discussed in a separate report.

the methacrylic acid content was less than 2%*. To prepare a viscous polymer solution with properties comparable to a mixture of 8% smoked sheet rubber dispersed in gasoline requires approximately 15 to 20% NR polymer. Since the polymer is the least readily available ingredient of gasoline gels, it is desirable to reduce the polymer content to a minimum. This has been accomplished by the use of soaps and fillers. In such soap fortified gels for each grade of methacrylate polymer there is an optimum concentration range below which the gels are unstable in storage and above which an increase in polymer content does not materially increase gel toughness. To cover the range of strengths required of gels for the various incendiary munitions, it is therefore preferable and, in part, necessary to alter the nature rather than the concentration of the polymer. A fairly complete coverage of the field is attained by the use of the four methacrylate polymers: NR polymer, AE polymer, 0.1 interpolymer and 0.3 interpolymer.

Gels prepared from NR polymer are in general soft pastes which show little tendency to string. They have excellent adhesion to wood but insufficient strength to resist disintegration when fired from internal burster well bombs. A high soap content is required to give them sufficient body. The toughness of the gel is less dependent on polymer content than gels prepared from other methacrylate polymers. Stable gels were prepared containing 5 to 6% soaps and from 2 to 7% NR polymer. The optimum concentration of this grade polymer appeared to be 5% as exemplified by the F-241 (IM-2) formula. An increase in the polymer content of this basic formula to 6 or 7% did not result in any marked improvement in gel strength or cohesion. When the polymer content was reduced to 2%, the resultant gels were stable but extremely weak, bearing a resemblance to cup grease.

The substitution of about 3/5 parts AE polymer for each part NR polymer in the above formulas allows a saving in polymer requirements without marked change in physical properties. Thus F-288 gels containing 3% AE polymer had firing characteristics in the M-69 bomb closely resembling that of F-241 (5% NR polymer) gels. In a similar manner nearly comparable gels of the F-614 (IM-3) formula could be obtained by using 2% AE polymer or 3.5% NR polymer. In gels of the same polymer concentration, however, AE polymer imparts enhanced strength, elasticity, and length as determined by the Eastman extensiometer. Thus direct replacement of AE for NR polymer in the

---

*<u>Note</u>: In the remainder of this report these materials will be referred to as NR polymer, AE polymer and X interpolymer where X% is the methacrylic acid content of the interpolymer.

F-241 formula (5% NR) yielded a gel (F-107) which was too tough for use in the M-69 incendiary but satisfactory for use in the M-47 internal burster well type bomb. It is therefore possible to prepare stable gels with a greater range of physical properties with AE than with NR polymer. Dependent on the impact strength desired, the optimum concentration of AE polymer lies between 2 and 5%. At high polymer concentrations (8 to 10% and even in some cases 5%), the gels were brittle or friable and tended to increase in stiffness during storage at ambient temperatures. The friability of such gels could be reduced by an increase in their naphthenic acid content but this also resulted in a decrease in gel strength so that no overall improvement was noted by reformulating 5% AE polymer gels to an 8 or 10% polymer content. The minimum polymer content is dictated by gel stability. Despite frequent attempts only one completely stable calcium soap fortified gel was prepared which contained less than 3% AE polymer. A stepwise decrease in the polymer content of the IM-1 formula from 5 to 3% (F-1400, 1404-5, 1433, 1419, Table 2) gave gels with a regular gradation in impact strength between 13 and 7 B scale units. A similar gel containing 2.5% AE polymer (F-1420) did not retain the lime particles uniformly dispersed and exhibited syneresis during storage. A further reduction in polymer content to 2% (F-1421) gave a mixture which separated into two phases immediately after gelation. The limit was less marked when the stearic acid, lime, and water content of the formula was increased but even with the F-1429 formula, room temperature syneresis occurred when the polymer content was only 2.5% (E-1429A) rather than 3%. The only lime gel (F-1422) containing 2% AE polymer which exhibited less than 1% syneresis after storage at room temperature for six months or -40°F. and 125°F. for one month was extremely fluid despite the presence of 5% of stearic acid. The stability limit of sodium soap fortified gels appears to be 1.5% of AE polymer. Many stable caustic gels containing 2% AE polymer were prepared and two (F-962 and F-614, IM-3) were recommended for use in the M-69. When the polymer content (2%) of the F-614 formula was reduced the syneresis observed after one month's storage at 125°F. rose steadily from 5% (average of three tests F-787, F-1525, F-1525A) at a polymer content of 1.5% to 10% (F-788) at a polymer content of 1.25%. The high temperature syneresis of gels containing only 1% AE polymer could be reduced by increasing the soap-forming acids content from 6% to 7 or 9% (F-727, F-730, F-733), but in such cases syneresis occurred during storage at room temperature.

Table 2

AE Methacrylate Polymer Lime Gels

Gel Number F-	1400	1405	1404	1433	1419	1420	1421	1422	1429	E-1429	E-1429A
Reference N.B.1049, pg.	74	75	75	88	83	83	83	83	88	Note 1	
Composition (Wt. %)											
AE Polymer	5.0	4.5	4.0	3.5	3.0	2.5	2.0	2.0	3.0	3.0	2.5
Stearic Acid	3.0	3.0	3.0	3.0	3.0	3.0	3.0	5.0	4.0	4.0	4.0
Lime	2.0	2.0	2.0	2.0	2.0	2.0	2.0	3.3	4.0	4.0	4.0
Water	1.25	1.25	1.25	1.25	1.25	1.25	1.25	2.1	2.5	2.5	2.5
Grade of Gasoline	Esso Winter Base									Quartermaster	
Physical Properties of gel 2 days after preparation						Note 2	Note 3				
Stormer Viscosity R/1000 sec./500 g.	1.9	0	3.0	10.0	47.0			0.4	0.25		
Parallel Plate Values (cm.)	8.2	8.5	8.7	9.1	11.9			7.7	6.8	7.2	7.4
Impact Strength C Units	13.0	11.8	11.2	9.6	7.0	4.8		8.6	15.5	14.8	9.8
Surveillance											
% Syneresis after 1 month											
at -40°F.	0	1.0				2.0		0	0	0	0
at 125°F.	1.0	0				0		0	0.5	0.5	0.2

Note (1) - E-1429 and E-1429A were prepared by the Chemical Warfare Service in their pilot plant at Edgewood Arsenal.

Note (2) - F-1420 not homogeneous.

Note (3) - F-1421 separated into two phases.

CLASSIFIED - CONFIDENTIAL

In formulation studies the transition from NR or AE polymer to one of the isobutyl methacrylate-methacrylic acid interpolymers results in a complete change in physical properties and optimum ingredient concentrations. Gels produced with interpolymers tend to be stronger but more resilient than those prepared with AE or NR polymer. Direct substitution of interpolymer for AE polymer in the best formulas developed for the latter material yields gels which in firing tests rebound from the target. The effect of this substitution on the properties of F-288 and F-614 gels is shown in Table 3. The presence of methacrylic acid in the polymer greatly increases the length or stringiness of the gels (Extensibility) and decreases their fluidity (Stormer viscosity) but does not markedly change the "yield point" or limiting shearing force at which flow becomes negligible (Parallel Plate value).

Table 3

Effect of Nature of Polymer on F-288 and F-614 Gel Properties

	Composition				Physical Properties		
Gel	Polymer %	Polymer Type	Equal Parts Stearic and Naphthenic Acids, %	No. of Gels Tested	Parallel Plate, cm.	Stormer Viscosity R.P.M./ 1400 g.	Extensibility in
F-575	3	NR	5	11	11.5-13.0	280-760	0.1
F-288	3	AE	5	3	9.0-10.5	72-150	0.4-1.3
F-1219	3	0.05 IP	5	1	10.1	17	1.8
F-1220	3	0.1 IP	5	1	8.4	2	8.0
F-1222	2	NR	6	3	12.0-13.0	450-475	0.1
F-614	2	AE	6	3	9.5-12.0	120-385	0.3-0.9
F-1227	2	0.05 IP	6	1	10.7	100	1.3
F-1228	2	0.1 IP	6	1	9.5	120	2.8

The use of only 1% of 0.1 interpolymer together with 6 or 7% soap-forming acids resulted in gels equal in strength to the typical 2% AE polymer gels. Gels containing only 1% of 0.1 interpolymer, however, all showed 15 to 30% syneresis during storage for one month at 125°F. Gels prepared with 1% of 0.3 interpolymer were even more unstable. The formulation of acceptable interpolymer gels is accomplished by reducing the soap-forming acids content and thus decreasing their rigidity. By the addition of toluene to the gasoline it is possible to prepare stable gels which contain no soap-forming acids and 3 to 6% of interpolymer. These soap-free fels (discussed in a separate report) are clear, moderately resilient materials.

Their rigidity, elasticity, and rate of heal are inversely proportional to the polymer content. Such gels containing only 2% interpolymer will break down at room temperature. When 15% toluene was present, the upper limit on polymer content was 6%. Gels containing 7, 8, and 9% of an interpolymer prepared from isobutyl methacrylate containing 0.3% methacrylic acid had impact strengths of 9.3 to 10.0 C units and synerized at room temperature, while the stable 6% interpolymer gel had an impact strength of 17.1 C units. The addition of from 1 to 3% of soap-forming acids to stable interpolymer gels will result in an increase in body and a slight increase in impact strength, but a marked decrease in their rate of combustion. Interesting soap-fortified 2% interpolymer gels were, however, obtained by the use of radically different mixtures of soap-forming acids as exemplified by the F-624 formula. In this case a stable fluid gel was stiffened by the addition of a filler to produce the M-69 incendiary filling which was judged the most effective of any tested in the fuel review of November 19-21 (1942). Such fluid mixtures susceptible to bodying by inert solids were generally observed only when interpolymer was the thickener. The use of 4 to 6% interpolymer in the more standard soap fortified methacrylate formulas yields gels which are not adhesive but have impact strengths greater than have been obtained with any other thickening agent. Thus in munitions where extremely high bursting or ejection forces are needed for proper functioning, the use of soap-fortified interpolymer gels may be required. A preferred solution would be adaptation of the munition to weaker gels of better incendiary quality.

Isobutyl methacrylate is the preferred methacrylate ester for the production of thickening agents. Fsters prepared from alcohols of lower molecular weight are gasoline-insoluble. There is no marked difference in gelling power between normal and isobutyl methacrylate polymers. n-Butyl methacrylate polymers, however, were given little consideration since their low softening temperature makes drying of the polymer emulsion difficult and might cause agglomeration of the flaked product during storage. The use of polylauryl methacrylate in the F-241 formula gave a very fluid weak gel which was inelastic but had excellent adhesion. Four per cent of an interpolymer of 2-ethylhexyl methacrylate and 0.2% methacrylic acid gave in gasoline-caustic mixtures a gel which was weak, soft and short. The addition of soaps resulted in weak paste-like gels. Interpolymers of 2-ethylhexyl methacrylate and 0.05% methacrylic acid gave stiff, fairly strong soap-fortified gels which, however, were friable.

In an attempt to extend methacrylate polymer, the condensation between the free acid groups in 1% interpolymer with the free hydroxyl groups in "Butacite" was studied. When esterification occurred, the products were not gasoline-soluble.

2. Nature and Concentration of Soap-Forming Acids

In an extensive search for bodying agents for methacrylate gels the only suitable compounds uncovered were gasoline-soluble organic acids. The various materials tested, a generalized statement of their value, and the source of the sample studied are indicated in Table 15 in the Appendix. Each new material was tested alone and in combination with the most promising soap-forming acids. Formulation of the six most effective materials was then studied more intensively in gels containing various combinations of 2 or 3 acids.

The soap-forming acids recommended for incorporation in incendiary gels perform one of two distinct functions. Stearic and oleic acids impart stiffness, body and high temperature stability to all types of methacrylate gels. They cause a slight increase in the strength of the gel. Naphthenic acid and dimerized soyabean oil acids act as plasticizers; when used in moderate concentrations they increase the fluidity and adhesiveness of stearic acid gels and prevent an increase in stiffness during storage without simultaneously reducing gel strength. Wood rosin and Turkey Red Oil normally function as plasticizing agents, but in certain combinations may fulfill both functions of soap-forming acids. It is significant that all strong stable gels uncovered in this study contained either or both stearic or oleic acid or their equivalents. Mixtures of plasticizing acids such as wood rosin and naphthenic acid or Turkey Red Oil and naphthenic acid invariably yielded weak fluid gels.

a. Gels Containing Stearic Acid

For a given gelling agent (NaOH, CaO, etc.) the optimum ratio of the different soap-forming acids to each other appears to be independent of the nature and concentration of the polymer while the total soap content required is inversely proportional to the polymer content and to the methacrylic acid content of the polymer. This is best demonstrated by the study of caustic gels containing stearic and naphthenic acids. A formulation study on this combination of acids was made for several munitions and for various polymer contents. In each case the proportions of each acid were varied widely but the gel chosen for field evaluation on the basis of stability and physical properties contained equal parts of the two acids (Table 4).

Table 4

Sodium Stearate-Naphthenate-Methacrylate Gels

Optimum Formulation for Various Polymer Contents

Gel No.	Composition				Intended Use
	Polymer		Stearic Acid, %	Naphthenic Acid, %	Incendiary Filling for
	%	Grade			
F-241	5	NR	2.5	2.5	M-69
F-288	3	AE	2.5	2.5	"
F-614	2	AE	3.0	3.0	"
F-720	1	0.3 IP	3.5	3.5	" (not sufficiently stable)
F-107	5	AE	2.5	2.5	M-47
F-1406	3	AE	3.0	3.0	"
F-519	4	0.1 IP	1.5	1.5	"
F-1941	5	0.3 IP	1.5	1.5	E-9 (too strong)

Gels containing stearic acid in excess were friable, stiff and tended to harden during storage. When naphthenic acid was present in excess the resultant gels were soft, fluid and unstable at 125°F.

While gels containing sodium stearate require high concentrations of plasticizer, those containing calcium stearate require little or no plasticizer. The IM-1 and IM-4 (F-1429) formulas represent stable lime gels which contain only stearic acid as the bodying agent. They are semi-solid, strong elastic gels which heal rapidly when cut but will not adhere well to wood. The effect of the incorporation of naphthenic acid into these two basic formulas is demonstrated in Table 5. The addition of 1% naphthenic acid (F-1440) to the naphthenic acid-free F-1429 formula markedly improves adhesion or "tack" of the gel without materially affecting gel strength or stability. The partial replacement of stearic by naphthenic acid in all cases reduces gel strength and body excessively. The stability of lime gels is much less sensitive than caustic gels to variations in the acids content. Of the gels listed in Table 3, only F-1416, in which replacement of stearic acid by naphthenic acid is nearly complete, shows more than 3% syneresis in either of the surveillance tests. The formulation of a lime gel containing stearic or stearic and naphthenic acids is dependent on the relative importance in the proposed use of gel strength and of adhesion.

Table 5

Calcium Stearate-Naphthenate-Methacrylate Gels

	Composition				Surveillance			Physical Properties*				
					% Syneresis After 1 Mo.				Parallel Plate		Impact Strength	
Gel No.	% AE Polymer	Stearic Acid, %	Naphthenic Acid,%	Lime, %	At -40°	at 125°	Adhesion to Wood	Stormer Viscosity	2 Days	1 Mo.	2 Days	1 Mo.
IM-1	5	3	-	2.0	2.6	0.5	None	1.9	8.2	8.6	13.0	15.6
F-1414	5	2	1	2.1			Good	40	9.5		8.3	
F-1413	5	1	2	2.3			Good	480	12.7		5.2	
F-1429	3	4	-	4.0	0	0.5	Unreliable	0.25	6.8	7.1	15.5	13.1
F-1440	3	4	1	3.47	1.0	0.5	Good	0.25	7.3	7.2	15.3	
F-1431	3	3	1	3.5	1.4	1.0	Good	2.2	7.9	8.7	10.3	9.7
F-1409	3	4	-	2.67	1.8	0	None	0.25	7.5		13.2	
F-1418	3	3	1	2.8	1.8	0	Good	3.3	8.5	8.8	8.4	8.1
F-1417	3	2	2	2.9	2.4	1.0	Good	42	10.2		4.2	
F-1416	3	1	1	3.1	7.0	3.0	Good	2490	14.8		2.6	

*Units: Stormer Viscosity: Revolutions per 1000 seconds under a 500 g. load.
Parellel Plate Value: (cm.)) Measure 2 days and 1 month after
Impact Strength: B Units) the gel was prepared.

CLASSIFIED - CONFIDENTIAL

Since the effect of fortifying agents is most apparent in gels of low polymer content, other organic acid combinations were principally studied in caustic gels containing 2% AE polymer or interpolymer and in lime gels containing 3% AE polymer. As demonstrated above, it is anticipated that the optimum acid combinations will be applicable to formulation studies at other polymer concentrations.

The Northern Regional Research Laboratory has developed a process to dimerize the methyl esters, principally linoleates, from soyabean oil. Hydrolysis of this dimer yields high molecular weight gasoline-soluble acids whose use as gel plasticizers has been appraised in only a preliminary manner. In combination with stearic acid, the dimerized soyabean oil acids yield moderately fluid non-elastic gels of high fuel content. The use of approximately equal parts of the two acids appears optimum. To improve stability and rate of heal in this type of gel it is necessary to use a 100% excess of caustic as gelling agent.

Based on the above observations, the preferred combination for use in the M-69 incendiary is the F-962 formula. This gel which closely resembles the F-614 gels in appearance has physical properties (parallel plate 8 to 9 cm., impact strength 5.5 to 6.0, C scale units) in the range found optimum for the M-69 bomb. The 33% lower solids content (5% greater fuel content)of the F-962 than that of the F-614 formula results in a 20% reduction in time of combustion. The burnt crust of the F-962 gel is harder and more tenacious than the residue from the F-614 gel. On the basis of an improvement in incendiary qualities, it is recommended that if manufacture of small tail ejection incendiaries should recommence, the F-962 formula should be evaluated in field trials. The use of AE polymer in place of 0.1 interpolymer in the F-962 formula yields stable gels (less than 1% syneresis in one month at -40 or 125°) which are, however, too weak (impact strength 3.7 B scale units) for use in the M-69. When the dimerized esters are partially hydrolyzed the resultant half ester-half acid yields much stronger but unstable gels. The soyabean oil dimerized acid mono esters of more complex alcohols, such as abietyl alcohol or octadecanediol, were without thickening action. In a few preliminary tests the dimerized soyabean oil acids appear promising as a plasticizer for lime gels. F-967 (3% AE polymer, 4% stearic acid, 2% dimerized acids, 3.5% CaO, 2.3% H_2O) was stable in laboratory surveillance tests and equals the IM-4 gel in strength. The use of dimerized soyabean oil acids might introduce manufacturing difficulties due to their high viscosity, 81 poises at 25°C., and congealing temperature, 0°C.

The presence of small amounts of Turkey Red Oil (sulfonated castor oil) in stearic acid gels will increase fluidity and adhesiveness but simultaneously reduce gel strength. The effect is most marked with lime gels. Thus, for example, the addition of 0.2% Turkey Red Oil (F-1447) to the IM-4 formula causes a reduction in impact strength from 13 to 9 C scale units and an increase in the Stormer viscosity value (i.e., a decrease in viscosity) from 0.25 to 16 revolutions/ 1000 sec./500 g. When sodium hydroxide is the gelling agent the preferred ratio of stearic acid to Turkey Red Oil is 3 to 1. Such gels are stable, fluid, and adhesive but appreciably weaker than naphthenic acid plasticized gels. At both higher and lower ratios the gels become stiff and friable so that in high concentrations Turkey Red Oil appears to act as a stiffening agent. Although the plasticizing action of Turkey Red Oil has not been successfully applied during the present formulation studies, its use with stearic acid warrants further consideration because of the adhesion and good low temperature properties it imparts. This is exemplified by gel F-2386 (5% ethyl cellulose, 3.5% stearic acid, 1.8% Turkey Red Oil, 15% acetone, 3.7% of 40% NaOH) described in Section VIII. Due to the nature of the viscolizing agent, this gel tended to scatter in M-69 static firing tests but all of the material remaining in the target area at both -40°F. (40%) and at 77°F. (53%) adhered to the target. This target adhesion at -40° is one of the highest observed in the M-69 field evaluations.

Methacrylate gels containing mixtures of stearic acid and wood rosin as the only soap-forming constituents separated and hardened during surveillance at 125°F. and lacked adhesion to wood. The soap-thickened fuel developed by the Standard Oil Development Company for use in the M-69 bomb contained 3.5% wood rosin and 1.75% stearic acid. When 2% of various grades of isobutyl methacrylate polymer was added to this formula, the gels synerized at room temperature and were stiff and weak. In the presence of 2% interpolymer the most stable gels contained equal parts of the two acids. The best gel in this series (F-2045) contained 1.5% stearic acid, 1.5% wood rosin, and 2.1% of 40% NaOH. In M-69 firing tests gel F-2045 exhibited moderate strength but negligible adhesion to the target. Gels fortified by mixtures of wood rosin and stearic acid showed in storage a greater decrease in parallel plate value (1.8 cm./mo. average of 10 gels) than did gels prepared from any other combination of soap-forming acids. The properties of the gels were not improved by the use of rosin derivatives (dimerized rosin and hydrogenated rosin).

Of the various combinations of three acids (including stearic but not oleic acid) studied, mixtures of wood rosin-stearic and naphthenic acids were the only ones to show improvement over the simpler two-acid systems. Gels containing equal amounts of these three acids were strong, elastic, fluid and stable in surveillance. This is the only combination of stearic acid and wood rosin that does not synerize at 125°F. and which apparently does not stiffen with age. The F-624 gel containing 2% of 0.1 interpolymer, 2% each of the three acids and 0.5% alpha-cellulose has been found to have the best incendiary characteristics as a filling for the M-69 bomb of any methacrylate gel tested. In the absence of filler (alpha-cellulose) the use of 2% of 0.2 interpolymer with the same soap-forming acids yields a gel which proved only slightly inferior in firing tests to the F-241 and F-624 formulas. Replacement of the naphthenic acid in the F-624 formula by Turkey Red Oil produced a stiff gel unstable at 125°F.

As indicated in appended Table 15, many other organic acids were found to be equivalent but in no way superior to stearic acid as gel fortifying agents. The ready availability of stearic acid therefore dictated its use. Based on surveillance studies, Hydrofol 51 was the grade of stearic acid most suitable for gel preparation. It was used as the standard stearic acid in laboratory formulation tests. Triple pressed but not double pressed stearic acid was nearly as satisfactory as Hydrofol 51 and was preferred when the gel contained wood rosin.

b. Gels Containing Oleic Acid

Gels containing oleic acid were softer, slightly stronger, and less stable at either -40°F. or 125°F. than equivalent stearic acid formulations. The only combination of oleic acid and a single other acid that was stable in surveillance tests was a mixture of oleic and naphthenic acids. The stabilization of other acid mixtures by the addition of stearic acid will be discussed in the next section. Gels in which oleic acid was the sole bodying agent were weak and very unstable. Gel F-2099 (2% of 0.3 interpolymer and 3% oleic acid) exhibited 20% phase separation after 3 days at 125°F. or 10 days at -40°F. The series of gels containing oleic and naphthenic acid were extremely strong, fluid and elastic. In consistency and feel they resembled a raw oyster. The stable formulas contained 2-3% oleic acid, 2% naphthenic acid, 2% of a 0.3 interpolymer, and 2.3 to 3.3% of 40% NaOH. Gels within this composition range were sufficiently strong to resist breakup when fired in an M-69 incendiary but target adhesion was always low. When the naphthenic acid content was increased to 3% the gel scattered badly when fired at -40°F. Due to excessive elasticity, these gels would not tolerate the presence of fibers. Gels containing alpha-cellulose or Darco were more unstable than unfilled gels and did not show improved adhesion in firing tests. Mixtures of oleic acid and Turkey Red Oil yielded soft rubbery methacrylate gels which could be stiffened by the presence of fillers but were very unstable at 125°F. All two acid gels containing oleic acid and wood rosin separated when stored at 125°F. The strongest gel in this series was F-2049 (2% of 0.3 interpolymer, 2% oleic acid, 3% wood rosin). Higher concentrations of wood rosin gave weak, grainy gels while lower concentrations of wood rosin made the gels fluid. In this series wood rosin appears to act as a stiffening rather than a plasticizing agent. Oleic acid-wood rosin mixtures were not gelled by bases other than caustic (CaO, $Zn(OH)_2$, Al_2O_3, $Ba(OH)_2$). The substitution of stearic by oleic acid in the F-624 formula (2% of 0.1 interpolymer, 2% stearic acid, 2% naphthenic acid, 2% wood rosin, 0.5% alpha-cellulose) yielded weak gels which broke down in surveillance at 125°F. This series of gels remained fluid and unstable when the wood rosin content was varied between 1 and 3%.

c. Gels Containing Both Stearic and Oleic Acids

Gels containing stearic and oleic acids but no plasticizing acid were stiff, friable, weak, and unstable. In order to reduce stiffness and to produce gels which would heal when cut, it was necessary to add a plasticizing agent in a concentration equal to or greater than the concentrations of the stearic and oleic acids combined. Such mixtures were in general fluid and were improved by the addition of fillers. The presence of oleic acid tended to reduce any change in gel physical properties that occurs with time but also reduced adhesion to wood.

In the series of gels containing mixtures of stearic, oleic and naphthenic acids, the optimum ratio of acids appears to be 1 to 1 to 3 (naphthenic). Such gels are fluid and elastic but when bodied by alpha-cellulose became inelastic and moderately stiff. F-615 (2% AE polymer, 1% stearic acid, 1% oleic acid, 3% naphthenic acid, 0.5% alpha-cellulose) was completely stable in surveillance tests. In M-69 firing tests this gel showed moderate strength at both 70°F. and -40°F. but adhesion to the target was low. The partial replacement of the stearic acid in F-615 formula by alpha-cellulose reduced stability and gel strength and increased resilience. All gels in this series containing 0.3 interpolymer as viscolizing agent were very unstable at 125°F.

The use of high concentrations of wood rosin allowed formulation of stable gels from stearic and oleic acids with interpolymer rather than AE polymer. The proportions of these acids yielding greatest strength without excessive stiffness was 1% stearic acid, 2% oleic acid and 3% wood rosin. When the viscolozing agent with these acids was 0.3% interpolymer (F-2052) or 0.2% interpolymer (F-622), the resultant gels showed no syneresis in surveillance tests and exceptional strength in M-69 firing tests at both 70°F. and -40°F. Adhesion to the target in M-69 firing tests was negligible. Due to its lower wood rosin content, another basic formula (F-623: 2% of 0.1 or 0.2 interpolymer, 1.5% stearic acid, 2% oleic acid, 1.75% wood rosin) which was stable in surveillance had moderate strength but was too stiff. The use of 2% each of the three acids, 0.5% alpha-cellulose and 2% AE polymer (F-563) gave a stable gel which was somewhat weaker and more elastic than its prototype F-624. Gelation of this mixture with lime produced a very fluid gel.

All gels prepared from mixtures of Turkey Red oil, stearic acid, and oleic acid were friable and weak.

d. Miscellaneous Bodying Agents

The addition of 5% of bodied linseed, soyabean or castor oils to soap-fortified gels did not cause any marked change in physical properties or firing characteristics.

Evaluation of lignin stearate (Meadol) as a bodying agent for methacrylate gels showed it to be without effect when used in normal concentrations (2.5-5.0%). A 10% solution of the ester in gasoline gelled with aqueous caustic is fluid, weak and inelastic. Up to 30% of the ester can be added to straight gasoline without significantly increasing its viscosity.

A gel prepared by substituting glyceryl tristearate for the stearic acid in the F-107 composition had a "setting up" time of 2 to 3 hours. The gel ultimately resembled a typical F-107 gel but was less stable at 125°F.

3. Gelation Agents

The gelation of gasoline solutions of methacrylate polymers and soap-forming acids was accomplished by the addition of aqueous solutions of strong bases, of which lime and sodium hydroxide proved most useful. The preparation of a satisfactory gel is strongly dependent on the proper choice of the nature and amount of the gelation agent and of the strength of the aqueous solution. With sodium hydroxide, a water-soluble base, best results were obtained when a 40% solution was added in an amount 1-1/2 times that required to neutralize the acidity of the soap-forming acids and the interpolymer. On the other hand, a sevenfold excess of ground lime appears optimum. Throughout the formulation study of soap-fortified gels, preliminary evaluation of other ingredients was based on these concentrations of gelling agent.

a. Sodium Hydroxide

In formulation studies on aqueous caustic as a gelling agent, the greatest variation in gel properties was observed when the strength of the caustic solution was varied. For gel stability the strength of the caustic solution must exceed 25%. For each strength of caustic solution there is an optimum range of gelling agent concentrations below which the gels tend to be hard, friable and unstable at -40°F. and above which the gels tend to be fluid and unstable at 125°F. The greater the concentration of the caustic solution, i.e., the less the amount of water present, the lower was the number of equivalents of NaOH at the optimum point. With 40% aqueous NaOH solutions approximately 1-1/2 equivalents of base per equivalent of organic acid in the gel were required while 1-3/4 equivalents of a 35% solution appeared optimum. The above generalizations on the importance and effect of the nature of the gelling agent were borne out in intensive formulation studies on the F-241 and F-614 formulas which are described in detail in Section VII A-1 and VII B-1, respectively. A similar effect was also noted in studies of gels containing the following combinations of acids: stearic-wood rosin, stearic-dimerized soyabean oil acids, and naphthenic-oleic acids. In all cases the point of optimum stability in surveillance coincided with the concentration yielding maximum impact strength and optimum body, i.e., neither fluid nor friable. A 40% solution of NaOH is about as concentrated a solution as can be conveniently handled. It freezes at approximately 5°C. An anhydrous system was obtained by neutralizing the organic acids with solid sodium methylate which is gasoline-soluble. In the absence of water neutralization of the stock solution with sodium methylate increased the viscosity but did not cause gel formation. If water were slowly added to this viscous mixture, gelation occurred. The best gels obtained in this manner contained base and water equivalent to a 40% NaOH solution. Since the presence of alcohol from hydrolysis of sodium methylate is known to affect the 125°F. stability adversely, this technique is not recommended.

b. Lime

In contrast to sodium hydroxide, the use of lime results in gels which are slow to set up and which are stable over a wide range of concentrations of gelling agent. While sodium hydroxide gels body approximately 30 seconds to 1 minute after the caustic is added and reach their ultimate strength in 24 hours, lime gels may require one hour to body sufficiently to keep the lime dispersed and require at least 48 hours to reach ultimate strength. Lime gels are prepared by dispersing the solid lime in the gasoline solution of polymer and soap-forming acids and then obtaining gelation by adding water with agitation. The major fraction of the lime never goes into solution but remains suspended in the gel. The final gels are somewhat fluid, resilient, and lack adhesion to wood unless a plasticizing acid is present. In formulation studies on lime gels containing 3% or more AE polymer, a large number of surveillance tests showed negligible syneresis at 125°F. and less than 2% syneresis at -40°F. The only exceptions were a gel prepared with lime which would not pass 60 mesh and a gel with very low water content.

While lime gel stability is insensitive to the concentration of the gelling agent, physical properties of the gels are strongly dependent on the amount of CaO in the gel, to a lesser extent on the size of the lime particles used, and to a very minor extent on the amount of water added. This is demonstrated by examples in Table 6. An increase in the lime content of a gel increases its strength and stiffness but reduces adhesiveness. As shown in Table 6-A, the impact strength of F-1429 (IM-4) gels increased from 5.7 to 17.4 C scale units as the lime content was increased from 1.67 to 5.0%. There was a simultaneous decrease in the Stormer viscosity and parallel plate values which determine the fluidity or body of the gel. In this series the ratio of the lime to water concentrations was kept constant and a standard graded lime was used. A series of IM-1 gels (Table 6-B) were prepared using as the gelling agent close fractions from a sieve analysis of ground lime. These fractions were recomposited as indicated to give a standard ground lime of uniform particle size for gelation studies. The finer the particle size the greater is the surface exposed and thus the greater is the effective concentration of lime in the gel. A decrease in the particle size, therefore, increases the strength and stiffness of the gel. The impact strength measured when the gels were two days old varied between 11.8 for the gel containing lime of 60-80 mesh to 17.0 when the lime passed 200 mesh. After a seven months period in storage the impact strengths of these IM-1 gels tended to approach each other but this was far from complete. In general, there are only slight changes with age in the physical properties of lime gels after the initial 48 hour gelation period. The rate at which gelation occurs is strongly dependent on the particle size of the lime. An IM-1 gel prepared with lime passing 200 mesh set up completely in 20 minutes while the gels in Table 6-B containing grades of lime of successively larger particle size

Table 6

Isobutyl Methacrylate AE Polymer Gels Containing Lime

Nature of the Gelling Agent

Gel	Composition: AE Polymer %	Composition: Stearic Acid %	Composition: Lime %	Composition: Lime Grade	Composition: H_2O %	Impact Strength C Units: 2 Days	Impact Strength C Units: 1 Mo.	Impact Strength C Units: 7 Mos.	Parallel Plate Value (cm.): 2 Days	Stormer Viscosity R/1000 Sec./500 g.: 2 Days
A. Effect of Lime Concentration										
F-1426	3	4	1.67	G*	1.0	5.7			9.9	70
1425	3	4	2.0	"	1.25	5.8			10.4	97
1424	3	4	2.3	"	1.4	7.2			9.2	26
1423	3	4	3.0	"	1.9	10.8			8.0	2.3
1429	3	4	4.0	"	2.5	15.5			6.8	0.25
1430	3	4	5.0	"	3.1	17.4			6.3	0
B. Effect of Lime Particle Size										
F-1395	5	3	2	35-60 mesh	1.25	2.4			13.6	5050
1397	5	3	2	60-80 mesh	1.25	11.8		12.6	9.3	3
1398	5	3	2	80-100 mesh	1.25	12.3		14.2	8.2	1.1
1399	5	3	2	100-200 mesh	1.25	14.0		-	7.7	0.9
1396	5	3	2	>200 mesh	1.25	17.0	16.8	16.4	7.5	0
1400	5	3	2	G	1.25	13.0	15.6	15.5	8.2	1.9
C. Effect of H_2O Concentration										
F-1428	3	4	2.67	G	1.33	9.4			8.6	5.0
1427	3	4	2.67	"	2.0	9.7			8.0	5.0
1452	3	4	2.67	"	5.0	11.1			8.5	3.0

*Analysis of Grade G Lime: 22% each of fractions 35-60, 60-80, 80-100 and 100-200 mesh and 12% finer than 200 mesh.

required from 1 to 20 hours to gel. The coarsest lime used (35-60 mesh) gave a gel which did not set up completely. As is shown in Table 6-C in the range of 1 to 5% the water content of IM-4 gels does not affect physical properties significantly. An increase in the water content yields a smoother, more uniform gel which exhibits slightly greater low temperature syneresis.

Gels of the same physical properties result whether calcium oxide and water or slaked lime, $Ca(OH)_2$, and water in an amount corrected for the quantity present due to slaking are used as the gelation agent. A group of 29 gels prepared with CaO or $Ca(OH)_2$ were studied in respect to aging characteristics and stability at outside storage conditions. Gels containing stearic but no naphthenic acid have constant parallel plate values over the period from 48 hours to 7 or 8 months (whether gelled with CaO or $Ca(OH)_2$), and show no syneresis. Those gels containing both stearic and naphthenic acid show no syneresis if CaO has been the gelling agent. When $Ca(OH)_2$ was used the gels show from 0.1 to 2.0% syneresis. The presence of naphthenic acid in lime gels tends to cause the gels to weaken with age. Gels containing 3% naphthenic acid and 1% stearic acid showed syneresis at room temperature within three weeks after preparation when $Ca(OH)_2$ was the gelling agent but no syneresis in 8 months when CaO was used.

Unsuccessful attempts to prepare lime gels in the continuous unit will be described in Section VII-D

c. Ammonia and Amines

Methacrylate gels containing ammonium hydroxide as gelling agent gave the most rapid burning rate and left the least residue on combustion of any gels tested. Despite a fairly comprehensive formulation study, however, no strong gels were prepared when either ammonia or amines were used as gelling agents. The complete data on this program are shown in Appended Table 16. The gels prepared from 27% aqueous ammonia were very fluid and synerized rapidly at 125°F. Gels containing organic amines and soap-forming acids retained the favorable burning characteristics of ammonium hydroxide gels but were weak and friable. Attempts to prepare soap-free interpolymer gels with amines as the gelation agent did not yield any mixture with sufficient strength even for use in frangible grenades. The soap-free interpolymer amine gels are stable at 125°F. but synerize at -40°F. Partial replacement of gasoline by toluene results in weaker but more stable and mobile gels at low temperature. Attempts to cross-link the polymer in interpolymer-gasoline solutions by the addition of diamines such as ethylenediamine did not result in strong gels.

d. Miscellaneous Gelation Agents

By proper choice of concentrations, other strong bases can be used as gelling agents in place of caustic or lime. The properties of both soap-free and soap-fortified interpolymer gels containing 40% NaOH could be duplicated by gelation with an equivalent number of moles of 50% aqueous KOH. A 40% KOH solution gave stiffer, more friable gels. In a few tests the use of lithium hydroxide in the F-241 formula or in the soap-free 5% interpolymer formula gave very fluid gels. In a similar manner barium hydroxide closely resembled calcium hydroxide as a gelling agent both as regards rate of gel formation and ultimate strength.

No gelation was obtained when weak bases or basic salts were added to interpolymer-gasoline or interpolymer-gasoline-soap-forming acid mixtures. The weak bases tested included zinc hydroxide, aluminum hydroxide, powdered ignited aluminum oxide, and basic aluminum acetate. Both sodium carbonate and sodium bicarbonate failed to react in interpolymer-gasoline mixtures.

The effect of the soaps of weak bases was therefore observed by adding polymer or a polymer solution to a gasoline solution of the preformed soap. Aluminum stearate could be dissolved in gasoline by boiling with reflux. Aluminum naphthenate, aluminum palmitate and Napalm went into solution at room temperature. Gels containing 6% aluminum palmitate and 3 to 5% interpolymer were tough, elastic and stable in surveillance. Due to the absence of excess gelling agent, the above high polymer and soap content was required to obtain a strong, stiff gel. A gel containing 4% aluminum palmitate and 5% interpolymer synerized at room temperature. Aluminum soaps could not be combined with sodium soaps since they are peptized by alkali. Lead soaps are not effective bodying agents but also are not affected by the presence of caustic. A 10% solution of the lead soap of cocoanut oil fatty acids in gasoline was not gelatinous. The addition of either AE polymer or interpolymers to the soap solution increased its viscosity to produce very fluid gels. An elastic, moderately strong gel was produced by adding 1% of 40% caustic to a gasoline solution containing 2% of 0.1 interpolymer and 5% lead soaps. The incorporation of stearic, oleic or naphthenic acids along with the lead soaps, followed by gelation with caustic, yielded stiff, grainy, friable gels.

Attempts to simultaneously fill and gel a polymer solution by dispersing alkali cellulose in it were technically unsatisfactory. Control of water and caustic content of the alkali cellulose was difficult. There was no indication that this technique caused the cellulose to bind the aqueous and gasoline phases or to act in any manner other than as an inert filler. The best of these gels resembled an alpha-cellulose filled gel prepared in the normal manner by addition of 40% caustic to the stock solution containing polymer and filler.

4. Fillers

The addition of inert solid materials imparts body and reduces resilience of fluid elastic gels. With certain combinations of soap-forming acids, this yields strong, adhesive, stable gels. The outstanding example is the F-624 gel.

Of the materials tested, alpha-cellulose ground in a micropulverizer appeared to be the most effective filler. Due to the low apparent density of this fibrous material, the maximum amount that can be dispersed in a gel is approximately 3% by weight. The optimum concentration appears to be 0.5%. Both Darco G-60 activated charcoal and crystalline sucrose in concentrations of 3% will stiffen a methacrylate gel to approximately the same extent as will 0.5% alpha-cellulose, but will not impart as great strength or adhesion.

The effective use of a filler requires a gel base which without filler is fluid and yet tough and therefore elastic and non-adhesive. Such gels were formed when the viscolizing agent was an isobutyl methacrylate-methacrylic acid interpolymer and plasticizing soaps were present in greater concentration than soaps which acted as bodying agents. In general gels containing such combinations of soaps were unstable at 125°F. For example, gels with excellent physical properties were obtained by filling stearic-naphthenic acid containing mixtures in which naphthenic acid was in excess. The best of this series contained 2% interpolymer, 1.5% stearic acid, 3.5% naphthenic acid and 1% alpha-cellulose, and completely broke down into two phases after 3 weeks at 125°F. The only soap combinations which were both stable in surveillance and susceptible to filling were: (a) 2% each of stearic acid, naphthenic acid and wood rosin, (b) 2% each of stearic acid, oleic acid and wood rosin, and (c) 0.5% stearic acid, 1% oleic acid and 3% naphthenic acid. Gels prepared from combination (b) were somewhat stiffer and weaker than those containing the acids in (a) (F-624). The mixture of acids (c) gave strong gels which in laboratory tests appeared the equal of the F-624 formula but which showed negligible adhesion to the target in M-69 static firing tests.

The optimum effect of a filler was obtained when there was a simultaneous reduction in the methacrylic acid content of the interpolymer in the base formula. Thus with acids combination (a) the optimum interpolymer was prepared from isobutyl methacrylate containing 0.2 and 0.1% methacrylic acid for unfilled (F-616) and filled (F-624) gels, respectively. In each series the use of a higher interpolymer gave tougher, somewhat elastic non-adhesive gels which were less stable in surveillance while a lower methacrylic acid content gave weak gels which were not improved by an increase in their alpha-cellulose content.

5. Fuels

As the major constituent of incendiary gels the importance of the fuel cannot be overemphasized. The nature of the gasoline is an important factor in determining gel strength and stability. The incendiary effect of gels is obviously due to the fuel; during combustion the other components of gels are only partially consumed. The rate of combustion and the total available thermal energy of the gels can be varied widely by appropriate choice of solvent. The presence of aqueous caustic or lime prevents the incorporation of pyrotechnic materials in methacrylate gels. Combinations of metals and oxidizing agents which intensified gel combustion also destroyed gel stability.

a. Gasoline

A study of the gasoline requirements of three (F-241, F-614, IM-1) specific gel formulas is described in Section VII. These data combined with the work of the group at the Standard Oil Development Company on F-241 and Napalm gels show a regular correlation between gasoline quality and gel properties. All gels tested showed an increase in syneresis during storage at -40°F. with an increase in the aniline point of the gasoline used in their preparation. For low temperature stability most methacrylate gels require a fuel with an aniline point below 105°F. A low aniline point of the gasoline tends to reduce the impact strength and increase the parallel plate value of a gel to a slight extent. This results in improved low temperature firing characteristics. Both rate of gelation and final gel strength, however, may vary widely when gasolines of identical aniline point are used. Blue Sunoco yields tougher, more rigid gels than does Purol gasoline of the same aniline point. It is believed that this is caused by the relative proportions of aromatics, olefines, naphthenes, and paraffins in the particular fuel. It appears desirable to obtain a low aniline point by blending with "cracked stock" which is largely olefinic rather than by adding aromatics.

At the present time the suitability of a particular source of gasoline cannot be predicted by the usual tests. It is therefore essential that the grade of fuel to be used in any gel preparation work be first tested in the laboratory as a component of a standard gel formula. The formulation studies described in this report were made with either Esso Winter Base or Purol Naphtha #2. These two fuels appear identical for this purpose.

b. Co-solvents

The availability and excellent incendiary qualities of gasoline dictated its general use as the fuel component, but a short formulation study with other solvents was undertaken. The partial replacement of gasoline by toluene to stabilize interpolymer gels has been reported separately.

Gels with desirable physical properties are only obtained in hydrocarbon solvents. Gels based on the F-614 formula were prepared in acetone, isobutanol, ethyl acetate, benzene, diisobutylene and cyclohexane. No gelation occurred in the first of these solvents and extremely weak pastes resulted from the use of the other two oxygen-containing solvents. Benzene gave very soft, fluid gels from which solid benzene separated below -20°F. Both cyclohexane and diisobutylene gave stiff, resilient gels which had fair strength. The hydrocarbon solvents which appeared to be available in sufficient quantities were toluene, turpentine, and higher boiling petroleum fractions. All of these materials have a greater heat of combustion per unit volume than does gasoline, so they allow an increase in the incendiary potency of a bomb filling. Higher boiling petroleum fractions have a lower vapor pressure and therefore in gels a slower burning rate than does gasoline. Although turpentine yields more viscous polymer solutions than does gasoline, turpentine gels were weaker than their gasoline prototypes. 5% AE polymer dissolved in gasoline, cyclohexane, and turpentine to give solutions whose viscosities were 1.25, 2.35 and 4.85 poises, respectively. F-614 (IM-3), F-1429 (IM-4) and F-1416 type gels were prepared using gasoline, turpentine or toluene as fuel. Those made with gasoline are the toughest, strongest and burn with the least amount of smoke. The use of toluene or turpentine in place of gasoline increases the burning rate of the F-614 caustic gel (3.0 or 2.9 vs. 3.6 minutes for 100 grams of gel). No significant effect was observed with the two types of lime gels. The turpentine gels took longer to ignite (for fire to spread over the complete gel surface). Partial replacement of gasoline by toluene was studied in a series of F-614 gels. As the toluene content was increased in 4% increments from 0 to 24%, the parallel plate value rose regularly from 9.7 to 10.9 cm. while the impact strength fell from 5.3 to 2.8 B scale units.

c. Solids

Due to the activity of other groups in this field, the effect of adding solid fuels or combustion acids was little investigated. When metals such as magnesium, zinc, lead, or aluminum were dispersed through the methacrylate gels, they did not ignite during combustion of the gel. Some metal-oxidizing agent combinations did increase the rate and intensity of gel combustion, but due to the presence of aqueous caustic or lime they were unstable. The presence in an F-614 gel of 50% of a mixture of equal parts of magnesium and sodium nitrate cut the burning time in half and gave a spectacular fire, but these two reagents reacted slowly, evolving ammonia and destroying the gel strength. Various other combinations of oxidizing agents including nitrates and barium chromate with powdered magnesium, zinc or sulfur were added to the F-614 gel. In all cases the metal did not ignite during gel combustion. The presence of oxidizing agents alone speeded up the combustion of the last quarter of the gel but did not affect the initial burning rate. The physical characteristics of the gels seem relatively unaffected as long as added solid content is kept below 30%. The presence of 25% sodium nitrate reduced the overall burning rate only 15%. Since this reduces the fuel and heat content of the gel, its use is not recommended. Liquid oxidizing agents such as nitrobenzene destroyed gel strength and stability.

B. Effect of Composition on Individual Gel Properties

The detailed information on gel formulation allows a few generalizations on the effect of the various components on gel properties.

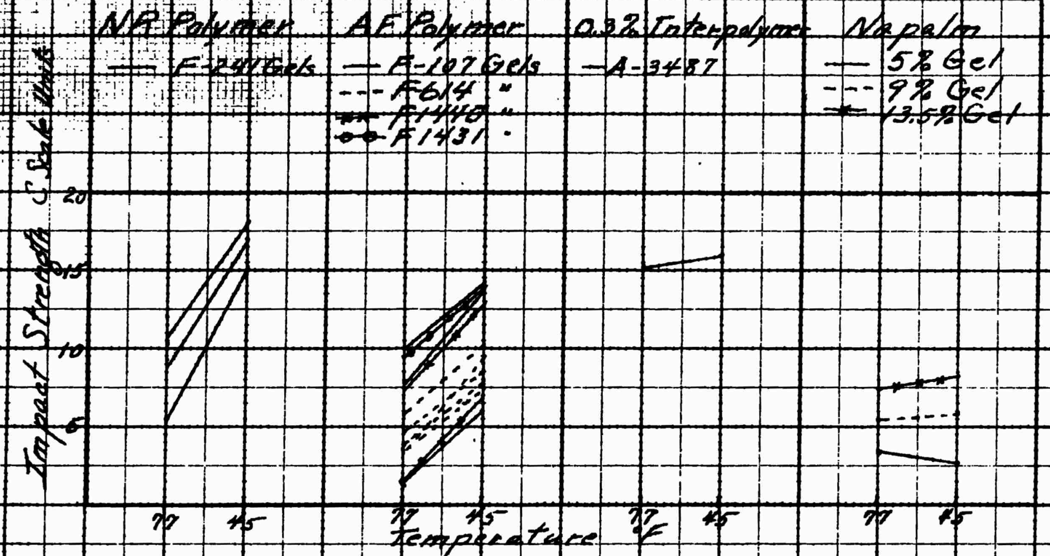
1. Consistency

The strength of the gel and its resistance to rupture when ejected from a bomb is largely determined by the nature and concentration of the polymer. The body of the gel on the other hand is determined by its soap content. Gels made with interpolymers tend to be elastic and tough, while the AE polymer produces more adhesive, slightly weaker, less elastic gels.

To demonstrate the variation in consistency with temperature the impact strengths of representative gels were measured at both 77°F. and 45°F. As shown in Figure 4, the gels fall into distinct classes which are determined by the nature of the viscolizing agent. It appears significant that

FIGURE 4

INCENDIARY GEL PROPERTIES: INFLUENCE OF TEMPERATURE

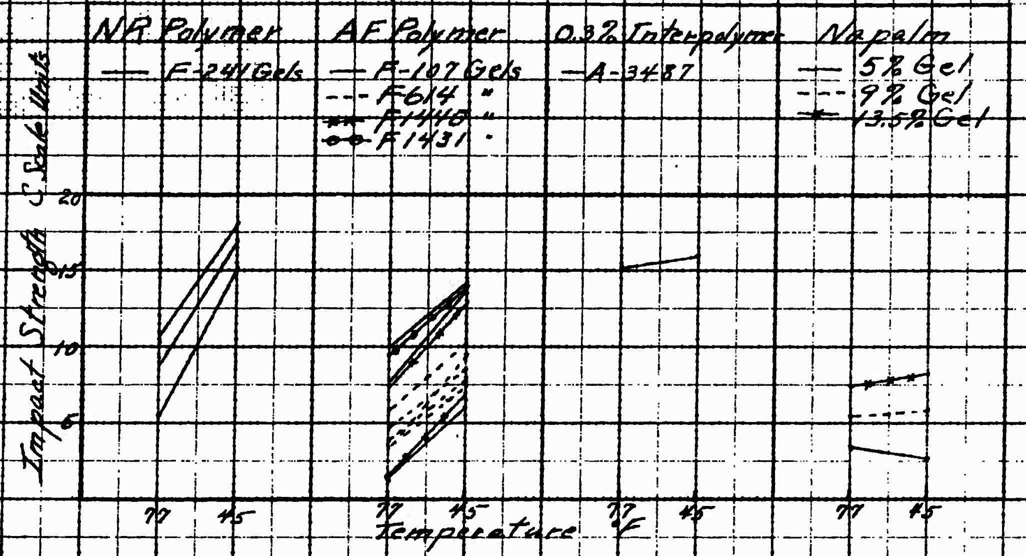


Weight Per Cent Composition of Gels

Code	F-241	F-107	F-614	F-1440	F-1431	A 3487
Components						
AF Polymer		5	2	3	3	
NR Polymer	5					
0.3% Interpolymer						4
Stearic Acid	2.5	2.5	3	4	3	
Naphthenic Acid	2.5	2.5	3	1	1	1
40% NaOH	3.0	3.0	4.5			1.5
Lime				3.5	3.5	
H_2O				2.2	2.2	
Alpha Cellulose						
Toluene						20
Gasoline	87	87	87.5	86.3	87.3	73.5

FIGURE 4

INCENDIARY GEL PROPERTIES: INFLUENCE OF TEMPERATURE



Weight Per Cent Composition of Gels

Code	F-241	F-107	F-614	F-1440	F-1431	A 3487
Components						
AF Polymer		5	2	3	3	
NR Polymer	5					
0.3% Interpolymer						4
Stearic Acid	2.5	2.5	3	4	3	
Naphthenic Acid	2.5	2.5	3	1	1	1
40% NaOH	3.0	3.0	4.5			1.5
Lime				3.5	3.5	
H_2O				2.2	2.2	
Alpha Cellulose						
Toluene						20
Gasoline	87	87	87.5	86.3	87.3	73.5

12 gels containing AE polymer and based on four diverse formulas all show impact strength versus temperature curves with substantially the same slopes. This series includes both lime and caustic gels, and gels containing from 2 to 5% polymer. The only factor that was not varied markedly was the total soap content. Based on this correlation, the thickners can be rated in order of their tendency to produce a gel whose consistency is insensitive to temperature in the following order: Napalm (best), 0.3 interpolymer, AE polymer, and NR polymer.

2. Incendiary Characteristics

The total heat of combustion of most of the gels under consideration is sufficiently uniform so that the incendiary characteristics of the gels are largely determined by their rate of combustion and the flow characteristics of the burning gel. Obviously the rate of combustion is influenced by the surface exposed and this in turn in practice will depend on the distribution of the gel from the munition. The burning rate tests give a comparative measure of incendiary properties which is independent of gel strength. Results are given in appended Table 17. The burning times are obviously relative and bear no direct relation to a particular munition.

In all cases combustion is incomplete. A residue of charred soap and polymer is apparent even during the early stages of combustion as a crust which coats the unburned portion of the fuel. Where this crust is strong it prevents the gel from flowing and spreading the fire. The heaviest crust is formed by lime gels and the weakest and most permeable crust by formulas gelled by aqueous ammonia. Caustic gels are intermediate. The quality of the crust is also dependent on the soap-forming acid content of the gel. Thus in 2% AE polymer gels a reduction in the soap content from 6% (F-614) to 4% (F-962) reduced the burning time 20%. The effect of soaps is further exemplified in a series of gels containing 4 or 5% of a 0.3 interpolymer. The addition of naphthenic acid caused a slight reduction in burning time while the presence of all other soap-forming acids tested increased the period of combustion appreciably. The effect of variation in the fuel fraction and the use of oxidizing agents have been discussed in the sections on co-solvents and solid fuels, respectively.

3. Stability

In methacrylate gels good low temperature stability is favored by a low polymer content, the use of polymer of low methacrylic acid content, a high soap content, a high concentration of gelling agent, a low water content, and the use of gasoline which has a low aniline point. With a reasonably stable basic formula, the most important factors appear to be the nature of the gasoline and the strength and concentration of the gelling agent. During plant manufacture based on two formulas, unstable gels were produced when the gasoline had an aniline point of 112°F. and when the strength of the caustic solution fell to 36%. These difficulties were eliminated and stable gels produced when a gasoline of 95°F. aniline point and a 40% caustic solution were used. In a similar manner the instability of the F-614 formula was corrected by increasing the amount of 40% NaOH in the gel from 3.6 to 4.5%.

Good high temperature stability is favored by a high polymer content, a high stearic acid content, and the use of concentrated solutions of the gelling agent, i.e., a low water content. The presence of oxygenated solvents will destroy high temperature stability. In general, lime gels are more stable than caustic gels and are less sensitive to variations in composition. Gels containing more naphthenic acid than all other soap-forming acids combined are extremely stable and, in general, the presence of naphthenic acid aids gel stability. Most rosin containing gels, on the other hand, exhibited syneresis. It is recommended that if rosin is to be used in new gel formulas, then the few stable rosin combinations described above be adhered to closely and the stability of the product be examined critically.

The results of all surveillance tests on laboratory gel samples are given in Table 18 in the Appendix.

VI. Evaluation of Gels in Field Trials

While physical testing techniques guided the work on gel formulation and eliminated many mixtures from further consideration, the orientation of the physical testing and the final choice of candidate fillings were based on field trials. Gels were assessed in this manner in four munitions of which the M-69 incendiary bomb was the application studied most intensively. In all cases this group merely prepared material for test by other groups in NDRC or CWS. Representatives of this group were frequently observers at such tests. Only the data pertaining to gel formulation are presented in this report. When firing tests were run on plant production or on experimental plant batches, the work was supervised by other groups and has been reported to them.

A. M-69 Incendiary Field Tests by Standard Oil Development

Forty methacrylate gels were submitted to the Standard Oil Development Company for evaluation as M-69 fillings. The static firing test against a plywood target served as a preliminary measure of gel properties. The best gels were then studied in more extensive tests including burning tests in attic structures, mortar shots, and open field range and ignition tests. Series of firing tests to standardize the properties of F-241, F-614, and F-624 gels will be described in Section VII. The results of a competitive examination of candidate M-69 fillings held November 19 to 21, 1942 are described in a fuel review report of November 27, 1942 by Prof. H. C. Hottel to Dr. R. P. Russell, Chairman, Division B-10, NDRC.

After establishing the merits of the F-241 gel as an M-69 filling the purpose of subsequent firing tests and formulation studies was to prepare a gel of equal value as an incendiary filling but of reduced methacrylate content. This polymer saving was accomplished in the F-614 and F-624 formulas containing 2% AE polymer as opposed to 5% NR polymer in the F-241 gels. As criteria of the value of these gels the per cent in the target area when the gel was fired at room temperature and at -40°F. and the room temperature adhesion to the target were chosen. A summary of the firing tests is shown in appended Table 19. With the exception of the F-624, no gel containing 2% of an interpolymer gave more than 35% adhesion to the target and in the majority of cases only 5 to 10% of the gel charge remained on the plywood target. This indicates the elastic and cohesive as opposed to adhesive properties of interpolymer gels. These gels also tended to scatter when fired at -40°F. If strength alone is considered and adhesion is ignored, the F-2052 (2% of 0.3% interpolymer, 2% oleic acid, 3% wood rosin and 1% stearic acid) would be chosen due to nearly complete absence of scatter at both -40°F. and 70°F. The lack of adhesiveness of interpolymer gels was corrected by the use of a filler in the case of the F-624 gel or by shifting to AE polymer and increasing the concentration of soap-forming acids as exemplified by the F-614 formula. We believe an acceptable M-69 filling will average less than 10% scatter at both -40° and 70°F. and will show at least 50% adhesion to the target at 70°F. The tests on the F-614 gels indicate that gels meeting these requirements will have impact strengths in the range of 4 to 10 B scale units.

The four ethyl cellulose thickened gels which underwent firing tests all showed excessive breakup. This may demonstrate the importance of the reaction between the gelling agent and the viscolizing agent in producing gel strength since methacrylate polymers react with caustic while ethyl cellulose is inert. Gel F-2386 (5% ethyl cellulose, 3.5% stearic acid, 1.8% Turkey Red Oil, 15% acetone) showed remarkable adhesion at both firing temperatures; the per cent adhering to the target nearly equaled the amount remaining in the target area. This suggests a further study of Turkey Red Oil as a plasticizer is warranted

If renewed production of the M-69 bomb is anticipated it is suggested that firing tests be made to compare the F-614, F-624 and F-962 gels as the incendiary material for this bomb.

B. E-9 Mortar Tests by Texas Company

Nineteen gels were submitted to the Texas Company for evaluation as a filling in the E-9 40 lb. incendiary. Composition of the gels and results of the firing tests are shown in Table 20 in the Appendix. The original nine samples were gel compositions which had previously been tested in the M-69 or M-47 bombs. The toughest IM-4 and the fastest burning gel (A-3461) from this group were fired and thought to be too weak for the E-9. A second series of five gels having a minimum impact strength approximately twice that of the first series was then submitted and tested. These gels were obviously too tough so that a final series of five gels with intermediate strength was submitted.

The most effective gels contained an isobutyl methacrylate-methacrylic acid interpolymer and 1% or less soap-forming acids. The properties of these gels have been reported separately. They indicate that the optimum filling for the E-9 bomb will have an impact strength of from 10 to 12 C scale units. Such gels when delivered into locations favoring incendiary action start rapid intense fires which consume all combustible material in that area.

The methacrylate gels containing high concentrations of soaps were difficult to appraise by the existing technique. Possibly due to slow ejection from the mortar they were deflected and aiming at the target was inadequate. This is reflected in the high concentrations of gel located beyond the target. Destruction data should be judged with this factor in mind. The five extremely tough gels (A-3942 - A-3946) when delivered to the target tended to bounce back and fall away from the base of the plywood. As the gels burned they did not flow. Gel A-3944 showed a very high target destruction (80%) but was judged inferior to soap-free interpolymer gels since it gave a much more slowly starting fire. The only gel which adhered in large amounts to the target wall was a gel (A-3461) which was so weak that it was ejected from the case as a spray. This spray covered the target and flash burned.

C. M-47 Tests by Chemical Warfare Service

In the early search for incendiary fillings for the M-47 bomb, two isobutyl methacrylate gels were submitted for test by the Technical Command at Edgewood Arsenal of the

Technical Division of the Chemical Warfare Service. These two gels (F-107 and F-70*) were tested by comparing their performance as a bomb filling with that of 8% smoked sheet rubber in gasoline when loaded bombs were released from an airplane at an altitude of 5,000 feet. Initial tests indicated the F-107 formula was superior based on ignition dispersion of gel and duration of fire. Flash in all cases was negligible. Manufacturing considerations led to adoption of a similar gel containing lime which was formulated by the Research Section of the Technical Command. This was standardized as the IM-1 gel.

A considerable number of soap-free interpolymer gels were also tested. The results are described separately. A gel of moderate soap content (F-519 - 4% of 0.1% interpolymer, 1.5% stearic acid, 1.5% naphthenic acid, 2% of 40% NaOH) gave encouraging preliminary test results but was not completely evaluated.

At the request of CWS, an attempt was made to reduce the methacrylate requirements of the IM-1 formula. Based on laboratory data, four formulas were suggested by this group. Two of these formulas and a modification of a third were then prepared and assessed in static firing tests at Edgewood Arsenal. The composition of the gels tested and the "fire index" obtained are listed in Table 7. The 1429 formula was considered at least equivalent to the IM-1 gel and was standardized as the IM-4 gel. Gel 1429A containing only 2.5% polymer appeared unstable and gave excessive scatter.

D. Frangible Grenade Tests by Chemical Warfare Service

Initial production of frangible grenades used as the incendiary material, gels based on the IM-1 formula in which polymer content was varied between 2 and 5%. These did not prove stable in surveillance. On July 29, 1943, CWS requested a temporary formula for an immediate production program. Four gels known to be stable were tested July 30, 1943 and two (F-1416 and F-1457**) were far superior to any previously examined. Only a very limited gel strength is required

---

* F-70 - 8% NR polymer, 4.5% rosin, 3% of 40% NaOH, 84.5% gasoline.

**F-1416 - 3% AE polymer, 1% stearic acid, 3% naphthenic acid, 3.1% CaO, 2.3% H_2O.

F-1457 - 3% AE polymer, 4.5% stearic acid, 0.5% naphthenic acid, 2.33% of 27% NH_4OH.

in grenade fillings. The gels should flow readily and burn rapidly without excessive flash.

Table 7

Isobutyl Methacrylate Gels as M-47 Fillings

Firing Tests at Edgewood Arsenal

Code	IM-1	F-1429	F-1429A	F-1431
Composition				
AE Polymer	5	3	2.5	3
Stearic Acid	3	4	4	3
Naphthenic Acid				1
Lime	2	4	4	3.5
Water	1.25	2.5	2.5	2.2
Gasoline	88.75	86.5	87.0	87.3
Impact Strength C Units		14.8	9.8	9.4
"Fire Index"	8-9	11	16	6

A formulation study was undertaken to improve F-1416 type mixtures. To determine the physical properties in a gel which will give optimum results, a series of eight gels were forwarded for appraisal. The composition of these gels and the results of the test are shown in appended Table 21. These data indicate that a suitable methacrylate gel must have a Modified Stormer viscosity of 40 to 200 R.P.M./800 g. and a Gardner Mobilometer value of 20 to 200 g./1.5 minutes/10 cm. Thicker gels did not spread and tended to roll off the target while thinner gels scattered badly.

The soap-fortified methacrylate gels which were sufficiently fluid for use as grenade fillings were too viscous at -40°F. Gasoline solutions thickened with Vistanex, synthetic rubber or with Napalm are superior on this basis.

VII. Development of Specific Formulas for Plant Manufacture

Before a gel formula which appeared satisfactory in firing tests could be accepted for standardization, it was necessary to outline suitable methods of manufacture and to develop means of plant control. Part or all of this work was accomplished on four gel formulas.

A. F-241 (IM-2) Gels

The requirements for production of good quality F-241 gel and methods of assessing gel quality were studied in detail. This work formed the basis for subsequent work on other formulas. The specifications on ingredients were based on data obtained with the assistance of the Technical Division of the Chemical Warfare Service and the Standard Oil Development Company. The development of control tests and the technique of continuous manufacture were accomplished in this laboratory.

1. Tolerance in Composition

The Chemical Warfare Service suggested that plant production of F-241 gels be allowed the following tolerances in concentration of ingredients:

5.0% ± 0.2%	NR Polymer
2.5% ± 0.3%	Stearic Acid
2.5% ± 0.3%	Naphthenic Acid
3.0% ± 0.1%	of 40% NaOH
87.0% ± 0.5%	gasoline

A series of gels were prepared in which the minimum allowable concentration of solid ingredients was combined with the maximum allowable quantity of gasoline, and vice versa. The parallel plate values of these gels differed by only 0.3 cm. and there was no apparent change in gel quality. It was concluded that the recommended composition limits were reasonable.

2. Quality of Ingredients

F-241 gels containing various grades of stearic acid were prepared in both Blue Sunoco and Esso Winter Base gasoline. Surveillance tests indicated that low temperature syneresis was excessive if the stearic acid had a high iodine number (a measure of unsaturation). The gels containing Hydrofol 51 exhibited exceptional stability, while the gel containing triple pressed stearic acid was the least stable at -40°F. (Table 8). This same phenomenon was observed in a series of F-107 gels.

Table 8

Stability of F-241 Gels - Stearic Acid Requirements

	Stearic Acid		% Syneresis After One Month	
Grade	Acid No.	Iodine No.	at -40°F.	at 125°F.
Hydrofol 46	201.5	25.9	14.4	40
51	199.0	6.0 max.	0.8	2
300	199.8	25.5	14.4	0
305	202.2	6.3	6.4	3
400	202.8	15.0	9.4	2
405	202.0	3.8	3.8	3
Snodette	196.0	2.2	1.2	-
Triple Pressed			14.6	0

The use of Hydrofol acids of high iodine value consistently resulted in less stable gels (cf. Hydrofols 300 and 305, 400 and 405, 46 and 51 - Table 8). The physical properties of a series of gels containing several brands of stearic acid which were considered for plant use were determined. Of six samples tested, only one, Neo-Fat 1-65, caused any marked variation in consistency (parallel plate 12.0 cm., Modified Stormer 270 R.P.M./700 g.); the other five, including Hydrofol 51, Hardesty beads and single-, double- and triple-pressed stearic acid, gave comparable results (parallel plate values 10.4 to 11.1 cm., Stormer viscosity 5-81 R.P.M./700 g.). All the gels appeared satisfactory in drop tests. It was suggested that the Federal Specification on stearic acid (U.S.A. #50-11-47) be supplemented by a requirement that the iodine value should not exceed 8. This was incorporated in CWS Specification 196-131-123. Hydrofol 51 was suggested as the preferred material.

Variations in quality of naphthenic acid have a very large effect on the consistency of F-241 gels (Table 9). As might be expected, very impure naphthenic acid, such as Standard Oil of California acid #30669R (acid number 100) gives a fluid gel which is entirely unsatisfactory. Standard Oil Company of California acids "C", "D", Naphthones "E" and "AA", and Shell "A" are on the borderline of acceptability while all other acids tested appear to be satisfactory. Nuodex #240 which was used as a standard, was one of the best for use in the F-241 formula. The above conclusions were borne out by qualitative examination of a similar series of gels prepared in Blue Sunoco rather than Esso Winter Base gasoline. These data and samples of the various naphthenic acids tested were forwarded to CWS for examination. Based on their analyses of

Table 9

F-241 Gels

Dependence of Consistency on Naphthenic Acid Quality

	Naphthenic Acid			Properties of F-241 Gel		
				Parallel Plate	Stormer Viscosities	
Gel No. F-	Grade	Calc. % Unsaponifiable	Acid No.	Value (cm.)	R.P.M. 700 g.	R.P.M. 1400 g.
1156	S.O.Cal.*30669R	42.5	100	14.0	-	870
1120	S.O.Cal.*"C" 29527R	9.0	200	13.7	550	-
1123	Shell "A"	10.5	237	12.8	39	-
1122	S.O.Cal.*"D" 21065R	20.8	160	12.3	420	-
1157	" "	20.8	160	11.9	-	720
1163	S.O.Cal.*Naphthone"E"	5.0	214	11.9	-	420
1164	S.O.Cal.*Naphthone"AA"	0.7	274	11.7	-	210
1159	S.O.Cal.*"C" 29527R	9.0	200	11.6	-	450
1158	Shell "A"	10.5	237	11.5	-	150
1121	S.O.Cal.* "CC"	-	-	10.8	40	-
1101	Harshaw "Processed"	7.0	241	10.8	-	-
1103	Stanco "	7.0	256	10.7	-	-
1124	Union Oil Co."Refined"	6.5	232	10.4	6	-
1104	Nuodex #240	5.5	233	10.4	-	-
1125	" "	5.5	233	10.4	5	-
1162	" "	5.5	233	10.3	-	200
1160	Union Oil Co."Refined"	6.5	232	10.2	-	175
1109	Nuodex #240	5.5	233	9.9	-	-
1161	Std.Oil La."Rectified"	6.4	242	9.8	-	180

Note: *S.O.Cal. = Standard Oil Company of California.

the naphthenic acids, the acceptability of the acids is somewhat in line with the "calculated % unsaponifiable", a value obtained from the acid numbers of the original acid and the separated saponfiable acid. Acids containing in excess of 10% calculated unsaponifiable matter yield gels of poor quality. For acids of the same acid number, there is an apparent relation between calculated unsaponifiable and parallel plate values. The optimum range of acid numbers appears to be 230 to 245. Despite its satisfactory acid number (237), Shell "A" naphthenic acid is properly eliminated on the basis of its high per cent calculated unsaponifiable matter. The modified Stormer test follows closely the apparent stiffness of the F-241 gels made with various naphthenic acids, but is not always exactly in line with parallel plate values. A combination of these tests might be valuable. Due to the uncertainties in specifying naphthenic acid by laboratory analysis alone, a test of the acid by preparation of an F-241 gel was included in CWS Specification 196-131-104. Such a test on Harshaw "rectified" naphthenic acid proved it less suitable than other available naphthenic acids. This was confirmed in static firing tests where scatter was excessive. Sodium hydroxide meeting Federal Specification P-S631 appeared satisfactory for use in F-241 gels.

Formulation studies indicated that sodium carbonate did not cause gelation of soap-forming acids-polymer-gasoline mixtures. When sodium carbonate was mixed with the hydroxide it behaved as an inert. Since the presence of carbonate in the hydroxide would reduce the effective concentration of caustic solution it was suggested that the limit of 5% carbonate allowed in Federal Specification P-S631 be maintained.

Attempts to develop specification tests for NR polymer based on the viscosity of cyclohexane, gasoline, or xylene solutions or on the effect of caustic upon such solutions were unsuccessful. There was no clear relation between the viscosity of polymer solutions and the gel forming ability of the polymers. When gasoline solutions of NR (or AE) polymer are treated with aqueous caustic, a weak gel structure is formed. Attempts to measure the viscosity of these soap-free gels failed because (1) the viscosity differences were small, (2) the measurements did not appear reproducible, (3) the gels tend to stratify, and (4) the gels tend to work-harden during any such measurement. The acceptability of a polymer sample was therefore judged by examining an F-241 gel prepared from it. To facilitate manufacture, it also appeared desirable to require the polymer to be sufficiently soluble to allow complete solution in gasoline within 1/2 hour. NR polymer quality is fixed by CWS Specification 196-131-119.

To study the gasoline requirements of F-241 gels, samples of 23 commercial motor fuels were procured and tested as gel ingredients. Low temperature syneresis of these gels varied between 0 and 8%. Two gasolines gave exceptionally soft gels and five gasolines yielded friable F-241 gels. Samples of these gasolines were analyzed by the Standard Oil Development Company. Based on these results and their observations on gels prepared in many additional hydrocarbon fractions they concluded that both gel consistency and stability can be correlated with gasoline aniline point. They proposed as specification limits an aniline point between 90 and 105°F. and an octane number greater than 80. An additional specification on the "acid heat value" was considered relevant but unnecessary.

3. Control Tests

A series of F-241 gels of varying consistency were prepared by altering quality of ingredients and by slight modifications in per cent composition. The gels were tested in M-69 static firing tests by the Standard Oil Development Company. Comparison of the physical properties of these gels indicates that the parallel plate measurement is most nearly in line with firing characteristics (Table 10). The gel with the highest parallel plate value (12.9), F-537, was on the borderline of acceptability. With this gel an aging period of two weeks was required to prevent excessive scatter. The gel with the lowest parallel plate value (8.3 cm.) did not spread out and did not adhere to the target. On the basis of these tests the parallel plate measurement was suggested as a simple control test and the specification limits proposed for consideration were 9 to 13 cm. As shown by gel F-579, the Stormer viscosity does not necessarily follow the order of the parallel plate measurements. This gel was thin but tough. Its firing properties were only determined at -40°C. and at this temperature was typical of standard F-241 gels.

4. Pilot Plant Manufacture

A large number of batches of F-241 gel were prepared without incident in the continuous unit described in Section IV-B. Capacity tests indicated that this unit could produce 10 pounds of gel per minute and that the product was uniform and of high quality. The product was discharged from the mixing pump under slight pressure and could be loaded directly into munitions. The continuous type of mixing rapidly disperses the caustic solution throughout the gasoline phase and yields a slightly tougher gel than is obtained by the more time-consuming batchwise process. F-241 gel was produced in this unit to fill

Table 10

F-241Gels: Behavior in M-69 Static Firing Tests

	Firing Test Results				Physical Properties of Gel 24 Hrs. After Preparation						
			Average % Gel							60' Drop Test	
Code	Age of Gel Days	No. Bombs Fired	Adhering to Target	In Target Area	Parallel Plate (cm.)	Stormer Viscosity RPM/700g.	Mobilometer Value g./10 cm./100 sec.	Extensibility Inches	ASTM Penetrometer cm.x10^2	Adhesion %	Area of Smear Sq.Ins.
F-537	3	2	20	40	12.9	34		4.5	375	100	250
	16	2	79	94							
F-2501	10	2	60	100	12.2	17	590	2.5	330	100	175
F-2502	10	1	90	95	11.1	13	640	0.75	316	100	315
F-579	4	1	0*	100*	10.4	53	500	1.25	350	60	315
F-2503	10	2	99	100	10.3	3	2700	14.0	352	100	200
F-2504	10	2	70	99	8.3	0.4		30.0	340	0	-

F-241 Formula: 5.0% NR grade Isobutyl Methacrylate Polymer
2.5% Naphthenic Acid
2.5% Stearic Acid
3.0% of 40% NaOH
87.0% Gasoline

*Fired at -40°F.

CLASSIFIED - CONFIDENTIAL

experimental M-47 and M-69 bombs. Operation of the unit was demonstrated September 16, 1942 to representatives of three companies which had received CWS contracts to load M-69 bombs on a large scale. The unit was then circulated to these organizations for further test.

B. F-614 (IM-3) Gels

Representatives of CWS and NDRC at the fuel review at Bayway, N. J., November 19-21, 1942, rated the performance of the F-614 formula (2% AE polymer, 3% stearic acid, 3% naphthenic acid) as equal to that of the F-241 formula (in production at that time). Since use of the newer formula would allow a 60% saving in methacrylate polymer, a pilot plant study of the new formula was requested. The original sample of F-614 gel contained 3.6% of 40% NaOH. Some batches of this composition and in particular an experimental batch produced in one of the M-69 loading plants under CWS contract exhibited syneresis during storage. When the formula was revised to include 4.5% of 40% NaOH, stable gels resulted. Laboratory gels were prepared to study variations in gel composition and effect of gasoline quality. Twenty-two batches of continuously mixed gels were then prepared in the pilot plant unit and submitted for static firing tests to study quality of ingredients.

Laboratory F-614 gels were prepared in Purol gasolines of 112° and 104°F. aniline point and in Esso Base gasoline by addition of 15, 25, 35, 40 and 50% aqueous caustic solutions in amounts such that the NaOH content of the gel was equivalent to that resulting from the use of 3.6, 4.0, 4.5, 5.0 and 6.0% of 40% NaOH. The free caustic content of each of these gels was determined and the gel discarded and re-prepared if the analytical value differed from the theoretical value by more than 0.05% NaOH. The gels containing 15 and 25% caustic solutions were all friable and broke down in surveillance. The gels containing 50% aqueous caustic all appeared fluid and somewhat elastic. The gels with the highest impact strengths and lowest Stormer viscosity values contained 4.5% of 40% caustic. These values were nearly met by gels prepared from 3.6% of 50% NaOH or 5.0% of 35% NaOH. The series of gels containing 40% aqueous caustic had physical properties that were the least sensitive to variations in composition. Even in gasoline of 112 aniline point, the use of 4.5% of 40% caustic yielded stable gels. No syneresis was observed during storage at -40°F. At 125°F. the gel softened and in a one month period a fluid phase representing approximately 5% of the total separated. When the gel was cooled to room temperature, the fluid phase was reabsorbed. In the above series of gels the parallel plate and impact strength values of gels of the same improves low temperature gel properties. When fired at -40°C. the five gels prepared in Purol 98° A.P. gasoline exhibited from 1 to 22% scatter with an average value of 16%, while the percentage scatter for ten gels prepared in Purol 112° A.P. gasoline varied between 14 and 85 and averaged 47%. When fired at 70°F. the adhesion of Purol 112° A.P. gasoline-containing gels was slightly superior to that of gels prepared in Purol 98° A.P. gasoline.

For gels containing 40% caustic solution, the optimum target adhesion was observed when 4.5% of the base was present in the gel. The gel giving greatest adhesion and the least scatter at ordinary temperatures contained 6% of 35% NaOH as the gelling agent. One gel containing only 5% soap-forming acids gave excessive scatter in firing tests even one month after preparation.

The range of physical properties of gels with satisfactory firing characteristics is given in Table 11. There was excellent agreement between the measurements on pilot plant samples and their duplicates prepared batchwise in the laboratory from identical ingredients. Only two physical measurements, parallel plate value and impact strength, appear significant.

Table 11

Inspection of Continuously Mixed and Laboratory F-614 Gels Rated Acceptable in M-69 Static Firing Tests

	Range of Observed Values			
	Continuously Mixed Samples		Laboratory Duplicates	
Physical Measurement	at 24 Hrs.	At Day of Firing	at 24 Hrs.	At Day of Firing
Parallel Plate, (cm.)	8.3-11.3	8.1-11.2	8.4-12.8(1)	7.9-10.8
Impact Strength B Scale Units	4.2- 8.8	4.4- 7.9(2)	3.4- 8.2	5.7-8.6
Extensibility(in.)	0.25-6.0		1.0- 8.6	

(1) Only one gel containing NR polymer had this high parallel plate value. The next highest value was 10.8 cm.

(2) The unsatisfactory interpolymer gels gave values of 9.4 and 10.3.

composition but in different gasolines did not vary beyond the accuracy of measurement.

F-614 gels were prepared at five temperatures between 10° and 50°C. Twenty-four hours after preparation the parallel plate values of these gels increased regularly (9.2-11.3 cm.) with gelling temperature. A low gelation temperature led to an initially tougher, stiffer gel with a slightly higher than normal free caustic content. After aging two weeks all gels approached a common consistency. The samples gelled at the higher temperatures exhibit the greater changes in parallel plate values with time. There was no significant change in physical properties during the period two weeks to two months after preparation.

The data on preparation of F-614 gels in the continuous unit are summarized in appended Table 22. The nature or concentration of the following gel components were all varied: polymer, gasoline, organic acids and caustic solution. Twenty-two of these gels were evaluated in static firing tests at -40°F. and 70°F. The physical properties of the gels were determined both 24 hours after preparation and at the time of static firing tests. Gels of similar composition were prepared batchwise in the laboratory and tested in a similar manner. The data on performance tests and pertinent physical measurements are given in Table 23.

Despite variations in caustic content and gasoline quality, all gels prepared from AE polymer and from NR polymer were rated satisfactory in firing behavior at room temperature. Gels prepared from interpolymers were lacking in adhesion.

The major factor determining minimum gel strength is apparently age at the time of test. When four gels were tested at room temperature 9 to 14 days after preparation, the amount of the gel which scattered from the vertical target varied between 15% and 40%. Save for a gel containing interpolymer which was so stiff that it "bounced" and a gel containing only 5% soaps, all gels fired 15 days or more after preparation gave less than 10% scatter at 70°F. One of the gels that had been tested when 14 days old was again fired when 31 days old; scatter from the target decreased from 37% to 8%. Examination of F-614 gels after five months' storage indicates that aging after the first three weeks period does not effect an increase in gel stiffness.

The firing test data indicate the use of Purol gasoline of 98°F. aniline point (A.P.) instead of Purol 112° A.P. gasoline, The extent of scatter from the target could be directly correlated with the impact strength. Gels which exhibited more than 10% scatter from the target had impact strengths of less than 5 B scale units. Two fluid adhesive gels prepared from NR polymer had impact strengths of 5.0 and 4.2 B units and gave only 6 and 2% scatter respectively. All other gels of which at least 90% remained in the target area had impact strengths in the range of 5 to 9 B scale units. Two gels containing interpolymer bounced from the target. They were distinguished from satisfactory gels by their high impact strengths but were comparable in parallel plate value to the good gels. The parallel plate value of the gel when 24 hours old gave a satisfactory estimate of the strength of aged AE polymer gels. There was also a correlation between parallel plate value and adhesion to the target. The F-614 gels which showed the greatest adhesion to the target (60-90%) had parallel plate values in the range of 9.0-10.0 cm. The above data were presented to the Technical Division of CWS with the suggestion that parallel plate specifications on F 614 gels be set at 7.5-11.5 cm.

Eighty M-69 bombs were filled with continuously mixed F-614 gels to be used in open field ignition tests at -40° and 70°F. by the Standard Oil Development Company. The ignition observed (detailed data shown at the bottom of Table 23) was 95% which equals the performance of F-241 gels in the same munition with the same ejection and ignition charge.

Surveillance tests on the continuously mixed gels were not conclusive due to failure of the temperature control equipment in the 125°F. bath. Despite exposure to temperatures above 125°F., the average syneresis after one month was 10%. This was the maximum value for gels containing AE polymer and 4.5% of 40% NaOH. These same gels upon cooling to room temperature showed about 2% separation after 2 hours. All 20 gels tested showed absolutely no syneresis after one month at -40°C. After the 125°F. bath had been repaired, 15 samples of F-614 gels were placed in surveillance. Separation after one month varied between 6 and 1%. An increase in polymer content increased stability but an increase in the stearic acid content was without effect. Upon cooling to room temperature the separated fraction in each mixture gelled completely. Batches of F-614 gel produced under CWS contract in a commercial loading plant were found in this laboratory to give 0 to 10% syneresis after one month at 125°F. The syneretic material was reabsorbed when the gels were cooled to room temperature.

Four runs each containing six states (FC-34 to 59, see Table 22) were made in the continuous pilot plant unit to study the effect of mixing speed. In each run the speed of the centrifugal mixing pump was varied in six steps between 165 and 1725 R.P.M. When F-614 gels of specified composition were prepared, a soft homogeneous gel whose physical properties were nearly independent of mixing speed resulted. Differences in impact strengths and parallel plate values tended to level out over a two weeks aging period. There was a minimum mixing speed below which slightly weaker gels resulted, as measured one day after preparation. This speed was proportional to the rate of throughput. When the caustic concentration was altered in a manner known to give stiff, friable gels, the above effects were more pronounced. Such a gel prepared at the fastest throughput rate (8 lbs./min.) and the slowest mixing speed (165 R.P.M.) contained a few tough areas poor in caustic. This was the only case where mixing was insufficient to give a homogeneous gel. A safe mixing speed is that rate which gives the centrifugal pump a capacity one and a half times as great as that of the metering gear pumps.

C. F-624 Gels

Despite its excellent incendiary characteristics, the F-624 gel was not chosen for use in the M-69 bomb due to lack of information on its stability and technique required in its manufacture. Since the six F-624 gels subsequently placed in surveillance all showed less than 1% syneresis after one month at either -40° or 125°F., and since there was no change in physical properties of the gels over this period, it seemed desirable to study its production in a continuous unit. The only new problem involved was dispersion and pumping of alpha-cellulose fibers.

Laboratory formulation studies prior to the incendiary evaluation had indicated that with proper dispersion 0.5% of alpha-cellulose in mixtures containing 0.1 interpolymer gave gels of optimum consistency. When 0.3 interpolymer was used as a thickening agent, the gels were tough, elastic and lacked adhesion in firing tests. With this interpolymer an alpha-cellulose content of 0.25% appeared optimum. The use of AE polymer gave gels which were lacking in strength.

Gels for initial firing tests were prepared batchwise. The micropulverized alpha-cellulose was added to the gasoline-acids stock solution and the mixture stirred 5 minutes. Polymer was then dissolved in the stock solution and the agitation increased to the maximum obtainable for 30 to 60 minutes before the gelling agent was added. If the resultant gel was weak it could be attributed to insufficient agitation at this point. Three batches of gels were appraised in static firing tests. The average per cent gel in the target area was 95 for seven shots at 77°F. and 94 for 4 shots at -40°F. Adhesion to the target was not as reproducible. Observed values varied between 10 and 65% while averaging 35%.

In a study of continuous gelation of F-624 gels the dispersion of the alpha-cellulose in the stock solution was prepared in the manner described for batchwise preparations. This stock solution could be pumped and recirculated through metering gear pumps without difficulty. When attempts were made to gel the mixture by addition of caustic in the regular centrifugal pump cotton fibers collected and eventually caused binding. Continuous gelation was feasible in a centrifugal mixing pump having an open type partial web impeller and internal clearances of more than 0.25 inch. Because fluid passage ways were more constricted pumps having open type full web impellers tended to plug.

At the request of the Standard Oil Development Company, 50 gallons of continuously mixed F-624 gel and 100 M-69 cases filled with this gel were prepared and forwarded for surveillance, static firing and ignition tests. Information on the results of these tests has not been reported to this laboratory.

D. Lime Gels

The IM-1 formula was developed by the CWS for use in the M-47 bomb. At their request, a study of continuous mixing of lime gels was undertaken. This work paralleled a formulation study of lime gels in an attempt to reduce the polymer content of the IM-1 formula. For this reason the gel chosen for study in the continuous unit was neither the IM-1 nor the IM-4 gel but rather the F-1431* formula which was proposed for simultaneous consideration with the F-1429 (IM-4) gel. The results on this gel indicate that by existing techniques the continuous preparation of lime gels is impractical.

The continuous method of gelation used to prepare caustic gels requires modification for use in lime gel manufacture since an aqueous lime slurry that can be pumped would contain prohibitive amounts of water. Two techniques: (a) adding water to a polymer-acid-slaked lime mixture and (b) adding a slurry of lime in gasoline to a modified stock solution, were studied. The first method resulted in breakdown of gel structure and production of viscous fluids. An

---

*F-1431: 3% AE polymer, 3% stearic acid, 1% naphthenic acid, 3.5% CaO, 2.2% H_2O.

inhomogeneous mixture of strings of gel suspended in a menstruum of thickened gasoline was obtained if rotation of the centrifugal mixing pump were stopped. When the stock solution was run through the gear pump and the centrifugal pump of the continuous unit without simultaneous water addition, there was a marked decrease in its viscosity. Recovery from the action of the centrifugal pump was not complete in 24 hours. A laboratory study of settling rates indicated slaked lime could be slurried in gasoline if the $Ca(OH)_2$ content was at least 25%. The settling rate of the slurry was not altered by the presence of AE polymer but was reduced by the addition of 2% stearic acid. The viscosity of this slurry increased from 675 to 1100 poises during the first 1.5 hours after preparation, but remained constant thereafter. Attempts to prepare F-1431 gels by metering in 1 part of this slurry to 5.25 parts of a stock solution were made by replacing the small Zenith gear pump used with caustic solutions with a larger Zenith gear pump and finally a Gould wide clearance gear pump. Compositions of the slurry stock solution and gel follow:

	Composition (wt. %)		
	Slurry	Stock Sol.	Gel
AE Polymer	-	3.57	3.0
Naphthenic Acid	-	1.18	1.0
Stearic Acid	2	4.25	4.0
$Ca(OH)_2$	25	-	4.0
H_2O	6	-	1.0
Gasoline 98° A.P.	67	91.0	87.0

As the speed of the centrifugal mixing pump was increased from 165 R.P.M. to 1725 R.P.M., the resultant gels became weaker and more fluid. Impact strengths decreased from 14 to 10 C units and the parallel plate values rose from 7.6 to 8.6 cm. The characteristics of this lime slurry, however, do not permit pumping at a constant rate with a gear pump. The large Zenith pump clogged immediately. The high lime content caused such severe abrasion of the Gould pump that it needed reconditioning every 10-12 hours of operation. Lime also tends to settle out and to plug the pipes. The use of a less abrasive less concentrated slurry would increase sedimentation. The consistency of continuously mixed F-1431 gels is not superior to that of material prepared batchwise. The preparation of lime gels by existing continuous techniques is therefore not recommended.

VIII. Methacrylate Substitutes

In a search for gasoline thickening agents other than polymers of methacrylic esters from isobutanol or higher alcohols, a formulation study of other commercial resins was undertaken. Modification of these resins and preparation of polymers not now in commercial production were investigated. A few attempts were made to prepare polymer-free gasoline gels.

A. Commercially Available Polymers

To be an acceptable substitute for methacrylates as gasoline viscolizing agents, a candidate resin should either be more readily available or yield superior gels. The original thickening agent, natural rubber, at this time cannot be procured for this purpose. The supply of the best synthetic rubbers, such as the Buna S type, was also considered critical. A search for gasoline-soluble polymers other than methacrylates revealed only the polyvinyl ethers, Vistanex (polyisobutylene), and the rubber substitutes derived from vegetable oils. Only polyvinyl ether resins proved satisfactory in formulation studies. The use of a co-solvent to solubilize polymers in gasoline was studied with especial emphasis on ethyl cellulose. Other techniques attempted include polymerization in gasoline solution and the preparation of water-in-gasoline polymer emulsions.

1. Polyvinyl Ethers

Polyvinyl ethers stated to be in pilot-plant production by the General Aniline and Film Corporation offer promise as methacrylate substitutes. Three laboratory and one semi-works prepared samples of this type polymer were received from the above organization. The samples were coded RE-302-D-29, RE-302-D-20, RE-412-D-100, and RE-302-D-19 (Semi-Works). Their exact composition was not revealed but they were reported to have intrinsic viscosities of 29, 20, 100, and 19, respectively. The sample coded RE-412-D-100, however, gave less viscous gasoline solutions and weaker soap-fortified gels than did the other three which were nearly equivalent. Its properties were therefore not considered further.

Due to the estimated moderate quantities of this material immediately available, it seemed desirable to place major research emphasis on its use as a thickener for flame thrower fuels rather than for incendiary fillings. The former objective involved a study of approximately 5% solutions in gasoline. Based on the data to be described, a 5% solution of RE-302-D-19 in gasoline, coded F-1951, was submitted to the Technical Division of the Engineering Department of E. I. duPont de Nemours & Company for test in 1/8" unignited jet experiments. Their results are reported in their Monthly Report to NDRC (Contract OEMsr-606) for the period ending November 15, 1943. Further tests are planned or are in progress by other organizations. The preparation of incendiary fillings was studied in a preliminary manner by formulating soap-fortified gels.

Ten per cent of the RE-302 type polyvinyl ethers dissolved in gasoline to give strong elastic gels. A reduction in polymer content results first in a weaker gel and ultimately in a viscous thixotropic solution. The viscosity of polymer solutions in gasoline is quite sensitive to concentration but is little affected by temperature changes. Surveillance tests of these solutions (5 and 10%) indicate no syneresis tendency at -40° or 125°F. The room temperature viscosity of a 5% solution was unchanged after the material had been held at 140°F. for one week. To determine polymer stability at elevated temperatures, 1% cyclohexane solutions of two of the samples were stored at 150°F. for four weeks. The viscosity of one (RE-302-D-20) showed substantially no decrease while the viscosity of the other (RE-302-D-29) decreased approximately 50%. Polymer degradation would be anticipated only if the acidic polymerization catalyst was not effectively removed.

A study of the viscosities and burning rates of polyvinyl ether RE-302-D-19-gasoline solutions and of Napalm-gasoline solutions was made as a basis for comparing the two types of mixtures as flame thrower fluids. The results are given in Table 12. It was observed that the Napalm solutions attained their maximum burning rate very shortly after ignition, whereas the polyvinyl ether solutions did not attain maximum burning rate until approximately 20 seconds after ignition. The Napalm solutions are nearly completely burned after 2 out of the total 3 to 3.5 minutes required for complete combustion, while the polyvinyl ether solutions burn at a fairly constant rate up until the last half minute. This suggests that with polyvinyl ether solutions less of the fuel will be consumed before reaching the target than with Napalm solutions. Based on the viscosity determinations given in Table 12, the maximum concentration of polyvinyl ether proposed for initial tests in flame thrower fuels is 5%.

The samples of RE-302 polyvinyl ether were assessed as thickeners by substitution for methacrylate polymer in the F-241, F-107 and F-614 formulas (5, 3, and 2% polymer, respectively, 2.5-3.0% stearic and naphthenic acids). Both the F-241 and F-107 formulas give gels which resemble their methacrylate prototypes in strength and stiffness but heal and flow more readily. The F-241 type gel prepared with the semi-works sample of polymer had a parallel plate value of 11.8 cm. and an impact strength of 5.7 C units. These properties are in the optimum range for M-69 incendiary fillings. The F-614 type gels made with these resins are too fluid to be of interest. In all formulas the three polyvinyl ether samples which differ in intrinsic viscosity give similar results. The F-241 and F-107 type gels gave no evidence of syneresis in -40° and 125°F. surveillance tests. Both appear to be excellent incendiary fillings.

Table 12

Polyvinyl Ether and Napalm Solutions in Gasoline

Comparison of Consistencies and Burning Rates

Composition	Modified Stormer Viscosity R.P.M./800 g.	Gardner Mobilometer Viscosity g./10 cm./1.5 min.	Burning Times Method A, min.
3% Napalm	769	20**	2.7
4% "	455	48**	3.0
5% "	425	117	3.3
6% "	400	249	3.4
4% RE-302-D-19*	480	59	3.8
5% "	400	96	4.1
6% "	300	165	5.1
7% "	160	220	5.7

* RE-302-D-19 - A polyvinyl ether prepared by General Aniline and Film Corp.

** Extrapolated values.

The polyvinyl ether samples all swell in turpentine. When used in 5% concentration RE-302-D-29 gave a strong gel structure.

2. Vistanex and other Synthetic Rubbers

The viscosity of 8% smoked sheet rubber in gasoline gels may be obtained with Vistanex (polyisobutylene produced by the Standard Oil Company of New Jersey and marketed by Advance Solvents and Chemical Company) solutions but concentrations of 30-60% are required. Twelve Vistanex soap gels containing 2-6% Vistanex (140,000) with combinations of stearic, oleic, naphthenic and rosin acids were prepared. They were all weak and inelastic.

To introduce functional groups capable of reacting with caustic, a sample of Vistanex was sulfonated by treatment with oleum. The product was charred and darkened on standing. Gasoline solutions of the sulfonated product showed only a very slight gel structure when treated with caustic. The use of the sulfonated Vistanex in place of AE polymer in the F-614 gel gave a very weak, friable gel.

Attempts to cross-link Vistanex in solution with di-isocyanates or with sulfur in the presence of accelerators and zinc oxide at 50°C. and 90°C. did not cause thickening of the polymer solutions. Similar reactions on 0.3 interpolymer were also unsuccessful. The introduction of functional groups into Vistanex either during or after polymerization warrants further study.

Several polyesters of ethylene glycol with dimerized linseed and soybean oil acids (Kempol 11, Norepol and precured Norepol, the latter two supplied by the Northern Regional Research Laboratory of the U. S. Department of Agriculture) were evaluated as possible polymer substitutes. Addition of caustic to gasoline solutions containing 10% of either Kempol 11 or Norepol resulted in no gelation. Addition of either 3% each of stearic and naphthenic acids or of 2% each of wood rosin, stearic acid, and naphthenic acid before gelation produced typical polymer-free soap gels--stiff, friable, weak, and tending to synerize at room temperature. Precured Norepol was insoluble both in gasoline and in a gasoline (80 parts by weight)-benzene (10 parts by weight) mixture.

A resin of undisclosed composition labeled T-132 Resin - Stanco Distributors - was found to be completely soluble in toluene, very nearly completely soluble in turpentine and cyclohexane, and insoluble but tending to swell strongly in gasoline. Five per cent solutions of the resin were slightly more viscous than the pure solvent and upon shaking work-hardened slightly. The addition of caustic caused strong gelation in toluene and turpentine solvents. Only a few soap-fortified gels were prepared and further work appears justified if this resin is available. The resin could be dissolved in gasoline-toluene mixtures containing a minimum of 20% toluene; on gelation with caustic, however, only weak soap-type gels were obtained.

3. Ethyl Cellulose

Considerable attention has been given to the incorporation of commercial ethyl cellulose into incendiary fillings.

While this material is not soluble in gasoline, viscous solutions could be obtained by adding ethyl cellulose to gasoline-organic solvent mixtures. The minimum amounts of various auxiliary solvents required to solubilize the resins were determined. Soap-fortified gels were then prepared by addition of organic acids and aqueous caustic to the polymer solutions. The physical properties and surveillance characteristics of these gels were determined and the four most promising gels underwent M-69 static firing tests. On the basis of these results it was concluded that without further modification ethyl cellulose does not impart sufficient strength to gasoline-soap gels to prevent excessive scattering in static firing tests and that the use of a water miscible auxiliary solvent results in poor high temperature gel stability and an increased variation in gel consistency with temperature.

a. Solubility Studies

The minimum amount of various solvents required to dissolve ethyl cellulose in gasoline was determined by tumbling for at least 24 hours gasoline-resin mixtures with graduated amounts of the solvent in question. It was found that for each solvent the minimum solvent requirement increased at most by 1% when the resin concentration in the mixture was increased from 2 to 10%. Furthermore, for samples of ethyl cellulose graded by the manufacturer as "200, 100, and 20 centipoises" and "7 secs.", the grade of ethyl cellulose did not influence the minimum solvent requirements by more than 1%. The minimum amounts of solvent in Esso Base gasoline which will solubilize 5% of 100 cps. ethyl cellulose are as follows:

Solvent	Minimum Required	Solvent	Minimum Required
n-Butanol	4%	95% Ethanol	23%
iso-Propanol	8%	Methyl Ethyl Ketone	26
Methanol	10	Benzol	35
Ethyl Acetate	19	Methyl Propionate	36
Acetone	20	Xylol	>50
i,n-Butyl Acetate	20	Turpentine	>>20
Acetic Acid	22	Ethyl Ether	>>20

In general, the addition of naphthenic acid reduced the auxiliary solvent requirement slightly.

b. Gel Preparation

Approximately 110 soap-fortified ethyl cellulose gels were prepared and examined. Of these, 50 used acetone as the auxiliary solvent, 40 contained low molecular weight alcohols (methanol to n-butanol), 10 were made in benzene-gasoline mixtures, and the remainder were made with miscellaneous solvents. These gels contained various combinations of the following organic acids: naphthenic, oleic, stearic, abietic, and Turkey Red Oil acids, and 40% aqueous caustic normally in an amount equal to 150% of that required for neutralization of the organic acids. As judged by visual observation, the spatula test, and in some cases physical measurements, the most promising gel formulas are listed in Table 13.

Table 13

Ethyl Cellulose Gels

Gel No., F-	2386	2344	2399	2369	2384
Composition, Wt. %					
Ethyl Cellulose 200 cps.	5	5	5	5	4
Acetone	15	15	20	-	20
Gasoline	71	73.2	65.7	80	67.1
Hydrofol 51	3.5	2	1	2	2.5
Naphthenic Acid	-	2	1	2	2.5
Turkey Red Oil	1.8	-	-	-	-
Oleic Acid	-	-	4	-	-
40% NaOH	3.7	2.8	3.3	3.5	2.9
Isopropanol	-	-	-	7.5	-
AE Polymer	-	-	-	-	1
Surveillance					
% Syneresis After					
1 month at -40°	0.5	1.0	0.7	0.3	0
1 month at 125°F.	0	0	40	6	6
Static Firing Tests at 70°F.					
% Adhesion	53	-	40	30	23
% in Target Area	53	-	70	35	50
at -40°F.					
% Adhesion	40	-	3	0	55
% in Target Area	50	-	60	60	65

When the formula included less than the minimum amount of auxiliary solvent required for solution of the resin, weak, friable jellies resulted. As the auxiliary solvent content was increased, the gels became softer and less friable. At the optimum solvent concentration stiff pastes resembling the F-241 gels were obtained. A large excess of organic solvent resulted in soft, weak creams. The minimum solvent concentration that would produce non-friable, stiff pastes rather than friable jellies could be brought down to the minimum solvent concentration required for solubility as determined in the preceding section by proper adjustment of the caustic concentration. Thus by increasing the caustic content in Hydrofol-naphthenic acid gels from 2.3% (50% excess NaOH) to 3.3%, the minimum solvent that would result in good gels was reduced from 20 to 15% in the case of acetone and from 40 to 10% in the case of isopropanol. The particular solvent used apparently influenced stiffness and friability but not strength of the resulting gels. Methanol gave very friable gels which separated at room temperature.

The samples of ethyl cellulose graded as 20 and 100 cps. gave more friable gels than did the 200 cps. material. This latter grade was used in most gel studies.

As fortifying agents the soaps of the following combinations of acids appear most promising: stearic-naphthenic; oleic; oleic-naphthenic; and Turkey Red oil with either or both stearic and naphthenic acid. Turkey Red oil and oleic acid both imparted unusual strength to ethyl cellulose gels.

c. Surveillance Tests

The stability and consistency of ethyl cellulose gels were observed after storage at -40°F. and +125°F. In general the consistency of these gels differed markedly at these temperature extremes, being quite stiff at low temperatures and soft at 125°F. The nature and concentration of the auxiliary solvent was a major factor in determining the surveillance characteristics of the gels.

All ethyl cellulose gels containing an alcohol as an auxiliary solvent which were put in surveillance broke down rapidly at 125°F. to thin liquids. Alcohols tested include methanol, isopropanol and n-butanol. Apparently at the high temperature alcohols completely dissolve resin and soaps into the gasoline phase.

Gels containing benzene failed at low temperature due to separation and freezing of the benzene.

While most acetone-ethyl cellulose gels have excellent low temperature properties, many of the 20 gels of this type tested were not stable at 125°F. The use of 200 cps. ethyl cellulose, a low caustic content and 15% rather than 10 or 20% acetone concentration all favor high temperature stability. Formulas F-2386 and 2344 (see Table 13) showed no separation or fluidity after one month at 125°F. The use of oleic acid invariably led to rapid breakdown of the gel at high temperatures.

d. Firing Tests

As shown in Table 13, three ethyl cellulose gels containing no methacrylate polymer were evaluated in M-69 static firing tests. A good methacrylate gel would give at least 70% adhesion and 95% remaining in the target area at room temperature and low adhesion, but at least 90% in the target area at -40°F. It is readily seen that ethyl cellulose gels which gave from 30-60% scatter even at room temperature are considerably weaker than the methacrylate gels. A result of interest is the excellent adhesion even at low temperatures exhibited by gel F-2386 containing Turkey Red oil. This is rated the best ethyl cellulose gel tested.

e. Ethyl Cellulose as a Methacrylate Extender

Twelve gels were prepared containing both ethyl cellulose and 1% isobutyl methacrylate polymer in an attempt to decrease methacrylate requirements for M-69 fillings below 2%. In strength and consistency the mixed polymer gels all resembled gels of a similar formula containing no ethyl cellulose. One such mixed polymer gel (F-2384, Table 13) gave very poor results in static firing tests. These data lend weight to the theorem that only resins which react with the gelling agent and thus bind the two-phase system will increase the strength of gasoline-soap gels.

f. Modification of Ethyl Cellulose

Treatment of ethyl cellulose in dioxane at the boiling point with phthalic anhydride gave a polymer whose viscosity in an inert solvent ($CHCl_3$) increased markedly on addition of aqueous caustic (Table 14). When this resin was dissolved in an 80-20 gasoline-acetone mixture, the viscosity increase on addition of caustic was slight and less than that observed with untreated ethyl cellulose. It was also found that the presence of 20% acetone in gasoline prevents gelation of caustic-isobutyl methacrylate-methacrylic acid interpolymer gels. These data indicate that water-miscible auxiliary solvents will interfere with the phase relationships in gasoline mixtures gelled with aqueous caustic. The decrease in viscosity of resin solutions caused by treatment with phthalic anhydride indicates that breakdown of the ethyl cellulose occurred.

Table 14

Modification of Ethyl Cellulose by Phthalic Anhydride

	100 cps. Ethyl Cellulose	Ethyl Cellulose-Phthalic Anhydride Modified
5% Solution in $CHCl_3$		
Viscosity, Poises	3.35	1.25
Viscosity when 1.2% of 40% NaOH added, poises	10.0	6.0
Viscosity Increase, %	199	475
5% Solution in 29.5% Acetone-70.5% Esso Base		
Viscosity, poises	0.85	<0.50
Caustic Viscosity, poises	3.85	2.10
Viscosity Increase, %	353	300

In dilute gasoline solutions treatment of ethyl cellulose with SO_2Cl_2 or $POCl_3$ did not introduce polar groups effective in strengthening the gel structure. Only weak gels were obtained on reaction of caustic with a 5-20-75 ethyl cellulose-acetone-gasoline mixture which had been treated with SO_2Cl_2 or with 5-20-2.5-2.5-70 ethyl cellulose-acetone-Turkey Red oil-stearic acid-gasoline mixtures which had been treated with SO_2Cl_2 or $POCl_3$.

4. Miscellaneous Polymers

Solubility measurements have been made on several representative types of polymers in gasoline and other available hydrocarbon solvents. While no polymer not previously discussed showed sufficient solubility in gasoline to be used as a direct substitute for polyisobutyl methacrylate, some of the polymers could be solubilized by an auxiliary solvent. The viscosities of mixtures of polymer solutions were found to be the arithmetic mean of the viscosities of each resin solution alone. The use of solvents other than gasoline did not produce encouraging soap-fortified gels with the polymers under consideration.

The effect of gasoline on representative polymers is shown in appended Table 24. In tests for suitable auxiliary solvents it was found that 35% of isopropanol or methanol would allow solution of 5% Butacite (polyvinyl butyl acetal) in gasoline. Soap-fortified gels were prepared in isopropanol-gasoline mixtures but they were friable and exhibited syneresis at room temperature. The solubilities of polyvinyl acetate and polyvinyl acetal in gasoline-solvent mixtures were determined using the same group of solvents as was used with ethyl cellulose (See Section 3-a). No clear solution of either of these two vinyl polymers was obtained in gasoline mixtures containing 40% or less auxiliary solvent.

To determine possible thickeners for petroleum fractions higher boiling than gasoline, the solibility of 7 polymers was measured in four refinery still cuts (22-95% aromatics). As shown in Table 24, polyisobutylene was soluble in the four solvents while polystyrene swelled in three and was soluble in the most highly aromatic mixture. Ethyl cellulose swelled in all but gelled only in the most aromatic fraction. Napalm, incidentally, swelled in these petroleum fractions to give gels more tacky and less friable than its gasoline gel. The remaining polymers tested were insoluble. At the request of the Permanenti Magnesium Company, the solubility of polymers in a Stoddard solvent-asphalt mixture used as the fluid base of their magnesium pastes was investigated. Only Vistanex would dissolve in this material.

The use of toluene and turpentine as base solvents further extends the list of soluble polymers. Butacite (polyvinyl butyral) and ethyl cellulose swell strongly in turpentine but do not form continuous gels. Dow Styron (polystyrene) is soluble in both toluene and turpentine. "Lucite" scrap (polymethyl methacrylate) was tested in toluene and mixtures of toluene with gasoline and turpentine. The most homogeneous solutions approaching gel like consistency, arranged in order of apparent increasing viscosity, were:

% Methyl Methacrylate	% Toluol	% Gasoline	% Turpentine
10	80	-	10
10	80	10	-
10	90	-	-
20	80	-	-

Dow Styron and "Lucite" scrap were formulated in the IM-2 and IM-3 type gels using toluene and turpentine as solvents. Styron gave extremely thin gels while "Lucite" scrap led to soft, weak gels. Turpentine discolors on addition of caustic.

Polystyrene, nitrocellulose, ethyl cellulose and Butacite were dissolved in suitable organic solvents and gasoline was added until an amorphous precipitate of polymer swollen by gasoline was formed. These precipitates did not swell to homogeneous gels when dispersed in gasoline.

5. Emulsions

Emulsification of polymer solutions in gasoline gave stable creams lacking in strength. Thus stable emulsions of 2-10% aqueous solutions of partly hydrolyzed polyvinyl acetate were obtained when the gasoline content of the emulsion was 86% or less. No emulsion was obtained when 12.5% solutions of polyvinyl acetate in methanol were dispersed with gasoline. In unsuccessful attempts the order of mixing was varied and glycol and glycerine were added. Emulsions of NR polymer could not be dispersed in gasoline even on incorporation of caustic.

6. Factices

Attempts were made to dissolve factices (polymerized natural oils) in gasoline or to prepare them in gasoline solution. No satisfactory gels resulted.

The following commercial factices obtained from Stanford Rubber Supply Company did not dissolve or swell in Blue Sunoco gasoline: Neophax A, AA White, Amberex Type B, and #15 Brown. Alkyd resins formed from linseed oil, which in 20% concentrations gave viscous gasoline solutions, were heated at 250-280° until gelation occurred. After cooling they were tumbled with gasoline. The products swelled in part but no homogeneous mixture could be obtained. Linseed oil was gelled by heating at 250-280°C. This gelled oil gave a grainy solution in gasoline.

In attempts to vulcanize oils and form factices in situ, 10 to 20% solutions in gasoline of corn oil, linseed oil, and oiticica oil were treated with S_2Cl_2 (40% by weight of oil). The solutions darkened but no gels or insoluble materials formed. When these solutions were evaporated and the residue treated with S_2Cl_2, a vigorous reaction yielded an insoluble factice. This indicates vulcanization did not occur in the dilute oil solutions. When 40% corn oil in gasoline was treated with S_2Cl_2, a jelly was obtained which reverted to an oil in one day.

B. Preparation of Polymers not now in Commercial Production

The review of existing commercial polymers did not reveal any material available in large quantities that met the requirements of a methacrylate substitute. A second approach was to study the synthesis of polymeric materials or the modification of existing products so as to produce a resin with characteristics which existing data indicate are desirable. This study was limited to reactions that could be accomplished in simple plant equipment.

Two basic types of polymers appear useful as gasoline thickening agents. The first is a linear polymer like isobutyl methacrylate whose fluid gasoline solutions gel on addition of aqueous caustic, and the second a cross-linked polymer such as rubber or the polyvinyl ethers which swell rather than dissolve to give viscous gasoline gels. On the basis of the work on methacrylates, ethyl cellulose, and polyisobutylene, it is believed that the ideal methacrylate substitute of the first type would be (a) gasoline soluble, (b) of high molecular weight, and (c) contain functional groups capable of reacting with the gelling agent. A substitute of the second type (requirement (c) not met) would involve considerably higher molecular weight resins.

The solubility criterion eliminates the polyamides and highly cross-linked polymers such as urea-formaldehyde, phenol-formaldehyde, and most of the alkyd resins. The major types of polymers which offer promise are the polyesters, vinyl esters, vinyl acetals, and cellulose ethers and esters. The initial problem is to incorporate substituents (long paraffin chains) on these polymeric chains that will produce gasoline solubility. The second phase of the problem is the introduction of reactive groups. Typical of the materials whose preparation was contemplated are cellulose stearate phthalate, butyl cellulose modified by etherification with hydroxyacetic acid, the vinyl acetal prepared from a mixture of 2-ethyl hexaldehyde and glyoxalic acid, and vinyl stearate salicylate. While gasoline soluble cellulose and vinyl resins could be prepared with difficulty, degradation occurred during introduction of functional groups so that only low molecular weight materials were obtained.

1. Cellulose Esters

Cellulose tristearate was successfully prepared by reacting at 125-140°C. alpha-cellulose with a large excess of stearyl chloride in xylene solvent in the presence of pyridine catalyst. The amorphous product was difficultly soluble or became swollen in hot gasoline and was soluble in naphthenic acid. The use of a quinoline catalyst containing a trace of iodine gave an exothermic reaction which could not be controlled; the low molecular weight product gave very fluid gasoline solutions. Since the high molecular weight cellulose esters proved only difficultly soluble and were readily degraded by further treatment, the modification of the triesters to incorporate acidic groups was not attempted.

The laboratory study of the preparation of cellulose esters is summarized in Table 25. Alpha-cellulose, sodium cellulose, rayon and acid pretreated cellulose were all studied as starting materials. Only alpha-cellulose gave high molecular weight products. The principal requirements on the solvent were that it dissolve the product and that it boil above 130°C. Xylene was preferred but tetrachlorethane was especially effective in converting the distearate to the tristearate. The only catalyst which caused the formation of a light colored product was pyridene. Both quinoline and alpha-picoline caused considerable decomposition and the formation of a dark residue. Acid catalysts were less effective.

Cellulose distearate and cellulose dilaurate were prepared by the use of lower temperatures (135°C.) and lower concentrations of catalyst and acid chloride than were ultimately found necessary for the preparation of the tristearate. The cellulose diesters did not swell in gasoline and did retain the fibrous shape of the alpha-cellulose while further substitution yielded an amorphous powder. The cellulose distearate could be converted into the tristearate by continuation of the reaction at higher temperatures. Attempts to solubilize the di or tristearate by treatment with $POCl_3$, stearyl chloride, or stearic acid tended to degrade the polymer.

A few gel preparations were made with two samples of cellulose tristearate. The high molecular weight material (S-21) was not sufficiently soluble to act as a thickening agent while the low molecular weight product (R-3) behaved as would stearic acid. Only weak, friable gels were obtained.

2. Polyvinyl Acetals

The synthesis of polyvinyl acetals is summarized in Table 26. A gasoline soluble polyvinyl acetal (Runs V, A-1, A-2) can be prepared by heating polyvinyl alcohol with 100% excess 2-ethyl-hexaldehyde (the longest chain aldehyde commercially available) for 19 hours at 70-80°C. in a mixture of butyl acetate and butyl alcohol with sulfuric acid catalyst. This product had an acetal content of 80% which is the theoretical maximum*. Experiments with other diluents and catalysts produced a gasoline insoluble type of acetal probably due to a lower (70%) degree of substitution.

Attempts to introduce acidic groups into the polymer by simultaneous or subsequent reaction with ortho-hydroxybenzaldehyde (A-3,4) or hydroxystearic acid (A-5) were unsuccessful. Apparently the large excess of 2-ethyl-hexaldehyde

---

*Random spacing of the acetal groups on the polyvinyl chain and consequent isolation of occasional hydroxyl groups make higher acetal contents improbable.

required to obtain a gasoline soluble product prevents reaction of the polymer with the other reagents.

The polyvinyl acetal of 2-ethyl-hexaldehyde did not have a sufficiently high molecular weight to gel gasoline when present in concentrations of 5-10%. The addition of aqueous caustic to these gasoline solutions caused a large increase in viscosity but no gelation. Gels made with 8% of this polymer combined with stearic and naphthenic acids had excellent adhesion but were too soft and weak to warrant further study.

3. Polyvinyl Esters

A number of reactions designed to yield polyvinyl stearate or oleate and employing polyvinyl acetate, polyvinyl alcohol, polyvinyl chloride and vinyl acetate monomer as raw materials were tested (Table 27). Only the reaction between polyvinyl acetate and stearic acid gave the desired product and in this one case (S-3) the reaction conditions were so severe (150-175°C. at 30 mm. pressure for 24 hours with o-toluene sulfonic acid catalyst) that excessive degradation occurred.

4. Polyhydroxystearic Acid

Samples of polyhydroxystearic acid having neutral equivalents of 2500 and 4600 were prepared by heating hydroxystearic acid several days at 200°C. in the presence of traces of adipic acid and octadecanediol as catalyst (procedure described in U. S. Patent 2,147,647). A sample of higher molecular weight was received from the Standard Oil Development Company. In moderate concentrations (10% or less), gasoline solutions of the two higher molecular weight samples do not gel on the addition of caustic, while the low molecular weight product gave a very weak soap gel. Fight gels made to appraise these polyesters, both as methacrylate substitutes and as soap-forming acids, proved to be substantially identical with equivalent formulations containing no hydroxystearic acid polymer.

C. Polymer-Free Gels

This section briefly outlines gelling experiments with miscellaneous materials. When gels were obtained they were weak and had a high solids content.

Gilsonite and other soft asphalts do not toughen rosin soap gels in gasoline. The addition of stearic acid to a gasoline solution of asphalt produced no gel structure. When cumar gum and cottonseed pitch were dissolved in rosin-gasoline mixtures the addition of aqueous caustic yielded weak, friable gels resembling the resin-free soap gels. The following materials were insoluble in gasoline: ester gum, pine tar, Rezyl #12 and #19, and Advagum (Advance Solvents & Chemical Company).

At 140°F. cyclohexanol stearate will dissolve in gasoline in concentrations up to 10%. When these solutions cool to room temperature, a soft, weak gel results.

As indicated in U. S. Patent 879,193, a mixture of 1 part sodium stearate to 4.5 parts sodium silicate will gel gasoline. To obtain a homogeneous gel, however, approximately a 50% solids content is required.

When kneaded into gasoline or gasoline-kerosene mixtures, active carbons will form heavy pastes. In this pigment type filled paste 40% carbon black is the minimum concentration that will yield a non-flowing mixture.

The reaction between indandione and laurylamine in gasoline solution yielded a weak, friable gel when the concentration of the organic reactants was 10%.

Attempts to prepare gasoline soluble materials by Friedel Crafts condensation of adipyl chloride with aromatic hydrocarbons gave only insoluble tars.

Prepared by ______________________
E. C. Kirkpatrick

Approved by John S Beekley
John S. Beekley
Official Investigator

6/6/44

APPENDIX A

Physical Test Procedures

The following detailed procedures supplement the discussion of test methods given in Section IV.

1. Parallel Plate Consistency Test

The apparatus consists of two pieces of plate glass, 12 inches x 12 inches, contained in an open-top box. It is convenient to have a paper with four lines crossing at 45° angles placed under the lower plate with the intersection directly below the center. A 2" circle may be centered on the upper plate. 5.0 cc. (±0.1 cc.) of gel are transferred to the center of the lower plate. A calibrated plug syringe as described below and in Figure 5 may be employed. The top plate is loaded centrally to bring its total weight up to 2.0 kilograms (±10 grams). One edge of the top plate is allowed to rest on an edge of the bottom plate; the other edge of the top plate is lowered gently until the sample resting on the lower plate is spread to a two inch diameter circle, and is then released. The extremities of four equally spaced diameters of the circle of gel are marked with India ink dots between 55 and 65 seconds after the top plate has been released. The plates are then separated and cleaned with methanol. The diameters are measured to the nearest millimeter, and the average is recorded as the result of the test. An average of the results of three parallel plate tests on a gel is taken as the final value. Any serious irregularities of the circle formed should be noted. The test should be run at a temperature of 25°C. ±3°C.

The plug syringe suggested for use with the parallel plate test is composed of two concentric pieces of glass tubing, a seal between the two being made by an annulus of neoprene tubing. Dimensions are given in Figure 5. The larger diameter glass tubing serves as a cylinder, which should be calibrated to deliver the required amount of incendiary filling. The smaller diameter tubing, to which a section of neoprene tubing is held by friction, serves as a piston to remove the incendiary filling from the syringe.

In order to withdraw a sample, remove the piston from the cylinder, insert end "A" of the cylinder into the material to be tested, and apply suction to the other end until the cylinder is about two-thirds full. Insert the piston in the cylinder, pushing the piston down to meet the surface of the sample, allowing the air to escape through the bore of the piston. (The piston operates more smoothly if moistened.) Push the piston in to the calibration mark on the cylinder, cut off the sample flush with the end, then place a finger over the opening at "B" and force out the sample onto the lower parallel plate. Any sample adhering to the piston can be transferred to the plate by means of a spatula.

2. Modified Stormer Consistency Tests

The equipment is described in an article by Geddes and Dawson (J. Ind. Eng. Chem., 34, 163 (1942)-). A sample of gel which has not been worked in twenty-four hours is transferred to the modified Stormer cup taking normal precautions to prevent inclusion of air. The cup is filled nearly to the top and the modified Stormer paddle is then immersed in the gel to a depth indicated on the paddle shaft. After the gel has rested 30 seconds, a load of 1400 grams, or for very thin gels 800 grams, is applied. This load gives the gel an initial working of 10 revolutions. The time required for a succeeding 100 revolutions or in the case of very tough gels the revolutions made in 5 minutes is then measured. This is reported as R.P.M./ 1400 g. With the F-241 and F-107 gels the rate is nearly constant throughout the 100 revolutions. With a gel which work-hardens, the paddle will speed up and slow down in an obvious manner many times during the course of a test. Some gels will break under the load. The rate of rotation will be very slow during the first approximately 10-20 revolutions and then will rapidly increase. A very large part of the time required for the 100 revolutions will have been consumed during the first slow period. This effect is most marked with gels containing high concentrations of soaps. Besides the numerical figure, the Stormer consistency may, therefore, bear the notation uniform, work-hardened, or broke.

3. Burning Rate Tests

The gel is burned outdoors in a draft-free area provided by an open topped "Transite" cubical box 1 yard to a side.

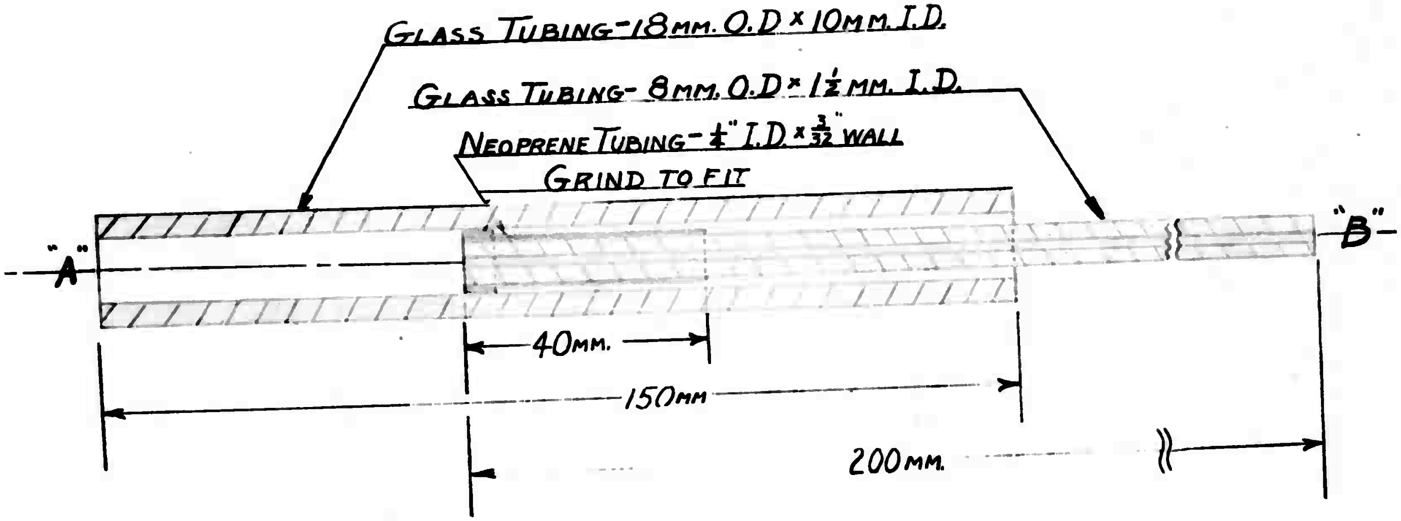
a. Method A

A 100 gram sample of the gel to be tested is spread uniformly over the inverted top of a 2 quart friction top can which is 7 inches in diameter. The top acting as a tray is placed in the center of the floor of the "Transite" box. The gel is ignited and the duration of the flame determined by means of a stopwatch. The average of three values is reported as the burning time. The weight of the residue was sometimes determined.

Figure 5

PLUG SYRINGE

CLASSIFIED - CONFIDENTIAL



CLASSIFIED - CONFIDENTIAL

b. Method B

Very strong gels cannot be spread in a reproducible manner over the 7 inch circular area. A 50 gram sample is therefore molded into a 2-3/4 inch cylinder by allowing the gel to stand in an ointment jar. The jar is then inverted and the molded sample placed in the center of the 7 inch can top and ignited. Not only the burning time but also the tendency of the gel to flow during combustion are observed. None of the gels spread sufficiently to completely cover the seven inch circle.

APPENDIX B

Summary Sheets - Tables 15-27

Information of a factual nature has been summarized in tabular form in this section. The tables are arranged in the order to which they are referred in the text. An overall view of the various gel ingredients tested can be gotten from Table 15 which lists the source and value of the materials tested and from Table 18 which includes the composition and stability of experimental gels placed in surveillance. In the tables the abbreviation I.P. for interpolymer is used.

The notebooks in which the original data have been recorded have been deposited in the Ammonia Department files and are available for reference. These are N.B. 884, 965, 971, 973, 978, 998, 1049, 1059, 1118.

CLASSIFIED - CONFIDENTIAL

Table 15

Materials Used in Gel Formulation Studies

Use	Composition	Trade Name or Grade	C.W.S. Specification	Source	Qualities Imparted to Gasoline Gels	Remarks
A. Materials Which Proved most Useful in Present Study						
Polymeric Thickener	Polyisobutyl methacrylate	NR Grade AE Grade	196-131-119 108	E. I. duPont de Nemours & Co., Ammonia Dept.	Toughness.	
	Interpolymers of isobutyl methacrylate with 0.1, 0.2 and 0.3% of methacrylic acid respectively.	0.1 Interpolymer 0.2 " 0.3 "		" " " " " " " " " " " "	Toughness & elasticity.	Experimental products.
	Polyvinyl ether	RE-302		General Aniline & Film Corp.	Viscosity - See Section VIII.	
	Aluminum soaps	Napalm		Nuodex Products Co.		
Soap-Forming Acids	Stearic acid (hydrogenated fish oil)	Hydrofol 51	123	Werner G. Smith Co., Cleveland, O.	Body, stability, hardness.	Other grades include Hydrofols 46, 300, 305, 400, 405.
	Stearic acid	Triple Pressed		E. I. duPont de Nemours & Co., Organic Chemicals Dept.		Double and single pressed.
	Naphthenic acid	Nuodex 240	104	Nuodex Products Co., Elizabeth, N. J.	Fluidity and strength.	
	Wood rosin	W.G.		Hercules Powder Co., Wilm., Del.	Fluidity and instability.	
	Oleic acid	U.S.P.		Merck & Co., Rahway, N.J.	Body.	
	Sulfonated castor oil	Turkey Red Oil		Hercules Powder Co.	Fluidity.	
	Dimerized soybean oil acids	Experimental			Fluidity and strength.	Prepared by hydrolysis of dimethyl esters from USDA Northern Regional Research Lab., Peoria, Ill.
Gelation Agent	NaOH	Technical		Mallinckrodt Chemical Works		
	Ca(OH)$_2$	U.S.P.		" " "		
	CaO	U.S.P.		" " "		
Fuels	Gasoline, commercial motor use	Pyrol Naphtha #2	124	Pure Oil Co., Heath Refinery, Newark, Ohio	Sect. V.	
		Esso Winter Base		Standard Oil Development Co., Bayway, N. J.		
		Blue Sunoco		Sun Oil Company filling stations.		
	Toluene	Toluol		E. I. duPont de Nemours	Stability.	
Filler	alpha-cellulose				Stiffness and inelasticity.	Ground in micropulverizer.
B. Materials of Possible Value for Future Study						
Polymeric Thickeners	Polyisobutylene	Vistanex 100,000		Advance Solvents & Chem. Co.	See Sect. VIII.	
	Ethyl cellulose			Hercules Powder Co.		
Soap-Forming Acids	Cottonseed oil acids			Woburn Degreasing Co. of N.J.	Same as by stearic acid.	
	Soybean oil acids			" " " "	" " " " "	
	Sardine oil acids				" " " " "	
	Tall oil acids	Ligro, Indusoil		W. Va. Pulp & Paper Co., N.Y.	" " " " "	
	Myristic acid	Myristilene		The Beacon Co., Boston, Mass.	" " " " "	
	Ricinoleic acid	U.S.P.		Merck & Co., Rahway, N.J.	" " " " "	Alan Baker's P-20 Grade.
	Dimerized wood rosin	Poly Pale Resin		Hercules Powder Co.	Same as by wood rosin.	
	Hydrogenated wood rosin	Staybelite		" " "	" " " " "	
	Unknown	T-132 Resin		Stanco Products Co., Elizabeth, N.J.	" " " stearic acid.	
Gelation Agent	Ca(OH)$_2$ (31% MgO)	Fertilizer Grade			Slightly inferior to U.S.P.	
	KOH	Technical		Mallinckrodt Chem. Works	Same as by NaOH.	
	LiOH	"		" " "	" " "	
	Ba(OH)$_2$	U.S.P.		" " "	" " " Ca(OH)$_2$	
Soaps	Aluminum naphthenate			Eimer & Amend, N.Y.	Body in the absence of base.	
	Aluminum palmitate			" " "	See Section V.	
	Aluminum stearate			" " "		
	Lead laurate	Experimental chemical.		From duPont Research Groups.		
Fillers	Activated charcoal	Darco G-60		Delaware Chemical Co.	See Section V.	
	Dextrose			Eimer & Amend, N.Y.		
C. Other Materials Tested						
Polymeric Thickeners	See Section VIII and Table 24.					
Soap-Forming Acids	Adipic acid	Laboratory reagent.		See Note.	Gasoline insol.	
	Benzoic acid				Stiff, non-adhesive, weak.	
	Phenol				Instability.	
	Phthalic acid				Gasoline insol.	
	12-Benzoyl hydroxystearic acid	Experimental Chemicals		From research groups within E. I. duPont de Nemours & Co.	Same as by stearic.	
	2,4-Dimethyl pentanoic acid				No gelation.	
	4,6-Dibutyl m-cresol				" "	
	Esters of dimerized soybean oil acids					
	Paraffin acids branched chain contg. 10-14 C atoms				" "	
	Sulfonic acids of C-6 to C-20 hydrocarbons				Weakness and lack of stability.	
	Sulfonamides " " "				" " "	
	Sulfonyl chlorides " "				" " "	
	Lignin stearate	Meadol		Mead Corp., Chillicothe, O.	No thickening action.	
	Terpene maleic anhydride adducts	Petrex Acids		Hercules Powder Co.	Insoluble.	
Oils	Blown linseed oil	ADM-100		Archer-Daniels-Midland Co.	Act as inerts.	
	Bodied linseed oil	OKO-M-7-1/2		" " " "	" " "	
	Bodied soybean oil	Heavy bodied		" " " "	" " "	
	Blown castor oil	40		Baker Castor Oil Co.	" " "	
Bases	NaHCO$_3$	U.S.P.		Baker or Eimer & Amend	See Section V.	
	Na$_2$CO$_3$	"		" " "		
	Zn(OH)$_2$	"		" " "		
	NH$_4$OH (27% soln.)	"			Instability, weak.	
	Tetraethanol ammonium hydroxide 40%			Carbide & Carbon Chem. Corp.	" "	
	All other amines (see Section V)	Experimental Chemicals			" "	

NOTE: When several grades of the same material were tested, only the most promising is listed.

Laboratory reagents are standard reagents procured from Mallinckrodt Chemical Works, Eimer & Amend, N.Y., or from Baker Chemical Co.

Experimental chemicals are materials obtained from other research groups within E. I. duPont de Nemours & Co.

CLASSIFIED - CONFIDENTIAL

CLASSIFIED - CONFIDENTIAL

Table 16

Isobutyl Methacrylate-Gasoline Gels: Ammonia and Amines as Gelling Agents

GEL F-	DATA BOOK	PAGE	COMPOSITION Polymer %	Amine %	Amine Name	Water %	Miscellaneous %	Miscellaneous Name	PHYSICAL PROPERTIES Viscosities, poise Gardner-Holdt Bubble Tube	Brookfield Synerolectric	SURVEILLANCE -40° F. %Syneresis	Days	125° F. % Syneresis	Days	REMARKS
			AE POLYMER AMINE GELS												
3010	1118	49	3.0	1.8	27 % Ammonia soln.		5.0	Stearic acid					25	3	Very soft gel.
1457	1049	94	3.0	2.33	" " "		4.5	" " 0.5 Naphthenic acid							2.5 Impact strength(B), 13.6 P.Plate
3018	1118	52	2.9	2.5	Diethanol Amine	5.4	5.7	" "					40	1	(Room Temp.). Very weak.
3019	"	"	2.9	2.3	" "	5.4	5.7	" "					10	1	(" "). Very weak.
3020	"	"	2.8	2.8	" "	5.4	5.7	" "					35	1	(" "). Very weak.
3000	"	48	2.1	2.1	Ethylene Diamine(68%)		3.1	" " 3.1 " "							No gellation.
3001	"	"	2.0	3.1	" " "		3.1	" " 3.1 " "					40	2	(Room Temp.). Very weak.
3002	"	"	2.0	5.0	" " "		3.0	" " 3.0 " "					60	2	(" "). Very weak.
3003	"	"	3.0	2.1	" " "	1.5	5.1	" " 1.5 " "							Separation. Very weak.
3004	"	"	2.0	2.0	" " "	2.5	3.0	" " 3.0							
3021	"	52	2.6	5.1	" " "	10.5	5.2	" "							White suspension. No gellation.
3022	"	"	0.0	5.4	" " "	10.7	5.4	" "							" precipitate " "
3023	"	"	2.8	5.4	" " "	11.0	0.0								2 layers, no gellation.
3005	"	48	2.0	2.7	Monoethanol Amine	3.1	3.0	" " 3.0 " "							White suspension. No gellation.
3006	"	"	2.6	5.7	" " "	11.1	5.1	" "							High syneresis, weak friable gel.
3007	"	"	2.7	4.8	" " "	8.4		5.4 " "							2 layers, no gellation.
3008	"	"	3.0	2.7	" " "			6.0 Turkey Red Oil							2 layers, slight " .
3009	"	"	2.5	2.2	" " "	9.9	7.7	40% NaOH 5.0 " " "							3 " , " " .
3011	"	52	2.8	5.8	" " "	2.5	5.7	Stearic acid					12	1	(Room Temp.). Very weak.
3012	"	"	2.9	4.0	" " "	3.8	5.7	" "					0	1	(" "). Very weak.
3013	"	"	2.8	4.0	" " "	4.4	5.7	" "					0	1	(" "). Very weak.
3014	"	"	2.9	2.2	" " "	5.1	5.7	" "					0	1	(" "). Very weak.
3015	"	"	2.9	1.6	" " "	5.7	5.7	" "					5	1	(" "). Very weak.
3016	"	"	2.8	2.5	" " "	6.3	5.6	" "					15	1	(" "). Very weak.
3017	"	"	2.8	1.8	" " "	7.5	5.6	" "					1	1	(" "). Very weak.
3024	"	"	0.0	3.0	" " "	5.5	5.8	" "					5	1	(" "). About as strong as 3011-7
3025	"	"	3.2	1.4	" " "	1.4	0.0	" "							No gelation.
			0.3% INTERPOLYMER AMINE GELS												
3048	1118	54	3.1	0.14	Ethylene Diamine	0.14									Clear, mod. viscous, work hardens.
3066	"	56	5.0	0.006	" "					1.25					
3067	"	"	5.0	0.011	" "					1.44					
3068	"	"	5.0	0.017	" "					1.43					
3069	"	"	5.0	0.023	" "					1.89					
3072	"	"	5.0	0.028	" "	0.035				3.28					
3070	"	"	5.0	0.034	" "	0.058				2.93					
3073	"	"	5.0	0.039	" "	0.035				3.13					
3071	"	"	5.0	0.045	" "	0.058				3.12					
3035	"	53	3.0	0.14	Monoethanol Amine	1.4									Very cloudy, sl. visc., work hardens.
3038	"	"	3.1	0.26	" "	0.54									" , " " , " " .
3037	"	"	3.1	0.13	" "	0.27									Clear , " " , " " .
3036	"	"	3.1	0.07	" "	0.10									" , " " , " " .
3033	"	"	3.1	0.6	" "	0.3									" , " " , " " .
3030	"	"	3.1	0.6	" "	0.6									" , " " , " " .
3031	"	"	3.0	0.6	" "	1.2									Cloudy, " " , " " .
3032	"	"	3.0	0.6	" "	2.4									Very " , " " , " " .
3034	"	"	3.0	1.3	" "	1.3									" , " " , " " .
3049	"	54	5.1	0.067	" "	0.067									3052 gave the clearest initial gel
3050	"	"	5.1	0.067	" "	0.13									of the 4 gels, 3049-3052. On
3051	"	"	5.0	0.067	" "	0.20									standing, water settled out of
3052	"	"	5.0	0.067	" "	0.27									3051 and 3052.
3045	"	53	5.0	0.13	" "	0.13									Clear, mod. visc., work hardens.
3029	"	"	5.0	0.6	" "	0.3									" , " " , " " .
3026	"	"	5.0	0.6	" "	0.6									" , " " , " " .
3027	"	"	5.0	0.6	" "	1.2									Cloudy, " " , " " .
3028	"	"	4.9	0.6	" "	2.4									Very " , " " , " " .
3043	"	"	3.1	0.14	" "	0.14	96.6	Turpentine							No thickening.
3046	"	54	3.0	0.40	" "	1.07	1.07	Stearic acid							Clear, weak gel, work hardens.
3044	"	53	5.0	0.13	" "	0.13	0.51	a-Cellulose							Viscous, but cellulose settled out.
541	984	185	2.0	3.14	Isobutyl Amine		2.0	Oleic, 5 Naphthenic acid							No gellation.
542	"	"	2.0	7.95	Tri-n-Butyl Amine		2.0	" 5 " "							" "
543	"	"	2.0	18.1	Tetraethanol NH_4OH		2.0	" 5 " "							Separated into 2 layers.
538	"	"	5.0	0.73	Isobutyl Amine										No gellation.
539	"	"	5.0	1.85	Tri-n-Butyl Amine										No gellation, but viscosity increase
540	"	"	5.0	4.22	Tetraethanol NH_4OH (50% aqueous)										Fluid, strong, v. elastic, adhesive
764	998	44	1.(0.1)	5.0	Isobutyl Amine	12.0	12.0	Benzene di-m-sulfonic acid							No gellation.
765	"	"	1.(0.1)	5.0	Tri-n-Butyl Amine		9.5	2,Naphthol,6,sulfonic acid							No gellation, but precipitation.

CLASSIFIED - CONFIDENTIAL

Table 16 (Sheet No. 2)

Isobutyl Methacrylate-Gasoline Gels: Ammonia and Amines as Gelling Agents

GEL F-	DATA BOOK	PAGE	COMPOSITION Polymer %	Amine %	Amine Name	Water %	%	Miscellaneous Name	PHYSICAL PROPERTIES Viscosities, poise Gardner-Holdt Bubble Tube	Brookfield Syncro-lectric	SURVEILLANCE -40° F. % Syn-eresin	-40° F. Days	125° F. % Syn-eresin	125° F. Days	REMARKS
			EFFECT OF TOLUENE IN INTERPOLYMER AMINE GELS												
3053	1118	54	5.0	0.1	Monoethanol Amine	0.2	94.7	Toluene	7.5		0	30			
3054	"	"	5.0	0.1	" " "	0.2	75.7	"	5.0		0	30			
3055	"	"	5.0	0.1	" " "	0.2	56.9	"	5.5		0	30			
3057	"	"	5.0	0.1	" " "	0.2	19.0	"	5.9		0	30	0	30	
3058	"	"	5.0	0.1	" " "	0.2	14.2	"	6.5		50	30	0	30	
3059	"	"	5.0	0.1	" " "	0.2	9.5	"	7.5		50	30	0	30	
3060	"	"	5.0	0.1		0.2	0.0	"	11.1		18	30	0	30	
3082	"	63	5.0	0.055	Tetrameth.Diamine	0.1	19.0	"	6.3		0	5			
3084	"	64	5.0	0.055	" "	0.1	14.2	"			3	5			
3085	"	"	5.0	0.055	" "	0.1	9.5	"			12	5			
3086	"	"	5.0	0.055	" "	0.1	4.7	"			30	5			
3087	"	"	5.0	0.055	" "	0.1	0.0	"			36	5			
			EFFECT OF GELLING AGENT IN INTERPOLYMER AMINE GELS												
3074	"	63	5.0	0.12	Glycine	0.6	18.8	Toluene	0.65						
3075	"	"	5.0	0.17	Diethanol Amine	0.1	18.9	"	1.80						
3076	"	"	5.0	0.15	Monoisobutanol Amine	0.1	18.9	"	1.30						
3077	"	"	5.0	0.16	Aniline	0.1	18.9	"	0.70						
3078	"	"	5.0	0.28	Diphenyl Amine	0.1	18.9	"	0.70						
3079	"	"	5.0	0.075	HexamethyleneDiamine	0.1	19.0	"	3.40						
3080	"	"	5.0	0.10	TriethyleneTetramine	0.1	19.0	"	2.40						
3081	"	"	5.0	0.10	EthanolamineFormal	0.1	19.0	"	3.00						
3082	"	"	5.0	0.055	Tetrameth.Diamine	0.1	19.0	"	6.30						
3083	"	"	5.0	0.11	DecamethyleneDiamine	0.1	19.0	"	2.10						
3057	"	54	5.0	0.10	Monoethanol Amine	0.2	19.0	"	5.90						
3092	"	64	5.0	0.067	Pentameth.Diamine	0.1	19.0	"	5.00						
4003	"	65	5.0	0.050	TrimethyleneDiamine	0.1	19.0	"	6.30						
			OTHER AMINE GELS												
71	884	33		5.0	Cyclohexylamine Stearate										Very fluid,stearate precipitated.
72	"	"		10.0	" "										Soft,rigid gel.
73	"	"		5.0	" "		2.6	Smoked Sheet Rubber							Very soft,sticky,non-fluid gel.
74	"	"		10.0	" "		2.4	" " "							Stiff,sticky,non-fluid.
91	"	36	5 NR	10.0	" "										Soft,pasty.

Note: Cyclohexylamine Stearate gels were prepared by dissolving the stearate at 75°C.,then cooling the resulting solution to 25°C.

CLASSIFIED CONFIDENTIAL

CLASSIFIED - CONFIDENTIAL

Table 17

Comparative Burning Rates of Incendiary Gels

GEL	DATA BOOK	PAGE	COMPOSITION Polymer %	Hydrofol %	Naph. Acid %	Miscellaneous %	Miscellaneous Name	Miscellaneous %	Miscellaneous Name	Toluene %	40% NaOH %	BURNING Time, min.	REMARKS
METHOD A (100 g. spread over 7" diameter circle).													
			STANDARD GELS (AE Polmer).										
IM-1	1118	3	5	3		1.25	Water	2.0	CaO(-40mesh)			7.0	
IM-3	965	135	2	3	3						4.5	3.6	
IM-4	1118	87	3	4		2.50	"	4.0	" " " "			5.6	
F-1416	965	144	3	1	3	2.30	"	3.1	" " " "			4.2	
			AE POLYMER GELS.										
1836	965	167	2	3	3					87.5	4.5	3.0	IM-3 made with toluene in place of gasolene.
1838	"	"	3	4		2.50	"	4.0	" " " "	86.5		5.2	IM-4 " " " " " " " .
1837	"	"	3	1	3	2.30	"	3.1	" " " "	87.6		4.1	IM-5 " " " " " " " .
1832	"	144	2	3	3					87.5Turp.	4.5	2.9	IM-3 made with turpentine in place of gasolene.
1833	"	"	3	1	3	2.30	"	3.1	" " " "	87.6Turp.		4.9	IM-5 " .
2521	"	115	2	2		2.0	Dimerized Soy Bean Oil Acid				2.1	2.9	Use of Soy Bean Oil Acid for Naphthenic Acid.
1746	"	153	3	5		3.0	27% NH₃,aqueous					2.6	Use of NH₄OH as gelling agent in place of NaOH or CaO.
1747	"	"	3	5		3.5	" " "					2.4	
			INTERPOLYMER GELS.										
3049A	1118	54	5-0.3 I.P.								0	2.7	5% Of 0.3 Interpolymer solution in gasolene, ungelled.
3049	"	"	5-0.3 "			0.07	Water	0.067	Monoethanol Amine			2.8	Use of an Amine Gelling agent in place of NaOH or CaO.
1846	965	158	3-0.2 "								1.0	2.8	1846-1849 Effect of concentration of polymer and type
1847	"	"	5-0.2 "								1.0	3.4	of interpolymer(percent Methacrylic Acid).
1848	"	"	3-0.3 "								1.0	3.1	
1849	"	"	5-0.3 "								1.0	3.7	
1946	"	192	4-0.3 "							15.0	1.0	3.4	Gels 1946,1947,1948 did not spread well on burning pan,
1947	"	"	5-0.3 "							15.0	1.0	3.0	hence the burning times are not very accurate. The effect
1948	"	"	6-0.3 "							15.0	1.0	3.2	of the 15% Toluene is very little.
1941	965	187	4-0.3 "	1.5	1.5					15.0	1.7	3.9	Use of low soap concentrations.
1939	"	"	5-0.3 "	1		2.0	T-132 Resin(Stanco Dist.).			20.0	2.0	4.0	Use of low soap and bodying agent concentrations.
			NAPALM AND RE RESIN*SOLUTIONS.										
4009	1118	66	3 Napalm									2.7	
4010	"	"	4 "									3.0	
4011	"	"	5 "									3.3	
4012	"	"	6 "									3.4	
1938	965	186	4 Resin									3.8	Re Resin:* A polymeric composition from the General Aniline
1935	"	"	5 "									4.1	and Film Corporation, Code No. Re 302-D-19.
1936	"	"	6 "									5.1	
1937	"	"	7 "									5.7	
METHOD B (50 g. in 2 3/4" diameter mold).													
			STANDARD GELS.										
IM-1	1118	86	5 AE	3		1.25	Water	2.0	CaO(-40mesh)			6.4	
IM-3	"	"	2 AE	3	3						4.5	5.5	
IM-4	1152	13	3 AE	4		2.50	"	4.0	" " "			6.5	
NP-1	1118	87	13.5 Napalm									5.8	
			INTERPOLYMER TOLUENE GELS.										
A-3489	1118	75	4-0.3 I.P.							20	1.0	2.8	Soap forming acids and other bodying agents increase the
A-3492	"	68	6-0.3 "							20	1.0	3.9	burning time of the gel except for Naphthenic Acid. This
A-3491A	"	75	5-0.2 "			0.5	a-Cellulose			20	1.0	3.6	acid produces a more fluid gel than the straight inter-
A-3491B	"	"	5-0.2 "	0.25		0.5	" "			20	1.2	4.2	polymer gel and has about the same burning time.
A-3488	"	"	4-0.3 "			0.5	" "			20	1.0	4.3	
A-3487	"	"	4-0.3 "		1					20	1.5	2.7	
A-3490	"	"	4-0.2 "	1						20	1.4	3.3	
A-3493	"	68	5-0.3 "	2						20	1.7	4.7	
A-3494	"	"	5-0.3 "			2.0	Turkey Red Oil			20	2.3	5.7	
A-3495	"	"	5-0.3 "	1		2.0	T-132 Resin(Stanco Dist.)			20	2.0	6.5	
A-3496	"	"	5-0.3 "	1.5	1.5					20	2.5	6.4	
F-1940	965	187	5-0.3 "	2.5	2.5					15	3.0	6.3	

CLASSIFIED - CONFIDENTIAL

TABLE 18

CLASSIFIED - CONFIDENTIAL

CLASSIFIED - CONFIDENTIAL

Surveillance Data

Gel No.	Notebook	Page	Composition of Gel, percent						Stability[a]		Parallel Plate Values, cm.		
F-			methacrylate		acids			remarks	days to breakdown				
			percent	grade					at -40°F	at 125°F	1 day	1 week	1 month
Hydrofol and naphthenic acid					Hydrofol 51	naphthenic	40% NaOH						
1017	965	5	5	0.1IP	1.5	1.5	1.8		(10.4)	(0.0)			
1005	965	3	5	AE	2.5	3.0	3.3		(8.4)	(0.0)			
1006	"	"	5	AE	2.0	3.0	3.0		(5.6)	(5.0)			
1007	"	"	5	AE	1.5	3.0	2.7		(1.2)	(0.0)			
1008	"	"	5	AE	1.0	3.0	2.4		(8.8)	(0.0)			
1001	965	3	5	AE	2.5	2.5	3.0		(13.4)	(0.0)			
1002	965	3	"	"	2.0	2.5	2.7		(10.0)	(0.0)			
1003	"	"	"	"	1.5	2.5	2.4		(10.0)	(10.0)			
1004	965	3	5	AE	1.0	2.5	2.1		31	26			
2015	973	4	5	NR	2.5	2.5	3.0		(0.8)	(2)			
2082	"	22	"	"	2.5	2.5	3.0		(7.4)	(5)	11.7	12.7	12.8
499	884	172	"	"	2.5	2.5	3.0	Hydrofol 46	(14.4)	26			
500	884	172	5	NR	2.5	2.5	3.0	Hydrofol 150	(0.4)	(0.0)			
501	"	"	"	"	"	"	"	Hydrofol 300	(14.4)	(0.0)			
502	"	"	"	"	"	"	"	Hydrofol 305	(6.0)	(3)			
503	"	"	"	"	"	"	"	Hydrofol 400	(9.4)	(2)			
504	"	"	"	"	"	"	"	Hydrofol 405	(3.8)	(3)			
505	884	172	5	NR	2.5 *	2.5	3.0	* Snodotte acids	(1.2)				
533	"	"	"	"	2.5	2.5	"	naphthenic acid "CC"					
1018	965	5	4	0.1IP	1.5	1.5	1.8		(0.0)	(0.0)			
658	998	18	3	0.035IP	2.5	2.3	3		(2.0)	(15.0)	9.7		10.2
1406	1049	75	3	AE	3	3	4.5		(3.0)	(2.0)			
662	998	18	3	AE	2.5	2.5	3		(5.7)	28	11.9		11.5
613	998	14	2	0.035IP	3.5	3.5	4.2		(0)	(5)	11.2	11.3	10.7
614	"	"	2	AE	3.0	3.0	3.6		(0)	(2)	10.3	9.8	9.2
659	998	18	2	AE	3.0	3.0	3.6		(0)	(15)	8.8		9.4
664	"	19	2	AE	3.0	3.0	3.6		(0)	(5)		11.2	12.0
1309	978	106	2	AE	3.0	3.0	4.5		(0)	(2)	9.8	8.8	8.8
2343	973	116	2	0.035IP	3.0	3.0		5% NaOCl	(0)	7		13.8	13.3
786	998	36	1.75	AE	3.0	3.0	4.5		(0)	(5)			
787	"	"	1.5	"	3.0	3.0	4.5		(0)	(1)			
788	"	"	1.25	"	3.0	3.0	4.5		(0)	(10)			
720	"	34	1	0.3IP	3.5	3.5	7.5		(0)	30	10.3	9.4	
750	998	42	1	0.2IP	3.5	3.5	7.5	0.25% α cellulose	(0)	7	9.5	9.2	9.6
719	"	34	1	0.1 "	3.5	3.5	7.5		(0)	7	10.7	11.1	
751	"	42	1	0.1 "	3.5	3.5	7.5	0.5% α cellulose	(0)	30	9.9	9.1	9.8
729	"	38	1	0.1 "	3.5	3.5	6.5		(0)	14	9.6	8.7	9.5
733	"	42	1	0.1 "	3.5	3.5	6.0		(0)	(15)	8.8	9.2	10.1
732	998	38	1	0.1 "	3.5	3.5	5.5		(0)	(15)		8.2 (2 wks.)	8.6
728	"	"	1	0.035IP	3.5	3.5	6.5		(0)	(10)	9.4	9.0	9.9
731	"	"	1	0.035 "	3.5	3.5	5.5		(0)	14	8.8	8.8	
727	"	"	1	AE	3.5	3.5	6.5		(0)	(15)	10.4	10.0	10.0
730	"	"	1	AE	3.5	3.5	5.5		(0)	30	8.6	8.7	
737	998	38	1	0.2IP	3.0	3.0	5.5		(0)	30			
736	"	"	1	0.1 "	3.0	3.0	5.5		(0)	(15)			
735	"	"	1	0.035 "	3.0	3.0	5.5		(0)	30	10.0	10.7	

a - Figures in parentheses give percent syneresis after 30 days.

CLASSIFIED - CONFIDENTIAL

TABLE 18 Cont.

CLASSIFIED - CONFIDENTIAL CLASSIFIED - CONFIDENTIAL

Surveillance Data

Gel No F-	Notebook	Page	Composition of Gel, percent: methacrylate percent	methacrylate grade	acids			40 % NaOH soln	remarks	Stability[a] days to breakdown at -40°F	at 125°F	Parallel Plate Values, cm 1 day	1 week	1 month
Stearic acid (triple pressed)					stearic									
412	884	120	5	AE	4				2% CaO; 1.20% H_2O	(0.8)	(0.0)			
414	"	"	5	"	4				2% CaO; 1.20% H_2O	(0.8)	(0.0)			
356	"	93	5	"	3			1.6		(1.0)	(1.0)			
345	"	92	3.5	0.1 IP	3			1.6		(1.0)	(0.0)			
401	"	128	3.5	0.1 "	2			1.2		(1.2)	(0.0)			
402	884	128	3.5	0.1 IP	1			1.0		(17.2)	(0.0)			
399	884	"	3	0.1 "	3			1.6		(5.6)				
403	"	"	2.5	0.1 "	3			1.6		(0.0)				
Naphthenic, wood rosin, or oleic acid					naphthenic	wood rosin	oleic							
1019	965	5	5	0.1 IP	2.5			1.5		(0.8)	25			
1020	"	5	4	0.1 "	2.5			1.5		(7.6)	20			
411	884	128	3.5	0.1 "		3.5		1.6		(17)	14			
1021	965	5	3	0.1 "			3	1.8		(6.0)	(3)			
467	884	150	2.5	0.1 "			1.75	0.93		(3.6)	25			
2099	973	24	2	0.3 "			3	1.6		10	3			
Naphthenic and Oleic Acids					naphthenic	oleic								
2063	973	14	2	0.3 IP	4	2		3.6		7			8.4	8.0
2069	"	16	2	0.3 "	3	3		3.5		7			8.1	8.1
2067	"	"	2	0.3 "	3	2		3.0		12				
574	884	192	2	0.3 "	2	3		3.0		(10)	(5)	10.0 (5 days)	9.6	
2119	973	34	2	0.3 "	2	3		3.0	5% Darco	7		7.4 (4 days)	8.0	7.2
2060	973	14	2	0.3 IP	2	2		2.3		(10.6)	(0)		8.4	7.9
2070	"	16	2	0.3 "	2	2		3.3		(2.2)	(0)			
2081	"	18	2	0.3 "	2	2		4.3		(0)	(0) one test 30 days 2nd test		10.0	10.1
2129	"	36	2	0.2 "	2	2		2.3		(0)	7	11.4 (2 days)	9.4	9.5
2061	"	14	2	0.1 "	2	2		2.3		(18.3)	7			
573	884	192	2	0.1 IP	2	2		2.3		(1)	(3)	11.9 (3 days)	11.0	10.8
2213	973	54	2	0.035 "	1.5	1.5		1.8	1% α cellulose	(0)	25		11.3 (2 wks)	11.2
Naphthenic, oleic, and stearic acids					naphthenic	oleic	stearic							
2100	973	24	2	0.3 IP	4	2	1	4.2		(7.8)	14	9.4 (3 days)	9.4	7.7
2101	"	"	2	0.3 "	4	2	0.5	3.9		(5.1)	14	9.0 (3 days)	8.5	9.0
2157	"	42	2	0.3 "	3	1	0.5	2.7		(0)	(7)		8.4	9.4
2079	"	18	2	0.3 "	2	2	1	2.9		(12.5)				
2186	"	48	2	0.1 "	3	1	0.5	2.7	1% α cellulose	(0)	(5)		10.7	10.0
2210	973	54	2	0.035 "	3	1	1	3.0	0.5% α cellulose	(0.1)	(0)		10.9 (2 wks.)	11.6
2201	"	52	2	0.035 "	3	1	0.5	2.7	1% α cellulose	(0)	25		11.9	10.8
Naphthenic, oleic, and Hydrofol 51					naphthenic	oleic	Hydrofol							
615	998	14	2	0.035 IP	3	1	1		0.5% α cellulose	(0)		10.7	9.8	9.5

a. Figures in parentheses represent percent syneresis after 30 days

TABLE 18 Cont.

CLASSIFIED - CONFIDENTIAL

CLASSIFIED - CONFIDENTIAL

Surveillance Data

Gel No. F-	Notebook	Page	Composition of Gel, percent: methacrylate percent	methacrylate grade	acids		40% NaOH soln	remarks	Stability[a] days to breakdown at -40°F	at 125°F	Parallel Plate Values, cm 1 day	1 week	1 month
Stearic and naphthenic acids					stearic	naphthenic							
462	884	156	5	0.1 IP	0.5	0.5	1.0		10	(7)			
1013	965	3	5	AE	2.5	3.0	3.3		28	(0.0)			
1014	"	"	5	"	2.0	3.0	3.0		(16)	(0.0)			
1015	"	"	5	"	1.5	3.0	2.7		(5.2)	(2.0)			
1016	"	"	5	"	1.0	3.0	2.4		(12.0)	(5)			
1009	965	3	5	AE	2.5	2.5	3.0		19	(0.0)			
386	884	113	5	"	2.5	2.5	3.0		(2)	(0.0)			
270	884	72	5	"	2.5	2.5*	2.7	* naphthenic acid "C"	(2.0)	(0.0)			
1010	965	3	5	"	2.0	2.5	2.7		(5.4)	(0.0)			
1011	965	3	5	AE	1.5	2.5	2.4		23	26			
1012	965	3	5	"	1.0	2.5	2.1		2	26			
1032	965		5	NR	2.5	2.5	3.0		(14.6)	(0)			
498	884	172	5	NR	2.5	2.5*	3.0	* naphthenic acid "CC"	(17.4)	(0.0)			
463	884	156	4	0.1 IP	0.5	0.5	1.0		19	(5.0)			
295	"	84	3.5	0.1 "	2.5	2.5	3.0		(4)	(0.0)			
408	884	128	3.5	0.1 "	1.5	1.5	2.0		26	(0.0)			
409	"	"	3.5	0.1 "	0.5	0.5	1.0		19	(1.0)			
406	884	128	3.0	0.1 "	2.5	2.5	3.0		(0.0)				
460	"	156	3.0	0.1 "	1.0	1.0	1.5		(8.4)				
407	884	128	2.0	0.1 IP	2.5	2.5	3.0		(0.4)	16			
2166	973	44	2	0.1 "	1.5	1.5	1.8		(5.1)	(1.0)	11.6	9.8	9.9
2204	"	52	2	0.1 "	1.5	1.5	1.8	1% Darco	(0.0)	(1.0)		10.6	9.5
2207	"	"	2	0.035 "	3.5	3.5	4.1		(0.5)	(1)		11.7	9.5
2171	"	44	2	0.035 "	0.5	3.5	3.0		(1)	30	13.4	11.6	12.2
2194	973	48	2	0.035 "	1.5	3.5	3.0	2% Darco	(6.4)	(0)		13.2	11.0
2193	"	"	2	0.035 "	1.5	3.5	3.0	1% α cellulose	(5)	25		11.5	9.5
2192	"	"	2	0.035 "	1.5	3.5	3.0	0.5% α cellulose	(6.2)	(1)		12.2	10.7
2169	"	44	2	0.035 "	3.0	3.0	3.5		(18)	(6)	13.5	12.2	10.5
2165	"	"	2	0.035 "	1.5	1.5	1.8		(5.8)	(10)	14.5	13.8	13.8
2191	973	48	2	0.035 "	1.5	1.5	1.8	1% Darco	(6.4)	(0)		12.4	10.7
Dimerized soybean oil acid					dimer acid	Hydrofol		CaO · water					
965	1059	163	3	AE	2	2		2.4 · 1.5	(0.2)	(0.0)			
967	"	"	3	AE	2	4		3.5 · 2.3	(0.0)	(0.0)			
962A	"	161	2	AE	2	2	2.8		(0.9)	(3)			
969	"	"	2	AE	2	2	2.6		(0.8)	(4)			
962	"	"	2	AE	2	2	2.1		(1.0)	(0.5)			
961	1059	161	2	AE	2	2	1.4		(0.2)	(15)			

a - Figures in parentheses give percent syneresis after 30 days.

TABLE 18 Cont.

Surveillance Data

Gel No. F-	Notebook	Page	Composition of Gel, percent: methacrylate percent	methacrylate grade	acids			40 % NaOH soln	remarks	Stability[a] days to breakdown at -40°F	at 125°F	Parallel Plate Values, cm. 1 day	1 week	1 month
Monomethyl ester of dimerized soybean oil acid					monomethyl ester	Hydrofol								
892	998	153	2	AE	2	2		2		(0.6)	21			
970	1059	161	2	AE	2	2		2		(0)	(6)			
964	"	"	2	AE	2	2		1.6		(0)	21			
963	"	"	2	AE	2	2		1.1		(0)	<14			
Wood resin - naphthenic acid - Hydrofol 51					wood resin	naphthenic	Hydrofol							
654	998	17	2	0.3 IP	2	2	2	3.7		(0)	14			
2214	973	56	2	0.3 "	2	2	2	3.7		(0.3)	(0)	10.5	9.8	9.8
616	998	14	2	0.2 "	2	2	2	3.7		(0)	(1)	11.4	9.8	10.2
660	"	18	2	0.2 "	2	2	2	3.7		(1.2)	21	11.8		10.3
2367	973	125	2	0.2 "	2	2	2	3.7	0.5 % α cellulose	(0)			10.7	9.8
655	998	17	2	0.2 IP	2	2	2	3.7	2 % Darco	(0)	7			
624	"	14	2	0.1 "	2	2	2	3.7	0.5% α cellulose	(0)	(2)	10.4	10.0	9.4
661	"	18	2	0.1 "	2	2	2	3.7	0.5% α cellulose	(0.6)		9.5		9.6
663	"	19	2	0.1 "	2	2	2	3.7	0.5% α "	(0.0)			11.4	11.3
2366	973	125	2	0.1 "	2	2	2	3.7	0.6% α "	(0.0)			10.7	9.8
FC33	1059	25	2	0.1 IP	2	2	2	3.7	0.5% α cellulose	(0.0)		11.2		
618	998	14	2	0.035 "	2	2	2	3.7	0.5% α "	(0.0)	(0.0)	13.4	13.8	12.4
2365	973	125	2	0.035 "	2	2	2	3.7	0.5% α "	(0.0)			11.8	11.2
617	998	14	2	0.035 "	2	2	2	3.7	2% Darco	(0.2)	(0.0)	12.1	12.0	11.3
2215	973	56	2	0.035 "	2	2	2	3.7	2% Darco	(0.0)	21	11.5	11.3	10.3
2364	973	125	2	AE	2	2	2	3.7	0.5 % α cellulose	(0.0)			11.7	11.6
2363	"	"	2	AE	2	2	2	3.7	0.5% α cellulose	(0.0)			11.4	11.9
Wood resin - naphthenic acid - stearic acid					wood resin	naphthenic	stearic							
575	884	192	2	0.3 IP	2	2	2	3.7		(0.1)	(5)	11.8	11.9	10.3
2102	973	24	2	0.3 "	2	2	2	3.7		(0.1)		11.4 (3 days)	10.6	8.9
2159	"	42	2	0.3 "	1	3	1	3.1		(4.0)	14		12.9	12.9
2162	"	"	2	0.3 "	1	2	0.5	2.2		(5.8)			16.0	16.1
678	884	190	2	0.2 "	2	2	2	3.7	0.5% α cellulose	(0)	(0)			
560	884	190	2	0.1 IP	2	2	2	3.7	0.5% α cellulose	(0)		9.6 (5 days)	8.2	8.5
2125	973	34	2	0.1 "	2	2	2	3.7	5% dextrose	(3.5)	3	9.7 (4 days)	9.4	8.4
2123	"	"	2	0.1 "	2	2	2	3.7	3 % Darco	(1.6)	(0)	9.1 (4 days)	9.7	9.0
2126	"	36	2	0.1 "	2	2	2	3.7	2 % Darco	(0)	(0)	11.5 (4 days)	9.8	
2127	"	"	2	0.1 "	2	2	2	3.7	1% Darco	(0)	25		10.9	8.6
2202	973	52	2	0.1 IP	1	2	0.5	2.2	1% α cellulose	(0.8)	2		11.8	11.3
557	884	190	1	0.3 "	2	2	2	3.7		(0)		9.9 (4 days)	8.0	very friable
489	"	168	1	0.3 "	1.75	2.5	2	3.8	1% aluminum palmitate	(0.2)	(10)			
490	"	"	1	0.3 "	1.75	2.5	2	3.8	1% al. palmitate; 1% al. stearate	(0.2)	(10)			

a- Figures in parentheses give percent syneresis after 30 days.

TABLE 18 Cont.

Surveillance Data

Gel No. F-	Notebook	Page	Composition of Gel, percent: methacrylate percent	methacrylate grade	acids			40% NaOH soln	remarks	Stability[a] days to breakdown at -40°F	at 125°F	Parallel Plate Values, cm. 1 day	1 week	1 month
Wood resin and Hydrofol 51					wood resin	Hydrofol								
2007	973	4	2	0.3 IP	2	2		2.4		(0.0)	15			
2046	"	12	2	0.3 "	2	2		2.4		(0.0)	(10)		7.9	7.2
2006	"	4	2	0.3 "	1.5	1.5		2.1		(0.6)	30			
2045	"	12	2	0.3 "	1.5	1.5		2.1		(0.0)	(2.0)		7.8	7.5
2005	"	4	2	0.2 "	1.5	1.5		2.1		(0.8)	(15)			
625	998	14	2	0.2 IP	1.5	1.5		1.8		(0.0)	(3)	10.6	10.8	9.8
656	998	17	2	0.2 "	1.5	1.5		1.8	0.5% α cellulose	(0.4)	(8)			
2016	973	6	2	0.1 "	1.5	1.5		1.8		(0.2)	14			
619	998	14	2	0.1 "	1.5	1.5		1.8		(0.1)	(5)	11.5	11.2	10.4
620	"	"	2	0.1 "	1.5	1.5		1.8	1% Darco	(0.1)	(2)	13.1	13.9	13.0
621	998	14	2	0.035IP	1.5	1.5			0.5% α cellulose	(0)	14	14.4	14.6	13.1
2017	973	6	1	0.3 "	1.5	1.5		1.8		(0)	25			
Wood resin and stearic acid					wood resin	stearic								
2139	973	38	2	0.3 IP	1.5	1.5		1.8		(0.4)		11.6	10.8	9.6
2145	"	40	2	0.3 "	1.5	1.5		1.8		(0.3)	(3)	12.0	10.3	9.6
2107	"	28	2	0.3 "	1.5	1.5		1.8		(0.4)	(0)	11.9	11.9	10.1
2138	"	38	2	0.3 "	1.5	1.5		1.8		(1.0)		11.9	10.9	9.2
2135	"	35	2	0.2 "	1.5	1.5		1.8		(0.1)		12.1	11.5	very friable
2137	973	38	2	0.2 IP	1.5	1.5		1.8		(0.3)		11.7	11.0	9.7
2146	"	40	2	0.1 "	1.5	1.5		1.8		(0.2)	7	11.5	9.6	<< 9.5
2136	"	38	2	0.1 "	1.5	1.5		1.8		(0.1)		12.1	10.4	8.6
473	884	158	1.75	0.3 "	1.75	3.5		3.5		(0.0)	(1.0)			
Wood resin and oleic acid					wood resin	oleic								
466	884	158	2.5	0.1 IP	1.75	3.5		3.0	1% aluminum palmitate	(0.0)	22			
2025	973	8	2	0.3 "	4	2		3.7		(0.0)	13			
2050	"	12	2	0.3 "	4	2		3.7		(0.0)	8	8.0	7.2	
2023	"	6	2	0.3 "	3	2		3.1		8	8			
2049	"	12	2	0.3 "	3	2		3.1		(0.0)	13	9.0	8.5	
469	884	158	1.75	0.3 IP	1.75	3.5		3.0		(14.0)	28			
2018	973	6	1.0	0.3 "	4	2		3.7		(0.0)	4			
2048	"	12	1.0	0.3 "	4	2		3.7		(0.0)	5	8.4	7.6	
2020	"	8	1.0	0.3 "	4	2		3.7	5% bodied linseed oil	(0.0)	34			
2051	"	12	1.0	0.3 "	4	2		3.7	5% " " "	(0.0)	18	10.0	8.3	
Wood resin, oleic, and naphthenic acids					wood resin	oleic	naphthenic							
2076	973	18	2	0.3 IP	3	2	2	4.3		(0.2)	7			
2074	"	"	2	0.3 "	1	2	2	3.0		(3.0)				
494	884	168	1	0.3 "	1.77	2.02	2.53	3.84	1.01 % aluminum palmitate	31	28			
Wood resin, oleic, and Hydrofol 51					wood resin	oleic	Hydrofol							
2218	973	56	2	0.3 IP	3	2	1	3.6		(0.1)	25	11.3		11.3
657	998	17	2	0.2 "	1.75	2	1.5	3.0		(0.1)	(1)			
623	"	14	2	0.1 "	1.75	2	1.5	3.6		(0)	(0)	10.9	11.6	11.3
2014	973	4	1.5	0.3 "	2	1	1	2.4		(0.0)	18			
2047	"	12	1.5	0.3 "	2	1	1	2.4		0.0	(12)	9.2	8.2	

a. Figures in parentheses represent percent syneresis after 30 days

TABLE 18 Cont.

CLASSIFIED · CONFIDENTIAL   CLASSIFIED · CONFIDENTIAL

Surveillance Data

Gel No. F-	Notebook	Page	Composition of Gel, percent: methacrylate percent	grade	acids			40% NaOH soln	remarks		Stability[a] days to breakdown at -40°F	at 125°F	Parallel Plate Values, cm 1 day	1 week	1 month
Wood rosin, oleic, and stearic acids					wood rosin	oleic	stearic								
2033	973	8	2	0.3 IP	3	2	1	3.6			(0.2)	(0)			
2032	"	12	2	0.3 "	3	2	1	3.6			(0.0)	(10)	8.9	8.8	
2034	"	8	2	0.3 "	1.75	2	1.5	3			(2.5)	(0)			
2053	"	12	2	0.3 "	1.75	2	1.5	3			(0.0)	30	10.4	9.8	
2163	"	42	2	0.3 "	1	2	1	2.3			(0.1)	(0)			
562	884	190	2	0.1 IP	2	2	2	3.7	0.25% α cellulose		(1.8)	(0)	9.0 (2 days)	8.3	7.8
563	"	"	2	AC	2	2	2	3.7	0.5% α "		(0)	(0)	9.6	8.7	8.5
Substitutes for wood rosin and for stearic acid					substitute for wood rosin	substitute for stearic	naphthenic		name of substitute for wood rosin	name of substitute for stearic					
2106	973	28	2	0.3 IP	1.5	1.5		1.8	polypale rosin	stearic acid	(7.8)	(0)	12.6	12.7	11.5
2144	"	38	2	0.3 "	1.5	1.5		1.8	Staybelite	" "	(0.3)		10.0	9.3	8.2
602	998	10	2	0.3 "	1.5	1.5		1.8	Petrex Resin 65	" "	(0.0)	(5)	6.5	7.1	6.7
2160	973	42	2	0.3 "	1.	0.5	3.5	2.2	polypale rosin	" "	(12)	7		13.1	11.9
2161	"	"	2	0.3 "	1	1	3	3.1	" "	double pressed stearic acid	(8.2)	14		12.9	12.3
2177	973	46	2	0.3 "	1.75	3.5		3.0	wood rosin	sardine oil acids	(0.3)		10.2		9.0
2183	"	"	2	0.3 "	1.75	3.5		3.0	" "	soybean oil acids	(9.0)	4	11.2	10.2	9.5
2100	"	30	2	0.3 "	1.5	1.5		1.8	" "	double pressed stearic	(0)	30	11.8	10.8	10.8
2109	"	"	2	0.3 "	1.5	1.5		1.8	" "	Hystrene	(0)	(0)	10.5	11.0	11.2
2269	"	84	2	0.2 "	1.75	3.5		3.0	" "	cotton seed oil acids	(0.1)	30		10.0	9.6
Tall oil acids					Indusoil	Ligro	stearic								
2142	973	38	2	0.3 IP	5			2.3			(0.8)			8.3	8.7
2174	"	46	2	0.3 "	3		1	2.6	1% wood rosin		(0.6)	(0)	10.0	10.9	9.1
2149	"	40	1.75	0.3 "	3.5			2.8	1.75% " "		(0.2)	7	9.9	13.9	12.9
2153	"	"	1	0.3 "	1.5		1.5	1.5			(0)	(10)	7.9	7.1	6.7
2143	"	38	2	0.3 "		5.0		2.1			(0.1)	7		9.6	8.8
2130	973	40	1.75	0.3 IP		3.5		2.6	1.75% wood rosin		(0.2)	7		14.1	13.9
2154	"	"	1	0.3 "		1.5	1.5	1.4			(0)	(0)	7.5	7.4	8.5
Ricinoleic acid					ricinoleic	stearic	naphthenic								
2140	973	38	2	0.3 IP	5			2.5	technical grade ricinoleic		22	1		15.4 (2 wks)	16.3
2141	"	"	2	0.3 "	5			2.5	Bakers' P20 "		(4.5)	(15)		8.8	7.9
2173	"	46	2	0.2 "	1	1	1	1.7	" P20 "		(0)	(5)	10.3	9.7	8.7
2152	"	40	1	0.3 "	1.5	1.5		1.6			(0.3)	(1)	10.1	9.3	7.6
Phenol					phenol	acid name	acid percent								
601	998	10	2	0.3 IP	4			6.36			13	1	10.6	10.9	11.8
591A	"	"	2	0.3 "	3.5	wood rosin	1.75	6.73			(2.0)	18	8.0	8.2	8.4
598	"	"	2	0.3 "	2	" "	1.75	4.35			(2.6)	20	7.6	8.2	8.4
600	"	"	2	0.3 "	2	oleic	2	4.24	bad syneresis in tube at room temp		(5.6)				
611	"	"	2	0.3 "	1.5	stearic	1.5	7.20			(0.6)	(5)		7.2	7.0

[a] Figures in parentheses give percent syneresis after 30 days.

CLASSIFIED - CONFIDENTIAL

TABLE 18 Cont.

Surveillance Data

Turkey Red Oil

Gel No. F-	Notebook	Page	Composition of Gel, percent: methacrylate percent	methacrylate grade	acids: Turkey Red Oil	acid name	acid percent	40% NaOH soln	remarks	Stability[a] days to breakdown at -40°F	at 125°F	Parallel Plate Values, cm 1 day	1 week	1 month
2278	973	90	2	0.2 IP	2	naphthenic	2	2.8		30	7		10.0	9.8
2280	"	94	2	0.035 "	2	oleic	2	2.6		(6.5)	7		15.3	15.4
2301	"	98	2	0.035 "	2	"	2	2.6	0.25% α cellulose	(2.2)	7		13.4	13.8
2300	"	"	1 1	0.1 " 0.035 "	2	"	2	2.6		(0.3)	7		12.1	11.1
2297	"	"	2	0.1 "	1.5	Hydrotol	1.5	2.0		(2.2)			10.8	11.0
2298	973	98	2	0.035 IP	2.5	Hydrotol	2.5	3.3		(0.2)	(1)		9.7	10.8
2323	"	111	2	0.035 "	2.5	"	2.5	3.3		(0.3)	(3)		10.7	10.7
2380	"	132	2	0.035 "	2.5	"	2.5	3.3		(0)	(10)		9.0	8.3
2279	"	94	2	0.035 "	1.5	"	1.5	2.0		(1)	25		13.2	13.6
696	998	28	2	0.1 "	2	"	2	3.9	2% wood rosin	(0.3)	(15)	9.4	9.1	9.2

Calcium oxide

Gel No. F-	Notebook	Page	methacrylate percent	methacrylate grade	acids (percent) 1	2	3	name of acids 1	2	3	CaO	H_2O	remarks	Stability at -40°F	at 125°F	1 day	1 week	1 month
571	884	188	2.	0.4 IP	3	2	1	wood rosin	oleic	stearic	2.02	1.30		20	7		12.2	10.2
589	998	2	2	0.3 "	3	2	1	" "	"	"	2.00	1.29		(1.6)	1		14.6	14.9
597	"	8	2	0.3 "	3	2	1	" "	"	"	2.02	1.29	0.5% α cellulose	(6.0)	1		12.2 (11 days)	10.7
547	884	188	1	0.3 "	3	2	1	" "	"	"	2.02	1.30		(0)	1		13.0	12.6
546	"	188	2	0.3 "	3	2	1	" "	"	"	2.0	1.30		14	3	11.0 (5 days)	10.4	11.1
593	998	8	2	0.3 IP	3	2		naphthenic	oleic		2.13	1.36	0.5% α cellulose	(0.5)			9.5 (2 weeks)	6.8
592	"	"	2	0.1 "	2	2		"	"		1.68	1.07	0.5% α cellulose	(0)			8.9 (2 weeks)	8.9
586	"	2	2	0.3 "	1.5	1.5		wood rosin	Hydrotol		1.10	0.76		(12.5)	1		15.2	15.2
587	"	"	2	0.3 "	1.5	1.5		" "	stearic		1.01	0.65		(9.4)	1		16.0	14.1
588	"	"	2	0.3 "	3	2		" "	oleic		1.74	1.11		(3.8)	1		15.7	15.6
590	998	2	2	0.3 IP	2	2	2	wood rosin	stearic	naphthenic	2.03	1.30		(5.5)	1		12.0	10.9
591	"	5	2	0.3 "	4			stearic			2.0	1.28		(2.7)	(0)		7.0	<<7.0

X104 aluminum soaps

Gel No. F-	Notebook	Page	methacrylate percent	methacrylate grade	X104	remarks	Stability at -40°F	at 125°F	1 day	1 week	1 month
523	884	176	2	0.3 IP	5.0		(0.0)	(0.0)			
515	884	174	2	0.2 "	5.0		(0.0)	(0.0)			
521	"	176	2	0.1 "	5.0		(0.0)	10			
516	"	174	1	0.4 "	5.0		(0.0)	(0.0)			
493	"	160	1	0.3 "	5.0		(0.0)	(0.0)			
2044	973	12	1	0.3 IP	5.0	5% bodied linseed oil	(0.0)	(0.0)	7.4	6.8	
522	884	176	1	0.1 "	5.0		(0.0)	10			
517	"	174	0.5	0.6 "	5.0		(0.0)	(0.0)			
534	"	176	0.5	0.3 "	5.0		(0.0)	30			
476	"	166	0.0		10.6		(0.0)	(0.0)			

Aluminum Palmitate

Gel No. F-	Notebook	Page	methacrylate percent	methacrylate grade	al. palm.	Stability at -40°F	at 125°F
418	884	133	5	0.6 IP	6	6	(0.0)
428	"	"	5	0.3 "	6	10	(0.0)
416	"	"	5	0.2 "	6	22	(0.0)
431	"	"	3	0.2 "	6	18	(0.0)

a. Figures in parentheses show percent syneresis after 30 days

TABLE 18 Cont

CLASSIFIED - CONFIDENTIAL

CLASSIFIED - CONFIDENTIAL

Surveillance Data

Gel No. F-	Notebook	Page	Composition of Gel, percent: ethyl cellulose percent	ethyl cellulose grade	acids			solvent	40 % NaOH soln	remarks	Stability[a] days to breakdown at -40°F	at 125°F	Parallel Plate Values, cm 1 day	1 week	1 month
Solvent - acetone															
Hydrofol and naphthenic acids					Hydrofol	naphthenic									
2303	973	100	5	200 c.p.s.	2	2		25	2.3		(2.7)	7	10.3	9.3	8.2(Subs)
2329	"	112	5	"	2	2		20	3.3		(13)	14		10.6	10.2
2289	"	96	5	"	2	2		20	2.3		(2.2)	(5)	10.0	9.5	9.4
2328	"	112	5	"	2	2		20	1.3		(0)	30			
2344	"	120	5	"	2	2		15	2.8		(1)	(0)			
2304	978	100	5	200 c.p.s.	2	2		15	2.3		(4.0)		10.7	9.7	very friable
2346	"	120	5	"	2	2		10	2.8		(0.3)	(10)		10.4	9.0
2305	"	100	5	"	2	2		10	2.3		(0)	30	9.2	9.1	
2306	"	"	5	100 c.p.s.	2	2		20	2.3		(0)	14	10.0	9.1	8.2
2321	"	110	5	"	2	2		20	2.3			(5)			
2334	973	114	5	20 c.p.s.	2	2		20	3.3		(0)	14		10.4	9.6
2307	"	100	5	"	2	2		20	2.3		(0)	7	9.1	8.7	7.6
2325	"	112	5	7 sec.	2	2		20	2.3		(0)	14			
2333	"	114	2	300 c.p.s.	2	2		20	3.3		(3.6)	14		10.7	9.7
2290	"	96	2	"	2	2		20	2.3		{syneresed room temp in 2 days}				
2385	973	114	5	300 c.p.s.	2	2		20		2% $NaOCH_3$	(0.3)			10.5	9.1
2341	"	116	5	"	2	2		20		2% $NaOCH_3$; 5% H_2O	(0)	10		10.1	10.0
2342	"	"	5	"	2	2		20		1% $NaOCH_3$; 3% H_2O	(0)	10		8.0	7.3
2352	"	120	5	"	2	2		20		2% $NaOCH_3$; 3% H_2O	(7.8)	10		13.6	11.6
2353	"	"	5	"	2	2		20		0.8% $NaOCH_3$; 3% H_2O	(1.4)	10		11.1	9.7
2384	973	132	4	200 c.p.s.	2.5	2.5		20	2.9	1% methacrylate AE	(0)	(6)		10.0	9.0
2320	"	108	4	"	2	2		20	2.3	1% " 0.3 interpolymer	(1.6)	(10)		9.9.	9.5
2318	"	"	4	"	2	2		20	2.3	1% " 0.1 "	(1)	14		10.2	9.1
2316	"	"	4	"	2	2		20	2.3	1% " AE	(2)	30			
2371	"	126	4	100 c.p.s.	2	2		20	2.3	1% " AE	(0.4)				
2319	973	108	2.5	200 c.p.s.	2	2		20	2.3	1% methacrylate 0.3 interpolymer	(0)	30		10.9	9.8(Subs.)
naphthenic, oleic, and Hydrofol acids					naphthenic	oleic	Hydrofol								
2379	973	130	5	100 c.p.s.		6		20	3.2		(1.3)	7		8.4	8.1
2374	"	128	5	200 c.p.s.	2	2		20	3.8		(18)	7			
2373	"	"	5	"	2	2		20	3.3		30	10			
2357	"	120	5	"	2	2		20	2.7		(11)	10		9.6	9.7
2331	"	114	5	"	2	2		20	2.3		30	14		11.3	10.8
2378	973	130	5	100 c.p.s.	2	4		20	3.4		(2.6)	7		8.9	8.4
2382	"	132	5	200 c.p.s.	1	4	1	20	3.3		(0.3)	20		8.3	8.8
2399	"	150	5	"	1	4	1	20	3.3		(0.7)	14	9.0	8.3	9.0
2368	"	126	5	100 c.p.s.	2	2	0.5	20	3.0		(0.2)	7			
Turkey Red Oil					Turkey Red Oil	naphthenic	Hydrofol								
698	998	28	5	200 c.p.s.	3		3	15	4.8			(0)	7.1	6.8	6.3
701	"	"	5	"	1.8		3.5	15	3.7		(1.0)	(0)	7.0	6.8	6.3
2386	973	132	5	"	1.8		3.5	15	3.7		(0.5)	(0)			
707	998	30	5	"	2	1	3	15	4.6		(2)	(0)		9.7(2 weeks)	7.5
706	"	"	5	"	1.5	1	2.5	15	3.8		(6.4)	(5)		9.2(2 weeks)	7.7

a- Figures in parentheses indicate percent syneresis after 30 days.

TABLE 18 Conc.

CLASSIFIED - CONFIDENTIAL

CLASSIFIED - CONFIDENTIAL

Surveillance Data

Gel No. F-	Notebook	Page	Composition of Gel, percent								remarks		Stability^a		Parallel Plate Values, cm.		
			ethyl cellulose		acids				solvent	40 % NaOH soln			days to breakdown				
			percent	grade									at -40°F	at 125°F	1 day	1 week	1 month
Solvent - acetone																	
Wood rosin, Hydrofol, and naphthenic acids					wood rosin	Hydrofol	naphthenic										
2356	973	120	5	200cps	2	2			20	2.8			(2.8)	(0)			
2332	"	114	5	"	2	2			20	2.4			(0)	(2)			
2338	"	116	5	"	2	2	2		20	4.0			(1)	10		10.1	10.0
Solvent - benzene					acid percent		name of acid										
					1	2	1	2									
2259	973	72	9.4	100 c.p.s.	1.9	1.9	naphthenic	oleic	37.6	2.2			(0)	(0)	8.4	7.2	
2263	"	"	4.7	"	1.9	1.9	"	"	32.9	2.2			(0)	(2)	9.0	9.4	syneresis
2261	"	"	9.3	"	1.85	2.8	wood rosin	"	37.0	2.7			(0)	(0)	10.7	8.6	7.8
2264	"	"	4.7	"	1.9	1.9	" "	Hydrofol	32.9	2.1			(0)	(0)	8.5	8.4	8.2
2265	"	"	4.6	"	2.3	2.3	naphthenic	"	13.9	2.8			(0)	28	8.5	syneresis	
Miscellaneous solvents					Hydrofol	naphthenic	wood rosin				name of solvent						
2336	973	116	5	200 c.p.s.	2	2			20	3.3	ethyl acetate		(0)	12		7.7	7.0
2287	"	96	5	"	2	2			20	2.3	" "		(0)	30		8.2	8.0
2337	"	116	5	"	2	2			10	3.3	isopropanol		(0.1)	12		8.6	8.2
2369	"	126	5	"	2	2			7.5	3.3	"		(0.3)	(10)		8.7	8.5
2348	"	120	5	"	2	2			7.5	3.3	"		(0.6)	12			
2351	973	120	5	200 cps.	2	2			5	3.8	isopropanol		(0.6)	7		10.3	9.0
2350	"	"	5	"	2	2			5	3.3	"		(1.1)	7		9.7	8.4
2330	"	112	5	20 c.p.s.	2	2			33	2.3	"			7			
2293	"	96	5	200 c.p.s.		2			10	2.3	"	2% oleic acid	(5.9)	7		10.8	12.2
2404	"	152	5	"	2	2			10	2.3	n-butanol		(0)	7	8.3		7.0
2401	973	152	5	200 c.p.s.	2	2			5	3.3	n-butanol		(0)	7	9.1		10.0
2403	"	"	5	"	2	2			3	3.3	"		(0)	7	9.9		10.2
2406	"	"	5	"	2	2	2		5	3.7	"		(0)	7	8.9		7.6
Vistanex (polyisobutylene) 140,000			Vistanex		Hydrofol	naphthenic	wood rosin										
			percent	grade													
2421	973	160	5	140,000	5					2.6			(0)	(2)			
2419	"	"	4	"		2.5	2.5			3.0			(0)				

a- Figures in parentheses give percent syneresis after 30 days

Table 19

M-69 Firing Tests at Standard Oil Development Company

	Composition of Gel, Per Cent									Static Firing Tests								
										at -40°F.			at Room Temp.			at 125°F.		
											Average % Gel			Average % Gel			Average % Gel	
	Methacrylate Polymer		Name of Acids			Per cent Acids												
Gel No.	Grade	%	1	2	3	1	2	3	Remarks	No. Bombs Fired	Adhering to Target	In Target Area	No. Bombs Fired	Adhering to Target	In Target Area	No. Bombs Fired	Adhering to Target	In Target Area
F-288	AE	3.0	Hydrofol	Naphthenic	-	2.5	2.5	-		3	4	95	3	55	100	2	60	63
288	AE	3.0	"	"	-	2.5	2.5	-		-	-	-	3	35	95	-	-	-
288	AE	3.0	"	"	-	2.5	2.5	-		-	-	-	5	28	94	-	-	-
613	0.035 I.P.*	2.0	Hydrofol	Naphthenic	-	3.5	3.5	-		1	5	55	2	10	70	-	-	-
614	AE	2.0	"	"	-	3.0	3.0	-	3.6% NaOH Sol'n.	3	7	93	3	55	80	3	10	35
614	AE	2.0	"	"	-	3.0	3.0	-	3.6% NaOH Sol'n.	-	-	-	3	57	90	-	-	-
614	AE	2.0	"	"	-	3.0	3.0	-	3.6% NaOH Sol'n.	1	2	82	2	8	80	-	-	-
616	0.2 Interpolymer	2.0	"	"	Wood rosin	2.0	2.0	2.0		3	15	55	3	35	90	3	20	45
616	"	2.0	"	"	" "	2.0	2.0	2.0		1	1	56	2	23	93	-	-	-
616	"	2.0	"	"	" "	2.0	2.0	2.0		-	-	-	3	47	93	-	-	-
624	0.1 Interpolymer	2.0	"	"	" "	2.0	2.0	2.0	0.5% α-cellulose	3	7	98	2	65	98	3	25	85
624	"	2.0	"	"	" "	2.0	2.0	2.0	0.5% α-cellulose	-	-	-	3	7	93			
624	"	2.0	"	"	" "	2.0	2.0	2.0	0.5% α-cellulose	1	1	91	2	35	95			
617	AE	2.0	Hydrofol	Naphthenic	Wood rosin	2.0	2.0	2.0	2.0% Darco	1	8	88	2	44	81			
575	0.3 Interpolymer	2.0	Stearic	"	" "	2.0	2.0	2.0	0.5% α-cellulose	1	10	30	1	2	100			
578	0.2 Interpolymer	2.0	"	"	" "	2.0	2.0	2.0	0.5% α-cellulose	1	5	75	1	2	100			
467A	0.1 Interpolymer	2.0	Oleic			1.75	-	-		1	5	10	1	7	93			
574	0.3 Interpolymer	2.0	"	Naphthenic		2.0	3.0	-		1	2	12	1	2	87			
573	0.1 I.P.	2.0	Oleic	Naphthenic		2.0	2.0	-		-	-	-	1	5	95			
615	AE	2.0	"	"	Hydrofol	1.0	3.0	1.0	0.5% α-cellulose	1	5	75	2	13	65			
2046	0.3 I.P.	2.0	Hydrofol	Wood rosin		2.0	2.0	-	Gel 5 days old	1	10	60	1	3	63			
2046	0.3 I.P.	2.0	"	" "		2.0	2.0	-	" 14 " "	-	-	-	1	0	30			
2045	0.3 I.P.	2.0	"	" "		1.5	1.5	-	" 5 " "	1	2	42	1	5	80			
2043	0.3 I.P.	2.0	Hydrofol	Wood rosin		1.5	1.5	-	Gel 14 days old	-	-	-	1	0	90			
619	0.1 "	2.0	"	" "		1.5	1.5	-		1	2	57	2	6	78			
620	0.1 "	2.0	"	" "		1.5	1.5	-	1% Darco	1	1	70	2	10	73			
621	AE	2.0	"	" "		1.5	1.5	-	0.5% α-cellulose	1	2	50	2	5	35			
2050	0.3 I.P.	2.0	Oleic	" "		2.0	4.0	-	Gel 5 days old	1	5	27	1	1	86			
2050	0.3 I.P.	2.0	Oleic	Wood rosin		2.0	4.0	-	Gel 14 days old	-	-	-	1	0	60			
2049	0.3 "	2.0	"	" "		2.0	3.0	-		1	5	35	1	15	90			
469A	0.3 "	1.75	"	" "		3.5	1.75	-		1	10	70	1	0	50			
2048	0.3 "	1.0	"	" "		2.0	4.0	-	Gel 5 days old	1	70	70	1	15	60			
2048	0.3 "	1.0	"	" "		2.0	4.0	-	Gel 14 days old	1	0	0	-	-	-			
2051	0.3 I.P.	1.0	Oleic	Wood rosin		2.0	4.0	-	5% bodied linseed oil	1	17	63	1	10	50			
622	0.2 "	2.0	"	" "	Hydrofol	2.0	3.0	1.0		1	5	55	2	5	70			
623	0.1 "	2.0	"	" "	"	2.0	1.75	1.5		1	2	7	2	7	82			
2047	0.3 "	1.5	"	" "	"	1.0	2.0	1.0	Gel 5 days old	1	10	85	1	10	60			
2047	0.3 "	1.5	"	" "	"	1.0	2.0	1.0	" 14 " "	-	-	-	1	0	60			
2052	0.3 I.P.	2.0	Oleic	Wood rosin	Stearic	2.0	3.0	1.0	Gel 5 days old	1	0	90	1	1	90			
2052	0.3 "	2.0	"	" "	"	2.0	3.0	1.0	" 14 " "	-	-	-	1	15	.25			
2052	0.3 "	2.0	"	" "	"	2.0	3.0	1.0	" 22 " "	-	-	-	2	10	100			
2052	0.3 "	2.0	"	" "	"	2.0	3.0	1.0	" 36 " "	1	15	100	-	-	-			
2053	0.3 I.P.	2.0	Oleic	Wood rosin	Stearic	2.0	1.75	1.5	Gel 5 days old	1	5	15	2	35	75			
2053	0.3 "	2.0	"	" "	"	2.0	1.75	1.5	" 22 " "	-	-	-	2	3	85			
2053	0.3 "	2.0	"	" "	"	2.0	1.75	1.5	" 36 " "	1	10	80	-	-	-			
2380	AE	2.0	Hydrofol	Turkey Red Oil		2.5	2.5	-		1	1	80	3	35	90			
612	0.3 I.P.	1.0	X-104			5.0	-	-		1	25	40	2	4	51			
2044	0.3 I.P.	1.0	X-104			5.0	-	-	5% bodied linseed oil	1	10	40	1	5	55			
	Ethyl Cellulose																	
2382	200 cps.	5.0	Hydrofol	Naphthenic	Oleic	1.0	1.0	4.0	20% acetone	1	3	60	3	40	70			
2386	"	5.0	"	Turkey Red Oil		3.5	1.8	-	15% "	1	40	50	3	53	53			
2369	"	5.0	"	Naphthenic		2.0	2.0	-	7.5% isopropanol	1	0	60	3	30	35			
3007	" AE Polymer	4.0) 1.0)	"	"		2.5	2.5	-	20% acetone	1	55	65	3	23	50			

I.P. = Interpolymer.

Table 20

Texas E-9 Mortar

Code of Gel	IM-4	A-5461	A-5942	A-5943	A-5944	A-5945	A-5946	A-5487	A-5488	A-5489	A-5490	A-5491B	A-5492
Composition, Wt. %													
Gasoline	86.5	90.7	73.0	71.3	70.7	70.0	69.0	73.5	74.5	75.0	73.6	73.05	71.3
Toluene	-	-	20.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0
AE Polymer	5.0	5.0	-	-	-	-	-	-	-	-	-	-	5.0
0.2 Interpolymer	-	-	-	-	-	-	-	-	-	-	4.0	5.0	-
0.3 Interpolymer	-	-	6.0	5.0	5.0	5.0	5.0	4.0	4.0	4.0	-	-	-
Stearic Acid	4.0	3.0	-	2.0	-	1.0	1.5	-	-	-	1.0	0.25	2.0
Naphthenic Acid	-	-	-	-	-	-	1.5	1.0	-	-	-	-	-
Turkey Red Oil	-	1.0	-	-	2.0	-	-	-	-	-	-	-	-
T-132 Resin	-	-	-	-	-	2.0	-	-	-	-	-	-	-
alpha-Cellulose	-	-	-	-	-	-	-	-	0.5	-	-	0.5	-
40% NaOH	-	2.3	1.0	1.7	2.3	2.0	2.5	1.5	1.0	1.0	1.4	1.2	1.7
CaO	4.0	-	-	-	-	-	-	-	-	-	-	-	-
H_2O	2.5	-	-	-	-	-	-	-	-	-	-	-	-
Physical Properties													
Parallel Plate (cm.)	7.2	10.6	6.8	6.4	6.6	6.2	6.3	10.0	6.6	7.5	8.0	6.8	7.6
Impact Strength - D Scale Units	4.5	-	13.7	8.9	14.2	10.7	16.0	11.5	11.4	10.2	11.7	13.8	8.4
Relative Burning Times - Method B	-	-	3.9	4.7	5.7	6.5	6.4	2.7	4.3	2.8	3.3	4.2	3.6
Mortar Firing Test													
Target	Steel backed		----------Cinder block backed----------										
Location of Gel													
On Target Vertical Wall	40	100	-	-	-	5	-	15 -	-	-	-	-	10
On Apron	-	-	95	90	85	5	10	- 35	95 20	90 80	15	10	25
On Ground	15	-	5	10	5	80	-	- -	5 10	- -	-	70	-
In Gun	5	-	-	-	10	10	20	5 5	- -	- -	-	10	-
Beyond Target	40	-	-	-	-	-	70	80 60	- 70	10 20	85	10	65
Time to Initiate Vigorous Wood Fire, Min.	3	-	1.5	6	4	6	-	- 1	1 2	1.5 1	5	5	-
Peak of Fire, Min.	-	1	4	-	5	10	10	1 3	2 10	3.0 2.5	-	-	2.5
% of Gel Burned in 10 min.	-	100	100	100	80	40	25	- 100	95 90	80 100	-	-	80
% of Fuel in Effective Location	40	100	95	90	85	10	10	15 35	95 20	90 80	15	-	35
% of Target Destroyed in 10 Min.	48	3 Gel shattered into spray; fire over at 5 min.	70	50	80	22	0	0 43 Flash fire charcoal wood only.	43 14	43 55	11	- Gel bounced off target to ground. Fire extinguished at 5 min.	30

Notes: Gasoline: Special Naphtha #2, Pure Oil Company.

AE Polymer: Isobutyl Methacrylate Polymer.

0.2 Interpolymer: Interpolymer of Isobutyl Methacrylate with 0.2% Methacrylic Acid.

0.3 Interpolymer: " " " " " 0.3% " "

T-132 Resin: Product of Stanco Products Company.

Table 21

Frangible Grenade Fillings

Gel No. F-	Composition (wt %)									Physical Properties				Observations in CWS Test at 70 °F	
	AE Polymer	Stearic Acid	Naphthenic Acid	Dimerized Soya Bean Oil Acid	Turkey Red Oil	CaO	H_2O	$Ca(OH)_2$	40% NaOH	Mobilometer Viscosity (g/10cm/1.5min) gel 3 days old	Mobilometer Viscosity (g/10cm/1.5min) at time of test	Stormer Value RPM/800g. gel 3 days old	Stormer Value RPM/800g. at time of test	Unignited Gel	Gel ignited by M-3 Ignitor.
1788	3	3	1	—	—	3.5	2.2	—	—	759	460	2	5	Too thick, did not spread	Did not stick or spread, too thick
1789	3	2	2	—	—	—	0.75	3	—	119	112	103	91	Spread well on impact ran down wall slowly	Very good. Spreads and flows well. Equal to F-1792.
1790	3	3	—	—	1.0	3.5	2.2	—	—	78	72	60	39	Spread fair.	Spreads and runs well. Equal to F-1789 + 1792
1791	5	1	2	—	—	2.3	1.7	—	—	144	123	17	29	Too thick Better than F-1788	Does not spread but runs well Gel slow burning
1792	3	1	3	—	—	3.1	2.3	—	—	82	20	74	104	Spread fair.	Similar to F-1789 + 1790
1794	3	1	3	—	—	2	1.5	—	—	5	5	90	290	Spreads and runs well	Too thin, scattered over wide area.
1795	3	1	3	—	—	3.1(1)	2.3	—	—	3	3	147	260	Does not adhere Poor spread.	Spreads and runs well
2553	2	2	—	2	—	—	—	—	2.1			400		Does not adhere.	Rolls off target.

Note (1) A coarse grade of lime was used in gel F-1795. Its analysis was 100% thru 40 mesh 30% thru 100 mesh screens.

Conclusions

Gels F-1789, F-1790, F-1792, and F-1795 are all equally good and approximately the same as F-1416. The requirements of the munition are met by gels with physical properties in the following range.

Modified Stormer Viscosity 40-200 RPM/800g

Mobilometer Value: 20-120 g/1.5min/10 cm

Table 22

Pilot Plant Continuous Preparation of F-614 Gels

Run F-C #	Notebook #	Page #	Composition: Type and Per Cent Ingredients							Operating Conditions			Physical Data on Product								Purpose
			Polymer				40% NaOH Solution						Impact Strength C Units		Parallel Plate (cm.)		Surveillance				
																	-40°F.		+125°F.		
			Type*	Per Cent	Stearic Acid	Naphthenic Acid	By Material Balance	By Analysis	Gasoline Type	Mixing Speed (RPM)	Throughput Rate (lb./min.)	Product Weight (lb.)	1 Day	2 Weeks	1 Day	2 Weeks	1 Week	1 Mo.	1 Week	1 Mo.	
8	973	179	AE	2	3	3	5.24	4.9	Purol 112" AP	1725	2.98	18.5	7.7	7.7	9.6	8.1	0	0	0	8	For static firing tests
9	973	179	AE	2	3	3	4.6	4.6	" "	1725	3.42	26.0	7.2	7.2	8.4	8.2	0	0	0	15	
10	973	179	AE	2	3	3	4.03	4.0	" "	1725	3.76	27.5	8.8	7.9	8.3	9.1	0	0	1	15	
11	973	182	AE	2	3	3	4.38	4.5	" "	1725	3.63	24.3	6.4	6.6	9.1	8.9	0	0	0.5	6	
12	973	182	AE	2	3	3	4.06	4.1	" "	1725	3.91	26.9	6.3	7.7	9.6	9.0	0	0	0.5	7	
13	973	183	AE	2	3	3	4.47	4.5	Purol 112" AP	1725	3.61	26.8	4.9	5.3	10.1	9.5	0	0	1	3	
14	973	183	AE	2	3	3	4.08	4.1	" "	1725	3.88	27.3	4.3	5.5	10.6	9.5	0	0	0.5	5	
15	973	185	NR	2	3	3	4.02	4.05	" "	1725	3.90	25.9	4.2	4.4	10.4	10.6	0	0	1	10	
16	973	185	NR	2	3	3	4.93	5.0	" "	1725	3.28	25.4	5.0	4.4	11.3	11.2	0	0	1	8	
17	973	187	0.1 I.P.	2	3	3	4.35	4.3	" "	1725	3.68	25.2	7.8	10.3	8.2	9.4	0	0	-	25	
18	973	187	0.1 I.P.	2	3	3	5.45	5.4	Purol 112" AP	1725	2.96	24.0	5.9	9.4	8.4	10.1	0	0	3	15	
19	973	188	0.07 I.P.	2	3	3	4.09	4.05	" "	1725	3.75	26.0	5.7	6.9	10.1	10.4	0	0	0	10	
20	973	189	AE	2	3	3	6.97 (34.5% NaOH)	6.54	" "	1725	2.38	24.0	6.9	7.3	9.1	9.4	0	0	0	5	
21	973	189	AE	2	3	3	4.32	4.25	Purol 98" AP	1725	3.72	24.2	5.7	6.0	9.1	9.0	0	0	0	5	
22	973	190	AE	2	3	3	4.82	4.9	" 112" AP	1725	3.32	24.6	4.8	4.8	9.1	10.2	0	0	1	10	
23	1059	1	0.07 I.P.	2	3	3	4.83	4.3	Purol 112" AP	1725	3.18	27.6	6.0	7.2	9.0	9.1	0	0	2	10	
24	1059	3	AE	2	3	3	5.03	4.7	" "	1725	3.17	27.4	5.8	5.1	9.4	9.4	0	0	0	20	
25	1059	3	AE	2	3	3	4.92	5.0	" 98" AP	1725	3.24	24.4	4.9	6.1	9.7	9.0	0	0	-	15	
26	1059	4	AE	2	3	3	4.72	4.9	" "	1725	3.40	24.5	5.1	4.9	9.5	9.6	0	0	-	10	
27	1059	4	0.1 I.P.	2	3	3	4.29	4.5	" "	1725	3.74	26.1	5.8	5.0	9.8	9.1	0	0	-	15	
28	1059	6	AE	2	3	3	4.72	5.1	Purol 98" AP	1725	3.40	26.6	5.0	-	8.6	-	0	0	0	10	
29	1059	6	AE	2	2.5	2.5	4.35	4.45	" "	1725	3.63	21.6	4.4	4.7	9.3	10.7	0	0	0	15	
30	1059	9	AE	2	3	3	4.45	4.45	" "	1725	3.62	27.2	4.2	3.8	10.2	9.9	0	0	0	2	
31	1059	13	AE	2	3	3	4.58	4.35	Purol 98" AP	1725	3.48	75.6	4.7	-	9.5	-	0	0	0	25	Ignition Tests
32	1059	16	AE	2	3	3	4.65	4.57	" "	1725	3.46	136.0	4.7	-	9.4	-	0	0	0	15	
34	1059	30	AE	2	3	3	4.58	-	Purol 98" AP	1100	3.46	9.5	6.0	6.3	8.9	9.0	0	0	0	0	To test effect of mixing speed.
35	1059	31	AE	2	3	3	4.55	-	" "	765	3.57	13.5	5.9	7.3	9.5	9.1	-	-	-	-	
36	1059	31	AE	2	3	3	4.43	-	" "	520	3.71	11.3	5.5	6.7	9.3	9.2	-	-	-	-	
37	1059	31	AE	2	3	3	4.43	-	" "	350	3.66	11.8	4.6	6.5	9.9	9.0	0	0	0	4	
38	1059	31	AE	2	3	3	4.36	-	" "	230	3.81	12.05	5.8	6.0	10.2	9.1	0	0	0	1	
39	1059	32	AE	2	3	3	4.33	-	" "	165	3.78	19.5	5.0	5.8	11.0	9.6	-	-	-	-	
40	1059	34	AE	2	3	3	4.52	(32.3% NaOH Sol'n.)	Purol 98" AP	1100	3.28	10.5	8.9	17.8	10.6	10.2	5.9	5.5	0	20	
41	1059	34	AE	2	3	3	5.25		" "	765	2.91	9.1	8.9	15.0	11.1	10.2	2.8	3.1	0	20	
42	1059	35	AE	2	3	3	5.36		" "	520	2.78	8.7	9.0	13.5	11.2	9.9	-	-	-	-	
43	1059	35	AE	2	3	3	4.86		" "	350	3.07	9.85	6.9	15.1	11.2	10.7	3.1	2.9	0.5	5	
44	1059	35	AE	2	3	3	4.66		" "	230	3.26	10.0	12.3	16.1	11.3	10.1	3.6	3.1	1	10	
45	1059	35	AE	2	3	3	4.81		" "	165	3.11	9.7	8.1	16.1	12.5	11.1	-	-	-	-	
46	1059	40	AE	2	3	3	3.63	-	Purol 98" AP	165	8.04	19.2	6.5	6.3	12.5	11.7	0	0	-	-	
47	1059	41	AE	2	3	3	3.54	-	" "	230	8.05	18.9	5.9	6.4	11.8	11.4	0	0	-	-	
48	1059	41	AE	2	3	3	3.71	-	" "	350	7.80	18.25	6.0	6.4	11.3	11.5	-	-	-	-	
49	1059	41	AE	2	3	3	3.70	-	" "	520	7.70	18.8	6.2	6.5	10.9	11.1	0	0	-	-	
50	1059	41	AE	2	3	3	3.63	-	" "	625	7.93	21.0	7.3	6.9	11.2	11.6	-	-	-	-	
51	1059	42	AE	2	3	3	3.59	-	" "	1100	8.17	21.5	8.0	7.6	11.3	11.6	0	0	-	-	
52	1059	42	AE	2	3	3	3.40	-	" "	1725	8.41	20.85	8.1	11.0	11.8	11.0	-	-	-	-	
53	1059	53	AE	2	3	3	4.65	-	Purol 98" AP	165	6.00	14.95	7.0	5.9	10.1	9.8	-	-	-	-	
54	1059	54	AE	2	3	3	4.78	-	" "	230	5.87	14.5	6.5	6.1	9.2	9.1	-	-	-	-	
55	1059	54	AE	2	3	3	4.75	4.56	" "	350	5.88	14.45	6.7	6.9	9.4	9.1	-	-	-	-	
56	1059	54	AE	2	3	3	4.11	3.92	" "	520	6.67	15.95	6.7	7.1	9.3	10.2	-	-	-	-	
57	1059	54	AE	2	3	3	4.00	3.74	" "	625	6.88	18.3	5.7	6.1	10.2	10.3	-	-	-	-	
58	1059	55	AE	2	3	3	4.25	-	" "	1100	6.55	16.05	4.9	5.4	10.8	10.2	-	-	-	-	
59	1059	55	AE	2	3	3	4.17	-	" "	1725	6.73	20.4	4.9	5.5	10.2	10.4	-	-	-	-	

*<u>Code</u>: NR and AE isobutyl methacrylate polymers. I.P. interpolymers of isobutyl methacrylate and methacrylic acid.

Table 23

Performance Tests on Pilot Plant F-614 Gels

Gel Number FC-	Sample Number A-	Composition			Firing Tests					Parallel Plate Values (cm)				Impact Strength B units				
		Polymer	Gasoline	% of 40% NaOH	-40°F		77°F		Age of Gel-days	Batch Lab. Sample		Continuous Sample		Laboratory Sample		Continuous Sample		
					% Scatter	% Adhesion	% Scatter	% Adhesion		at 24 hrs	Day of Test	24 hrs	Day of Firing	24 hrs	Day of Test	24 hrs	Day of Firing	
8	3055	AE a	Purol 112°	5.0	48	2	5	30	18			9.6	8.1			7.7	7.7	
9	3056	"	"	4.5	48	2	5	87	18			8.4	8.2			7.2	7.2	
10	3057	"	"	4.0	35	0	7	70	18			8.3	9.1			8.8	7.9	
11	3058	AE b	"	4.5	48	2	10	82	15			9.1	8.9			6.4	6.6	
12	3059	"	"	4.1	48	2	5	20	15	8.4	7.9	9.6	9.0		8.8	6.3	7.7	
13	3060	AE c	P 112°	4.5	85	5	37	7	14	10.2	8.3	10.1	9.5			4.9	5.3	
13	"	"	"	"			8	25	31	10.2	8.3	10.1	9.8			4.9	6.5	
14	3061	"	"	4.1	65	5	15	20	14	9.4	8.9	10.6	9.5			4.3	5.5	
15	3066	NR	"	4.0	30	10	6	40	23	10.8	10.8	10.4	10.6	6.0	5.0	4.2	4.4	
16	3067	NR	"	5.0	40	10	2	50	23	12.8	10.2	11.3	11.2	3.4	4.2	5.0	4.4	
17	3068	I.P 0.1%	P 112°	4.3	20	5	20	5	19	8.2	8.2	8.2	9.4	8.6	5.5	7.8	10.3	
18	3069	IP 0.1%	"	5.4	45	10	3	10	19	8.6	9.3	8.4	10.1	8.6	4.9	5.9	9.4	
19	3070	IP 0.07%	"	4.0	28	2	5	20	17	9.2	9.2	10.1	10.4	7.4	3.9	5.7	6.9	
20	3071	AE a)	"	note*	20	15	2	98	16	8.6	9.4	9.1	9.4	8.1	7.7	6.9	7.3	*6.2% of 35% NaOH
21	3072	AE a	Purol 98°	4.3	1	1	5	40	16	8.8	8.9	9.1	9.0	8.1	7.8	5.7	6.0	
22	3073	AE c	P 112°	4.9	14	1	10	50	16	10.3	8.3	9.1	10.2	6.5		4.8	4.8	
23	3076	IP 0.07%	"	4.3								9.0	9.1			6.0	7.2	
24	3077	AE b	"	4.7	55	10	0	90	16	9.1	8.5	9.4	9.4	7.4	7.8	5.8	5.1	
25	3078	AE b	P 98°	5.0	20	5	5	50	16	9.1	8.5	9.7	9.0	7.9	8.3	4.9	6.1	
26	3079	AE c	"	4.9	20	15	10	53	15	8.9	8.6	9.5	9.6	6.3	5.7	5.1	4.9	
27	3080	IP 0.07%	P 98°	4.5	10	5	10	27	15	9.2	8.7	9.8	9.1	6.6	8.3	5.8	5.0	
29	3081	AE c	"	5.1	22	8	38	43	11	9.8	9.1	9.3	10.7	5.5	6.3	4.4	4.7	} Only 5% Organic Acids
29	"	"	"	5.1	-	-	42	12	32	9.8	9.1	9.3		5.5		4.4		
30	3082	"	"	4.5	15	15	38	15	9			10.2	9.9			4.2	3.8	
30	"	"	"	4.5	-	-	17	56	16			10.2				4.2		
31	3084	AE b	P 98°	4.6	5	5	25	40	7			9.3				4.2		
32	3084	"	"	4.6	23	2	10	40	24			9.1				4.6		

10 Foot Drop Ignition Tests

Gel No FC-	Age days	Temperature	No. of Cases Tested	% Ignited
31	7	-40°F	20	97
31	7	70	10	100
32	7	70	10	90
32	24	-40	19	100
32	24	70	20	85
			Average	95

CLASSIFIED - CONFIDENTIAL

Table 24

CLASSIFIED - CONFIDENTIAL

Action of Solvents on Representative Synthetic Polymers

	Solvent	Gasoline	Benzene Gasoline Mixture	Toluene	Turpentine	Petroleum Fractions				Stoddard Solvent.
	* Aromatic Content % S.O.D. Sample number initial b.p. °F final b.p. °F		39			22 3618C 555 587	32 3616A 448 492	36 3617C 497 544	95 3619D 430 520	
	Polymer Content Tested.	5	3	5	5	10	10	10	10	10
Composition of Polymer	Polymer Trade Name									
Polyisobutylene	Vistanex 100,000	soluble.			soluble	soluble	soluble	soluble	soluble	soluble
Ethyl cellulose	Ethocal	insoluble	soluble		swells strongly	insoluble	slight swelling		—	insoluble
polystyrene	Styron	insoluble.		soluble.	soluble	swells	—	—	soluble	
polymethylmethacrylate	"Lucite"	insoluble.		soluble		insoluble	—	—	—	—
polyvinyl butyral	~~Butacite~~	insoluble.			swells strongly	insoluble	slight swelling		swells	
polyvinyl acetate		insoluble								
Cellulose acetate propionate		insoluble			insoluble	—	—	—	—	—
Cellulose acetate butyrate		insoluble			insoluble	—	—	—	—	—
Cellulose acetate stearate		insoluble	some gel formed.							
Cellulose acetate phthalate		insoluble	insoluble							
Cellulose caproate.		insoluble	insoluble							
Polyvinyl acetal	Alvar.	insoluble.								
Polyvinyl campholate		3% soluble.								
Polyvinyl alcohol		insoluble								
[illegible] ethyl Cellulose		softens	insoluble							
[illegible] methyl Cellulose		softens.	some gel formation							
Cetyl ethyl Cellulose		softens	swells slightly							
Casein.		insoluble.								
Isobutylene-vinyl acetate-fumaric anhydride interpolymer.		insoluble.								

* Petroleum fractions were procured by the Standard Oil Development Co. from the Bayway Refinery of the Standard Oil Company of N.J.

CLASSIFIED - CONFIDENTIAL

Table 25

Preparation of Cellulose Esters

Run No.	Type of Reactions	Reference N.B.	Reference Page	Source of Cellulose Type	Source of Cellulose g.	Reagents, g. 90% Stearyl Chloride	Reagents, g. Stearic Anhydride	Reagents, g. Acetyl Chloride	Reagents, g. Acetic Anhydride	Reagents, g. Lauryl Chloride	Solvents, g. Benzene	Solvents, g. Xylene	Solvents, g. CH_2Cl_2	Solvents, g. Stearic Acid	Solvents, g. Acetic Acid	Catalyst Pyridine	Catalyst Quinoline	Catalyst Iodine	Catalyst Picoline	Catalyst Sulfoacetic Acid	Catalyst $POCl_3$	Reaction Conditions Time Hours	Reaction Conditions Temperature Range °C.	Product Yield g.	Product Yield %	Product Color and Form	Product Solubility In Gasoline	Product Solubility In Naphthenic Acid	Literature Reference to Reaction Attempted	Remarks
S-1	A	1059	69	Sodium Cellulose	2	60					81											1 3	50-53 25	-	-				U.S.Patent 2,294,925	
S-2	A		72	"	2	60					81											1 3	50-55 25	-	-				"	
S-3	A		76	"	2	60					81											3 1	60-66 25	-	-				"	
S-4	C		78	"	2			60			81											1.5 3	48-52 25	0.3	15	Yellow flakes			"	
S-5	C		84	"	2			60			81											2 2	50-55 25	0.32	16	"			"	
S-6	A		91	A.C.	5	33						195		30		25						20 5	94-96 134-8	22.2	445	Tan fiber	Swells		Br.Patents 283.181 & 305,947	Probably distearate.
S-9	D		101	"	3					14.3		87				12.3						20.5 2	92-96 133-138	5.7	190	"	Sl.swells	Insol.	Br.Patent 297,766	Probably dilaurate.
S-10	E		103	S-6	7.5							65									8.4	1-1/4	100-115	0.2	-	Black	Insol.		Br.292,929	
S-11	A		106	A.C.	5	35						150				25						20-3/4 4	93-96 133-4	19.8	396	Tan fibers.	Very sl. swelling	Sl.swelling	See S-6.	
S-12	C		108	"	5				29		76				18					3.5		120	25	8.7	174	White fibers.	Insol.		Ger.Patent 663,154	Cellulose pretreated. 8 g. HOAc in vacuum.
S-14	E		112	S-11	5							48									0.55	2	90	4.8	-	Tan powder.	Sl.swelling.	Sl.swelling.	See S-10	
S-15	A		113	A.C.	10	67						260				50						21	90-100	48.2	482	Cream colored fibers.	Swells.	Partly sol.	See S-6	Largely distearate product.
S-16	B		115	S-15	5	24							100			25						4	130-145	7.25	145	Coarse fibers.	Swells.	Partly sol.		No improvement over S-15.
S-17	A		117	A.C.	5	33								100		25						21	95-99	4.77	-	Fibers.	Insol.	Insol.		
S-18	A		120	"	5		75					110			9					3.5		360	25	2.7	-	White fibers.	Insol.	Insol.	See S-12.	Pretreated cellulose as in S-12.
S-19	B		123	S-15	5	20							50			20						3	126-143	10.67	213	Brown fibers.	Sl.swelling.	Sol.		
S-20	E		125	S-15	5	2							60									4	144-6	4.9	-	Fibers.	Strong " .	Insol.		
S-21	A		132	A.C.	5	56						81				50						7	125-8	28.8	576		" " .	Sol.	H.Pringsheim: Cellulosiechemie 13, 119 (1932)	Largely tristearate.
S-22	E		134	S-15	5									75								5	195-208	1.7	-	Grey powder.	Sol. hot.	Insol.		
S-23	A		135	Rayon	5	56						83				50						7	129-136	20.9	419	Fibers.	Swells.	Sl.swelling.	See S-21.	S-21 product preferred.
S-24	A		140	A.C.	5	56						83					50					3.5	147-8	2.9	-	Dark powder.	Insol.	Insol.	See S-21.	" " "
S-25	E		141	S-15	5	3							50									5.5	145-9	5.1	-		Strong swelling.	Sol. hot.		
S-26	A		144	A.C.	5	56						83							50			2	140-2	4.7	-	Black powder.	Insol.	Insol.		
S-27	E		146	S-21	5	3							50									5	147-8	4.55	-	Tan powder.	Strong swelling.	Sol. hot.		
R-3	A	1049	171	Rayon	23.5	220					9						150	0.1				0.3 8	70 130-160	-	-	Brown wax.	90% sol. 10% swells.			Gas. solution of low viscosity.

*A - Preparation of tristearate from cellulose.
B - Preparation of tristearate from distearate.
C - Preparation of triacetate.
D - Preparation of trilaurate.
E - Solubilization of di- or tristearate.

A.C. - alpha-cellulose.

Significant Runs - Cellulose Distearate S-6, S-11, S-15.
Cellulose Tristearate S-21, R-3, S-25, S-27.
Cellulose Dilaurate S-9.

CLASSIFIED - CONFIDENTIAL

TABLE 26

CLASSIFIED - CONFIDENTIAL

Polyvinyl Acetals

Run No.	Page in nbk 998	Reaction Conditions, First Step								Reaction Conditions Second Step						Results
		2 ethyl hexaldehyde mole/mol pv alcohol	other aldehyde or alcohol name	mol/mol pv alcohol	solvent	temp. °C	time, hrs	catalyst	stirring	2 ethyl hexaldehyde mol/mol pv alcohol	salicylal gr./gr. polymer	solvent	Temp. °C	Time hrs.	catalyst	
Completely hydrolyzed polyvinyl alcohol (RH 391, 1.4 % pv acetate)																
A-1	155	1.0			butyl acetate butyl alcohol	70-80	19	H_2SO_4	yes		0.039	butyl acetate butyl alcohol	70-80	19	H_2SO_4	first step - soluble C_6H_6 + gasoline, 82% acetal second step - insoluble common solvents
A-2	158	1.0			butyl acetate butyl alcohol	70-80	19	H_2SO_4	yes							gasoline soluble
A-3	172	1.0	salicylal	.005	butyl acetate butyl alcohol	70-80	19	H_2SO_4	yes							salicylaldehyde not introduced into polymer
A-4	180	1.0	salicylal	.025	butyl acetate butyl alcohol	70-80	72	H_2SO_4	yes							salicylaldehyde not introduced into polymer; some degradation
A-5	181	1.0	hydroxy-stearic acid	.050	butyl acetate butyl alcohol	70-80	72	H_2SO_4	yes							hydroxystearic not introduced, some degradation
A-6	158	1.0			butyl acetate 2 ethyl hexanol	70-80	19	H_2SO_4	yes							soluble in C_6H_6; partially soluble gasoline 67% acetal
A-7	155	0.5			acetic	70-80	19	H_3PO_4	yes							C_6H_6 soluble; insoluble gasoline
A-8	145	0.96			acetic	60-80	16	H_3PO_4	yes	0.125		benzene	boiling	1		first step - insoluble C_6H_6, 27% acetal second step - partial cross-linking
A-9	156	0.5			acetic	70-80	64	$Cl_3C-COOH$	some							soluble in C_6H_6, insoluble in gasoline
A-10	157	1.0			acetic	70-80	19	H_2SO_4	yes							soluble in C_6H_6; swells in gasoline
A-11	157				ethanol - water	25	48	HCl	no							polymer - water - ethanol mixture too thick to be handled
Partially hydrolyzed polyvinyl alcohol (RH 403, 28% pv acetate)																
A-12	137	0.5			acetic	60-70	2	H_3PO_4	yes	0.125		benzene	boiling	2½	H_3PO_4; p-tol. sulfonic	second step - soluble C_6H_6, insoluble gasoline some cross-linking.
A-13	109-110	0.5	salicylal	0.020	acetic	75-95	5	H_3PO_4	yes	0.137		benzene	boiling	5	H_3PO_4	first and second steps - C_6H_6 soluble, second step - 58.6 % acetal, both gasoline insoluble.
A-14	107	0.5	salicylal	0.020	dioxane	75-80	5½	H_3PO_4	yes							no acetal formation
A-15	108	0.5	salicylal	0.020	dioxane	boiling	7	H_3PO_4	yes							no acetal formation

CLASSIFIED - CONFIDENTIAL

Table 27

CLASSIFIED - CONFIDENTIAL

Polyvinyl Esters

CLASSIFIED - CONFIDENTIAL

Run No.	Page in n'bk. 99	Reaction Conditions: other component name	mol/mol p.v. derivative	solvent	temp. °C	time, hrs	catalyst	pressure mm. Hg	remarks	Results
From polyvinyl acetate										
S-1	83	stearic acid	2.00		170 - 200	136	H_3PO_4	760	nitrogen-blanketed	polymer degraded
S-2	128	stearic acid	1.00	xylene, benzene	boiling	10	$ZnCl_2$	760	nitrogen-blanketed	Thick, rubbery polymer insoluble in hot xylene
S-3	129	stearic acid	2.00		150-175	4	o-toluene sulfonic ac.	30		61 % theoretical acetic recovered. Polymer badly degraded
S-4	159	stearic acid	1.00		~ 175	6		30		very little reaction
S-5	161	stearic acid	1.00		200	48	litharge	30		very little reaction
From polyvinyl alcohol (RH 403, 28% p.v. acetate)										
O-1	74	oleic acid	2.00	dioxane	boiling	7	H_3PO_4	760		no esterification
O-2	74	oleic acid	2.00	dimethyl formamide	boiling	7	H_3PO_4	760		no esterification
O-3	74	oleic acid	2.00	"triclene" (trichloroethane)	boiling	7	H_3PO_4	760		no esterification
S-6	127	stearic acid	1.00	benzene	boiling	6	H_2SO_4	760		no esterification
S-7	127	stearic acid	1.00	benzene	boiling	8	H_2SO_4 $Hg(C_2H_3O_2)_2$	760		no esterification
S-8	122	stearyl chloride	1.00	benzene, pyridine	50-60	24		760		no esterification
S-9	126	stearyl chloride	1.00	benzene	boiling	5	H_2SO_4	760		no esterification
S-10	131	stearic anhydride	0.50	pyridine-pyridine hydrochloride	boiling	3		760		little or no esterification
S-11 A	124	sodium	1.00	liquid ammonia	-33	96		760		sodium polyvinylate obtained. reacted with stearyl chloride as described below.
* S-11 B	124	stearyl chloride	1.10	benzene	boiling	8		760		polymer insoluble in common solvents
From polyvinyl chloride										
S-12	160	sodium stearate	1.10	dioxane	boiling	7		760		no reaction
From vinyl acetate monomer										
S-13	84	stearic acid	0.50		boiling	8	H_3PO_4 $Hg(C_2H_3O_2)_2$	vacuum topped		Some reaction, product not worked up
S-14	91	stearic acid	1.12		89-180	23	H_3PO_4, H_2SO_4, + $Hg(C_2H_3O_2)_2$	760		reaction 40 % complete. No method of isolating pure vinyl stearate obtained.

* Stearyl chloride reacted with sodium polyvinylate

CLASSIFIED - CONFIDENTIAL

REEL – C

1344

A.T.I.

31510

CONFIDENTIAL

TITLE: Synthetic Polymers as Gasoline Thickening Agents

OVER U

AUTHOR(S): Kirkpatrick, E. C.
ORIGINATING AGENCY: E. I. du Pont de Nemours and Co., Wilmington, Del.
PUBLISHED BY: Office of Scientific Research and Development, NDRC, Div 11

ATI- 31510
REVISION
(None)
ORIG. AGENCY NO.
(None)
PUBLISHING AGENCY NO.
OSRD-4202

DATE	DOC. CLASS.	COUNTRY	LANGUAGE	PAGES	ILLUSTRATIONS
Oct '44	Confd'l	U.S.	Eng.	113	tables

ABSTRACT:

A laboratory formulation study was undertaken to develop new gasoline incendiary mixtures containing polymeric viscolizing agents. Major emphasis was placed on combinations of isobutyl methacrylate polymers with diverse soap forming acids, strong bases, fillers, and gasoline with or without other fuels. The optimum type and amount of each of the basic gel ingredients were determined. Based on physical studies and surveillance tests, 80 gel formulas were chosen for evaluation in performance tests in one of five munitions. Three methacrylate gel formulas have been accepted and manufactured. Problems of commercial gel manufacture were investigated in a continuous pilot plant. A series of gel formulas of graded toughness, representing the compositions prepared during this study and most suitable for use as incendiary fillings, are listed.

DISTRIBUTION: Copies of this report obtainable from Air Documents Division; Attn: MCIDXD

DIVISION: ~~Ordnance and Armament (22)~~ 57
SECTION: ~~Chemicals and Incendiaries (11)~~ 3

3
3

ATI SHEET NO.: C-22-11-65

SUBJECT HEADINGS: Incendiaries - Development (50960.6)

Air Documents Division, Intelligence Department
Air Materiel Command

AIR TECHNICAL INDEX

Wright-Patterson Air Force Base
Dayton, Ohio

CONFIDENTIAL

CLASSIFICATION CANCELLED
(RF) By authority OSRD List #3,
Dated 2-11 January 1946
By , USCO

18 SEP 1950

P 19/1/1

(23)

* Incendiary mixtures
fuel thickeners