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Report on "Comparative Tests of Various Incendiary Mixtures - Part IV. - Improvements in the Test Procedure, Evaluation of Different Types of Rubber"

to
November 28, 1941

by
L. F. Fieser

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Report on "Comparative Tests Of Various Incendiary Mixtures." (C.W.S.-21)

Endorsement (1) From Dr. L. F. Fieser, Member Section B-7-D, to Dr. E. P. Stevenson, Chairman, Section B-7. Forwarding report.

(2) From Dr. E. P. Stevenson, Chairman, Section B-7 to Dr. Roger Adams, Chairman, Division B. Forwarding report and noting:

> "This is a report of the progress since the previous report of November 3, 1941. The material covers improvements in test procedure, evaluation of different types of rubber, and comparison of the incendiary effect on three different kinds of wood."

(3) Twenty-one copies forwarded to Dr. Irvin Stewart, Secretary of the National Defense Research Committee, as Progress Report under Contract (B-186, NDCrc) with Harvard University.

Roger Adams, Chairman
by Harris M. Chadwell
Technical Aide

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COMPARATIVE TESTS OF VARIOUS INCENDIARY MIXTURES

PART IV. IMPROVEMENTS IN THE TEST PROCEDURE, EVALUATION OF DIFFERENT TYPES OF RUBBER

by

LOUIS F. FIESER

ABSTRACT

The procedure of the wood-burning test has been revised and improved in several details. The standard for comparison has been changed from a weight to a volume basis, and the results are now referred directly to a standard volume ([illegible] cc.) of the test sample equivalent to that of 85 g. of a 7% solution of smoked sheet rubber in benzene. Because of this change, and also because the burning is now done indoors in a completely quiet atmosphere, the present results are assigned to a "New Series" and are not directly comparable with the data of Parts I-III. Such tentative conclusions as were reached on the basis of the earlier results are confirmed by the more accurate data of the present report.

The investigation of gums of varying concentration of rubber of three types with both benzene and naphtha as the solvent has indicated that solutions of very low rubber content and low viscosity have inferior incendiary characteristics. As the rubber concentration increases the performance improves for a time and then reaches a maximum. The amount of rubber required to give maximum effectiveness

varies considerably with the type of rubber, that is, with the extent of processing to which it has been subjected and the consequent degree of depolymerization. The concentrations indicated by the present results as required to give fully effective gums in either benzene or naphtha are as follows: pale crepe rubber, 5%; latex, 5-5.5% (actual content of pure rubber); smoked sheet rubber, 7%. Reclaimed rubber is wholly unsuitable for the purpose.

Gums compounded from light naphthas are rather inferior in quality, but with petroleum fractions in the heavy solvent-naphtha group gums can be obtained which are fully effective and equal to the best gums compounded from benzene or other solvent. With a suitable blending of about 10% of the light naphtha to provide for ignition even at low temperatures, such gums prepared with the use of either crepe rubber or latex provide the best filling as yet considered for the oil bomb.

Miscellaneous mixtures studied include solutions of polybutene in naphtha, mixtures of rubber-benzene with sodium nitrate and with nitrocellulose, and celluloid. A few comparative tests on structures made of hemlock, spruce, and oak are reported.

REPORT

The test method described in Part I of this series appears to provide a reasonably satisfactory means of appraising the incendiary efficacy of hydrocarbon gums, and indeed of incendiaries in general. It was noted in Part III, however, that the procedure as originally outlined is subject to sources of error and uncertainty which make it difficult to differentiate between materials showing only subtle differences in performance. The procedure has now been overhauled completely and refined in several details. The original specifications of the test structure have been retained, but the conditions of experimentation and the basis of comparison have been revised to such an extent that the data from this point on constitute a new series not comparable with the old series previously reported.

Gibbs Test Room. - Burning tests conducted in an outdoors screened court (Part I) were inevitably subject to variable drafts. An almost completely closed and covered outdoors shack was then constructed and provided with an adjustable air duct and a forced draft chimney, but this proved unsatisfactory because of its too limited air space and because of difficulties associated with variable weather.

The present test room on the top floor of the Gibbs laboratory is an interior, completely closed and shielded room, which formerly housed Professor [illegible]. T. Richard's precision analytical balances. The room is 12 1/2 ft. by 10 1/2 ft., and 14 ft. high, and has doors on two opposite sides. It is glass-windowed on two sides, and a third side has a glass-windowed door, and hence full observation can be made from the outside. A ventilator duct centered about 3 ft. below the ceiling makes connection to a ventilator provided with a powerful fan. Trial burnings with the ventilator in operation and with admission of air to the room proved unsatisfactory, for under these conditions variable drafts, accompanied by a certain fanning of the flame, are inevitable. Very satisfactory results were obtained, however, by conducting the burning in an entirely quiet atmosphere without the admission of air and with no ventilation. The flame invariably rises vertically without vortex, and a satisfactory reproducibility of results is secured. This method of conducting the test perhaps simulates the most significant set of conditions to be encountered in practice.

In conducting the burn, the operator lights the charge on the test structure, placed on the floor at the center of the room, withdraws, and closes the door. The room fills up with smoke but, at least with an 85-g. charge, the fire continues without supression from the smoke and is easily observed. The time of the aggressive burn is clocked as in Part I but, in the procedure now adopted as standard and employed in all of the present experiments except Tests 1-8, the afterburn is not allowed to continue until it dies out of itself but is soon extinguished. A 30-sec. period is allowed after the aggressive burn for observation of any noteworthy behavior, and for confirmation of judgment on the termination of the main burn, and the structure is then pulled over to one of the doors by means of a previously attached wire and extinguished. The fan is then turned ~~on~~ to clear the room of smoke, one door being left partly open. The structure is withdrawn through the other door to a scraping room equipped with motor driven wire and fibre brushes and with a powerful blower and hood assembly for taking off the char and dust through a conduit leading directly to the roof. The wood pieces are scraped down to a clean, sound surface, weighings are made on a direct-reading Toledo scale accurate to 0.5 g.

Standard Test Charge, and Technique of Measuring the Sample.

The earlier procedure specified the use of an 85-g. sample, and comparison of different incendiary gums on the more significant volume basis was made by calculation from assumed densities. A change has now been made to the use for the test of a standard volume of the gum. As a purely arbitrary standard, we have taken the volume at 25° of a sample of 85 g. of a fully homogeneous solution of 7% smoked sheet rubber in pure benzene. This sample (Test 8) has a viscosity of 4 min., 14 sec. (falling ball method) and the density of 0.871 g./cc. (both at 25°), and hence the standard volume is 97.7 cc.

The earlier procedure of dispensing the test sample from a tared can with a paddle was recognized as subject to error and uncertainty associated with variable evaporation and changing viscosity during the necessarily slow and awkward transfer (see Part III). A suitable method has now been found in dispensing the charge from a modified grease gun. An Alemite gun (model 1585, 9-oz. capacity) can be adapted to the purpose by removing the compression mechanism and the distributor plate inside the barrel; this leaves a nozzle 7/16 in. in diameter. For filling, the discharge tip is closed with a cork stopper, the plunger is withdrawn, and the gun is filled through the open end with somewhat more than enough gum to provide two test samples. Air bubbles are largely eliminated by holding the gun in a vertical position and working out some of the excess gum. By making notch marks on the plunger shaft, the gun can be calibrated to deliver two samples of approximately the required standard volume of 97.3 cc., but the actual amount delivered is determined accurately by weighing the gun before and after each ejection. Thus the gun is filled, the excess is pushed out until the proper notch is reached, the stopper is put in place, and the gun is weighed to 0.5 g. The charge is then distributed from the nozzle as evenly as possible over the area inclosed by a 14-cm. circle drawn at the center of the baseboard, the stopper is replaced, and the half-full gun is weighed when convenient. After the second sample has been dispersed, the gun is cleaned most conveniently by squeezing out excess gum, removing and wiping the plunger, and allowing the barrel to drain overnight; when the solvent has evaporated the rubber skin is easily torn away and leaves a clean metal surface. A supply of three guns proved adequate.

Since the volume is controlled only approximately and may deviate slightly from the standard volume, a correction factor is applied to the values found for the total "loss in weight" and the total "surfaces charred" (but not to the figures for the individual pieces or to the time of the burn). Thus:

$$\text{Factor} = 97.3 \text{ (Stand. Vol.)} \times \frac{\text{Density of sample}}{\text{Weight of sample}}$$

In the present series of tests the factors usually fell in the range 0.98-1.04.

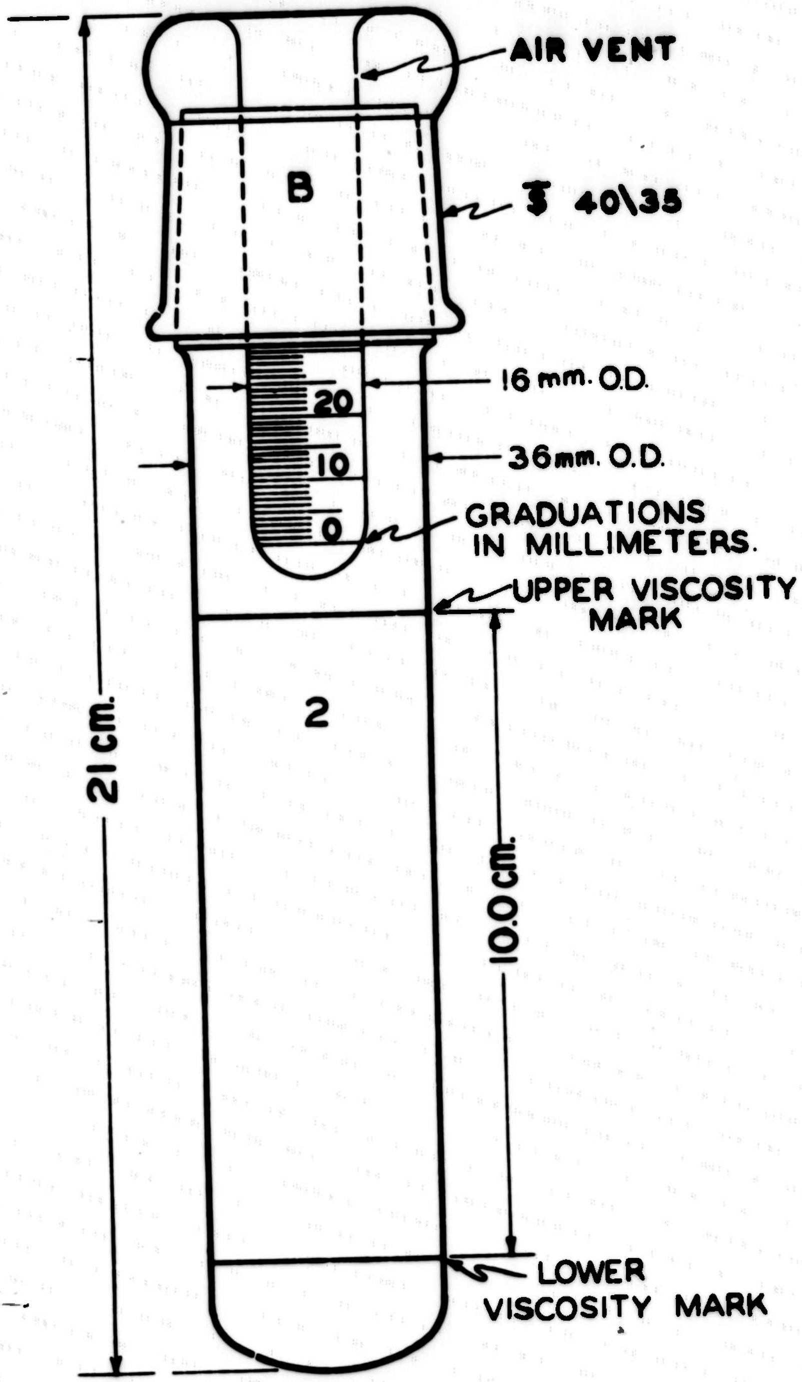
The copies of typical data sheets to be found at the end of this report illustrate the method of recording the results.

<u>Determination of Density and Viscosity.</u> - Both determinations were made with the use of the combined pycnometer-viscometer shown in

Fig. 1. A tube which is to contain the highly viscous gums must be of large diameter if the gum is to be poured in with reasonable ease and with sufficient rapidity to obviate extensive evaporation of the solvent. As a means of circumventing the large error attending a volume reading in a wide tube, the tube pictured is provided with a cap carrying a wide plunger having an etched scale on which the volume reading can be made.

The tube proper was constructed by sealing the inner member of a 45/35 standard taper joint to form a round-bottomed tube 20 cm. long and 35 mm. in outside diameter. Two lines were etched around the circumference of the tube, one about 1.5 cm. from the bottom and the other exactly 10 cm. above the lower line. The cap was made by drawing down the female member of the ground glass joint just above the ground section and joining this by means of a ring seal to a 17-mm. Pyrex tube which was then blown to a rounded end at a point 4 cm. below the seal, as shown in the Figure. A pinhole near the top of the inner tube allowed for equalization of pressure during centrifugation. Three pycnometer tubes (I, II, III) and two caps (A, B) were constructed and calibrated at 25° by filling them with water to various scale readings and taking the weights; calibration tables were prepared for the combinations: IA, IIB, IIIA.

The density and viscosity determinations were carried out in a room held at 25 ± 1° C. by the electrical thermostat system, and the gum samples usually were stored in this room for at least a day prior to use. In conducting a determination the gum is poured through a funnel of very wide stem into one of the calibrated tubes to a point which will give a reading on the scale when the cap is inserted. The



COMBINATION DENSITY-VISCOSITY APPARATUS

tube is stoppered with a cork and centrifuged for one hour at about 1000 r.p.m. to eliminate air bubbles and to remove traces of suspended material from the zone between the etched lines. The cap is then put in place and the tube is centrifuged for two minutes at 1000 r.p.m. in order to produce an even meniscus. The centrifugation results in a slight warming of the gum, and the tube must be allowed to stand in the constant-temperature room for at least two hours in order for it to come to the required temperature. The volume is then read, the tube is weighed and the weight of sample obtained by difference, and the density (g./cc. at 25° C.) is calculated. The temperature of the gum is checked after the final weighing. With transparent gums, the pycnometer scale can be read easily to 0.5 mm. (0.3% error) and, since the weighings on a triple beam balance are accurate to 0.2 g., the density determinations are subject to an error of no more than about 0.5%. With opaque solutions, the scale reading is more difficult but the error probably is not over 1%.

The viscosity of the gum is determined on the same centrifuged sample by timing the fall of a 5/32 in. steel ball between the two etched markings. Usually three determinations are made, the temperature is checked after the last one, and the values are averaged. The ball ordinarily is dropped at the center of the tube to avoid retardation due to a wall effect, but with very opaque solutions the determination is made with the ball close to the wall in order to provide an at least approximate indication of the viscosity. Successive balls should not be dropped in the same spot without first stirring the gum, for otherwise there is a grooving effect which accellerates the fall. This effect is particularly noticeable with solutions of crepe rubber.

With the precautions noted, duplicate viscosity determinations on a transparent gum invariably are in excellent agreement. Difficulty frequently has been experienced, however, in attempting to prepare duplicate samples of a given gum. Even though the quantities are carefully measured and the rubber taken from the same or similar stock, the resulting gums may vary rather widely in viscosity. In many instances this variability very probably is due in large part to incomplete solution of the rubber, and indeed some of the gums are seen on careful inspection to fall short of complete homogeneity. Since slight deviations in this respect do not seem to influence the burning characteristics, we frequently have proceeded with the burning tests after having obtained a reasonably uniform solution and have not delayed the work for the purpose of obtaining full homogeneity. In those instances where the gum is recognized as being imperfectly dissolved, the viscosity value is inclosed in parentheses. The gums used in the earlier experiments probably fell short of full homogeneity, for it was not recognized at the outset that, when the compounding is done without adequate mechanical disintegration, the final breakdown of the rubber-gel structure may require a period of several days. The viscosity values given in Part III are thus for the most part fallacious and much too high.

It is also entirely possible that a certain variability is associated with the rubber, and that this may vary in average molecular weight from lot to lot, or even from piece to piece. It will be noted below that the swelling characteristics of the rubber hydrocarbons vary enormously with the processing to which the material has been submitted.

It is further possible, but in our opinion rather unlikely, that the viscosity of the gum is dependent to some extent on the amount of oxygenation which occurs in the course of the compounding. Rubber technologists whom we have consulted differ in opinion on this point and also on the possible role of oxygen as an accellerator for the process of swelling. We carried out parallel experiments in which strips of smoked sheet rubber were rotated with benzene in a one-third filled glass bottle. In the first, the bottle was swept thoroughly with nitrogen before being sealed, and in the second the bottle was flushed with pure oxygen and this gas was admitted at a slight positive pressure throughout the experiment through a hole drilled in the base of the bottle and connected to an oxygen cylinder by way of a flexible tubing working through a mercury seal. No difference was noted in the rate of solution, and the resulting gums had essentially the same viscosity.

Preparation of Rubber Gums. - The gums from smoked sheet or crepe rubber were compounded by rotating the mixtures on a roller mill either in 2-quart Ball jars or in a 12-liter wide-mouthed glass bottle. The mouth of the large bottle was ground to a smooth surface and covered with a Neoprene gasket and an iron plate held in place with wing-nut clamps. It is well to rotate the mixture from the start, for troublesome lumps may form if it is allowed to stand for a preliminary period. When smoked sheet rubber is mixed with benzene and the rolling started at once, all discrete lumps usually disappear in about 3 days but it may take some 7-9 days before a fully even solution is attained and traces of gelatinous material have disappeared. Pale crepe rubber dissolves much more rapidly. The lumps disappear in about 1 day (with benzene as solvent) and after 2 days the solution appears perfectly clear when viewed in bulk; when, however, observation is made of a thin film flowing down a glass wall, a fine gel structure is still apparent.

It should be noted that this laboratory method of preparing gums is not one which would be considered for manufacturing purposes. Commercial rubber cements (e.g. Grippit) are manufactured in a disintegration of the dough-mixer type. The rubber, usually pale crepe, is first introduced along with a very small amount of the solvent; small additional amounts of solvent are added as the disintegration proceeds, but the bulk of the solvent is added only after the original gel structure has been fully broken down. A thoroughly uniform, true solution is obtained, and the whole process can be completed in a period of a very few hours. This process would appear to be well adapted to the rapid, large-scale production of incendiary gums. A disadvantage, although one of relatively minor significance, is that the process of mechanical disintegration is attended with a certain heat effect and results in some depolymerization of the rubber hydrocarbon; slightly more rubber would therefore be required to reach a given viscosity.

Gums were compounded from latex by a procedure communicated to us by the Standard Oil Development Co. group (1st procedure). In a typical case, 75.1 g. of concentrated latex (Esso R D No. 567-41) was added during 60 seconds to 918 g. of benzene, which was stirred mechanically in a tin can; this gave a fairly even dispersion which became so thick after continued stirring for 3 1/2 min. that the mixing then became inefficient. A 9-g. charge of oleic acid (12% of the latex) was then added and the mixture was worked with a paddle for 15 min., during which time the temperature was observed to drop from 20-22° C. to 15° C. The odor of ammonia disappeared at first but came back after 1 hour. The oleic acid noticeably improved the smoothness of the mix and increased the viscosity. The gum contained 7.5% of the concentrated latex; we initially assumed this to contain 65% of rubber and on our data sheets recorded the actual concentration of rubber as 7.5 x .65 = 4.9%. From further information it appears more likely that the latex contains 60% of total solids of which about 95% is rubber hydrocarbon, and we therefore consider the true rubber content to be 7.5 x .60 x .95 = 4.3%.

When latex was similarly stirred into naphtha it gave a suspension which settled at once on stopping the stirrer. On the addition of oleic acid the material soon began to disperse and dissolve, but the process was slower than with benzene and the thickening was complete only after about one hour. A temperature drop (e.g. to 9° C.) was again observed.

The latex gums which we have prepared have shown definite signs of syneresis on standing for no more than a week. Such alteration may constitute a serious disadvantage and will merit further investigation.

Rubber-Benzene Gums. - Beyond the typical data sheets appended to this report, the results of the present series of burning tests will not be reported in full detail but rather recorded in summary form, as in Table I. The pairs of figures in the last three columns refer to the first and second burn respectively. The figures for the loss in weight of the structure and the number of surfaces charred represent the corrected totals referred to a standard test charge occupying a volume of 97.3 cc.

The second burn, since it is made with the upper parts of the structure in a dried-out condition, normally should be more severe than the first burn, and in all but one of the 17 tests reported in Table I this is the case. On the average, the loss in weight in the second burn is 12.4% greater than in the first burn, but in extreme cases the ratio varies over the wide limits of from -17% to 36%. This variability demonstrates the difficulty in attaining accuracy in burning tests even under carefully controlled conditions and emphasizes

Table I

Rubber-Benzene Gums

A. Smoked Sheet Rubber

Rubber concn.	Viscosity, min.:sec.	Density	Test No.	Loss in wt., g.	Surfaces charred	Time of burn, min.:sec.
5%, plus 1% asbestos	1:00	0.877	14	387, 491	17.7, 17.6	2:50, 2:15
6%	1:25	.869	15	348, 348	15.0, 11.4	1:58, 2:00
6.5%	2:05	.869	16	352, 490	16.5, 18.	2:01, 2:18
7%	4:14	.874	1	515, 531	14.8, 17.	2:25, 2:35
			2	456, 473	14.5, 17.5	2:45, 2:28
			3	402	14.0	2:50
7.5%	5:15	.875	9	456, 473	14.2, 16.5	2:37, 2:17
8%	(10:11)	.876	10	570, 475	14.9, 16.5	2:47, 2:00
			11	434, 467	14.7, 18.9	2:50, 2:08

B. Pale Crepe Rubber

5%	1:21	.870	25	497, 517	14.7, 16.9	2:31, 2:30
5.5%	(7:21)	.873	26	446, 565	14.6, 17.4	2:26, 2:27
6%	8:57	.874	20	516, 565	15.6, 18.4	2:28, 2:27

C. Latex

4.3%	(55:00)	.869	20	522, 477	14.5, 16.	2:13, 2:10
5.1%	(80:00)	.876	22	498, 551	13.4, 16.9	4:05, 2:55

(cont'd on next page)

D. Heat-Treated Rubber ("Milled Smooth")

7%	0.15	.875	[illegible]	311, 41[illegible]	15.[illegible], 18.[illegible]	1:30, [illegible]:05

the importance of drawing conclusions only from a rather extensive body of data. The three independent tests of the 7% solution of smoked sheet rubber in benzene provide some further index of the degree of accuracy of the present method; the maximum deviation in the results for the first burn amounts to 1[illegible]%, and the average is 487 ± [illegible]1 (4%).

Inspection of the data for the surface coverage leads to the conclusion that with incendiary gums of the same general type the area attacked by the flame is so nearly the same that this factor does not provide a very significant index of performance. Differences in the time over which the aggressive burn continues appear to be significant, and in general the figures for the first and second burn are in good agreement.

Comparison of the various gums is best made by reference to the averages summarized in Table II. In the series of gums compounded

Table II

Comparison of Rubber-benzene Gums

(averages of results for all 1st and 2nd burns)

Concn.	Type of rubber	Viscosity	Wt. loss, g.	Time
6%	Smoked Sheet	1:[illegible]5	70[illegible]	1:59
6.5%	" "	[illegible]:05	[illegible]	[illegible]:07
7%	" "	[illegible]:11	[illegible]	[illegible]:[illegible]7
7.5%	" "	[illegible]:15	[illegible]	[illegible]:[illegible]7
8%	" "	(10:11)	[illegible]	

(cont'd on next page)

Concn.	Type of rubber	Viscosity	Wt. loss, g.	Time	
5%	Crepe	4:[illegible]1	507	2:30	← Max.
5.5%	"	(7:[illegible]4)	505	2:[illegible]6	
6%	"	9:57	541	2:32	
4.[illegible]%	Latex	(35:00)	405	[illegible]:1[illegible]	
5.1%	"	(30:00)	5[illegible]4	3:00	← Max.
7%	Heat-treated	0:15	361	1:16	

from smoked sheet rubber the thinner solutions are definitely inferior in incendiary effectiveness to the more viscous ones and a maximum performance is reached in the 7% solution showing a viscosity of 4 min., 14 sec. It is well known to rubber technologists that the viscosity of rubber solutions is a function of the molecular weight of the rubber and that even the mildest processing of the native material may result in a partial depolymerization and hence in a lower viscosity per given amount of total solids. The smoking process is known to be attended with some depolymerization, whereas the conversion of latex into crepe, being accomplished largely by washing and with a minimum of warming, leaves the rubber hydrocarbon more nearly in its native state. Thus a 5% solution of crepe rubber in benzene has about the same viscosity as a 7% solution of smoked sheet rubber in the same solvent. The burning tests (Table II) indicate that a maximum of effectiveness is reached in this 5% solution and that it is fully equal to the 7% smoked sheet solution. It is evident that for the production of incendiary gums crepe rubber is definitely more economical of resources than smoked sheet rubber; a further advantage is that it swells more rapidly in solvents than the smoked material.

A comparison of gums from crepe and from latex on the basis of viscosities is somewhat illusory, for the apparently very high viscosities of the latex gums at low rubber concentration is probably due in large part to the soap-effect of the ammonium oleate in combination with the small but significant amount of water derived from the latex. The latex gums appear, indeed, to be in the category of gels, and this is demonstrated by their tendency to undergo syneresis. The burning tests with the two latex gums demonstrate clearly that a high viscosity alone is not sufficient for satisfactory performance. The solution containing 1.7% of rubber as latex is very viscous but is inferior as an incendiary to the best of the gums produced from the coagulated rubbers. The 5.1% solution, however, reaches the maximum of performance noted in the other cases. It is of interest and of practical significance that the same degree of performance is attained with about 5% of rubber either as crepe or as latex.

The sample of heat-treated rubber (milled smooth) evidently consisted of rather extensively depolymerized material, for a 7% solution showed a very low viscosity and performed feebly in the burning test. A rubber sample which had been submitted to even more severe heat treatment (R. S. No. 2) gave an even thinner solution. Unmilled smoked sheet, [illegible] rubber and milled cryovac scrap were also examined and found equally unpromising. The samples of reclaimed rubber were examined and found unsatisfactory. One, an ordinary reclaimed type, swelled somewhat in benzene but did not dissolve. The other had been processed in such a way as to give material which could be dispersed to a "synthetic latex"; this largely dissolved in benzene but gave a solution no more viscous than the solvent alone. Since reclaimed rubber inevitably consists in highly depolymerized material, it is wholly inadequate for the preparation of incendiary gums.

Trials are in progress on the possible thickening of solutions of low rubber content by vulcanization. Thus a 2% benzene solution of rubber as latex was treated at room temperature with sulfur in carbon bisulfide and the low-temperature accelerator CPB (di-butylxanthyl disulfide), but as yet little thickening has been observed. Such experiments are not viewed with much enthusiasm in view of the opinion from a competent rubber technologist to the effect that any adequate thickening is likely to result in a merely transitory gel which will be subject to fairly rapid syneresis.

Rubber-Naphtha Gums. - The naphtha samples employed were supplied by the Process Division of the Esso Laboratories and are characterized by the following inspections:

Table III

Description of Naphtha Samples

PDL No.	18812	18820	[illegible]	18817
Gravity °API	66.5	58.6	[illegible]	44.5
I.B.P. °F.	113	198	313	308
10%	145	208	[illegible]	[illegible]
50%	178	221	340	343
90%	[illegible]	[illegible]	368	378
F.B.P. °F.	[illegible]	270	393	[illegible]
R.V.P. - lbs/sq. In.	6.8	[illegible]	0.0	[illegible]
Flash °F.	-Below 20° F.-		103	104
Fire °F.	-Below 20° F.-		115	120
Pour °F.	----------Below -76° F.----------			

Table IV

Rubber-Naphtha Gums.

Naphtha 18518, b.p. 113-285 °F

Rubber	Viscosity	Density	Test No.	Loss in wt., g.	Srfc. chrd.	Time
7% Smoked Sheet	0:50	0.710	31	387, 399	12.8, 15.7	2:34, 2:08
6% Crepe	(4:00)	.707	34	406, 398	14.0, 14.3	2:40, 2:35
5.7% Latex	1:42	.721	28	340, 360	11.8, 10.7	2:35, 2:06

Naphtha 18820, b.p. 198-270 °F

7% Smoked Sheet	1:45	.716	33	451, 470	14.0, 16.8	2:25, 2:35
6% Crepe	(6:07)	.717	39	438, 460	15.5, 16.0	3:05, 2:45
5.7% Latex	(27:40)	.750	27	403, 378	15.2, 16.7	2:30, 2:55

Naphtha 18530, b.p. 313-397 °F

5.9% Latex	(9:37)	.785	42	427, 471	12. , 11.8	3:30, 3:50*

90% Naphtha 18530 + 10% Naphtha 18518

4% Latex	0:09	.776	50	345, 410	17.4, 18.2	2:30, 2:05
4.5 %	0:33	.784	49	409, 458	11.1, 17.1	2:40, 2:35

Naphtha 18817, b.p. 303-407 °F

7% Smoked Sheet	4:15	.800	37	510, 520	17.8, 17.5	3:56, 3:10*
6% Crepe	(15:15)	.805	38	497, 570	11.6, 17.3	3:45, 3:55*
5.7% Latex	105:00	.806	29	477, 484	13.1, 16.5	3:40, 3:11*

90% Naphtha 18817, 10% Petroleum Ether (b.p. 20-40 °C)

6% Crepe	9:55	.786	15	505, 530	17.8, 17.5	3:24, 3:10
5.1% Latex	(7:07)	.797	17	517, 531	14.9, 16.8	3:00, 3:15+

* 30-40 sec. lag in ignition of whole charge

\+ Prompt ignition, but very low flame for 5-10 sec.

Gums were made with the use of smoked sheet, crepe, and latex rubber, for the most part merely at a concentration judged from the above results to be in the range of maximum effectiveness. The compositions are shown in the summary of results recorded in Table IV. The two high-boiling naphthas (15858 and 18517) have flash points too high for satisfactory ignition and hence in some of the tests they were blended with 10% of either petroleum ether or the roughly equivalent light naphtha 18813, which gave the desired ignition characteristics and did not materially alter the burning effectiveness. Similarly in field tests it was found that when a 5-lb charge of a gum from naphtha 18517 and latex was ejected from a test bomb with a charge of 50 g. of black powder the material was distributed satisfactorily but failed to ignite; when, however, the same naphtha was blended with 10% of benzene, the gum was ignited satisfactorily. In the latter test the the bomb was fired in a wooden structure built to simulate the corner of a room. A considerable amount of the burning gum was plastered over the ceiling area where it burned very vigorously and set a fire which continued until extinguished.

In the 14 tests recorded in Table IV the loss in weight in the second burn is higher than in the first burn in all but two instances. The average increase is 7.1%, and the extreme limits of deviation are from -8% to +20%.

Comparison of the results for the different gums is best made by reference to the overall averages summarized in Table V. The effect of varying the rubber concentration was investigated only for the case of solutions of latex rubber in one of the two high-boiling naphthas (No. 15858). The relationship is similar to that observed

Table V

Comparison of Rubber-Naphtha Gums

(Average values found for loss in weight in all burns with the naphthas as such or with 10% of petroleum ether)

	Naphtha No. and principal b.p. range, °F			
	18818	18850	[illegible]	18817
Rubber	112-2[illegible]	190-270	212-30[illegible]	300-[illegible]
7% Smoked Sheet	393	451		575
6% Crepe	[illegible]	440		[illegible]
4% Latex			[illegible]	
4.5% "			[illegible]	
5.2-5.4% Latex	[illegible]	390	[illegible]	[illegible]

with benzene solutions, namely that the incendiary effectiveness increases progressively on raising the concentration (calculated for the actual content of rubber hydrocarbon) from 4% to 4.5% and to 5.2%. From the results with the other naphthas it appears that gums containing 5.2-5.4% of rubber introduced in the form of latex are very nearly as effective as gums containing 7% of smoked sheet rubber or 6% of crepe rubber. From the observations made with benzene solutions it would seem likely that full effectiveness could be attained with 5% crepe rubber solutions, but this remains to be established.

The naphthas numbered 18818, 18850 and 18817 represent fractions of progressively increasing boiling range, and the results for the three sets of gums indicate very consistently that the burning efficiency increases with increasing boiling point. The naphtha of very low boiling point is at a distinct disadvantage as compared with other solvents because of the low density. Thus the 87.7-cc. test charge of 7% smoked sheet gum weighs only 69 g., or 81% of the weight of the standard 7% smoked sheet rubber-benzene, and the destructiveness in the burn amounts to 80% of that observed with the standard. The 7% solution of smoked sheet rubber in the high-boiling naphtha No. 18517 approaches the standard in density (92%) and is slightly superior to it in burning effectiveness on either a weight or volume basis. On the whole, the gums of adequate rubber content compounded from a high-boiling solvent naphtha base are very nearly equivalent in performance to the best benzene gums. Since there is no choice with respect to incendiary effectiveness on the volume basis, preference can be given to naphtha as the solvent because of its greater availability and because, when properly blended, it will ignite at sub-zero temperatures at which a benzene gum would be solid and show no flash point.

Other Hydrocarbon Gums. - Among other miscellaneous mixtures investigated (Table VI), trial was made of a turpentine solution containing enough rubber to give an adequate viscosity. Since the average amount of destruction (407 g.) is only about 79% of that obtainable with a good naphtha gum (517 g. av.), and since the turpentine supply is at present required for other purposes, there seems to be little reason for considering the material further.

Two naphtha solutions of synthetic polybutene (Stanco's Vistanex, high-molecular weight sample) were examined. The results recorded for the 5% solution are rather illusory, for, although a considerable amount of wood was eventually burned, the flame was very feeble and ineffective at the outset and became really significant only

Table VI

Miscellaneous Mixtures

Material	Density	Test No.	Loss in wt. g.	Srfc. chrd.	Time
7% Smoked sheet rub.-turpentine, viscosity (3:40)	0.862	16	367, 418	14.3, 18.1	3:55, 4:02
18% Polybutene (Vistanex) in VPM naphtha, b.p. 200-300°F, viscosity 1 min., 28 sec.	.760	4, 6 8	552, 477 590, 519	15.2, 17.1 14.2, 17.4	3:02 3:04
35% Polybutene in VPM naphtha, viscosity 41 min., 7 sec.	.788	7	[illegible]	15.0, 17.8	3:02 (Ineffective in first 2 min.)
7% Smoked sheet rub.-benzene + 1/5 part $NaNO_3$	.866	17 19	366, 430 396, 457	13.8, 14.2 9.7, 15.7	3:43, 3:18 3:55, 3:30
5% Crepe rub. + 1/5 part $NaNO_3$	.971	25	373, 379	11.3, 14.6	2:55, 3:40
5% Crepe rub.-benzene + 20% nitrocellulose	.977	46	427, 514	10.4, 16.7	3:55, 3:04
6% Crepe rub. in naphtha 13817 with 10% petroleum ether + 25% BDO-propellent (120 g. sum.)	[illegible]	48	530	19.1	2:55
Celluloid cylinder, 10 cm. x 2.3 cm. diam., 140-147 g.	1.37	47	718, 487	9.6, 11.1	3:55, 4:03

after a delay of at least [illegible] minutes; such material could easily be extinguished. The 18% solution had a viscosity not much below that of the standard rubber-benzene solution and in the burning tests gave a performance (518 g., av.) very close to that obtainable with the best naphtha gums. The material, however, has no advantage over a naphtha-rubber gum and, since some 18 parts of the expensive synthetic polymer are required where 5 parts of crepe rubber will serve equally well, there is no call to give further consideration to a substance which at present is produced to the extent of only about 50 tons per month and which is required for other, more specific purposes.

<u>Nitrate Mixtures</u>. - Results are recorded in Table VI for two mixtures of rubber-benzene gums containing 1/5 part of finely powdered sodium nitrate (ground with the solvent). The damage done, in terms of the loss in weight of the wood structure is not impressive, for in the two cases it is only 59% and 62%, respectively, of that observed with an equal volume of the gum alone. The attack, to be sure, is more confined and is concentrated on the lower parts of the structure, and the time of burn is distinctly longer. Although nitrate mixtures do not at present seem particularly promising, the rather unique burning characteristics justify the investigation of mixtures of other types and proportions. A trial with nitrocellulose fibres in crepe rubber-benzene was disappointing, for the average burn amounted to only 93% of that obtained with the gum alone and the rubber seemed to suppress the burning of the nitrocellulose. An even poorer performance was observed with a mixture of a very vigorously burning SPC-nitrate compound with a rubber-naphtha gum.

The last entry in the list of miscellaneous results (Table VI) refers to a test made on solid cylinders of celluloid corresponding to the standard volume (37.5 cc.). The average loss in weight of the structure was 401 g., or 73% of that observed with the naphtha gums. This relationship suggests the possibility of enhancing the effectiveness of the present celluloid incendiary leaves by preparing the leaves in the form of hollow shells and filling them with a gum incendiary.

Test Structures of Hemlock, Spruce and Oak. – The test method as applied in the above experiments included comparisons based on the performance of incendiaries against a fresh structure of a whitewood base and hemlock uprights and against a structure with a fresh base but with dried-out uprights. It seemed desirable to conduct at least a few exploratory comparative tests with still different woods. Structures of the standard type were therefore constructed of unplaned spruce and of planed oak, and burning tests were made with three of the same [illegible] employed in the above series of experiments. The results are recorded in Table VII.

In representative tests, determinations were made of the total amount of char scraped off of the burned surfaces. With the hemlock structure (Part I), the char in the first burn amounted to 16.1% of the total loss in weight of the wood, and in the second burn the char was 1[illegible].5% of the total. With the oak structure the percentages were very nearly the same, namely 14.3% and 20.1% in the first and second burn, respectively (see appended data sheets). With spruce, the values found in the trial experiment were somewhat higher: 18% for the first burn and 25.[illegible]% for the second. The differences observed in these isolated experiments are hardly great enough to be of much moment. Perhaps the most noteworthy variation from the performance of hemlock is that with the oak structures the amount of wood destroyed was very much greater in the second burn than in the first ([illegible]3%, [illegible]7%, and 48% greater).

Table VII

Tests with Different Woods

Material	Test No.	Loss in wt., g.	Mois. cont.	Time
Spruce Test Structures				
7% Smoked sheet rub.-benzene	[illegible]	460, [illegible]	14.[illegible], 15.1	[illegible]:01, [illegible]:10
6% Crepe rub.-naphtha 18817	[illegible]	517, [illegible]	15.3, 17.[illegible]	[illegible]
7% Sm. Sh. rub.-benz. + 1/5 part $NaNO_3$	[illegible]	567, [illegible]	17.6, 16.[illegible]	[illegible]:56, [illegible]:13
Oak Test Structures				
7% Smoked sheet rub.-benzene	[illegible]	[illegible]	17.[illegible], 11.4,	[illegible]:06, [illegible]
6% Crepe rub.-naphtha 18817	[illegible]	[illegible], [illegible]	17.8, 12.[illegible]	3:30, [illegible]
7% Sm. sh. rub.-benz.+ 1/5 part $NaNO_3$	21	[illegible], [illegible]	[illegible].6, 15.8	3:16, 3:07

Table VIII

Summary of Tests with Different Woods

(Average values for loss in weight)

Material	Hemlock	Spruce	Oak
7% Smoked sheet rubber-benzene	[illegible]	451	[illegible]
6% Crepe rubber-naphtha 18817	[illegible]	510	[illegible]
7% Smoked sheet rubber-benzene + 1/5 part $NaNO_3$	[illegible]	[illegible]	[illegible]

Averages of the three sets of results obtained with hemlock, spruce, and oak structures are summarized in Table VIII. The performance of the first two incendiary mixtures does not vary much from one of the three woods to another (maximum deviation, 16%). The third mixture gives very nearly the same results on hemlock and on oak but there is an apparent fluctuation with the spruce structure. No very far-reaching conclusions could be drawn without a much more extensive body of data, but it appears from this exploratory series of tests that no pronounced variation is likely to be encountered in the relative efficacy of gum incendiaries as estimated by tests on different types of woods. An expansion of the present brief series of comparisons therefore does not seem justified.

Test No.: 12

Operators: FCH, MM, GCH

Date: 11/6/41

Observers: LFF

Material: 7% Rubber-Benzene (Spruce Structure)

Viscosity test: 4 min. 14 sec.

Gun, full 959 g.

" half 869 g.

1st sample 90 g. Factor 0.945

Gun, empty 790 g.

2nd sample 79 g. Factor 1.08

1st Burn

	A	B	B′	C	C′	D	E	Total	Corr. Tot.
Initial wt., g.	1290.5	1128.5	1132.5	1147	1194	524.5	316		
After 1st burn, g.	1218.5	1051	1029	1101	1126	441.5	279		
Loss in wt., g.	72	77.5	103.5	46	68	83	37	487	460
Surfaces charred	0.8	2.0	2.6	1.0	1.5	4.0	3.8	15.7	14.8

Burn: 2 min. 1 sec.

Afterburn: min. 30 sec.

Wt. of char = 90.0 g. (18.5% of total loss in wt.)

2nd Burn

After 2nd burn, g.	1156	975	953	1062	1068	377	248		
Loss in wt., g.	62.5	78	76	39	58	64.5	31	409	442
Surfaces charred	0.8	2.5	2.5	1.1	1.8	4.0	4.0	16.7	18.1

Burn: 2 min. 10 sec.

Afterburn: min. 30 sec. xt.

Wt. of char = 105 g. (25.7% of total loss in wt.)

Test No.: 25 **Operators:** FCN, GCH, [illegible] **Gun, full** 973.5 g.

Date: 11/6/41 **Observers:** LFF **" half** 886 g.

Material: 7% Rubber-Benzene (Oak Structure)

1st sample 87.5 g. **Factor** 0.971

Gun, empty 809 g.

Viscosity test: 4 min. 14 sec.

2nd sample 79 g. **Factor** 1.08

1st Burn

	A	B	B'	C	C'	D	E	Total	Cor. Tot.
Initial wt., g.	1579	1136	1153	1063.5	1127.5	537	304.5		
After 1st burn, g.	1528	1058	1073	1015.5	1101	460	275		
Loss in wt., g.	51	78	80	48	26.5	77	29.5	390	378
Surfaces charred	0.6	1.7	1.6	1.0	0.7	3.9	3.1	12.6	12.2

Burn: 2 min. 6 sec. **Afterburn:** min. 30 sec. Ext.

Wt. of char = 65.5 g. (16.8% of total loss in wt.)

2nd Burn

After 2nd burn, g.	1468	980	990	950	1065	379	244		
Loss in wt., g.	60	78	83	65.5	36	81	31	451.5	464
Surfaces charred	0.6	1.5	2.1	1.8	1.2	4.0	3.2	13.4	14.4

Burn: 2 min. 30 sec. **Afterburn:** min. 30 sec. Ext.

Wt. of char = 92.0 g. (20.7% of total loss in wt.)

Test No.: 22 **Operators:** [illegible]CH, JCH

Date: 11/13/41 **Observers:** [illegible]

Material: 5.8% Rubber (as Latex)-Benzene

Viscosity test: 20 **min.** **sec.** (approx.)
Density: 0.87[illegible] g./cc.

Gun, full 969 **g.**
" half 887.5 **g.**
1st sample 81.5 **g.** **Factor** 1.05
Gun, empty 806.5 **g.**
2nd sample 81.0 **g.** **Factor** 1.05

1st Burn

	A	B	B′	C	C′	D	E	Total	Corr. Tot.
Initial wt., g.	1211	685	671.5	789	806.5	377	233.5		
After 1st burn, g.	1106	610	587.5	751.5	751.5	294	199		
Loss in wt., g.	105	75	84	37.5	55	83	34.5	474	498
Surfaces charred	0.8	1.4	2.3	1.1	1.1	4.0	3.1	13.8	14.5

Burn: 3 **min.** 05 **sec.** **Afterburn:** …… **min.** 30 **sec.** Ext.

2nd Burn

	A	B	B′	C	C′	D	E	Total	Corr. Tot.
After 2nd burn, g.	995	531	493	692.5	693.5	210	159.5		
Loss in wt., g.	111	79	94.5	59	58	84	39.5	525	551
Surfaces charred	0.8	2.1	2.9	1.3	1.2	4.0	3.8	16.1	16.9

Burn: 2 **min.** 55 **sec.** **Afterburn:** …… **min.** 30 **sec.** Ext.

Test No.: 25 **Operators:** FCN, MM, GCH

Date: 11/12/41 **Observers:**

Material: 5% Crepe Rubber in Benzene

Viscosity test: 4 **min.** 21 **sec.**
Density: 0.870 g./cc.

Gun, full 972.5 **g.**
" half 890 **g.**

		Factor
1st sample	82.5 **g.**	1.03
Gun, empty	**g.**	**Factor**
2nd sample	83.5 **g.**	1.01

1st Burn

	A	B	B′	C	C′	D	E	Total	Corr. Tot.
Initial wt., g.	1346.5	888	913	729	811.5	461.5	201.5		
After 1st burn, g.	1247	802	831	677	762.5	381	167.5		
Loss in wt., g.	99.5	86	82	52	49.0	80.5	34	483	497
Surfaces charred	0.7	1.8	1.8	1.4	1.3	3.9	3.4	14.3	14.7

Burn: 2 **min.** 31 **sec.** **Afterburn:** **min.** 30 **sec.** Ext.

2nd Burn

After 2nd burn, g.	1134	709	749	613	710	304	137		
Loss in wt., g.	113	93	82	64	52.5	77	30.5	512	517
Surfaces charred	0.8	2.9	2.8	2.1	1.5	4.0	3.9	18.0	18.2

Burn: 2 **min.** 30 **sec.** **Afterburn:** **min.** 30 **sec.** Ext.

Test No.: 35 **Operators:** FCN, GCH, MM **Gun, full** 1006 g.

Date: 11/17/41 **Observers:** Gibbs conference group **" half** 911.5 g.

Material: 5 Parts 5% Crepe Rubber-Benzene
1 Part $NaNO_3$

1st sample 94.5 g. **Factor** 1.00

Gun, empty 820 g.

2nd sample 91.5 g. **Factor** 1.04

Viscosity test: min. sec.

Density 0.974 g./cc.

1st Burn

	A	B	B′	C	C′	D	E	Total	Corr. Tot.
Initial wt., g.	1044	927	933	869.5	815	466	199		
After 1st burn, g.	944	862	861.5	831	777.5	411.5	186.5		
Loss in wt., g.	100	65	71.5	38.5	37.5	54.5	12.5	379.5	379.5
Surfaces charred	0.8	1.0	2.0	0.9	0.9	3.1	2.0	11.3	11.3

Burn: 2 **min.** 55 **sec.** **Afterburn:** **min.** 30 **sec.** Ext.

2nd Burn

	A	B	B′	C	C′	D	E	Total	Corr. Tot.
After 2nd burn, g.	836	795	782	795	728	332	155		
Loss in wt., g.	108	67	79.5	36	49.5	79.5	31.5	461	479
Surfaces charred	0.7	1.4	2.2	1.2	1.3	4.0	3.2	14.0	14.6

Burn: 3 **min.** 40 **sec.** **Afterburn:** **min.** 30 **sec.** Ext.

Test No.: 42 **Operators:** GCH, [illegible], FCN **Gun, full** 947.5 **g.**

Date: 11/21/41 **Observers:** **" half** 872.0 **g.**

Material: 5.7% Rubber as Latex in 9 Parts Naphtha (PDL-18817), b.p. 309-402° F and 1 Part Petroleum Ether, b.p. 20-40° C.

1st sample 75.5 **g.** **Factor** 1.02

Gun, empty 798 **g.**

2nd sample 74 **g.** **Factor** 1.04

Viscosity test: 3 **min.** 7 **sec.** (approx.)

Density: 0.793 g./cc.

1st Burn

	A	B	B′	C	C′	D	E	Total	Corr. Tot.
Initial wt., g.	1162	959	791	820	845	477	191		
After 1st burn, g.	1051	877	711	770	795	385.5	154		
Loss in wt., g.	111	82	80	50	50	91.5	37	501.5	512
Surfaces charred	0.8	1.5	1.8	1.2	1.1	4.0	3.2	14.6	14.9

Burn: 4 **min.** 00 **sec.** **Afterburn:** **min.** 30 **sec.** Ext.

Prompt ignition, but low flame for 5-10 sec.

2nd Burn

After 2nd burn, g.	944	782	626	704	745	289	112		
Loss in wt., g.	107	95	85	66	50	96.5	42	541.5	564
Surfaces charred	0.8	2.2	2.7	1.4	1.4	4.0	3.7	162	16.8

Burn: 3 **min.** 45 **sec.** **Afterburn:** **min.** 30 **sec.** Ext.

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ABSTRACT:

Studies were made on improvements in wood burning test procedure, evaluation of different types of rubber, and comparison of the incendiary effect on three different kinds of wood. The standard for comparison was changed from a weight to a volume basis, and the results are now referred directly to a standard volume (97.3 cc) of the test sample equivalent to that of 85 g of a 7% solution of smoked sheet rubber in benzene. The investigation of gums of varying concentration of rubber of three types, with both benzene and naphtha as the solvent, has indicated that solutions of very low rubber content and low viscosity have inferior incendiary characteristics. Miscellaneous mixtures studied include solutions of polybutene in naptha, mixtures of rubber-benzene with sodium nitrate and with nitrocellulose, and celluloid.

(23) Incendiary mixtures, Rubber, Incendiary effects

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