The Scientific Method

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THE SCIENTIFIC METHOD

A Personal Account of Unusual Projects in War and in Peace

By Louis F. Fieser

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Preface

IN ATTACKING a problem of the usual type a scientist studiously looks for a clue or for a working hypothesis, carries out experiments planned to exploit the clue or to test the hypothesis, carefully observes and analyzes the results, and plans further experiments for advancement of the problem. Experiences cited in this book demonstrate that the scientific method is effective also in the solution of problems outside the domain of ordinary scientific research. Several of the chapters record episodes during World War II when I became involved in some unusual projects requiring construction of incendiary devices of assorted and unconventional types. Postwar projects include development of devices for improvement of student experimentation or teaching; one involved Sunday research at home. Whether the project was to design a package device for the ignition of oil slicks on water, an efficient fractionating column for students, or a squirrel-proof bird feeder, the scientific method of approach proved to be invaluable. Some of the problems were important, others trivial, but each had points of special interest. Perhaps this account of the steps by which each problem was solved will provide an entertaining and unorthodox demonstration of the scientific method.

The wartime work on incendiaries and on medicinal chemistry was conducted with the able cooperation of three research groups, and I heartily thank these co-workers for contributions spelled out or implied in the text. I also extend cordial regards to other wartime friends and acquaintances, as well as to former students, and I hope that they will take pleasure, as I have, in recalling stimulating experiences out of the past.

Cambridge, Massachusetts Louis F. Fieser

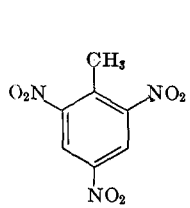
Contents

1. Before Pearl Harbor 9
2. Napalm 25
3. White Phosphorus Burster 34
4. A Fight to the Finish with Industrialists 45
5. The Harvard Candle 54
6. Pocket Incendiary 59
7. The 500-Pound Bomb 76
8. The City Slicker and the Paul Revere 81
9. Ignition of a 1,000-Gallon Oil Slick 96
10. Production of the First Thousand Paul Reveres . . . 100
11. Sabotage of Motor Vehicles 113
12. Antitank Grenade 114
13. Bat Bombs 121
14. The Book 135
15. Destruction of Documents in Danger of Confiscation . 138
16. The E-19 Bomb 144
17. Cortisone 159
18. Antimalarials 163
19. Training Manual for OSS Operators 192
20. Alsos Mission 202
21. Equipment for Students 205
22. The Movie 223
23. Of Cats and Birds 228

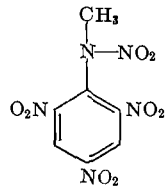
1. *Before Pearl Harbor*

AT A TIME in 1940 when German armies were overrunning France at such a pace that in the German view the war against England and France seemed nearly over, a few key leaders of science in the United States foresaw that our country was seriously threatened with involvement in what might well become a protracted war on a greatly expanded scale. Foreseeing also the possibility that science might play a decisive role in determining the eventual outcome of the conflict, they drew up plans for immediate indoctrination of civilian scientists in an extensive program of research on war projects designed to supplement the all-too-limited program in operation on a peacetime scale by the armed forces. And so, with the active cooperation of President Roosevelt, the National Defense Research Committee came into official existence on July 2, 1940, a year and a half before the Japanese attack on Pearl Harbor. Vannevar Bush, President of the Carnegie Institute, was chairman. Original committee members, in addition to representatives of the armed forces and the government, included Roger Adams, head of the department of chemistry of the University of Illinois, Karl T. Compton, President of Massachusetts Institute of Technology, James B. Conant, President of Harvard University, F. B. Jewett, President of the National Academy of Sciences, and R. C. Tolman, Dean of the Graduate School, California Institute of Technology.

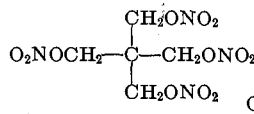
One division set up under the National Defense Research Committee, or NDRC, was for work on bombs, fuels, poison gases, and chemical problems, and this division, under the chairmanship of Dr. Conant, was the first to get under way. A first meeting of potential investigators was set for October 23, 1940 at the home of Roger Adams, and I was one of some 20 university professors invited. Dr. Conant outlined the organization and aims of the NDRC, obtained immediate assurance of cooperation from all those in attendance, and outlined problems on which each of us was to work. My problem was the synthesis of new nitro compounds for evaluation as possible explosives. The chief explosives then available were the polynitro compounds shown in the formulas. TNT had been known since



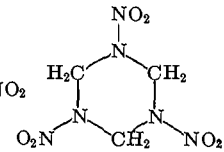
TNT
100



Tetryl
125.5



PETN
145.8

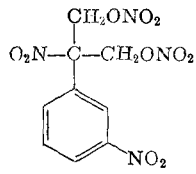


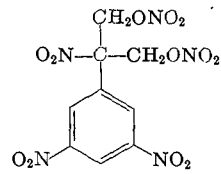
Cyclonite
150.2

Relative ballistic strength

1863; TNT, tetryl, and PETN were standard military explosives. Cyclonite had found limited use in World War I (British RDX) and was recognized as an explosive of superior quality, but improved methods were needed for the production of the chemical and for techniques for compounding it into shells; several NDRC groups were set up to investigate these problems. Other groups established at the Explosives Research Laboratory at the Bureau of Mines Experiment Station at Bruceton, Pennsylvania, facilities for evaluation of explosives, and the figures for relative ballistic strength given under the formulas were determined at Bruceton.

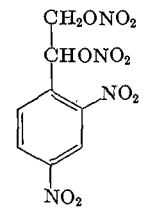
For my program, which was one of several aimed at the discovery of new explosives, I enlisted the following young chemists (I was 41): R. C. Clapp,[1] W. H. Daudt,[2] W. von E. Doering,[3] and M. Gates.[4] These men worked in secrecy in two basement laboratories. The program proceeded satisfactorily and without incident, and two of the new polynitro compounds





114 126

Ballistic strength relative to TNT



Too labile to test

[1] Richard C. Clapp, b. 1915 Boston; A.B. Bowdoin College, 37; Ph.D. Harvard (Fieser), 41; Quartermaster Research and Development Center, Natick, Mass.

[2] William H. Daudt, b. 1916 Washington, D.C.; B.S. Haverford College, 37; Ph.D. Harvard (Fieser), 41; Dow Corning Corp., Midland, Mich.

[3] William von E. Doering, b. 1917 F. Worth, Texas; B.S. Harvard, 38; Ph.D. Harvard (Linstead), 43; Columbia Univ., Yale Univ.

[4] Marshall Gates, b. 1915 Boyne City, Mich.; B.S. Rice Institute, 36; Ph.D. Harvard (Fieser), 41; Bryn Mawr College; Univ. Rochester; Editor, *Journal of the American Chemical Society*.

formulated proved superior in ballistic strength to TNT. The results were published in 1946.

At an NDRC conference on explosives at Chicago on May 28, 1941, each principal investigator reported on the results obtained by his group. Dr. Conant then gave an account of a series of explosions which had occurred in a du Pont plant for the manufacture of divinylacetylene, a hydrocarbon formed by combination of three molecules of acetylene and marketed as a

$$\underset{\text{Acetylene}}{HC{\equiv}CH} + HC{\equiv}CH + HC{\equiv}CH \longrightarrow \underset{\text{Divinylacetylene}}{H_2C{=}CH{-}C{\equiv}C{-}CH{=}CH_2}$$

synthetic drying oil for paints. The substance is extremely sensitive to atmospheric oxygen, which converts it in part into reactive peroxides. The peroxides initiate the joining together of many molecules of hydrocarbon and of peroxidized hydrocarbon to form an inert, stable, polymeric substance. Thus, when a surface is painted with a layer of pigment suspended in liquid divinylacetylene and exposed to the air for a suitable period, the liquid sets to a tough, adhesive, protective film. In the du Pont plant for the manufacture of divinylacetylene all known precautions had been taken for exclusion of oxygen. However, the devastating explosions that had occurred indicated that something had gone wrong and suggested that divinylacetylene is either an explosive or a spontaneously flammable substance. If so, it might have some military application. We conference members were asked if one of us would undertake to investigate this possibility. I volunteered.

I volunteered chiefly because I had available in my peacetime research group a man ideally qualified to experiment with and evaluate a hazardous chemical. Dr. E. B. Hershberg,[1] known as "E. B.," had had one year of postdoctoral experience in Switzerland and 5½ years with me at Harvard. A masterful experimentalist in organic chemistry, he was also versed in engineering, in mechanical drawing, in carpentry, in machining, in glass blowing, and in photography, and he had invented and constructed a number of laboratory devices which have found wide use, for example, the Hershberg stirrer, the Hershberg stirring motor, the Hershberg melting-point apparatus. Furthermore Hershberg, as a reserve officer of the Chemical Warfare Service, was experienced in the handling of military explosives, fuzes, poison gases, smoke pots, and grenades. The new and

[1] Emanuel Benjamin Hershberg, b. 1908 Lynn, Mass.; B.S. Massachusetts Institute of Technology, 29; Ph.D. M.I.T. (Huntress), 33; Director, Chemical Research and Development Division, Schering Corp.

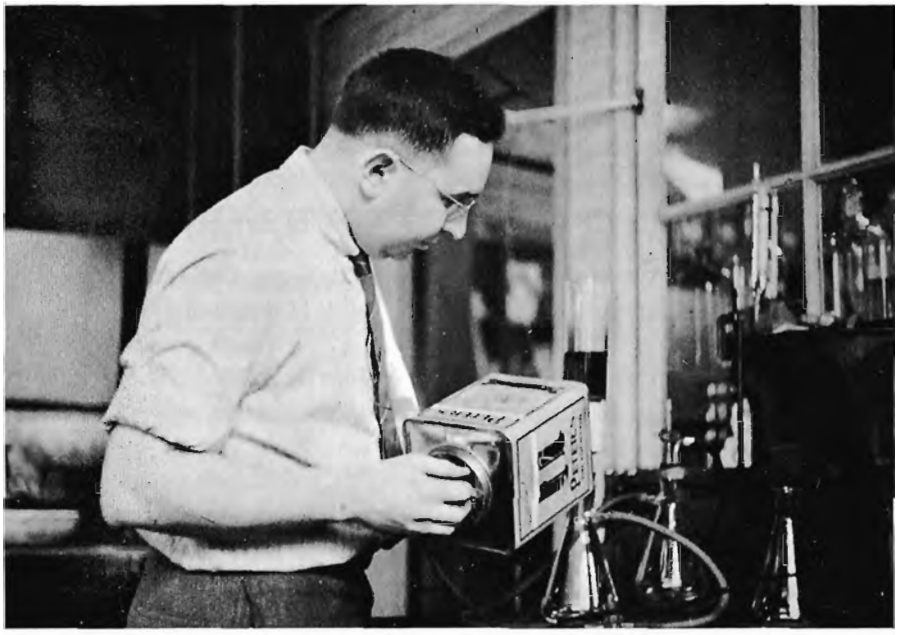


Fig. 1.1. — E. B. Hershberg Inspecting a
Chromatography Column under Ultraviolet Light (1937)

challenging problem appealed to him and work started promptly under a new 4-month contract starting June 15, 1941.

In a visit to Wilmington I had obtained all information available from du Pont chemists on the manufacturing process and concerning the explosions. In his corner laboratory in the basement of Converse, Hershberg prepared successive batches of divinylacetylene, and he and I exposed samples to air for varying periods of time in pans placed in the outside window well and noted the gradual change from a mobile liquid to a gel of viscosity which increased with time. We poked at the gels with sticks and dropped stones onto them in an effort to produce an explosion or a fire, but without encouraging results. At day's end we usually destroyed the gels of peroxidized hydrocarbon by setting fire to them with a match. Because of the presence of considerable peroxide, they burned with an impressive sputter and sparkle. We noticed also that when a viscous gel burns it does not become fluid but retains its viscous, sticky consistency. The experience suggested the idea of a bomb that would scatter large burning globs of sticky gel. Hershberg made up improvised bombs of peroxidized divinylacetylene in tin cans fired by a charge of black powder,

and when we tried them out in a remote section of Everett they seemed promising.

A report to NDRC of our interest in incendiaries resulted in a visit in August from Major Gerrard M. Rambaut of the British Air Ministry. Major Rambaut, who had played a part in the development of the British magnesium bomb, viewed our experiments with interest and advised us to set up a test for the quantitative evaluation of the incendiary effectiveness of our gels which would permit comparison of different gels and comparison of gels with magnesium. In acting on this obviously sound suggestion, we designed the standard test structure shown in Fig. 1.2. A standard amount of incendiary material is placed on the baseboard, ignited, and let burn

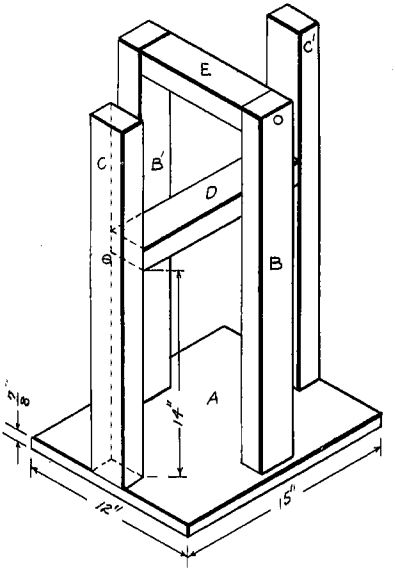


Fig. 1.2. — Burning-Test Structure
(The baseboard of whitewood and uprights and cross bars of hemlock are brought, in storage, to a standard moisture content.)

until the fire dies out, which happens with the best of incendiaries. The first burns were conducted in Hershberg's outside window well, since this was shielded from observation from the street. After a burn, we scraped the charcoal off of each piece of wood with a wire brush and weighed the total charcoal produced. My appearance at the end of a day presented a puzzle to my wife Mary, to whom I had not disclosed the nature of my war work, but she was let into the secret a few weeks later when she met

Major Rambaut at the airport and he asked: "How is your husband's incendiary project progressing?"

During the summer of 1941 the NDRC program had been reorganized and I had been instructed to terminate work on explosives and to work instead on poison gases, vesicants. This reallocation did not please me. Use of poison gas seemed to me inhumane. Furthermore, I doubted very much that vesicants would be used in the war that seemed increasingly imminent, and I would much prefer to work on something of practical value to the war effort. But I swallowed my personal feelings and engaged a new group of men to start research on vesicants in the fall. However, when these men reported the program was delayed for a time to permit installation of special ventilator hoods that were required for experimentation with toxic chemicals in comparative safety.

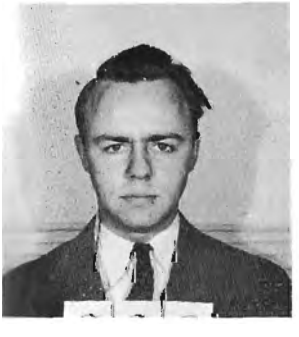
By this time the idea of a gelled-fuel bomb had become increasingly attractive and so, in the interim period of waiting, I made a canvass of activities in progress on incendiaries. The NDRC had not as yet initiated any work in this area. In a visit to the Chemical Warfare Service at Edgewood Arsenal, Maryland, I found that the only military men assigned to work on incendiaries were Capt. Julius Rex Adams, a former reservist, and Capt. R. Bruce Epler, an Air Force pilot; about six civilian members of the CWS staff gave part-time attention to incendiaries. The main concern was over the status, improvement, and production of two small bombs. One, a counterpart of the British magnesium bomb, was rated as probably unsatisfactory and in any case incapable of large-scale production because of limited supplies of magnesium. The bomb favored was a 4-lb. thermite bomb in which ignition of a flare mix initiates a reaction between aluminum and iron oxide which produces a puddle of molten iron. I suspected from the start that the molten iron would have little power to start a fire and hence that the bomb was a flop. No burning tests had been applied and the only basis for evaluation was qualitative observation of the firing of bombs in the absence of combustible material. Captains Adams and Epler expressed interest in our burning test and our ideas about incendiary gels, but deplored the fact that shortage of manpower ruled out work along these lines at the Arsenal.

On my return from this visit I appealed by telephone to my NDRC chiefs Roger Adams and Walter R. Kirner for permission to use the new manpower and funds for work on incendiaries rather than on vesicants. The proposal was approved on the spot. I also arranged to extend Dr. Hershberg's participation in our program on a flexible basis which would permit him to lend his talents to the incendiary project and also to perfect

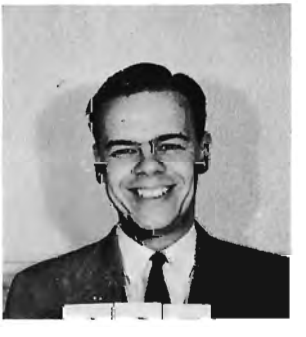
E. B. Hershberg

George C. Harris

Frederick C. Novello



Morley Morgana



Stearns T. Putnam

Fig. 1.3. — War Boys

a new type of rocket propellant which he had compounded from a mixture of sodium nitrate and ammonium picrate with peroxidized divinylacetylene as binder. A succession of formulations were evaluated at the Explosives Research Laboratory under a group headed by Professor Louis P. Hammett of Columbia. For a time Hershberg's entry made a good bid for

acceptance, but I believe that a propellant developed by a Monsanto group eventually took the lead of propellants of the type which at the time were under consideration.

By early October the new work on incendiaries was in full progress. Besides the two senior members, the group included George C. Harris,[1] Morley Morgana,[2] Frederick C. Novello,[3] and Stearns T. Putnam[4] (Fig. 1.3). Our six-man group remained intact for four years. Coded at Harvard as "Anonymous Research No. 4, OEMsr-179," the NDRC contract on the incendiary project alone totalled $359,125. I was obliged to give up both undergraduate and graduate teaching, as well as ordinary research, early in the school year 1941–42, and my job was taken over by Hans Heymann,[1] informally at first and then by appointment. Douglas M. Bowen [2] took over the lectures in the last war year. I do not recall what arrangements were made, but the University records indicate that I was not granted a leave of absence and that I continued to receive my regular salary. Apparently, for nearly four years, my services were contributed by Harvard, off the record, to the war effort.

Our first concern was to improve the burning test for evaluation of incendiaries, for the early outdoors tests had been disturbed by variable drafts. We moved into the third floor of the Gibbs Laboratory and installed the test in a glass-inclosed room-within-a-room which had been built to accommodate the precision balances for Theodore W. Richards' Nobel Prize-winning atomic weight determinations. A large hole was cut in the roof to house a powerful fan with which smoke from one burn could be sucked out of the room to ready it for the next burn. Thus incendiary materials and bombs could be allowed to burn in the complete absence of drafts and the experiments could be viewed from all sides through the glass windows. Since a gelled fuel would be carried in a bomb of capacity limited by volume, rather than by weight, we set as the standard for com-

[1] George C. Harris, b. 1916 Thessaly, Greece; A.B. Harvard, 38; Ph.D. Harvard (W. P. Campbell), 42; Hercules Powder Co., Wilmington, Del.

[2] Morley Morgana, b. 1918 Detroit, Mich.; A.B. Harvard, 39; Ph.D. Harvard (W. P. Campbell), 42; Ethyl Corporation; pres. South. Land Prods. Co.; v. pres. Morley Develop. Co., Baton Rouge, La.

[3] Frederick C. Novello, b. 1916 Somerville, Mass.; S.B. Harvard, 38; Ph.D. Harvard (Fieser), 41; Sharp and Dohme Div., Merck and Co., West Point, Pa.

[4] Stearns T. Putnam, b. 1917 Springfield, Vt.; Sc.B. Brown, 38; Ph.D. Harvard (Fieser), 42; Hercules Powder Co., Wilmington, Del.

[1] Hans Heymann, b. 1915 Cologne, Germany; A.B. equiv. Munich, 38; Ph.D. Harvard (Fieser), 41; Instructor and Lecturer, Harvard, 42–46; Univ. of Washington; Ciba Pharmaceutical Co., Summit, N.J.

[2] Douglas M. Bowen, b. 1917 Wellesley, Mass.; A.B. Harvard, 37; Ph.D. Harvard (Fieser), 40; Instructor, Harvard, 41–45; Dartmouth College, 45–.

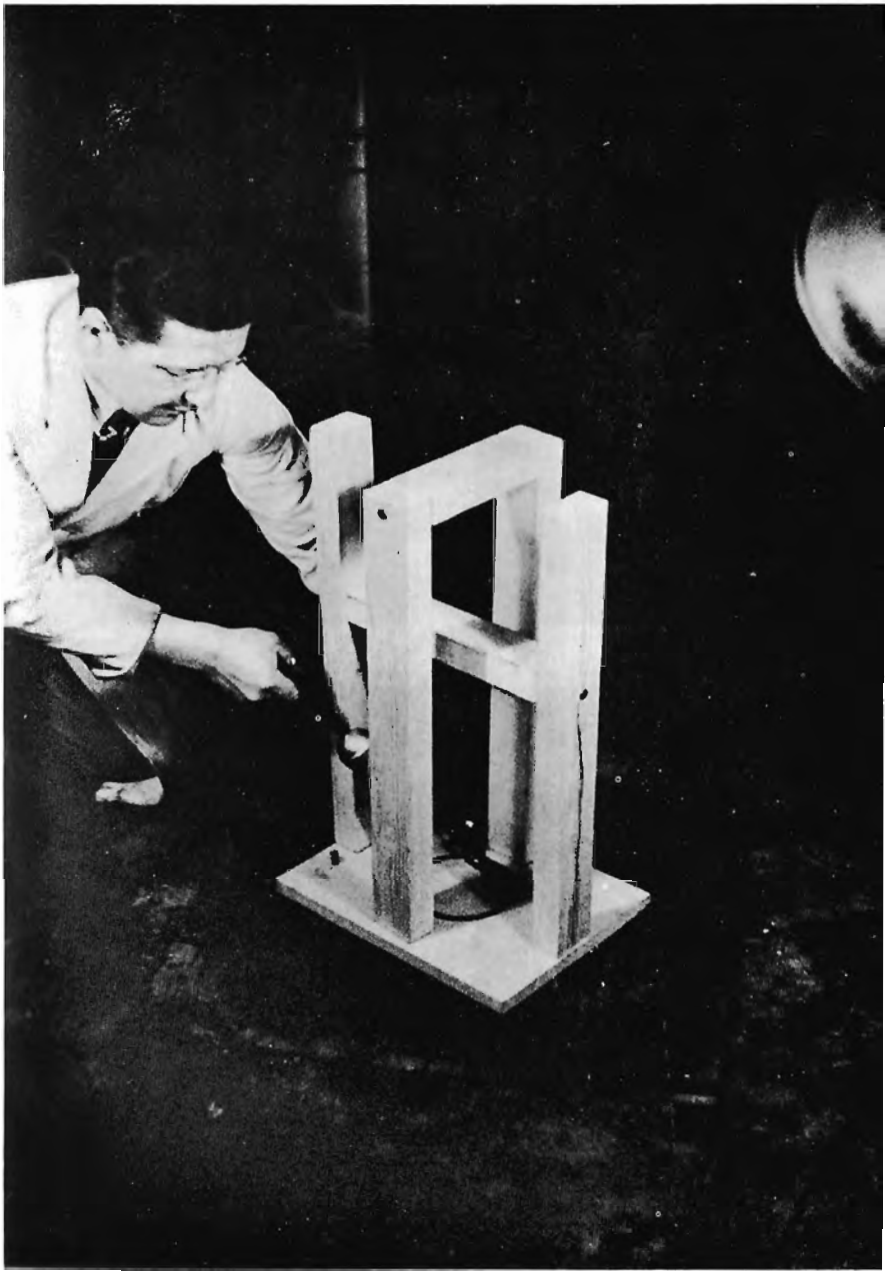


Fig. 1.4. — Dispensing a sample of Gel

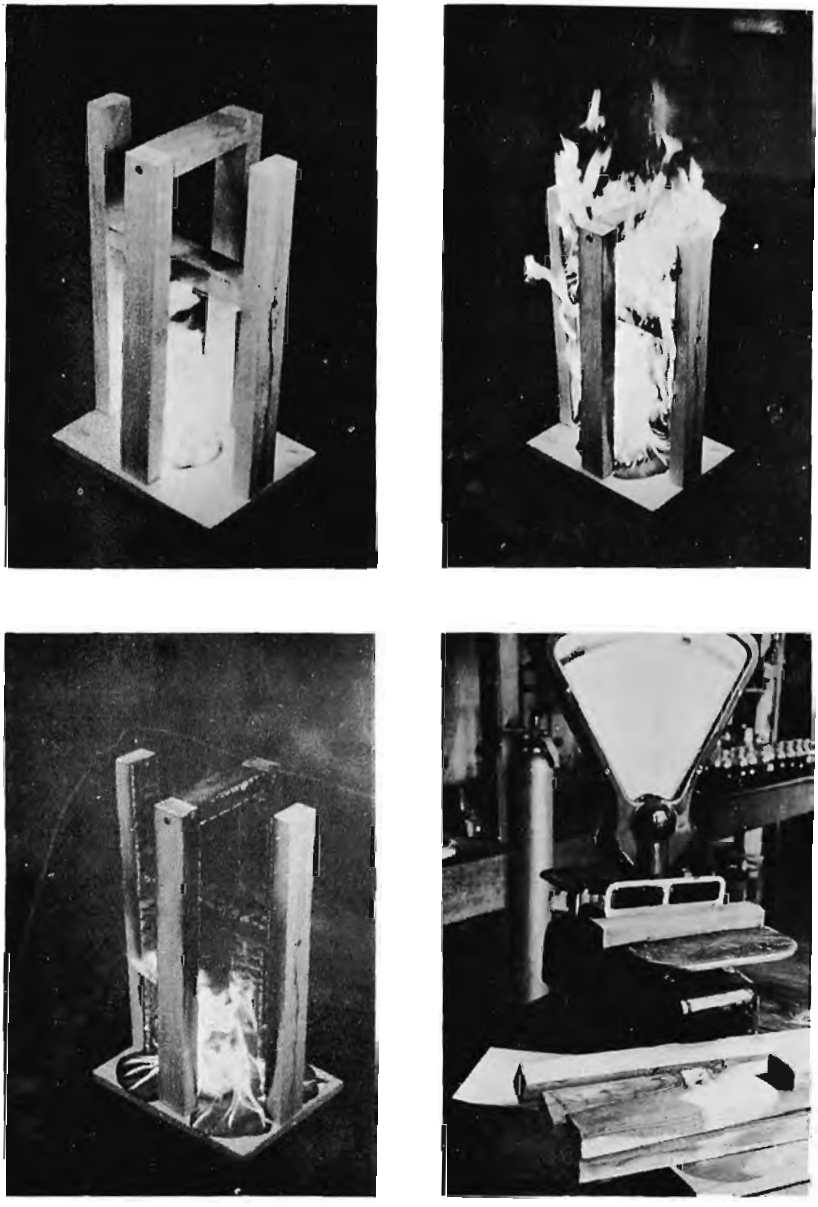
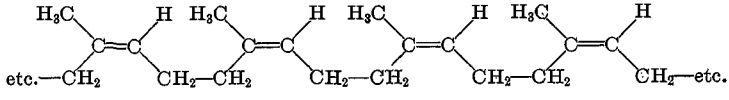


Fig. 1.5. — Gibbs Burning Test

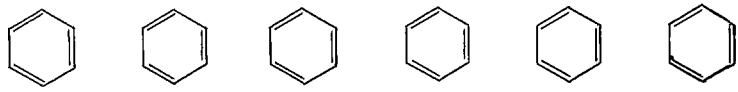
parison the volume of gel contained in a modified grease gun from which it is delivered by expression. Fig. 1.4 shows George Harris dispensing a standard-volume sample of gel (97.3 milliliters) onto the baseboard of a test structure. Fig. 1.5 shows successive stages of the fire in a draft-free environment. The initial weight of the structure less the weight of the charred pieces gave a measure of the incendiary effectiveness of the sample tested. Results were reproducible with accuracy, and hence we were in a position to evaluate different materials.

Rubber cements, such as Sanford's Grippit, are viscous adhesives made by dissolving raw rubber in a nonflammable solvent (e.g. carbon tetrachloride). Rubber also dissolves in flammable hydrocarbon solvents, and if the concentration is high enough the result is a viscous gel of the type we sought. Major Rambaut had stated that the British had experimented with rubber–benzene gels but had not developed a completed bomb. We prepared a number of rubber-benzene gels and from burning tests found

Rubber:



Benzene:



that they have high incendiary effectiveness. The amount of wood destroyed in the test increased markedly when the concentration of rubber was increased from 4% to 8%; further increase produced little further change. We found some correlation of incendiary efficiency with gel density and viscosity, which we determined in the apparatus shown in Fig. 1.6; this was designed and constructed by E. B. Hershberg, who also made the drawing. Peroxidized divinylacetylene gel burns in a manner that is spectacular and impressive, but quantitative burning tests showed that the incendiary value is far below that of a rubber–benzene gel of comparable viscosity. The relationship might have been anticipated: combination of the hydrocarbon with oxygen, prior to burning, decreases the energy of combustion with oxygen in the process of burning. An incendiary mixture compounded by Professor Morris S. Kharasch's NDRC group at Chicago from peroxidized divinylacetylene and sodium nitrate was more spectacular in appearance but had a low rating in the quantitative burning test (however, a batch of this material took off in the Chicago laboratory and did considerable damage). In the Gibbs evaluation, thermite made a particularly poor showing: about 1/7 the effectiveness of an 8% gel of rubber

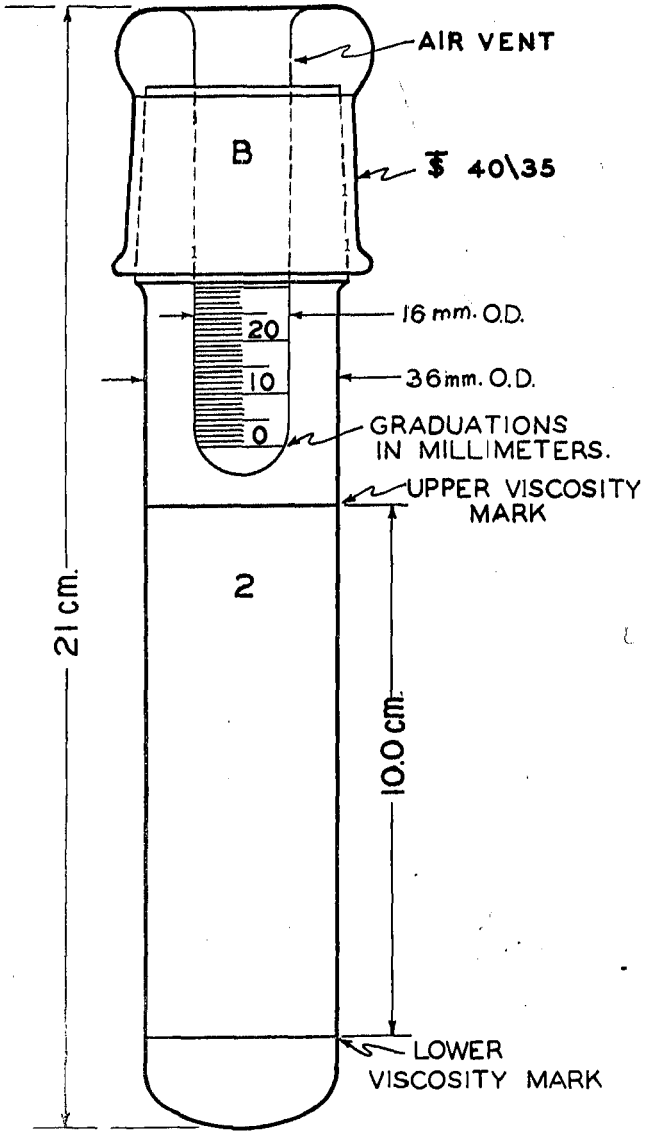


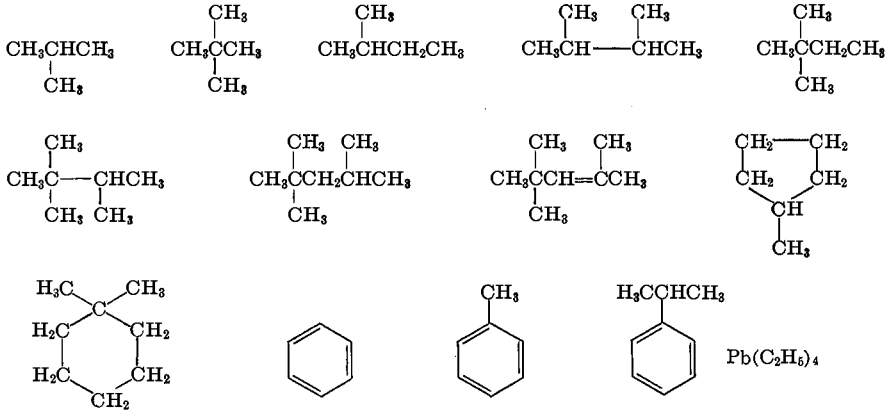
Fig. 1.6. — Combined Determination of Density and Viscosity. The glass tube is filled with gel to an appropriate point, stoppered with a cork, and centrifuged to remove air bubbles. The cap B is inserted, the tube is centrifuged briefly to produce an even meniscus, and the volume is read on the graduated scale. The assembly is then weighed, and the density is calculated from the weight, empty and filled, and from the volume. The viscosity of the gel is determined by timing the fall of a $\frac{5}{32}$-in. steel ball between the upper and lower "viscosity marks."

in benzene. Several subsequently established NDRC incendiary groups confirmed our evaluation of the Chemical Warfare Service thermite bomb as a dismal failure, but CWS abandoned the bomb only after it had been in production for two years. The delay assuredly was not due to lack of evidence or to delay in its presentation. Each set of tests or experiments by our group was described in a report which went out to all interested military and NDRC groups within a day or so after completion of the work; my secretary routinely made 35 copies of each report.

The following report summarized our findings:

> "Magnesium leads the field for overall incendiary effectiveness. The gum incendiary takes a reasonably good second place and, as an accessory substance having a special characteristic not found in magnesium, merits careful consideration."

We prepared and tested gels of rubber in a variety of solvents, including gasoline. Rubber–gasoline gels had the desired toughness and stickiness and rated nearly as high in incendiary effectiveness as gels in much less available solvents, and we settled on gasoline as the solvent of choice.



Typical Gasoline Components

Three forms of raw rubber were available from the rubber tree plantations of the Malaya Peninsula, Indonesia, and Ceylon, depending on the method of collection and processing: amber-colored smoked sheet rubber, pale crepe rubber, and rubber latex, or milk of rubber, a stabilized emulsion. Each of these forms of raw (natural) rubber proved satisfactory, and we determined the concentration of each required to produce gels of maximal incendiary value in gasolines of various types. Vulcanization destroys the

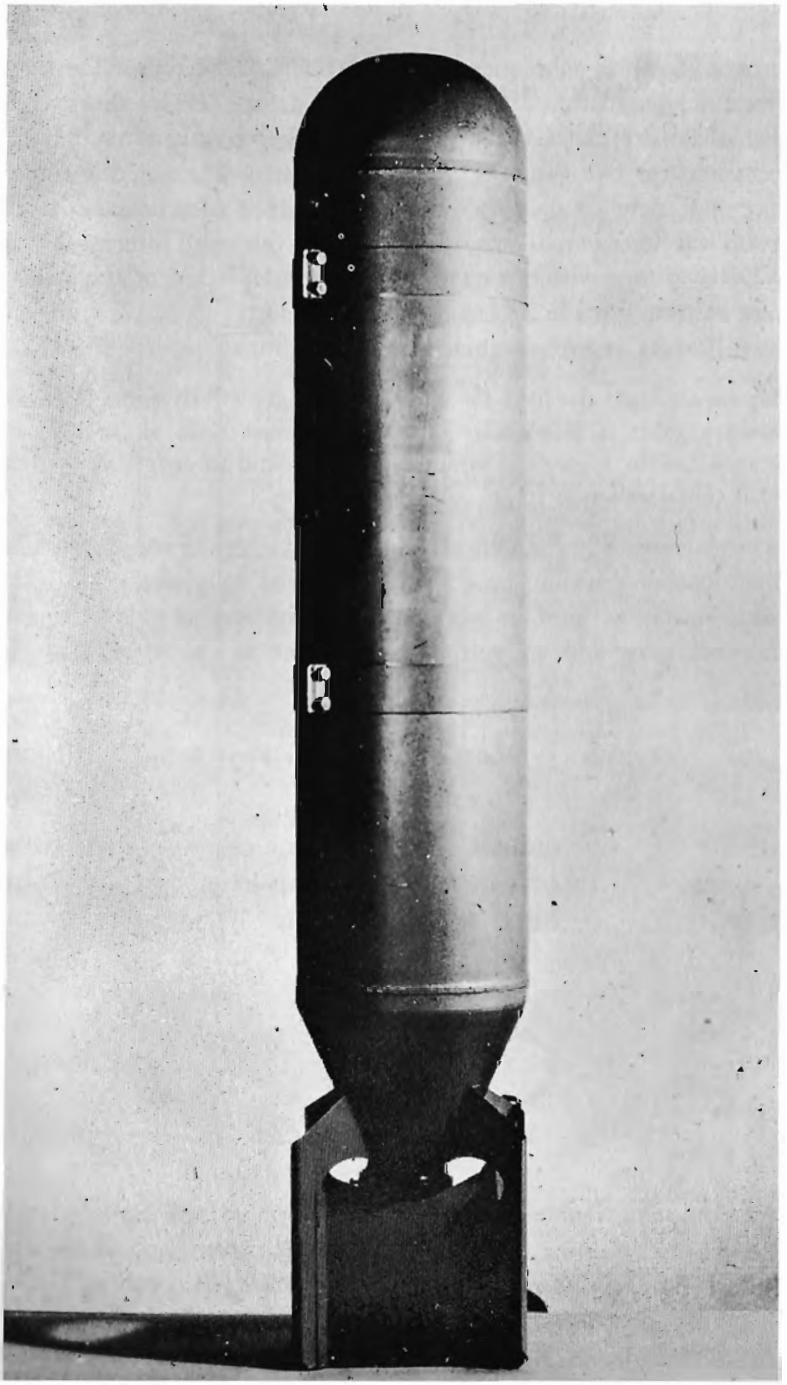


Fig. 1.7. — M-47 Bomb

ability of rubber to dissolve and form a gel, and we found all forms of vulcanized and reclaimed rubber to be unsatisfactory.

Having found a type of gel that is easily compounded from two abundantly available materials, we set out to develop a bomb. Fortunately Chemical Warfare Service had in production a bomb case, the M-47, which probably would serve as container. The M-47 bomb (Fig. 1.7) is 45 inches in overall length and the steel case of 18-gauge metal is 8 inches in diameter. The bomb was designed to be loaded with mustard gas, or other vesicant, and fired with a charge of black powder contained in a burster-tube which is screwed into a hole in the nose of the bomb and which has a $1\frac{5}{16}$-inch steel well extending to the bottom. An impact nose fuze screwed into the open end of the burster ignites the powder and the explosion rips open burster tube and bomb casing and distributes the charge over the target area. We obtained a truckload of bomb cases and started an exciting line of experimentation. Bombs were loaded with gel in the Gibbs Laboratory and taken for static firing with an electric squib to an area in back of the Harvard Stadium, from the top of which we could make observations and take photographs and movies (a film to be shown to the military had to be edited carefully for removal of an occasional small-boy spectator). Data recorded included area of distribution, average size of the globs of gel, and estimated percentage ignition of gel. If the globs were too small, we increased the concentration of rubber or decreased the explosive charge, or both. Once the adjustments had produced a bomb of reasonably satisfactory performance, we recorded the results in a film and I set off for Edgewood Arsenal with a demonstration bomb which I transported in a lower berth on the Federal Express. When the porter carried the closed package on to the train he said "It feels heavy enough to be a bomb."

I was to find from experience after experience that gaining acceptance of a new device by the military usually is a long and difficult operation, however good the device. A whole succession of individuals and groups have to be convinced and won over. Our project received impetus from prompt adoption of an improvement introduced by CWS Lt. Gordon Ponder consisting in using in the burster tube a charge of black powder and magnesium; we referred to this in reports as the PONDER MIX. The going was much easier in this first venture than later, when the CWS staff was greatly expanded. Actually success came sooner than expected. On November 27, I was asked to dictate by telephone to the CWS office directions for the filling of a shipment of 10,000 M-47 bombs that were en route to Manila. After consulting with Bradley Dewey on the types of rubber likely

to be available in Manila, I made a recommendation along the following lines:

> Cut *X* lbs. of smoked sheet rubber or *Y* lbs. of pale crepe rubber into strips and insert these through the opening in the nose of a bomb. Place the bomb upright and run in gasoline until the level is 3 inches from the top (in order to provide a 5% void). Screw in the burster tube securely and rest the bomb on its side. To ensure even mixing, rotate each bomb 180° at three 1–2 hr. intervals.

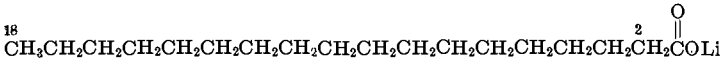
But the bombs never reached Manila. They were lost at sea by enemy action. On December 7, 1941, the Japanese had attacked Pearl Harbor. By this action, the Japanese not only destroyed a large part of our fleet but gained control of all the important sources of natural rubber.

2. *Napalm*

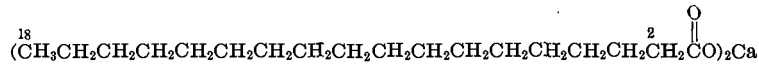
AT THE OPENING of the CWS Laboratory at Massachusetts Institute of Technology a few days after Pearl Harbor, Earl P. Stevenson [1] and I discussed the situation with Col. M. E. Barker, Chief, Technical Service, CWS, who said "Now find us something to use in place of rubber." Our search for a substitute thickener, launched the following day, had to be conducted along purely empirical lines, but it was a good example of efficient team work. Every member of the group made significant contributions, and everyone joined in drives to develop each successive formulation that offered promise. The Gibbs burning test was invaluable as a guide to satisfactory incendiary performance, and we learned to make preliminary evaluation of toughness and stickiness by manipulations with the fingers. The requirements set by CWS were severe. The gel must not thin out at 150° F. (operations in the tropics) or become brittle at −40° F. (or C.), the temperature usually reached in a bomb bay; it must be tough enough to withstand the blast of an explosive charge and not shatter; it must not deteriorate in storage; it must be adaptable to a simple field-filling operation. Actually the plan that bombs would be loaded at the theater of operation was subsequently abandoned and all gel-filled bombs were filled at the site of manufacture or in local arsenals, but the early insistence on field filling turned out to have been a fortunate circumstance when gelled gasoline came into use in flame throwers and in belly tanks.

We tried all available synthetic rubbers but none appeared at all promising. The only other clue was the knowledge that certain metal soaps are used for thickening lubricating oils to form greases. Lubricating oil is a mixture of long-chain hydrocarbons, and a metal soap such as lithium stearate or calcium stearate resembles a lubricating oil in having one or more long hydrocarbon groups. These metal soaps are insoluble in water and not detergents but, in accordance with the principle that like dissolves like, they dissolve in lubricating oil and form greases. A grease, of course, is brittle, and neither strong nor adhesive and, as expected, combinations of gasoline with

[1] Earl P. Stevenson, b. 1893 Logansport, Ind.; B.S. Wesleyan, 16; M.S. Mass. Inst. Techn., 18; Arthur D. Little Co.; Nat. Defense Res. Committee.

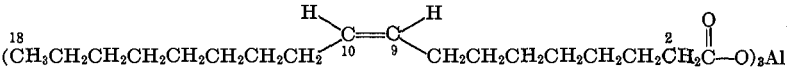


Lithium stearate



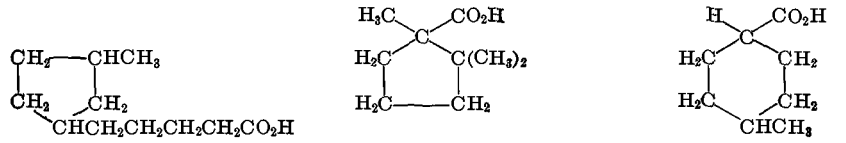
Calcium stearate

lithium or calcium stearate, or with aluminum, cobalt, magnesium, or nickel stearate were all completely unpromising. But there are other metals and other acids, and we secured for evaluation samples of several metal soaps that were available commercially. Aluminum oleate seemed the most interesting of the soaps tried initially, but it fell short by a good bit of being satisfactory.



Aluminum oleate

With entry of our country into the war other NDRC incendiary groups had been set up at the A. D. Little Co. in Cambridge, the du Pont Co. at Wilmington, Del., and the Standard Oil Development Co. at Bayway, N.J. We exchanged information and visits with these groups and in the early stages of search for a thickener we were in particularly close contact with H. G. Billings and others at A. D. Little in Cambridge. This group soon introduced us to aluminum naphthenate. Aluminum naphthenate can be represented approximately by the noncommittal formula $(RCOO)_3Al$, but it is a mixture of a great many components. The acid from which it is derived, naphthenic acid (or acids) is a constituent of petroleum which, in the refining operation, is removed by extraction with alkali, followed by acidification. The extracted material, a sticky dark brown tar, is a mixture of



Constituents of Naphthenic "acid"

acids derived from cyclic hydrocarbons present in petroleum and known as naphthenes. A few of the component acids which have been characterized are shown in the formulas. Petroleums refined in the U.S.A. afford only 0.1–0.3% of naphthenic acid, but the volume of production is so gigantic

that naphthenic acid is available in large quantity. Aluminum naphthenate is a commercial product manufactured by Nuodex Products Co., Elizabeth, New Jersey and by the Shepherd Chemical Co., Cincinnati, Ohio. It is an almost black, sticky tar which cannot be incorporated into gasoline by stirring. However, the Little group found two methods for incorporation of aluminum naphthenate into gasoline. They produced an 8% gel by a heating process, and a 5% gel from alcohol-washed aluminum naphthenate without application of heat. We thought these gels promising and used them in demonstrations at Bayway on January 3, 1942. The heating process might be adaptable to a manufacturing operation but was at the time ruled out by the requirement for a field-filling process. The Little chemists consulted with Arthur Minnich of Nuodex about the alcohol process and were informed that the alcohol washing could not be conducted on a manufacturing scale except at great loss and expense. Hence our group, in addition to continued trial of other metal soaps, sought simpler methods for utilizing commercial aluminum naphthenate. Impressed by the fact that aluminum naphthenate, a complex mixture, seemed superior to all the single-component soaps we had tried, I suggested to my co-workers that, if a given soap seemed unsatisfactory, it be mixed with one or two other soaps and tried again.

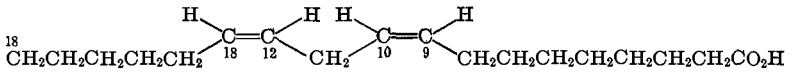
One new soap tried was a powder obtained from Metasap Chemical Co., Harrison, N.J., under the name "aluminum palmitate"; the name is put in quotation marks for a reason which will become apparent later. When stirred into gasoline in concentration of 5–10%, this soap dissolved readily and formed gels, but all gels tried were slushy and weak and generally inferior. We found however, that "aluminum palmitate" has the distinctive property of inducing low-temperature gelation of aluminum naphthenate and aluminum oleate. On Feb. 14, we reported to NDRC development of two lines of gels that could be prepared by stirring with gasoline at room temperature. To one, made from aluminum *na*phthenate and "aluminum *palm*itate," I gave the name Napalm. Two of the first formulations are as follows:

Napalm gels: Gasoline +

(a)
- 5% "Aluminum palmitate"
- 5% Aluminum naphthenate (Shepherd)
- 1% Wood flour

(b)
- 5% "Aluminum palmitate"
- 3% Aluminum naphthenate (Nuodex)
- 0.5% Lampblack

The minor additive, wood flour or lampblack, increased the value in the burning test from 440 g. to 530 g. and 536 g. The gels were strong and tough, but they had been made by mixing the components with gasoline in a gear pump, a manufacturing rather than a field-filling operation. Hence attention focused on gels of a second type based on observation that the weak gel formed by "aluminum palmitate" alone can be strengthened considerably by addition, as plasticizer, of unsaturated ("enic") acids, for



Linoleic acid

example a commercial mixture of oleic acid and linoleic acid. These we called Palmene gels. A typical formulation is:

Palmene Gel: Gasoline + { 7% "Aluminum palmitate"; 4% Oleic-Linoleic acid; 1% Lampblack }

A Palmene gel can be made by simply measuring the components into a bomb and shaking. I described and showed samples of these gels at a CWS-NDRC conference at Edgewood Arsenal on February 26, 1942 and was asked when we could load bombs for trial. I said "tomorrow" and telephoned instructions to Putnam in Cambridge to bring down the ingredients on the night train. Our supply of "aluminum palmitate" was exhausted, but Putnam persuaded Metasap Co. to put 25 lbs. on his Pullman at Newark and he fulfilled his mission.

The next day Putnam and I worked in Smoke Shop No. 2 loading ten M-47 bombs with Palmene (use of lampblack made this dirty work). In Smoke Shop No. 3 Dr. N. F. ("Slim") Myers and Dr. G. L. Matheson of the Standard Oil Development Co. loaded bombs with SOD's rival gel known as Formula 122 and compounded from 81% gasoline and a mixture of stearic acid, rosin, castor oil, sodium hydroxide, water, and kerosene. That was Saturday. Tests carried out the following Monday by dropping the bombs from 10,000 ft. onto Maryland mud were not very conclusive. I rated the Palmene gel as definitely better than SOD's Formula 122, which became identified privately by the Harvard group as apple sauce, but felt that our gel was not strong enough to stand the excessive charge of Ponder's black powder — magnesium mix which the CWS officers had insisted on using. We therefore turned to the Napalm type gel and sought to devise a field-filling process.

But first a word about Metasap's "aluminum palmitate." When we rec-

ognized this material as a promising compounding agent and felt that a satisfactory gelling agent could be developed from it, we took steps to learn if it could be made available in a quantity estimated at 500 tons per month. Companies other than Metasap listing aluminum palmitate included American Cyanamid, Harshaw Chemical Co., Mallinckrodt Chemical Works, and possibly others. Letters of inquiry went out to all known suppliers of the soap and to Armour and Co., supplier of palmitic acid and of Neo-fat mixed acids. We secured samples of the aluminum soaps, and asked Metasap to prepare for us aluminum soaps from samples of Armour's palmitic acid and related Neo-fats. A given soap might vary with the acid source or with the manufacturing process, and we wanted to make sure that all the samples would perform reasonably well. However, none of them proved to be even remotely promising. The behavior in contact with gasoline resembled more the behavior of aluminum stearate than the behavior of Metasap's soap. Something seemed fishy about the Metasap product, or rather about the name. Indeed, in a visit to Metasap I learned that their product is not a palmitate at all but the aluminum soap made from the total acids of coconut oil. Coconut oil is characterized by a high content of lauric

$$\overset{18}{C}H_3CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2\overset{1}{C}O_2H$$

Stearic acid (C_{18})

$$\overset{16}{C}H_3CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2\overset{1}{C}O_2H$$

Palmitic acid (C_{16})

$$\overset{12}{C}H_3CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2\overset{1}{C}O_2H$$

Lauric acid (C_{12})

acid, a C_{12}-acid which accounts for 48% of the total acid content; the amount of palmitic acid is only 8.2%. Palmitic acid (C_{16}) is indeed more closely related in structure to stearic acid (C_{18}) than to lauric acid (C_{12}). The shorter hydrocarbon chain of lauric acid is evidently required to give a soap of the specific desirable properties noted. The opinion of the Metasap chemists that coconut oil could be made available in adequate quantity was later confirmed in a letter from William Yandell Elliott, Harvard Professor of Government, then serving as Chief, Stockpile and Shipping Branch, War Production Board:

> "We have taken steps to have an immediate freezing of more than enough coconut oil to take care of your requirements."

Furthermore palm kernel oil and babassu oil have about the same lauric

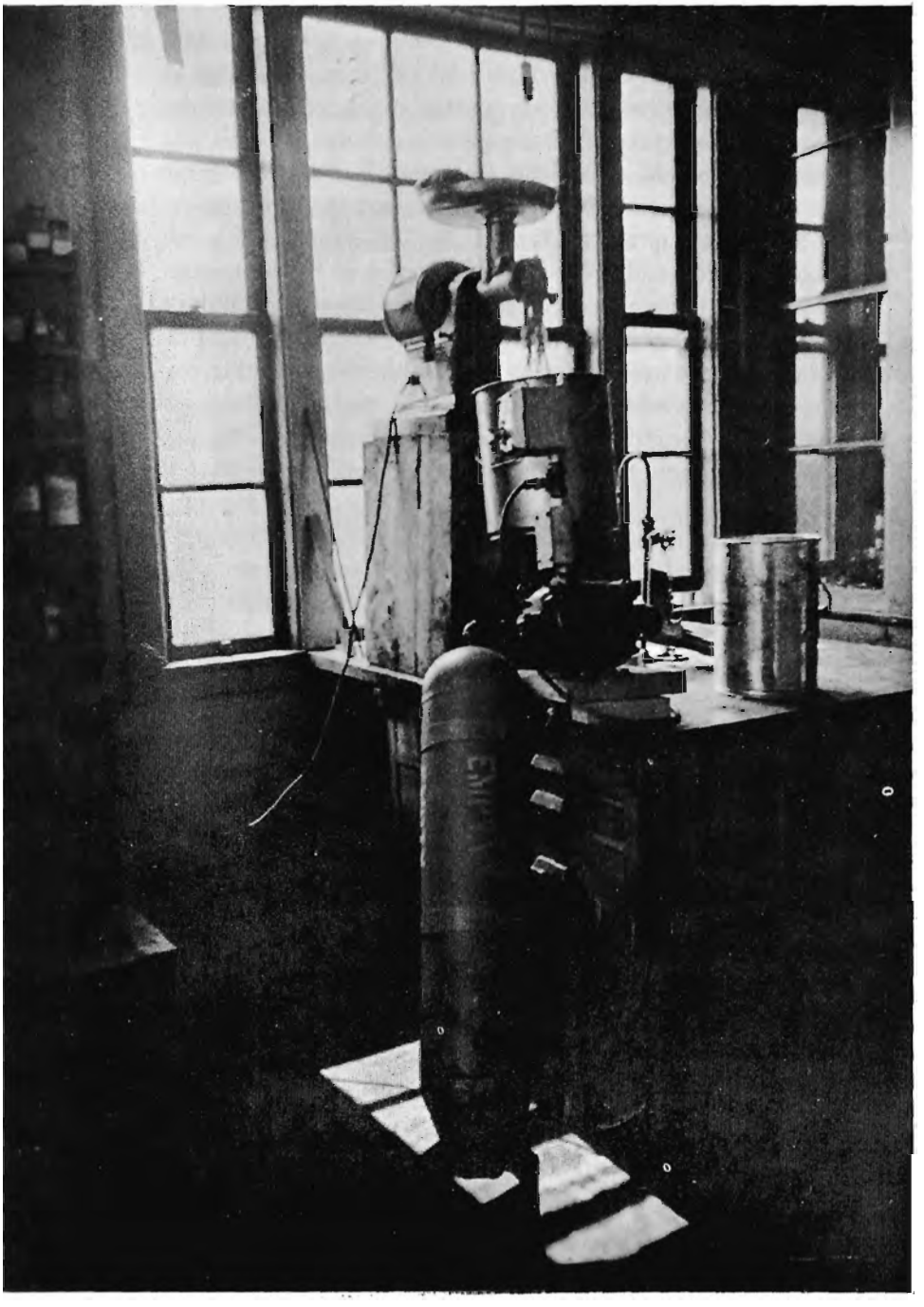


Fig. 2.1. — Preparation and Loading of Napalm Polymer Gel

acid content as coconut oil, and we made aluminum soaps from the split acids of these oils and found them fully satisfactory.

But the name Napalm, now seen to be nondescriptive, was there to stay. It was amusing later to receive a clipping from the *New York Sun* of July 30, 1945 giving the Empire City racing results: a horse named Napalm had been scratched. Possibly the owner got into trouble for using a word that was a military secret, if a nicely deceiving one.

Further work of our group through the spring months was greatly assisted by the cooperation of many companies in supplying high-lauric fatty acid fractions from a variety of sources which provided abundant material for an eventual 10-page patent with 15 claims: U. S. Patent 2,606,107. Incendiary Gels (1952). We did not, ourselves, find a variation of Napalm thickener suitable for field filling, but such a development came about in the following way. About March 22, 1942 we developed a material designated Napalm Polymer gum made by incorporating 1 part of Metasap's aluminum coconut oil acid soap and 1 part of aluminum naphthenate into 2 parts of kerosene at 100° C. in a steam-jacketed mixer at the Dewey and Almy plant, which we were kindly allowed to use under the direction of Hershberg, the brains of this engineering process. This material was incorporated into gasoline with the apparatus shown in Fig. 2.1. The tough gum was passed through a meat grinder and the spaghetti-like strands let drop into a mixer containing gasoline agitated by passage through a gear pump. The resulting gel was of superior quality and showed the high value of 588 in the burning test without an added filler. Static firing tests in Cambridge and bombing tests at the Edgewood and Huntsville Arsenals showed the gel to be distinctly tougher and more resistant to the shattering action of an explosive charge than any of the earlier gels. A request to fill 20 bombs for another series of tests overtaxed our capacity, particularly since our use of the Dewey and Almy mixer was limited to odd hours, and we decided to call for help from the Nuodex Products Co., makers of aluminum naphthenate. I visited Arthur Minnich at Nuodex on March 31 and he was most cooperative. He agreed to do the job in a pilot plant, which would be operated by a picked crew at night in secrecy. He also agreed to try out certain variations in the Napalm line and send us samples for evaluation. The aluminum naphthenate manufactured by Nuodex was made by a fusion process, but Mr. Minnich introduced into the discussion the important information that aluminum naphthenate can be produced as an only slightly sticky solid by precipitation from a strongly alkaline solution by addition of a solution of aluminum sulfate. This seemed extremely interesting, and we agreed upon a series of three coprecipitated

Napalm-type soaps which Nuodex would prepare and which we would test. The most interesting sample, Napalm X-104, arrived on April 13, 1942. It was made by the precipitation method from 2 parts of coconut oil acid, 1 part of naphthenic acid, and 1 part of oleic acid. The material is a brownish, dry, nonsticky powder. When an amount of Napalm powder sufficient to produce a 12% solution is poured into gasoline and given one stir, solvation occurs rapidly and the swollen solvated particles soon fill the container with material of applesauce consistency, which is pourable (Fig. 2.2). After aging for a few hours without attention, the gel reaches its

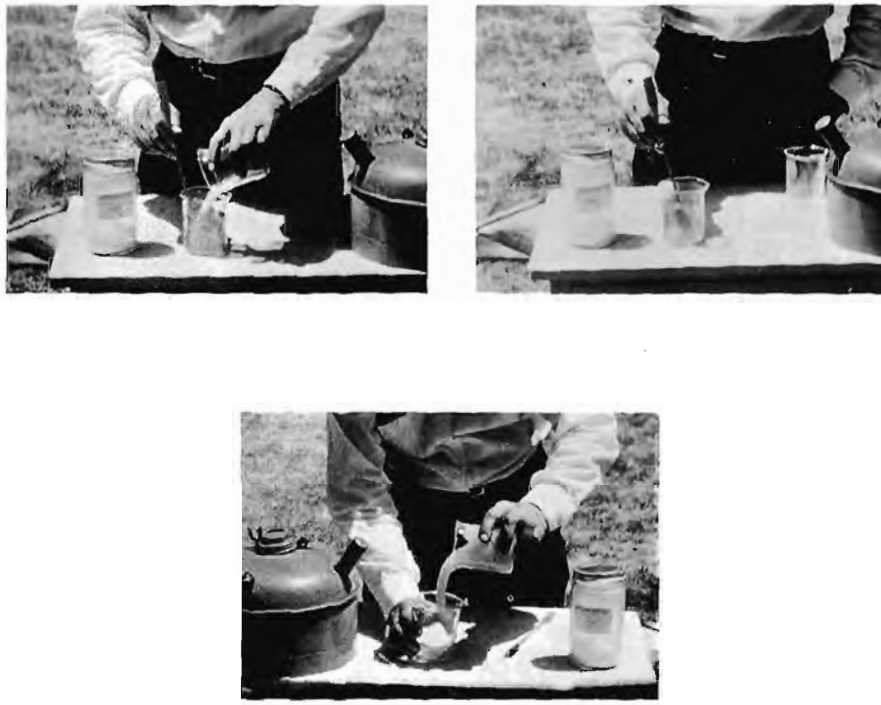


Fig. 2.2. — Solvation of Napalm Powder

final form, in which it is tough, strong, and sticky (Fig. 2.3). These desirable properties are retained at +150° F. and at −40° F. The value in the burning test is comparable to that of Napalm Polymer gel, and indeed little difference was discoverable in the quality of the new gel that is easy to make and the one requiring a manufacturing operation. Would Napalm

Fig. 2.3. — Aged Napalm Gel

X-104 gels be stable in storage? We applied such expedited aging tests as aging at 125°, recycling between −40° and 25°, vibration, and agitation with a paddle and discovered no weakness. We could not be sure of complete stability in prolonged storage, but the prospect seemed good. Actually two samples prepared in 1942 looked just as good in 1963.

3. *White Phosphorus Burster*

THE PERFORMANCE of an incendiary gel is determined not only by the quality of the gel but also by the type and construction of the bomb in which it is used and by the nature and amounts of materials used for opening the bomb and igniting the contents. In the early stages of World War II, officers at the Edgewood Arsenal evaluated performance by observing what happened when a bomb was dropped from a plane at an altitude of 10,000 ft. onto the ground, which usually was wet and often muddy. I witnessed several "tests" in which bombs plowed into the mud so far that they were largely underground at the time of firing. Even under the best of circumstances it was very difficult for an observer stationed at a safe distance to judge whether a poor performance was due to an improper charge of explosive, poor ignition, or poor quality of the gel. Our group had relied from the start on observation of the results of firing bombs statically on the ground, as already described. We could measure the pattern and area of distribution, estimate the average size of the incendiary globs, and make a rough estimate of the percentage ignition. Also, the results could be recorded on film. This method was applied to the first tests of M-47 bombs filled with rubber–gasoline gels and continued through the development of a succession of Napalm and Palmene gels. Irregularities not evident when bombs are released from a plane as at Edgewood become obvious when such variables as depth of penetration, rocks, and weather were eliminated by static firing of the bomb.

Our observations soon indicated that use of either black powder or Ponder's black powder–magnesium mix results in very irregular distribution and in poor ignition of gel. Our expert gadgeteer E. B. Hershberg thought up a new scheme. He reasoned that black powder on ignition builds up pressure slowly, and that this pressure is relieved, all at once, by forcing a hole at the weakest seam of the bomb. Often only a portion of the gel is ejected and the remainder burns in a segment of the torn casing. A high explosive such as TNT or tetryl produces an immediate, sharp explosive wave which should open both burster and bomb casing evenly over the whole length. Since a high explosive produces no flame, Hershberg's idea

was to surround a column of high explosive with a well filled with white phosphorus, which ignites on contact with air. Other igniters tried and rejected as less satisfactory than white phosphorus were magnesium, sodium, potassium, sodium–potassium alloy, diethylzinc, ethylsilica, and pyrophoric metals. The construction of the new burster is shown in Fig. 3.1. The burster well, which screws into the nose of the M-47 bomb, is loaded with the proper amount of molten white phosphorus, and the steel inner well

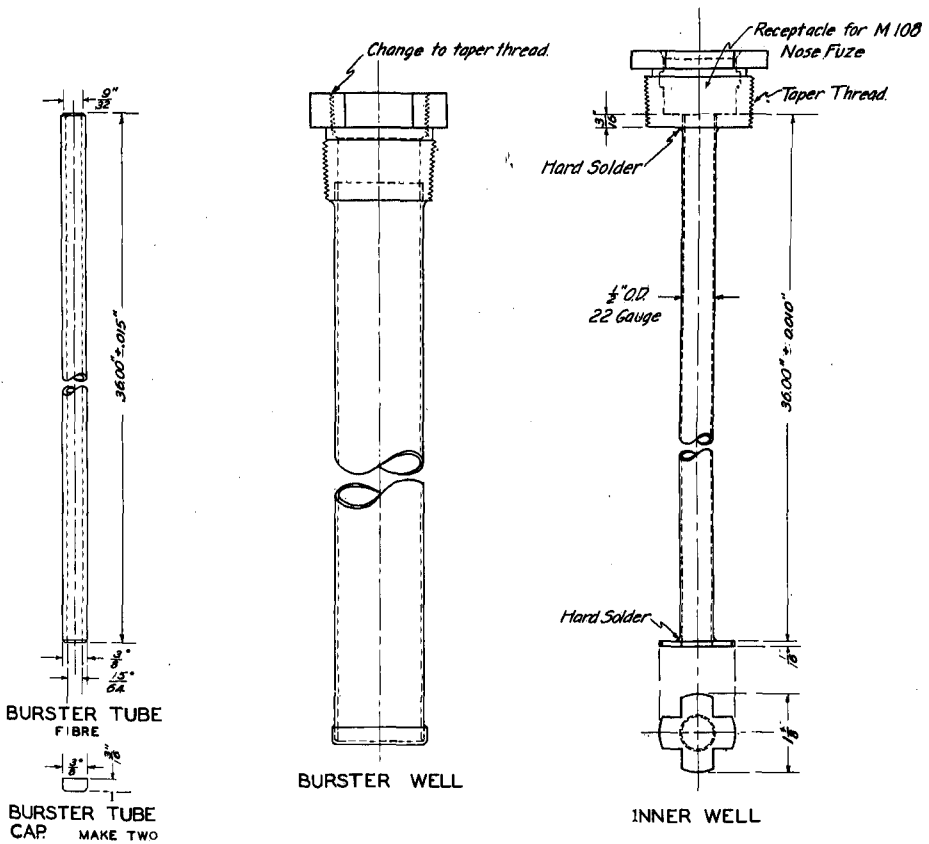


Fig. 3.1. — White Phosphorus–TNT Burster

shown on the right is inserted and screwed into place; the tapered screw-thread forms a completely tight seal. The star shaped disc soldered onto the end of the inner well centers this well so that it is surrounded evenly with phosphorus. The fiber burster tube shown on the left is charged with pellets of high explosive and inserted in the inner well and the assembly is ready to be screwed into a gel-filled M-47 casing. Insertion of a nose fuze readies the bomb for use. For safety in transportation, the gel-filled bomb, burster well charged with phosphorus, fiber tube containing the high explosive, and nose fuze can be shipped separately.

Hershberg arrived at the dimensions shown in the drawing from orienting trials with improvised units. In developing a practical firing assembly meeting Service requirements, he consulted with experts at the Ballistics Research Laboratory, Aberdeen, Md., and at Picatinny Arsenal. For production of a trial lot of the new bursters, we turned to Noblitt-Sparks Industries, in Columbus, Ind., since this company manufactured the M-47 bomb casing and the burster well. Edmund Ludlow, able engineer of this company, proved to be highly cooperative and helpful, details were worked out in visits back and forth, and it was not long before the bursters arrived and tests could be made.

The performance, from the start, was most impressive. The high explosive cuts the inner well into ribbons and opens the casing down the entire length. Pieces of phosphorus are driven into the gel, and large, burning globs are distributed evenly over a circular area about 50 yards in diameter. Extinguished with carbon dioxide or water, the phosphorus-containing gel may later reignite.

Objective comparisons of the new with the old bursters were made in two ways. In one Professor Harold E. Edgerton of M.I.T. took high-speed photographs of bombs filled with the same gel, one fired with a black powder–magnesium burster and the other with Hershberg's burster. On viewing at slow motion films taken at about 1,000 frames per second, one could see exactly how the two bursters operate and why one is immensely superior to the other.

The second comparison required a shallow water pond, and the flat Harvard soccer field beside the Business School was selected as a suitable site (Fig. 3.2). The Maintenance Department was persuaded to construct a circular earthen dike 30 yards in radius and two Cambridge fire engines obligingly flooded this with water to a depth of 4–9 inches. The bomb was mounted vertically on its nose on an iron-clad platform in the center of the pond and distances were marked out in four directions with stakes. An

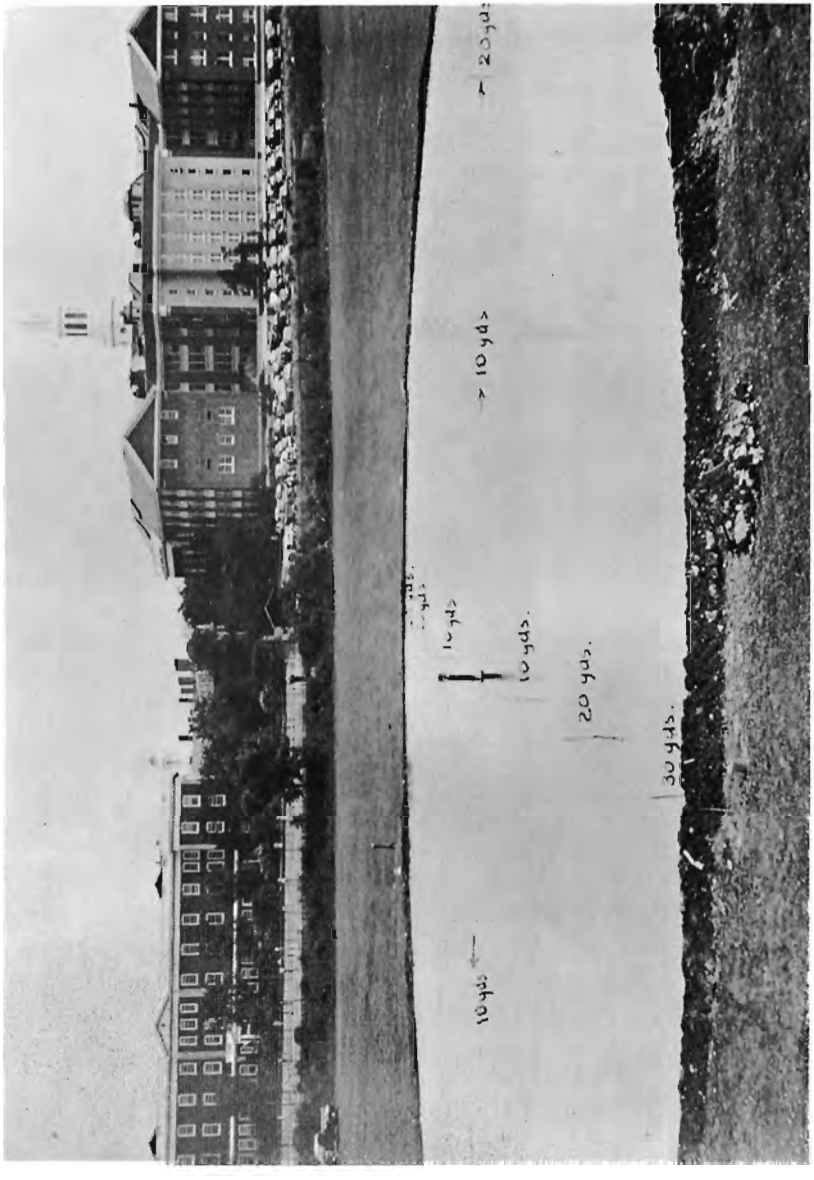


Fig. 3.2. — Test Pond with Bomb and Markers

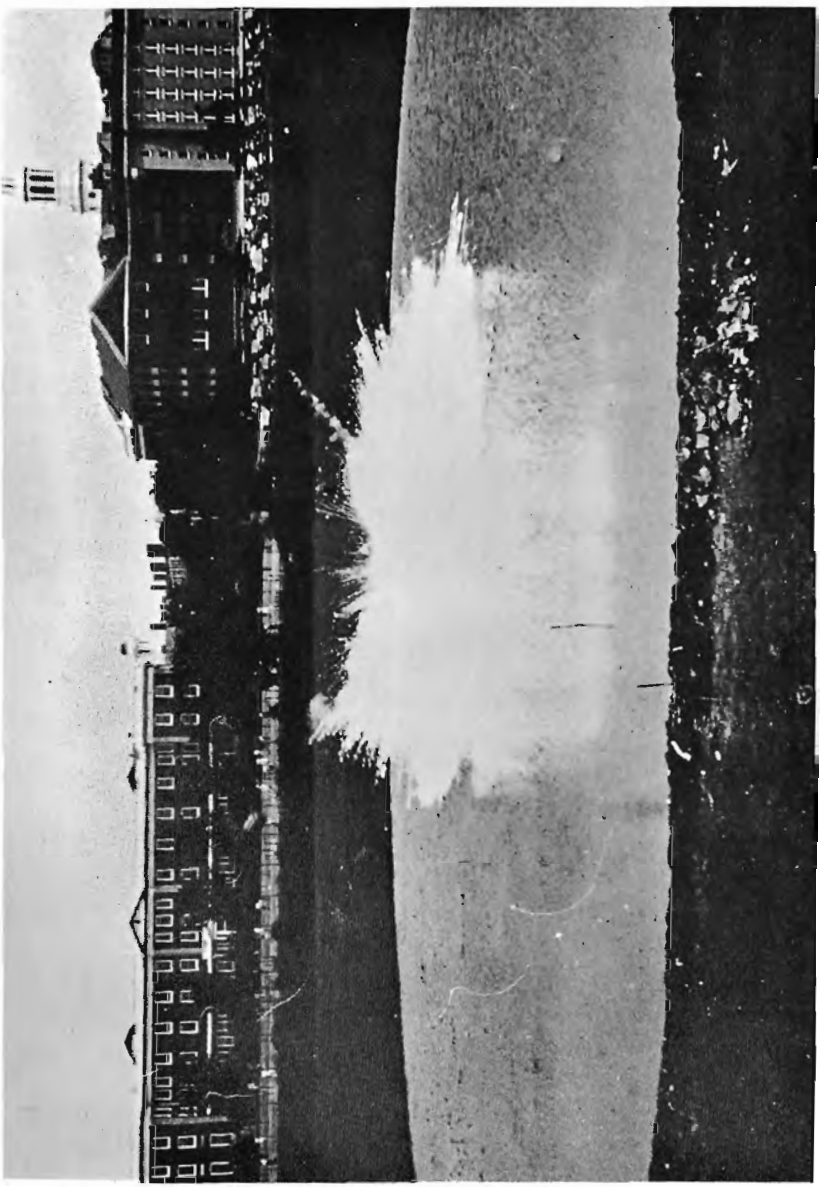


Fig. 3.3. — An Electric Squib Fires the Bomb

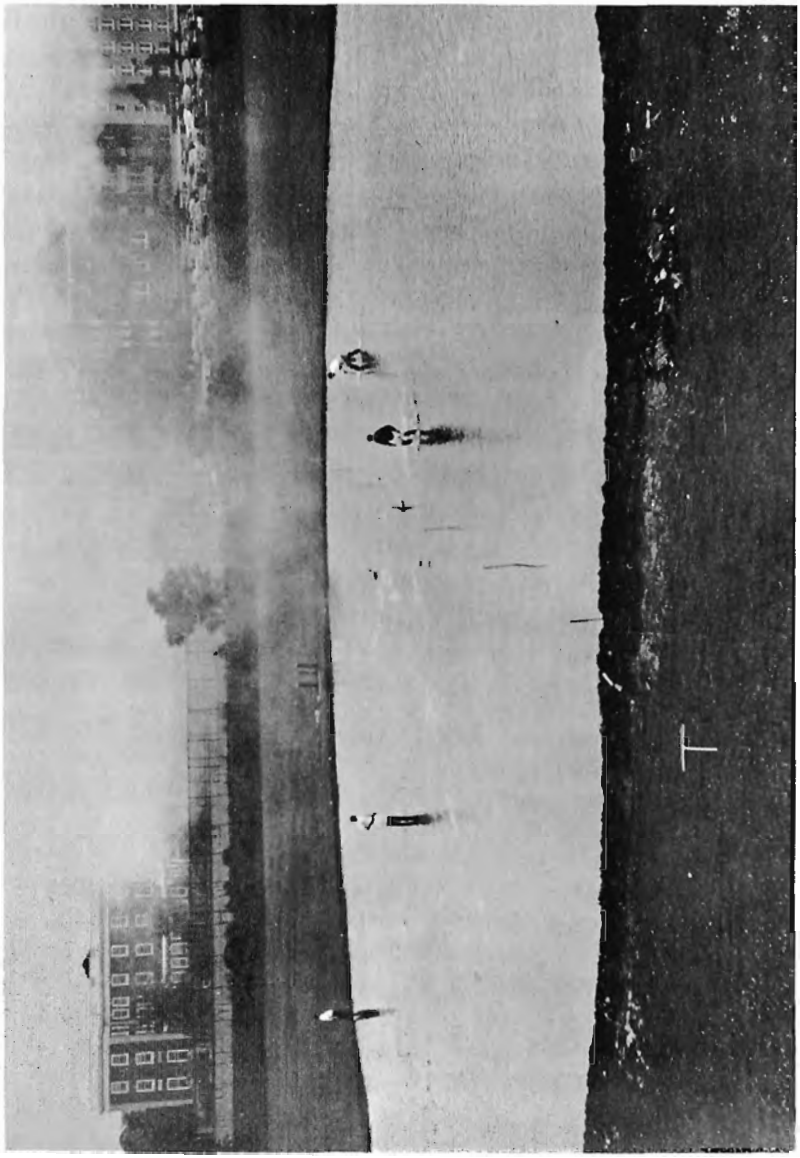


Fig. 3.4. — Fires Extinguished after One Minute

operator was placed at a safe distance from the North, East, South, and West stations, and each had a bucket of water at the edge of the pond for extinguishing the few fires going beyond the dike. The flight of the bomb casing, which presented the only hazard, is almost invariably in the direction opposite to the longitudinal seam of the bomb. Following the burst (Fig. 3.3) the four operators rushed in and splashed water on any pieces of burning gum. The photograph of Fig. 3.4, taken approximately one minute after the burst, shows that the fires could be extinguished quickly; loss from burning appeared to be very minor. The area of distribution and size of the pieces could be studied in detail. The floating pieces were submerged to such a point that evaporation was not extensive, and a nearly quantitative collection of clean Napalm gel was made by transferring pieces by hand (rubber glove) to a tared bucket. The collected gel was drained free from all but traces of water and weighed on the field. The difference in the weight of gel loaded into the bomb and that recovered gave a measure of the amount of fuel wasted in a "flash burn" of atomized gel. The relative effectiveness of the two bursters is evident from the data for Bombs 1 and 2.

BOMB 1

Napalm X-104 (42 lbs.)

White Phosphorus–TNT Burster

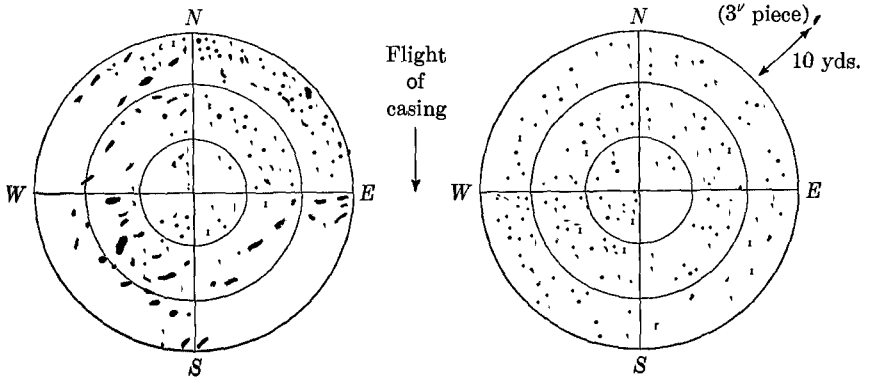
(2–2.2 lbs. WP, 49 g. TNT + 1 g. Tetryl)

Fires extinguished: 1 minute.

Distribution: Good. Several large, many medium pieces, only 2–3 lbs. found in small fragments.

Gel recovered: 40 lbs., 2 oz. (95.6%).

Large pieces: 16 pieces weighing 13 lbs., 2 oz. (31.3%) average weight 0.82 lbs.



Bomb 1 Bomb 2

BOMB 2

Napalm X-104 (42 lbs.)

Black powder–Magnesium Burster
[363 g. of powder (70 parts)-Mg (30 parts) with
2% castor oil binder]

Fires extinguished: At once. There was no noticeable burning of gel on the water. The slow-motion color photographs strongly suggest that the gel was blown downwards through the water and thus extinguished.
Distribution: Poor. The entire surface of the pond was covered with a spattering of very fine particles of gel (nearly all less than 2″).
Gel collected: 25 lbs. (59.5%)
Large pieces: None

Tests of four bombs were carried out on July 4, 1942. Although well aware that the soccer field was not intended as a place for the firing of bombs, I was surprised with the repercussion expressed in the following letter:

NAVY SUPPLY CORPS SCHOOL
Harvard University
Soldiers Field, Boston, Mass.

July 30, 1942

Dean Donald K. David
Harvard Graduate School of Business Administration
Boston, Massachusetts

Dear Dean David:

As you will recall, a few weeks ago some bomb experiments were carried out on the soccer field. A circular embankment approximately 175 feet in diameter was thrown up, which remained for about two days. This rendered about one-fourth of the drill area useless for that time. Also, after the explosions, irritating vesicant fumes clung to that corner of the field for several days. Effects of these fumes were still visible in the eyes of one officer six days after exposure. In addition, some seventeen officers required treatment during the night.

The soccer field has been designated by the University as a drill field for the Navy Supply Corps School, and makes possible an important part of our indoctrination. In addition to a student body of approximately six hundred, two companies of Business School students start participation in our drill periods on August 3. This number cannot be accommodated on the field unless its entire extent is available, nor, except for Sundays, is the field left idle any day of the week.

It has come to my attention that further experiments on this field are

contemplated. If so, they will seriously hamper the training and athletic program at the Navy Supply Corps School. It is earnestly requested that another site for these tests be selected.

Yours very sincerely,
K. C. McIntosh

HARVARD UNIVERSITY
Department of Chemistry

12 Oxford Street
Cambridge 38, Massachusetts

August 7, 1942

Captain K. C. McIntosh
Navy Supply Corps School
Harvard University
Soldiers Field, Boston, Mass.

Dear Captain McIntosh:

I greatly regret that the bomb firing experiments which we carried out on July 4th on a directive from the Chemical Warfare Service took up some of the space on the drill field area for a few days. Your comments on the effectiveness and persistence of fumes from the bomb may possibly represent an interesting commentary on the efficacy of this new munition, but I find it difficult to believe that there could have been a direct connection with the disabling of some of your personnel. There were eight men in all in my group of operators and we not only spent the day near the embankment but ran directly into the water-covered area immediately after each of the four explosions and spent practically the whole time making measurements and collections at the site of the bomb crater. A number of workers and firemen were exposed to some extent and there was no instance of any illness with any of these individuals or among my operators.

As for the future, we have no plans for further experiments calling for the soccer field and thus should cause you no further concern.

Sincerely yours,
Louis F. Fieser

Attempts to interest the CWS group in the burster had been initiated two months earlier, but progress was slow. Pellets of tetryl had been used as the explosive in the first bursters demonstrated and described in reports, but Edgewood officers expressed concern about the hazard of using an explosive that is more sensitive to impact than TNT and which might be set off by the impact of a bullet. So Hershberg, after suitable firing tests to determine the proper charge, devised a modification employing TNT pellets as the main explosive and with a ¾-inch section of pelleted tetryl as

booster at each end of the fiber burster tube (to make the ends interchangeable). The modified burster operated as satisfactorily as the original one and the small amount of tetryl offered no more hazard than the charge of lead azide in the nose fuze. But, argued someone at Edgewood, a hit on the steel well containing phosphorus might ignite the bomb. A group of us went out from the Arsenal on a warm day in May, supported a bomb horizontally, as it would be in a bomb bay, and had a soldier fire 50-calibre armour-piercing machine gun shells at it at a range of 40–50 yards. On the second shot a direct hit on the phosphorus well grazed the TNT tube and laid the phosphorus well open cleanly over a 2½-inch section. At the site of emergence, the shell had ripped out an irregular hole casing 3–4 inches in diameter. There was no fire. Observers stationed downwind smelled oxides of phosphorus; evidently some phosphorus had been driven into the air in the form of a fine dust which had undergone combustion without producing a flame in the vicinity of the gel. The phosphorus in the torn burster well seemed fully exposed to air, but it did not ignite in the period of about 10 minutes devoted to inspection and photography. Since the phosphorus was white, and did not have the yellowish appearance of exposed phosphorus, it may have been covered by a very thin protective film of gel. A possible minor objection had been eliminated. Why had not the same objection been raised to the CWS-favored black powder–magnesium burster, which is over twice as large a target and more easily ignited?

The original plan called for loading the phosphorus into the burster well under warm water and screwing in the inner well at a temperature above the melting point of phosphorus. But, said the officers, phosphorus in long contact with water produces "phossy water" which is corrosive. Inquiries addressed to Dr. H. Adler of Victor Chemical Co. and to Mr. C. A. Thomas of Monsanto Chemical Co., both experts with extensive experience in the manufacture and handling of phosphorus, brought expressions of complete confidence in the proposed method of loading and sealing the phosphorus. Dr. Adler stated further that it would be entirely practical to run dry molten phosphorus into the burster well at a temperature of about 50° C. and to introduce a small amount of inert carbon dioxide gas at the time of screwing in the inner well. "We are certain," he said, "that it will be possible to work out a loading procedure adaptable to mass production."

Would the bomb operate and ignite satisfactorily at the bomb bay temperature of −40° C.? A bomb was cooled overnight in the storage room of a Cambridge dry ice company; just prior to firing at Soldier's Field the average temperature of the gel filling was −36° C. The bomb operated

satisfactorily and the ignition was estimated as 75–80%; at 20–25° C. ignition is in the range 95–100%.

But mass production was a long way off. Many more bombs had to be dropped into the muddy fields at Edgewood Arsenal in preparation for the slightly more significant bombing operations described in the next section. Thus the high-speed movies and the water pond tests described above had been carried out in an effort to convince military and NDRC personnel of a conclusion which all members of our group had reached in April, 1942, namely that the new bomb was a fully perfected and highly effective munition. But there were delays, doubts, and new people to be satisfied. Edgewood and Washington vacillated. Edgewood officers thought it hazardous to ship gel-filled bombs fitted with the burster well containing phosphorus, even though the tube of explosive, as well as the nose fuze, were shipped separately. Eventually, without doing any experiments, or asking us to do them, they prepared drawings and specifications for a modified burster, the M-13, in which the phosphorus–TNT combination could be shipped separate from the gel-filled bomb. The CWS-modified burster had one more steel tube to be blasted away than Hershberg's fully tested model but specified the same explosive charge. It is hardly surprising that the M-13 was not a great success. However, Hershberg's principle was assuredly sound and it was adopted with better luck in bursters designed at Edgewood, with guidance from experimentation, for the 500-lb. "goop" bomb and for the Napalm-filled gasoline drums and belly tanks which were used with great success in the Pacific area.

4. *A Fight to the Finish with Industrialists*

My records show that on October 23, 1941 I visited Bayway, N.J. to report what I knew about incendiaries at the first meeting of a new NDRC group established at the Standard Oil Development Co. under the leadership of Robert P. Russell (M.S. M.I.T., 1922), then Vice President of the company. During the winter a du Pont incendiary program was set up under Dr. John G. Woodhouse (Ph.D. Harvard, 1927). The first objective of both industrial groups was to find a thickener for gasoline. The three groups exchanged information, met frequently at conferences and at Edgewood and other Arsenals, and each hoped to be the first to find the best solution of the problem. The Standard Oil Development group, or SOD group, before long set as a second objective the development of a gel-filled incendiary bomb of a new type, a tail-ejection bomb. This second project was eminently successful and resulted in a munition, the M-69 bomb, which was used by the millions with devastating effect in Japan. Two versions of the SOD bomb are shown in Fig. 4.1.[1] The casing is hexagonal and flat at each end so that many bombs can be put up into a cluster and released all at once. The usual tail for ballistic stabilization is replaced by cloth streamers, which save space and weight. Mounting the time-delay nose fuze horizontally rather than vertically saves further space for fuel. Use of a thin metal case as the container saves weight and simplifies production. In ideal operation the bomb penetrates only the roof, and the time-delay fuze prevents firing until the bomb has entered the target structure. When the fuze does operate, the gelled fuel is ignited and ejected out of the tail and splattered against wall, ceiling, furniture, etc. Preliminary tests in simulated attic structures had shown that small amounts of fuel placed at favorable distances from the eaves are highly effective in starting fires. The bomb shown on the left carries a charge of white phosphorus for production of a smoke screen to hamper fire fighting.

[1] Reproduced with permission from W. A. Noyes, Jr., "Science in World War II. Chemistry," Little, Brown and Company, 1948.

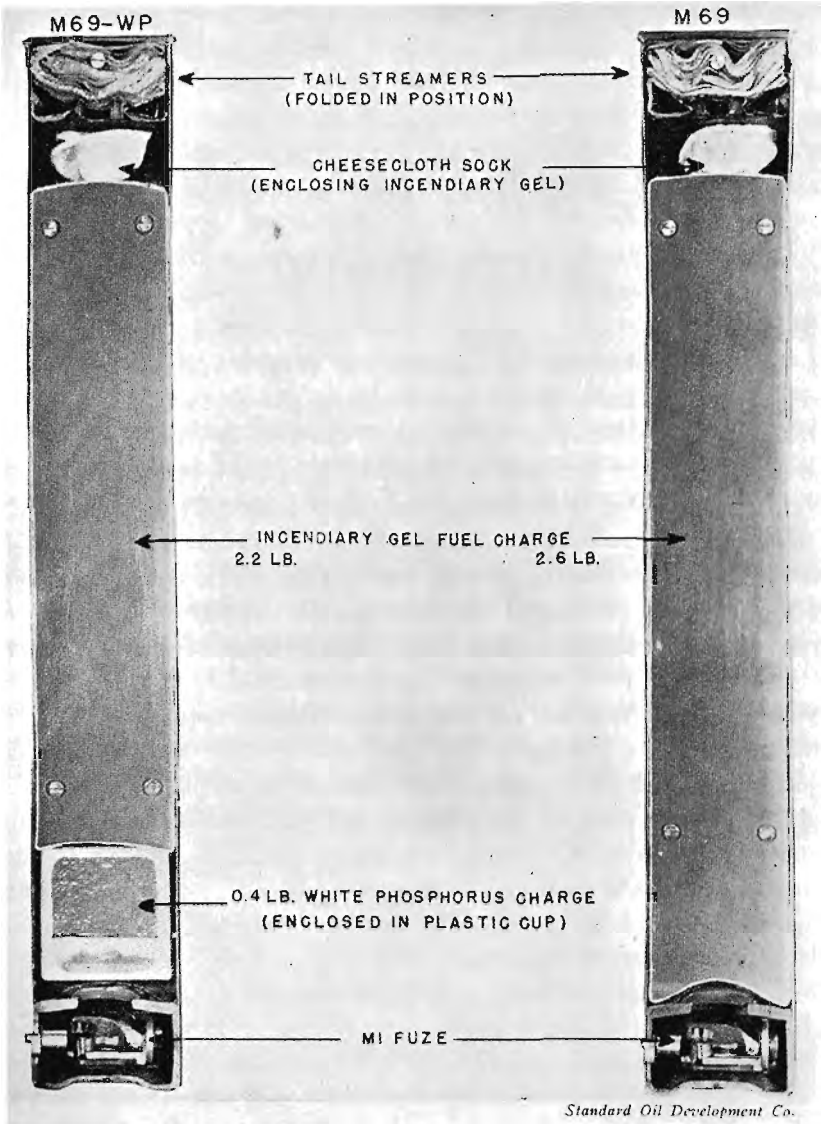


Fig. 4.1. — Sectional models of AN-M-69 and M-69-WP bombs

The groups at du Pont and Harvard followed the development of the M-69 bomb with appreciation and with considerable personal interest, for here was another munition requiring a satisfactory incendiary gel. My group secured from SOD successive batches of bomb cases, ran tests with them filled with our currently best gel and experimented with variations

that might be helpful to the SOD group. For example, a letter addressed to Dr. H. A. Richards, Jr. read in part as follows:

April 25, 1942. —

"Novello and Harris will arrive at Bayway on the usual train on Monday morning and deliver 7 small bombs and 7 large ones, each loaded with 100 grams of white phosphorus and with Napalm Polymer Gum prepared in gasoline doctored up as nearly as we could to give it a 12-lb. Reid vapor pressure. We would want you to try these out in any way you like, but I would very much appreciate it if you would use a few for the following severe test of ignition in direct comparison with the best igniter you are now considering: free firing in the open with no target and with the bomb placed unrestrained at an elevation of 15–30°. Another condition would be to maintain a gum temperature of −40° C., for this test provides a means of telling just how good different ignition systems are.

"I sent some pictures of the bursting of these bombs to Bob Russell and mentioned the process used in loading. The boys will demonstrate this method further to you, but for completeness . . ." (see Fig. 4.2).

The SOD group naturally started off using as incendiary their applesauce Formula 122, and I suspect that the drawing of Fig. 4.1 represents an early model because the gel is inclosed in a cheesecloth sock, an expedient by which reasonably good performance could be secured with a gel of inferior strength. However, the du Pont and Harvard groups both had gels which they thought needed no sock. The du Pont gel was based on use of isobutylmethacrylate; the preferred formulation was:

du Pont's IM-gel:

- 5% Isobutylmethacrylate, $CH_2{=}C(CH_3)C(=O)OCH_2CH(CH_3)_2$
- 2.5% Stearic acid
- 2.5% Naphthenic acid
- 3% Sodium hydroxide solution (40%)
- 87% Gasoline

This gel looked good to us, almost as good as Napalm. May 3, 1942 was set as the date for a series of comparative tests at Bayway for selection of the gel best suited for the M-69. Our best formulation at the time was:

Napalm gel:

- 6.75% Aluminum naphthenate
- 6.75% Aluminum coconut oil acid soap
- 13.5% Kerosene
- 73% Gasoline

To be included were static tests in attic structures, ejection tests from 30

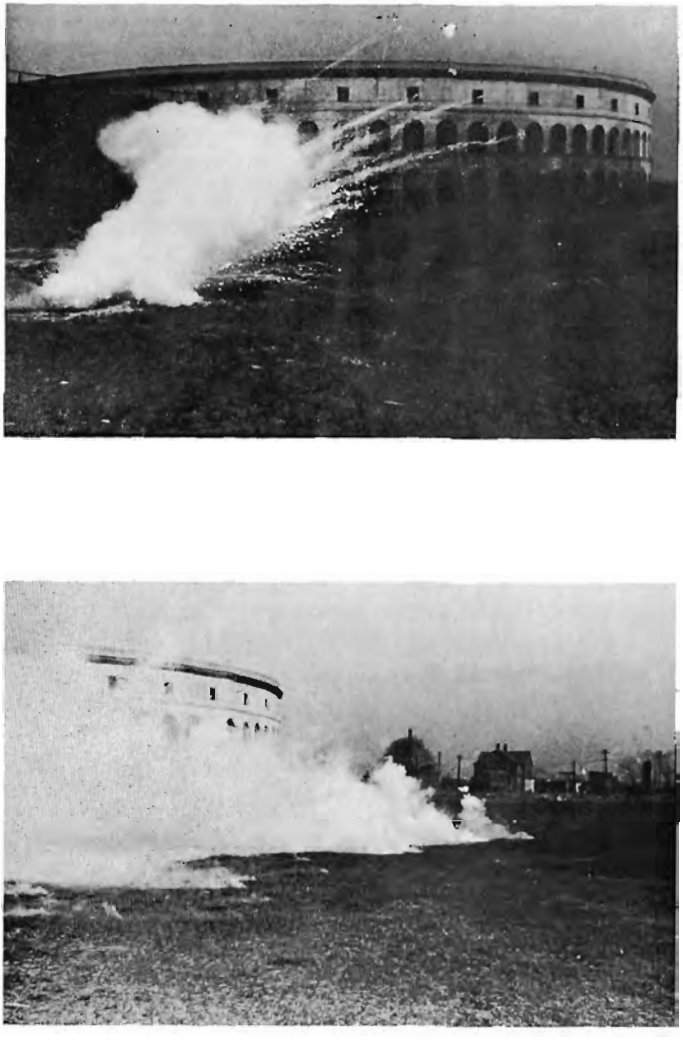


Fig. 4.2. — Use of White Phosphorus in the Napalm-Filled M-69 Bomb to promote Ignition and Provide an Obscuring Screen (Harvard Stadium, 1942)

ft. against a target wall at −40°, 77°, and 125° F., ejection tests into attic structures, tests in which bombs were fired from a mortar at a velocity of 210 ft./sec. An NDRC committee was to supervise the tests and evaluate the results. CWS officers would attend as observers. Personnel of the three

competing groups were to load and ready their bombs at Bayway in advance. The six members of the Harvard group were all needed.

I can describe the results of the Bayway Shoot in no way better than to quote from a diary recording all out-of-town trips:

> May 2–3, 1942. Bayway, N.J. — "NDRC tests to select gel for the M-69. We entered Napalm in competition with the then favored du Pont IM. The boys and I worked at Bayway the day before until 5 a.m. trying to determine the optimum powder charge and concentration for the mortar tests and were still ill-prepared (and ragged tired) at 9 a.m. when the program began. Even so, we thought that NP made as good a showing as IM. However, the judges, Hottel and Stevenson, much to our disappointment, ruled IM the winner. That evening at the Biltmore men's bar: disgust session with the boys."

This summary makes no mention of one entry for the reason that SOD's Formula 122, even when supported in a sock, took a low third place and was not heard from again. Napalm had been rated No. 2 for the M-69 but was still in contention for the M-47; in combination with the Hershberg burster it should have a good chance of winning. CWS officers eventually realized the futility of "tests" involving the dropping of bombs onto mud and set up a program of tests on M-47s and M-69s at Huntsville Arsenal, Alabama, where, we were told, bombs could be dropped on actual and combustible structures. Actually the targets were rickety old abandoned farmhouses, barns, and settlers' shacks, and little or nothing was gained from the effort. The diary record is:

> June 3–7, 1942. Huntsville Arsenal, Ala. — "Static and bombing tests of incendiaries on settlers cabins, etc., conducted by CWS and observed by N. F. Meyers (SOD), D. E. Strain (du Pont) and myself. M-69s seemed to me to make a better showing with a Napalm charge than when filled with du Pont's IM-gel. The bombing tests were so delayed by weather, engine trouble, etc., that I did not stay (to see mainly near misses)."

A much better test area was then located at the Jefferson Proving Ground, Madison, Indiana. A large tract of land taken over by Ordnance included a number of substantial houses in village and farm groups which had been evacuated recently and which were in good condition. A big CWS-Air Force test of all available incendiaries was scheduled for July 11–21, 1942. In arranging to supply the 100 M-47s requested, we had the able cooperation of Edmund Ludlow of Noblitt-Sparks, where the bursters and inner wells were manufactured, of Howard Adler of Victor Chemical Co., where the wells were dry-loaded with phosphorus, of Arthur Minnich of

Nuodex, where Napalm X-104 was compounded. National Fireworks Co. pelleted the high explosives and loaded the fiber burster tubes.

The operating personnel of 15 for the Jefferson tests included skilled fliers and bombardiers. Those present included representatives of:

Jefferson Proving Ground
Office of the Chief of CWS
Office of the Assistant Chief of Staff
Army Air Forces
Headquarters, Services of Supply
British Air Commission
Joint New Weapons Commission
National Defense Research Committee
Bureau of Ordnance, Navy Department
Wright Field
U.S. Marine Corps
Edgewood Arsenal
Standard Oil Development
(N. F. Myers, W. P. Knox)
du Pont Co.
(D. E. Strain)

For some reason I was listed not as of Harvard University but as one of nine representatives of NDRC, which included Vannevar Bush and Roger Adams.

The program was well planned and the tests went off nicely. B-25s and Dive Bombers delivered bombs on target with remarkable precision. Napalm competed, in M-47s, with an Edgewood version of du Pont's IM-gel; the phosphorus–TNT burster fought round by round with Ponder's magnesium burster. We had some lucky hits and beautiful fires. Fig. 4.3 shows target areas where results were particularly exciting. In target Group 158 a Napalm bomb fired statically in house No. 2 burned the house down in 2–3 minutes. In group 159 a direct hit by a Napalm bomb on large barn 2 set a fire which leapt, in succession, to sheds 5 and 4. Another NP bomb hit in front of house 1, and gel splattered under the tin eaves slowly set fire to the house and three sheds.

It was a real pleasure, on my return, to tell the boys that, in my opinion at least, Napalm had won out easily over du Pont's IM-gel and that Hershberg's burster had proved far superior to the Ponder mix. There had been one curious incident. A static test for comparison of the Napalm and IM bombs had been scheduled in a place some 20 miles distant where

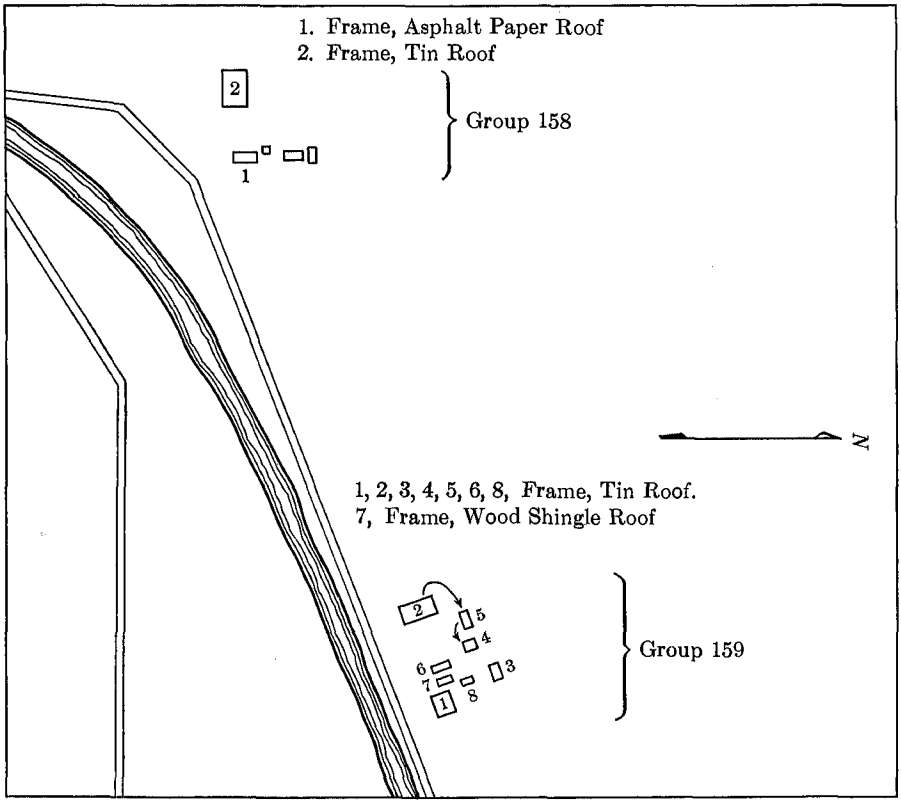


Fig. 4.3. — Jefferson Proving Ground Tests

there were two almost identical, very large barns. Bombs and observers were trucked over the rough road and a Napalm bomb was fired first in one of the barns. It is difficult to imagine what happens when 42 lbs. of burning gel is plastered all over the inside of a sturdy wooden barn: flames bursting out of the windows, blasting open the door, belching forth at the eaves and then through the roof. In a matter of minutes what remained of the structure collapsed into a burning heap. The du Pont bomb was duly placed in the second barn and fired. There was a loud blast, which blew out the windows and doors, and one flash of light. Then all was quiet and there was no fire. The result was what might be expected of a bomb filled with unthickened gasoline. A replacement bomb had not been taken along, and the schedule did not permit arrangements for another trial. The officers in charge felt sure that someone had erred in leaving the IM-thickener out of

this particular bomb, and there was a good bit of speculation as to who was to blame.

On relating this incident to my co-workers, E. B. Hershberg expressed the opinion that the du Pont IM-gel had been partially broken down, or caused to undergo syneresis, by the vibration involved in trucking the bombs over 20 miles of rough road, and that the force of the explosion had completely destroyed the residual gel structure. He had found that the IM-gel when worked mechanically at a low temperature, as by prolonged agitation with a paddle, could be broken down with separation of an upper layer of gasoline. A report of the experiment went unheeded. The IM-gel tested by Hershberg was an early sample, not identical with that tested at Jefferson or with the gel soon standardized for large-scale manufacture of M-69 bombs. But Hershberg had discovered a real fault which was recognized only after several million IM-filled M-69s had been manufactured and shipped to England. Bombs which had been fully inspected and found satisfactory on shipment performed unsatisfactorily on arrival. Vibration, in shipment, had caused partial syneresis to a point such that the explosive charge used to fire the bomb completed the process, with the result that the bomb fired in an ineffective flash burn.

A hasty switch was made to the No. 2 choice, Napalm, and the Napalm-filled M-69s developed no fault and scored a high record of success in the bombing of Germany, and a superb record in the Pacific Theater of Operations. Shortly before V-J day, which marked the end of the war with Japan, I had a visit from a group of British officers who had been assigned the problem of inspecting a cache in England of some 4 million M-69s and of picking out and destroying those in which the gel had started to "turn to water." The bombs all carried the same marking but some contained IM-gel and some contained Napalm. Sample inspection had shown that the Napalm-filled bombs were still satisfactory, but that practically all of the IM-filled bombs had deteriorated to such a stage that they should be discarded.

Participation of our group in the Napalm development did not extend much beyond the tests at Jefferson Proving ground. Other NDRC groups figured importantly in work on standardization, stabilization, trouble-shooting, and evaluation in cooperation with a number of eventual manufacturers. By war's end production of Napalm thickener had reached a scale of about 75 million pounds per year; approximately 30,000,000 M-69 bombs had been produced.

Our group played no part in the adaptation of Napalm gel to use in flame throwers, but it was gratifying to us to learn that the SOD and other

groups found this particular gel ideally applicable to such use. A gel of about 6% concentration can be forced through a nozzle under gas pressure and ignited, with the result that a burning, rod-like stream is ejected and that some 70% of the burning fuel can be delivered onto a distant target. Flame throwers tried in World War I utilizing gasoline as the fuel were of some slight value for their psychological effect if the wind was in the right direction. In World War II, thickened fuel led to development of highly effective tank-borne and manually operated flame throwers. The du Pont IM-gel is unsatisfactory for this purpose because it undergoes syneresis under the pressure required for ejection; it is thixotropic. Gels of raw rubber in gasoline suffer from the same defect and would not have been satisfactory for use in the M-47 or M-69 bomb, or in flame throwers. Hence Japan's early acquisition of the major supplies of raw rubber turned out to have been a blessing to the allied nations.

5. *The Harvard Candle*

In a letter addressed to Earl P. Stevenson of NDRC, Col. Barker of CWS stated in part:

> December 18, 1941. — "We have the requirement set up for a little incendiary munition which will weigh not over three or four ounces, which can be carried safely in a man's pocket, which will be completely waterproof, and which can be used by a soldier under very cold weather conditions to light a fire when the wood is not too dry, and which can be used also for starting fires in light constructions."

Actually the requirement had come from the Army Air Force, interested in developing a survival kit for use by a flier forced down in a remote area. In addition to an air mattress, traps, fishing tackle, and other gadgets, the kit was to contain a fire starter. Earl Stevenson evidently had passed on the word to my group. I soon wrote to Col. Barker as follows:

> January 6, 1942. — "We have hit upon a very efficient method of burning either thickened or unthickened oils which is particularly well suited to the purpose of a small incendiary munition to be carried in the pocket. This consists in loading the oil into a hollow cylinder of thick-walled celluloid of such quality as to leave an appreciable ash. The cylinder burns first and the oil then burns in a porous shell which is something like a welsbach mantle. The result is a long and very effective burn.
>
> "I am sending you 6 pocket incendiaries constructed on this principle. The casing, made for us by the Plastics Division of Monsanto Chemical Co., is 4 in. long by 1⅝ in. outer diameter . . .
>
> "This gadget, which we refer to as the Harvard Candle, works particularly well when filled with a thickened oil . . .
>
> "We buried one unit in dry ice for three hours and found that it ignited and burned satisfactorily at an initial temperature of −65°C. We made burns on our standard test structure and found that a candle containing aluminum naphthenate gel burns about 7 minutes and destroys 751–796 g. of wood."

Celluloid, which is nitrocellulose plasticized with camphor, is an ideal

container because of its high flammability. However, the wick principle turned out to be unimportant. A later model filled with Napalm gel in kerosene burned satisfactorily whether or not the porous ash-mantle is broken. It is perhaps worth noting that if the cylinder is filled with a rubber gel, this gel plugs the openings and burns only if the ash is broken. Col. Barker was favorably impressed with the first Harvard Candles, but noted that the highly flammable celluloid case might be set on fire in a soldier's pocket or pack. An answer to this objection was found in providing the cylinder with a thin film of nonflammable vinylite lacquer. Demonstration that the flame of a match could be applied to the treated cylinder without igniting it satisfied Edgewood.

The final form of the unit is shown in Fig. 5.1 and details of the assembly

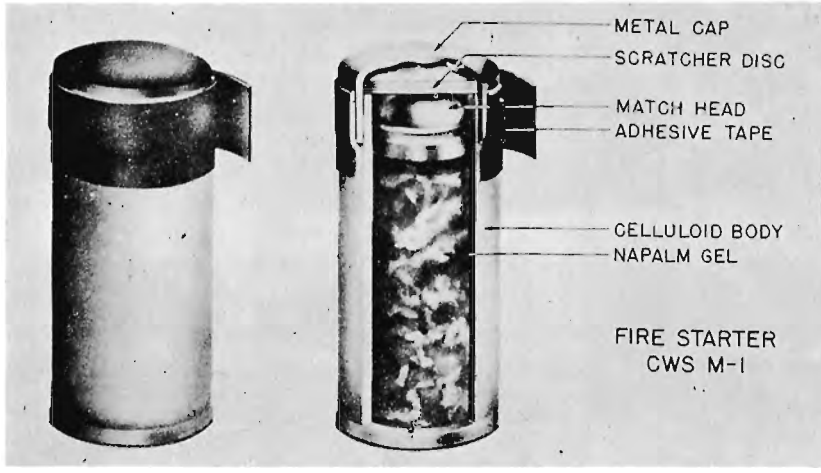


Fig. 5.1

are shown in Fig. 5.2. As supplied by Monsanto, each cylinder had a recessed top sealed in place (A, center). A cylinder is inverted and filled with gel either with a grease gun (B) or under a slight air pressure. The celluloid base plate is cemented on with acetone (C), with care to avoid entrance of a drop or two of acetone, for this will slowly peptize the Napalm gel and cause syneresis. A match-head composition (Universal Match Co.) is covered with a perforated kraft paper disc and molded into a button flush with the top (D). A milk bottle pull cap to serve as a scratcher disc is coated on one side with a striker composition and placed upside down (for safety) on top of the cylinder (F). A metal slip cap is put in place (E, F) and secured with waterproof tape (G). The unit is then dipped in vinylite lacquer (H)

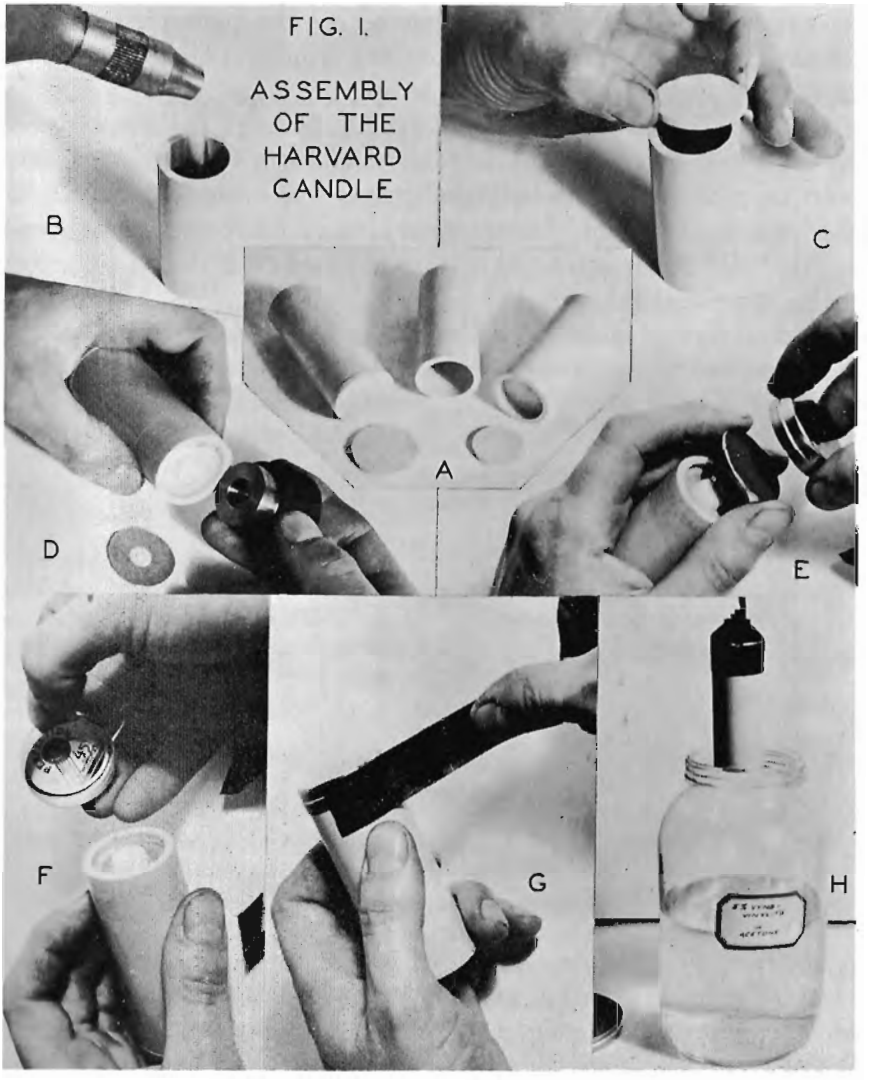


Fig. 5.2. — Harvard Candle

and dried. For operation of the munition, the tape is removed and the striker disc is held in the fingers and drawn across the match head. Ignition occurs even in a high wind or at a very low temperature. The candle as supplied is completely waterproof; it floats on water. A candle assembled in 1942 was tested twenty years later and found to function perfectly.

Chemical Warfare Service set November 30, 1942 as a date for a program of tests for evaluation of all candidate fire starters. We entered the Harvard Candle with confidence, for we had already made the comparison shown in Fig. 5.3 with a competing device developed by a group headed by Norman J. Thompson at the Factory Mutual Research Corporation. The Kharasch group at Chicago entered two fire starters into the competition. Chemical Warfare Service, prior to receipt of the first report on the Harvard Candle, had placed an order for 250 plastic cases with screw caps. But since the plastic selected was cellulose acetate, which is essentially nonflammable, the Edgewood entries were far from impressive.

The Chicago group was represented at the tests by Professor Frank H. Westheimer, the Harvard group by Dr. E. B. Hershberg. The results are summarized as follows in a report to General W. B. Kabrich by Dr. H. M. Chadwell,[1] NDRC Technical Aide and former roommate of mine in the Harvard graduate school:

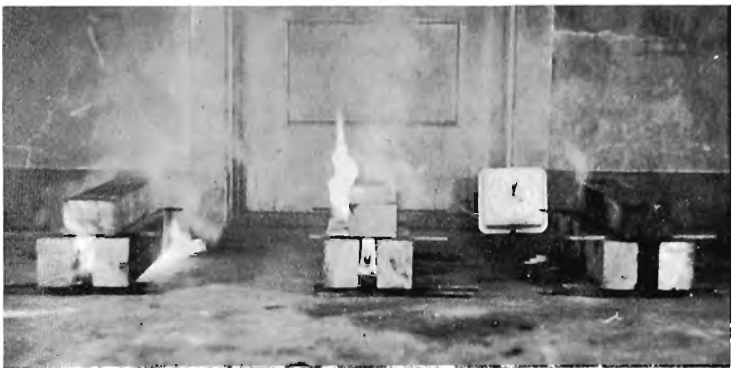
> "The general conclusion of this demonstration was that the Harvard Candle was superior to any of the others under test."

The device was promptly adopted and standardized as the M-1 Fire Starter (M for MARK). Our final report included all information needed for the drawing up of specifications and for submission of orders, as well as an estimate of a cost per unit of 22.37 cents on a production of 1 million units.

The fire starter impressed me particularly when I made a personal trial in the course of a two-day vacation at a boyhood wonderland at Indian River, Michigan, incidental to an NDRC conference at Buffalo on September 6, 1942. After spending a day fishing for trout in the beautiful Sturgeon River, I had no trouble starting a fire in the rain with nothing but a Harvard Candle and rain-drenched twigs and sticks. The Boston Herald for December 16, 1959 carried an account of two officers from an Air Force base in Bangor who had bailed out at night in the northern Maine woods in zero weather and foot-deep snow. These men credited Air Force survival training and equipment with making possible their rescue. One found a cabin and was "quite comfortable" sleeping on the air mattress. The other "trudged all night through the woods, stopping occasionally to light a fire for warmth."

[1] H. Marshall Chadwell, b. 1898 Amesbury, Mass.; B.S. Dartmouth, 19; Ph.D. Harvard (T. W. Richards, 24); Tufts College; Rockefeller Found.; U.S. Embassy, London; U.S. Atomic Energy Comm.

Comparative Tests with Wood Soaked in Water for 24 Hours

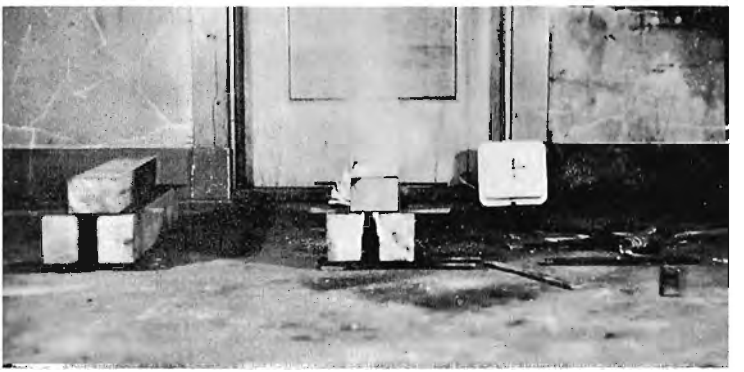


Factory Mutual Long Candle (Candle burned 2 min. 17 sec., wood fire out in 4 min.)

Harvard Candle

Factory Mutual Short Candle (Candle burned 1 min. 40 sec., wood fire out in 3 min.)

THREE MINUTES



Harvard Candle burned 8½ min.; permanent wood fire in 13 min.

FIFTEEN MINUTES

Fig. 5.3. — Comparison of Fire Starters

6. *Pocket Incendiary*

THE NEXT problem was presented by the Office of Strategic Services and fell under the jurisdiction of NDRC's cooperating Division 19. Since the highly secret operations of OSS were outside the scope of ordinary warfare, the nature of this organization may not now be generally appreciated. The following account was written by Dr. Warren C. Lothrop,[1,2] one of two Technical Aides to Division 19; Dr. H. M. Chadwell was the second Aide.

"Carried on the second or later pages of many of the country's newspapers during the war years could be found, from time to time, accounts

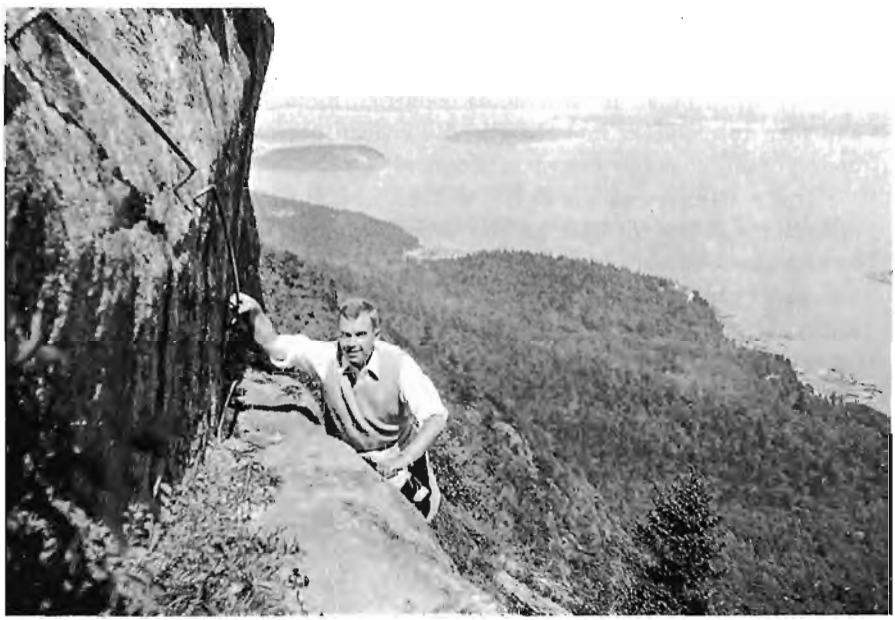


Fig. 6.1. — Warren C. Lothrop

[1] Warren C. Lothrop, b. 1912 Brookline, Mass.; A.B. Harvard, 33; Ph.D. Harvard (Fieser), 37; Trinity College; Williams College; Arthur D. Little, Inc.; Armour and Co.

[2] "Science in World War II. Chemistry," pp. 431–432, Little, Brown and Co., Boston 1948; reproduced with permission of author, editor, and publisher.

of patriot action, mysterious underground attack, or sabotage against German and Japanese military control. These could not, of course, be considered as militarily decisive, but their combined and increasing effect of psychological and material harassment wore on the nerves of the enemy and diverted part of his attention and resources to essentially unproductive effort.

"The situation was a strategic one in that the aggressions of Germany and Japan had resulted in placing under their authority many peoples who showed to a varying degree their appreciation for 'culture' or 'coprosperity.' The United Nations were thereby provided with a new weapon in the form of countless allies well placed in the enemy's camp, and the British and the Americans were not slow to grasp the opportunity. Their effort took a number of forms.

"Most spectacular were the large military raids made along enemy-held coasts by Commandos and later gathered together under the Combined Operations of the British, and under the Rangers and similar groups in the Army and Navy of the United States. On a less ambitious scale, but bearing close resemblance, were the so-called *coup-de-main* parties or small groups of specialists trained and dispatched for attack on some closely guarded but vital enemy target. Frequently these groups were composed of patriots who had escaped to England and, after outfitting and training, were returned by air to their home countries, there to organize the attack and then to disappear into the friendly civilian population and perhaps by underground channels return to England.

"It was necessary for the success of these men, as well as for the maximum disturbance of the enemy, that the underground groups of patriots should be organized into effective units, supplied with and instructed in weapons such as they would have occasion to use. This meant the supply, particularly to the continent of Europe, of numbers of men who had facility in the languages of the different countries and were thoroughly trained in strategy and operations. It is easy to visualize one of these brave men parachuting at night from a low-flying Halifax bomber into some remote part of Yugoslavia, Poland, Greece, or France, there explaining his mission to the natives, enlisting their support and through their co-operation avoiding the ever-present Gestapo, while he organized a resistance group and arranged by wireless for its supply from Allied planes with the explosives, incendiaries, and special devices calculated to annoy the enemy and to interrupt and destroy his transport and matériel.

"The increasing numbers of these resistance cells meant increasing variety and increasing quantity of devices, and the problem of supply and the problems of development became increasingly serious. Thus during the year 1944 alone, the Office of Strategic Services, the American group charged with this type of operation, dropped in occupied Europe alone over

8500 tons of material which included all manner of destructive devices as well as food, medical supplies, and regular Army or Navy equipment. The result of this was not only the immediate loss to the enemy of many badly needed power stations, ships, railway tunnels, warehouses, etc., but also the psychological boost to the morale of the conquered peoples, who by these means were able to feel that they were tangibly contributing to their own liberation.

"The British, because of their earlier entrance into the war and their proximity to operations, naturally assumed the original burden both in manufacture and in development of the implements best suited for this unorthodox type of warfare. Their severely taxed manpower, the rapidly expanding scope of resistance operations, and the entry of the United States into the War all contributed to the establishment of a similar group in America, which was created by Executive Order, named the Office of Strategic Services, placed under the direction of Colonel (later Major General) William J. Donovan, and organized under the Joint Chiefs of Staff. To this branch of the Services the original British groups operating in this specialized field assigned permanent liaison officers, and a joint program of procurement and development was initiated. The latter part became, at the request of OSS, nearly the sole responsibility of Division 19 of the National Defense Research Committee."

The Office of Strategic Services saw an urgent need for an incendiary for use by underground operators for the sabotage of enemy warehouses, factories, ships, railroad and truck cargoes, rail and road bridges. With this objective in mind, Stanley P. Lovell of OSS, in company with an NDRC–CWS group, had attended the tests of fire starters held at Edgewood Arsenal on November 30, 1942 and had had additional experiments done for evaluation of the small fire starters as weapons of sabotage. The Harvard Candle seemed to be a satisfactory starting point, and a conference was scheduled for December 18, 1942 at the Gibbs Laboratory. In attendance were representatives of the Plastics Division of the Monsanto Co., the Standard Pyroxoloid Corp. of Leominster, Mass., Factory Mutual, and Harvard. Mr. Lovell outlined the problem; his further comments are indicated in the following quotation from his report of the conference:

December 18, 1942. — "It was stated by Mr. Lovell that three objections were found to the Harvard Candle as it was produced:

1. It rolled and did not stay in position.
2. It was not a convenient and inconspicuous pocket size.
3. It lacked a time-delay mechanism.

It was agreed that the elimination of these faults would be entrusted to Dr. Fieser, and that the preferred size was ¾ x 2¾ x 5 inches. Such a case

is estimated to contain two or three times as much incendiary mixture as the Harvard Candle . . .

This office is to furnish 3 dozen black-mark time-delay pencils each to Dr. Fieser and to William H. Lane, Standard Pyroxoloid Corp."

A time-delay device, by which the incendiary could be set to go off after a predetermined number of hours, was all-important. An operator could then plan his mission in such a way as to establish for himself an alibi at the time of firing, or to arrange for the unit to go off after a ship was at sea or a plane in flight. Fortunately an NDRC group had already developed for OSS a very satisfactory time-delay pencil for firing explosives. It would be our problem to modify this for ignition of an incendiary.

The flat shape agreed upon seemed to call for a case made of thin-walled celluloid. Filled with Napalm gel, the casing would ignite easily and set fire to the gel. In this project the minor hazard to the operator was not even mentioned. The Standard Pyroxoloid plant was in nearby Leominster and the specialty of the company was in manufacturing soap boxes, and other containers and gadgets, by stamping out parts from sheet celluloid. Mr. Lane said that the incendiary case could be made very easily by the same technique and that he could supply samples in a matter of days. The cases would be called "soap boxes" in discussions with plant personnel. Prompt progress is evident from a letter to Warren Lothrop:

December 22, 1942. — "I just had a call from Mr. Lane stating that he is coming up tomorrow to bring some sample 'soap boxes.' We still have not received the pencils or we could move right up on this problem. Would you mind giving me Mr. Lovell's address and telephone number? We not only want to finish up this problem but find out about some more. His stuff is most interesting and offers a good field for E. B.'s genius."

Development of a satisfactory PI, or pocket incendiary, complete except for the time-delay element, ran into only one snag. Two halves of the case are stamped out by one operator (Fig. 6.2), cemented together in a press by another, and trimmed by a third. In first trials an 8% gel of Napalm in kerosene (a refined grade) was run in through a small hole in one end under pressure, the hole was cleaned, and a celluloid plate cemented over the hole. But the girl operators at Leominster had difficulty cleaning the gel from the outer surface in a manner satisfactory for the cementing on of the plate. We obtained samples of uncemented cases made of transparent plastic and soon found a solution of the problem. The proper amount of Napalm powder is measured with a scoop into one member of the case, the

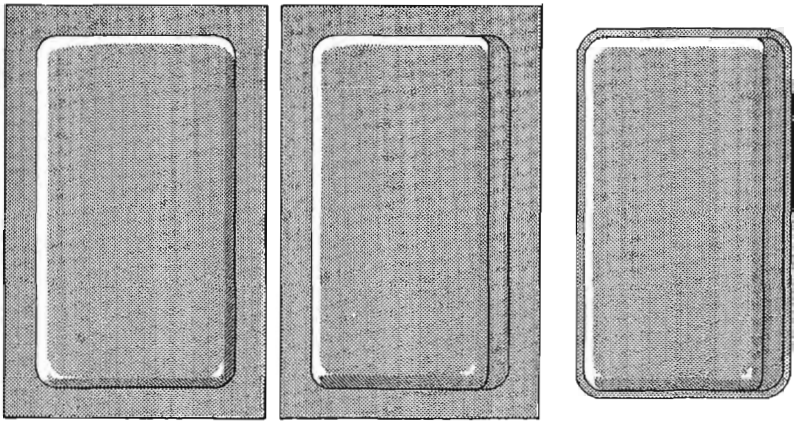


Fig. 6.2. — Construction of the Case

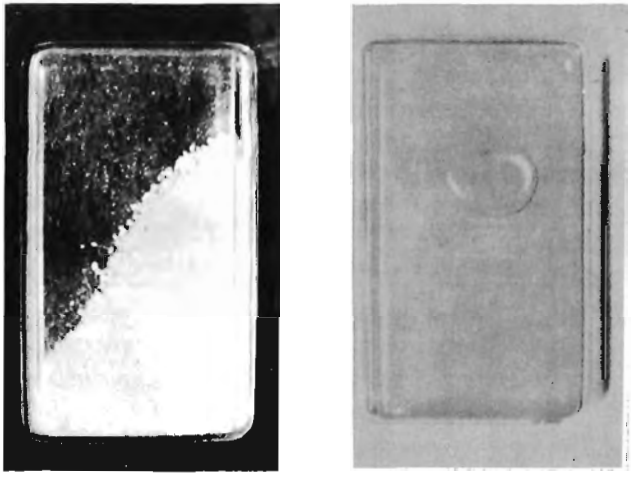


Fig. 6.3. — Before and After Addition of Kerosene

other member is cemented on and the case trimmed (Fig. 6.3), kerosene is flowed in, the sealing disc is cemented in place, and the unit is placed in a flat position for an hour or two. Experiments with the transparent cases showed that a fully uniform gel results, and this simple method of filling proved satisfactory for the eventual manufacture of millions of units. The size of the air bubble in the photograph of the filled case shows that the outage is very small.

The problem of modifying the time-delay element was much more difficult, but E. B. Hershberg eventually found a solution. The original firing device had a percussion cap at the end of a brass tube which when hit by a firing pin goes off with a loud report and will set off a blasting cap or a Bickford safety fuze that has been pushed into a spring snout. Hershberg first developed a satisfactory unit utilizing the pencil in unmodified form, except for the omission of the spring snout, in which the explosion of the percussion cap ignites a combination of black powder and commercial match mixture. However, the loud report made by the detonator was an obviously serious disadvantage, and so work continued. Hershberg's next idea was to provide the striker with a pinpoint which would pierce the light cover of a celluloid cylinder containing the head of a strike-anywhere, or allway, match.[1] Other adjustments had to be made, but the successful design shown

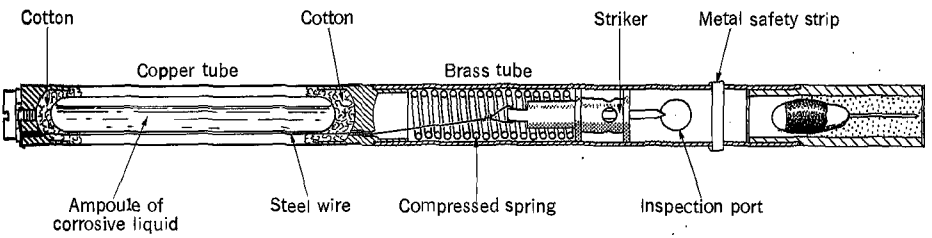


Fig. 6.4. — Time-Delay Pencil

in Fig. 6.4 eventually emerged. The match head igniter, described in more detail below, fits into a brass tube containing a spring which is held in compression by a steel wire connecting the striker to a brass screw at the end of a copper-tube section of the pencil. The copper tube is closed at the end

[1] "The idea of the igniter came from an old trick that Bob Coburn used to play and with which many a child has burned the family homestead. If a strike-anywhere match is thrown against a wall it will ignite if it hits squarely head-on. The game is to see how many one can throw in succession without a miss. If, now, the match were held stationary and a pin driven into the head, we reasoned that it would ignite equally well. The experimental combination of a dressmaker's pin and the common match showed a reliability that was astounding: several hundred successive firings without a failure." — E. B. Hershberg

facing the spring, but the end has a hole to accommodate the steel wire. A glass vial containing a solution corrosive to steel is placed in the copper tube alongside the steel wire and between plugs of cotton at each end. The brass tube at the right has an inspection port for determination if premature firing has occurred, and also a colored metal safety strip. The color marked on the safety strip denotes the length of the time delay, which ranges from 10 minutes (black) to 6½ hours. The copper tube of the pencil is of such thickness that the tube can be compressed by a strong squeeze with the thumb and fingers. This squeeze crushes the glass ampoule and the liquid is absorbed by the cotton and starts corroding the steel wire. The safety strip is withdrawn and the pencil is ready to operate. When the steel wire is eaten away, the spring is released and drives the striker down the tube and the pinpoint impinges on the matchhead and starts ignition. Note that the striker is provided with two guiding rings; these fit snugly enough into the metal sleeve to ensure centering of the pin even at a point where the striker has partly emerged from the sleeve. Without these sleeves, inspection of failures showed that the pin had been driven in off center and missed the match head. Gases are formed when the pin hits the match head, and the pressure if not relieved would blow the celluloid igniter out of the brass tube. Hence, for pressure release, the striker is provided with a hollow core and four blowout holes.

Details of the construction of the match igniter assembly are shown in Fig. 6.5. A commercial match head is cut off and mounted on a steel pin to provide for conductance of heat through the powdered potassium chlorate, and pin and match stick are coated with a booster paste of celluloid cement. The pin is then cut off and the unit is inserted in the celluloid tube, and the space below the match is packed with potassium chlorate, tamped in by hand pressure. Celluloid plates cemented at each end of the tube render the igniter unit waterproof and complete the assembly.

The casing had been provided with a recess to accommodate the pencil, which was held in place by a cemented-on band of celluloid. The completed pocket incendiary (Fig. 6.6), or PI, performed beautifully, and there were very few failures when igniters for initial tests and demonstrations were turned out by a production crew consisting of three Ph.D. chemists under Hershberg's supervision. It was easy to demonstrate significant characteristics of the PI (Fig. 6.7): strength, high heat content, and ability to burn while floating on water. The unit fires satisfactorily after immersion in water for days. Ability to start fires was evident from demonstrations such as those shown in Figs. 6.8 and 6.9. The "packing cases" shown were actually boxes used for shipment of M–47 bombs. Other trials included

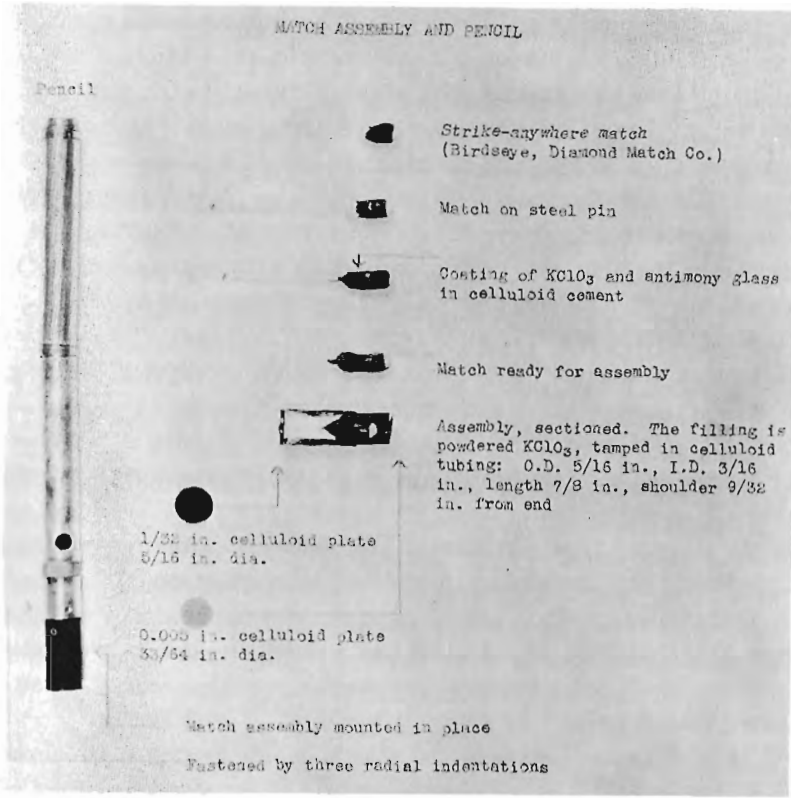


Fig. 6.5. — Igniter and Pencil

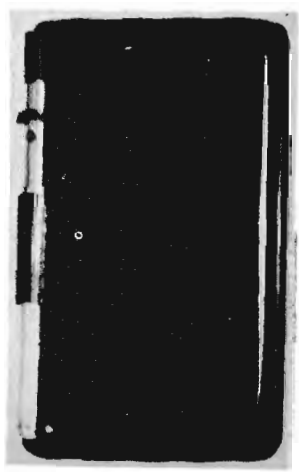
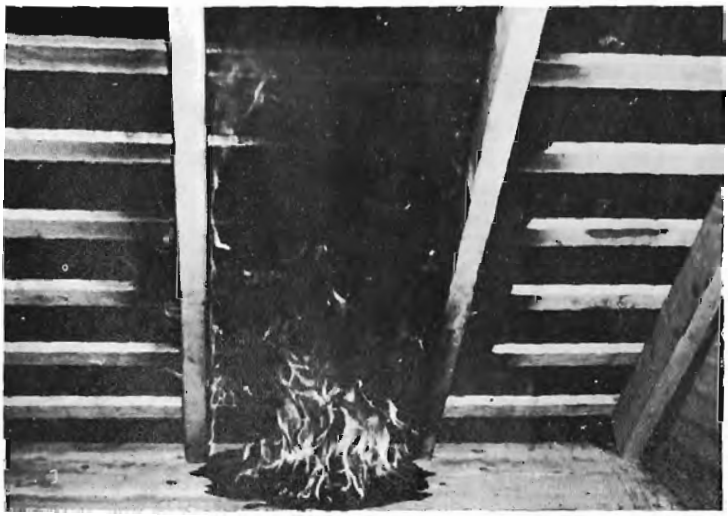


Fig. 6.6. — Pocket Incendiary

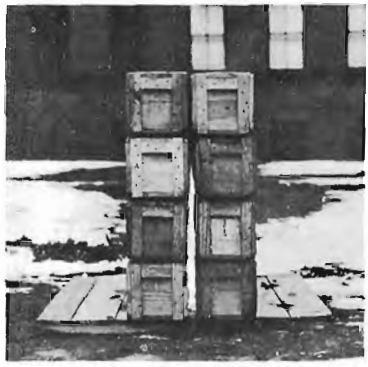
Fig. 6.7. — Characteristics of the PI

3 minutes

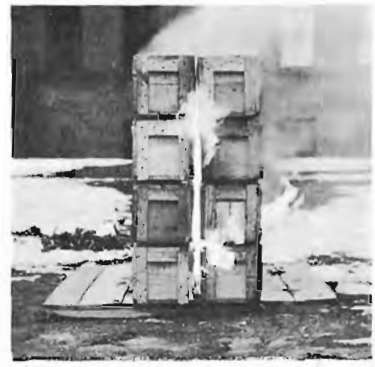


4½ minutes

Fig. 6.8. — PI in Factory Mutual Attic Structure



½ min.



3 min.

4 min.

5 min.

6 min.

Collapse

Fig. 6.9. — Sabotage of Packing Cases with a Pocket Incendiary

dropping from aircraft both regular PIs and PIs equipped with time-delay pull-fuze igniters. They withstood impact well, even on a concrete slab.

An acceptable version of the incendiary had been worked out in a matter of weeks and the PI was in production in a matter of months. Mr. Lane's Standard Pyroxoloid Corporation in Leominster could easily handle production and filling of the cases, tools were available for construction of new metal parts, and Hershberg had already consulted with Mr. A. F. of the X Match Company about production of the igniter.

OSS, being new and small, could move with much more speed than CWS, and they put more trust in their collaborators in NDRC. In the case of the PI, our group initially had the actual responsibility of writing the specifications and of lining up and controlling the manufacture; at one stage, when our laboratory work on the igniter element was just a shade ahead of the production at X Match Co., we had to authorize a change in production several weeks before the specifications could be officially amended, and in the meantime some of the army officers fairly frothed at the mouth on discovering the discrepancy. The delay was occasioned largely by a transfer of the procurement to CWS, a change that resulted in other troubles. CWS felt obliged to rewrite the specifications and introduced some Edgewood "improvements," one of which was to employ gasoline instead of the high-boiling kerosene which we had selected because of superior fuel value and low vapor pressure. Gasoline is not merely inferior, but simply cannot be used; we had filled a few of the thin-walled celluloid cases with a gasoline gel and found that they became swollen and distorted when subjected to even moderately elevated temperatures. The necessity of rectifying such mistakes by desk officers tended to divert attention from some very real problems that remained. One was to build up a 100% reliable igniter starting with a 97% reliable commercial match. The familiar strike-anywhere match made by the X Match Co. in two plants is not 100% perfect. A few fail, and a few explode. How eliminate the 3% duds? When production of igniters was undertaken by X Match Co. early in 1943, A. F., in charge of the project, made every effort to boost the performance to 100%. Matches were selected from the production lot with care, the assembly steps were checked by inspection, and sample lots of finished igniters were tested by firing them in a gig. Tests of igniters mounted in 10-minute pencils were made both at Leominster and in the Gibbs Laboratory. Soon, all groups agreed that the performance was miserable. Through Drs. Jacob Fine and Arnold M. Seligman of the Beth Israel Hospital, Hershberg and I arranged to have sample production lots X-rayed, with the results illustrated in Fig. 6.10. The 176-igniters in each group were then fired in pencils and

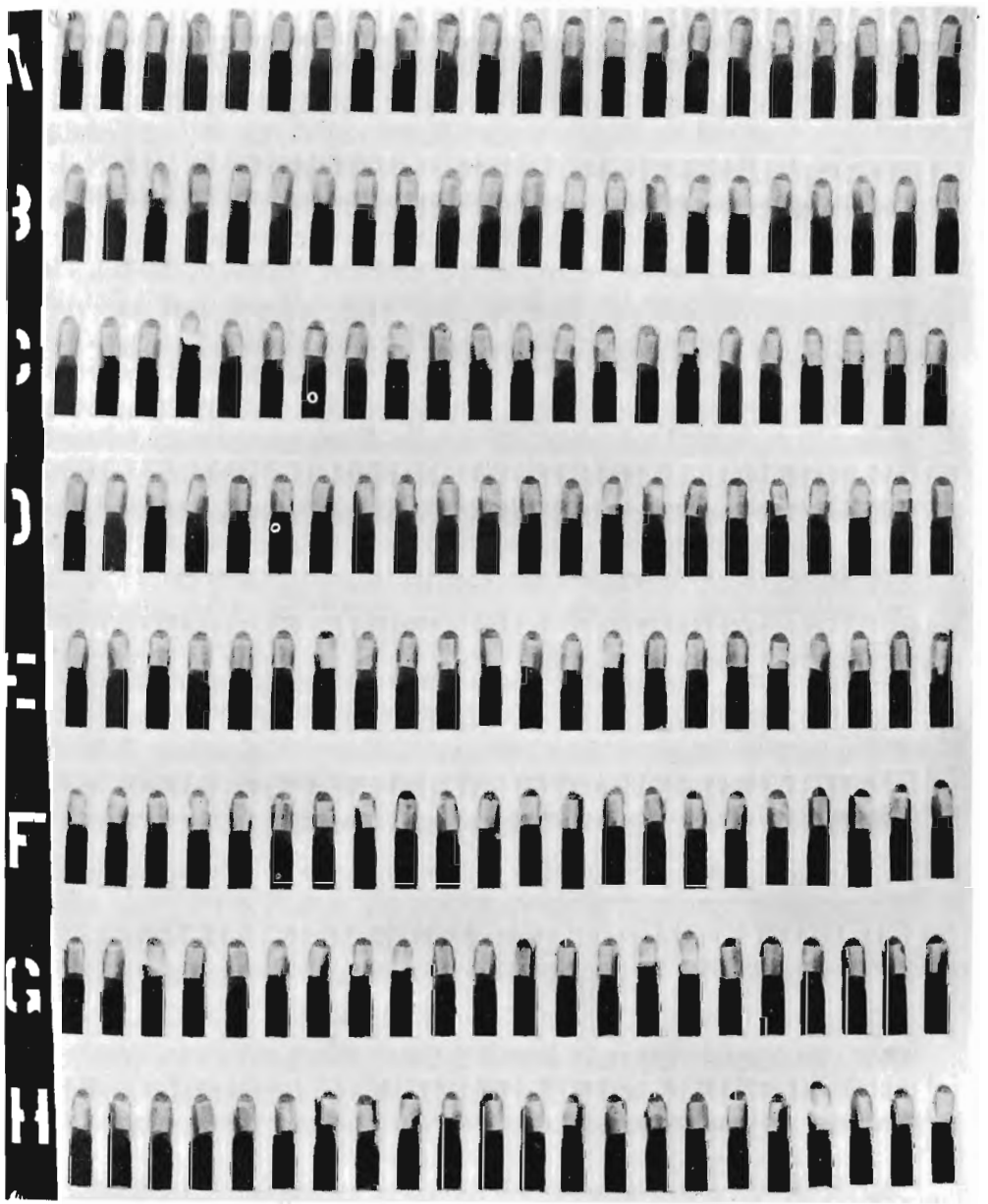


Fig. 6.10. — X-ray Inspection of Igniters

each igniter in the photograph given a score: normal (N), explosion (X), or misfire(O). Unfortunately the only satisfactory photograph now available (Fig. 6.10) is not marked to indicate the score. This batch had come from plant A and the score was: failures, 10.6%. A second batch X-rayed at the same time had been made in plant B, and scored 21% failure. Careful study of the photographs and scores led to better operation in plant A and to some improvement in plant B. But the performance of the igniter was still short of that of the match, and way below the goal set.

Then Lane of Standard Pyroxoloid pulled a rabbit out of the hat. He suspected that the fault lay not in the igniter but in the construction of the pinpoint of the striker; the angle should be less sharp and the surface roughened. A. C. Gilbert Co., makers of the pencil, supplied a trial batch of strikers with the angle changed from 60° to 45° and with the striking surface Parkerized. They worked beautifully. Everyone was much relieved, and on switching to the modified striker performance went up very close to 100%. But by this time 400,000 PIs had been manufactured. Some had been used in tests and a few had gone out to operators (one had taken a Japanese ship). But when various testing groups reported failures averaging 9%, distribution was stopped and the bulk of the 400,000 units impounded. This degree of misfunction could not be tolerated in a munition of such a nature that an operator on going out on a mission almost invariably risks his life. Thus salvaging of the PIs with the faulty igniters became a major problem for all participating groups.

Another major problem which had arisen was that of moisture, as expressed in a letter to Dr. Lothrop.

> July 31, 1943. — "Our report of February had a good bit of sales talk in it. In our ignorance of plastics we assumed that, if water would not go through celluloid, water vapor would be retained. This certainly is not the case . . ."

The British had ordered a quantity of PIs but had specified that they must pass a murderous cyclic humidity test in which units are placed in a humidifier at 150° F. and 90% humidity. After a certain period of time, the units are removed and let stand at room conditions for another period. The process is then repeated. Tests were conducted first by a group at the Maryland Research Laboratory in Washington, D.C., headed by Dr. Kenneth S. Pitzer, now president of Rice University. Later a Columbia testing group was set up under Professor Colin C. Fink and Dr. Henry B. Linford. In the Linford humidifier the test was made more severe than called for by the British specifications by circulating the air. The best PIs at

hand failed miserably in even the milder version of the British test. The groups at Plant A, Leominster, and Cambridge first investigated all available lacquers for protection, particularly for protection of the window of thin celluloid over the match head. Some were fair to good, but even with the best lacquer and with a thicker window the requirements of the British test could not be met. Someone thought of electroplating, and a cooperative pair of workers (Eddie and Bud) at Commonwealth Plastics in Leominster made several batches of samples. A celluloid igniter is given a lacquer dip, surfaced with graphite, and electroplated. The first copper-plated igniters may have been moisture proof, but when fired they failed to ignite the PI case. Apparently the copper shell remained intact and the fire all issued through the hole made by the striker pin. Lighter coatings of copper and of silver failed to provide adequate protection from moisture. A. F. at X Match Co. initiated an attempt to develop an igniter made of magnesium rather than of celluloid, but progress was slow and time was pressing.

By midsummer of 1943 lacquered units were available which did not fall very far short of meeting the British test and many of us felt that the best solution of the problem was to abandon this requirement for U.S. units and to try to persuade the British to change the specifications. Lt. Paul W. Sampson of OSS reported the results of a survey showing the worst possible worldwide conditions of temperature and humidity. Tests were set up in which 100 pencils were exposed to each of three sets of conditions, recycled, and fired on removal from the humidity cabinet. The results

Humidity Tests (Sept. 24, 1943)

Conditions of	Temperature	Humidity	Failures
Guadalcanal	100° F.	90%	1%
Tobruk	135° F.	30%	1%
Kiska	−40° F.	100%	3%

shown in the table seemed to most of us very good, but the British requirement was still operative.

An eventual solution of the humidity problem followed a line suggested by Professor James L. Crenshaw, with whom I had served on the staff at Bryn Mawr College, and whom I had persuaded to spend the summer working with my group. Crenshaw had suggested coating the igniter with a layer of wax by dipping. Dewey and Almy Co., experienced in thermoplastic waxes, supplied a first sample which when applied to igniters seemed promising. So 100 igniters were dipped, fitted into pencils, and dispatched

to Columbia for submission to the British test at 150° F. and 90% humidity. The score: 88% functioned normally. This Wax I seemed a little too brittle, and experiments were started on two modified waxes.

OSS had considered packaging the PIs in moisture-proof containers and there had been some talk of using tin cans, if procurable, or possibly wax-dipped boxes. The promising results obtained with waxed-dipped igniters led me to explore the possible use of a waxed-dipped cardboard carton. From discussions with box manufacturers in Boston and in Leominster it became evident that the designing of a carton that can be glued 100% tight and that is suitable for the waxing operation is quite a specialty and that the Robert Gair Co. in Piermont N.Y., was generally regarded as leader in this kind of design. I visited the Gair Co. and found the personnel very able and very cooperative. They explained some of the intricacies of box construction and the significance of what would appear to the novice to be trifles. They understood the problem, took the specifications for the size of a box to hold two units, and agreed to design a carton and submit samples soon. In a matter of a few days the samples arrived. We glued them up according to advice from Gair, loaded them with PIs and with igniter-pencil units. After application of two improved Dewey and Almy waxes, the units went off to be tested. It was a pleasure, on October 1, 1943, to write the report: "Igniter Humidity Tests, Memorandum No. 6" and present the following data:

RESULTS OF SERIES XVIII: British Test, 150°, 90%

a. 53 Pencils, igniters treated with Wax II
   92.5% fired normally
b. 98 Pencils, igniters treated with Wax III
   95% fired normally
c. 99 Pencils with treated igniters sealed into wax-dipped cardboard cartons
   98% fired normally
d. 3 Pairs of PI units with igniters treated with Wax III sealed in wax-dipped cartons (total of 6 PIs)
   100% fired normally

If a 95% perfect unit were to be packed in a 98% perfect carton, the expected performance in the British test would be 99.9%. The supply of boxes on hand permitted trial of only six units but the score of 100% matched expectations.

The waxed-dipped cartons provided a simple solution to the problem of salvaging the 400,000 PIs which showed 9% failure: wax-dip the igniters, cement two units together, place the double unit in a carton, and wax-dip

the box. After activation of both pencils, the expected performance would be 99.82%. Or the score could be raised by cementing each old PI to a new one.

Two incidents of the PI work may be worth recording. At a time when humidity tests were being done by the Pitzer group in Washington, a batch of PIs shipped to Washington from Leominster took fire in the South Station, Boston, and destroyed a certain amount of property in the baggage room. A charred remnant of the package carried my name, and I was on the spot. However, on receipt of a telephone call from Washington asking for an account of the incident, I replied that an accident had occurred in the course of work on a secret project, the nature of which could not be disclosed. There were no further questions.

Early in the PI-work (April, 1943) I was pleased to comply with a request to submit samples for testing at the Mountain and Winter Warfare Board, Camp Hale, Colorado. A report by this Board dated June 28 was somehow delayed in transit to me, but I acknowledged receipt of the document in the following letter to Dr. Lothrop:

> September 14, 1943. — "I am writing to comment on a report from the Mountain and Winter Warfare Board. One comment is that June 28 is so far in the past that perhaps few people are interested in the matter. The Board concludes that the PIs are ineffective in setting fires under mountain and winter conditions and suggest that a thermite incendiary would be more effective. All I can say is that I am dead sure that the Board is wrong. I do not believe that the Colorado officers know how to start fires, and think they fail to appreciate that the fundamental principle is to pile your logs properly. We spent some little time making careful comparative tests of different types of units, and I challenge anyone to show that there is anything wrong with the procedures used or the conclusions reached. I also would like to challenge anyone in the State of Colorado to a fire-starting contest."

7. *The 500-Pound Bomb*

On APRIL 30, 1943 I attended a second series of dropping tests at Edgewood Arsenal on the E-19 bomb (E for Experimental), then known also as the "Harvard–Factory-Mutual" penetration bomb. This project, under development by E. B. Hershberg, Norman F. Thompson, and others will be described later, but I may note that my diary records the following commentary on the tests: "Victory!" In the course of the day there was

Fig. 7.1. — Second Test of the E–19 Bomb [1]

considerable discussion of a new requirement of the Army Air Force for a 500-lb. incendiary bomb of penetration power superior to that of any existing bomb. A structure built at Edgewood on H-field for testing pene-

[1] The author's identification is a cane, used as travelling companion and not for support. On many trips I taped the tip of a fishing rod on the cane and put butt and reel in my suitcase, just in case an opportunity should arise.

tration had a roof area of 200 x 200 ft. and was two stories high. The roof was quartered into sections of different construction: reinforced concrete, concrete tile, metal sawtooth, and another one not named in my records. Witnessing tests of M-47s on this structure had been exciting, for the best method of observation was to crouch in a 5-ft. cellar under the very heavy first floor until a bomb had hit either the structure or the ground nearby and then rush out to see what had happened.

The M-47 penetrated the toughest target, the 6-in. reinforced concrete roof, probably from the combined force of impact and explosion. The Air Force wanted a bomb that would penetrate the roof and also secondary obstructions before functioning. A steel bomb casing of minimum wall thickness of 0.3 in. was in procurement and would be tried out with various fillings. Napalm might not be strong enough to withstand the burst of an explosive charge adequate for opening the very heavy casing. The CWS had heard that Permanente Metals Corporation in San Jose, California was manufacturing magnesium by a process that produces crude magnesium in so finely divided a form that it is pyrophoric and has to be wetted down immediately with a high-boiling petroleum fraction to permit processing in the next operation. The intermediate pasty magnesium-oil mixture known as goop might offer possibilities as an incendiary. General Kabrich asked me to serve as CWS representative on a visit to Permanente to see if the goop offered any promise as a filling for the 500-lb. bomb. I was already scheduled for a trip to Los Angeles two weeks later for work on the Adams Plan, but the General thought this too much of a delay and so I made a first journey to California on May 3, returned on May 6 for a demonstration of the E-19 bomb to CWS, AAF, and British Air Commission officers ("very successful tests in Thompson's bedroom structure"), and flew out to California again on May 16. In those days air travel was sometimes delayed by appearance of a passenger with a higher priority, and in any case several relays were required on a transcontinental flight. I arrived in San Francisco late, tired, and sticky, and when told at the St. Francis that the room reserved for me had no bath, I blew up and appealed to the manager. He did nothing, of course, and on returning to the desk I found that my room had been given to a more considerate traveller. I spent the night on a cot in the Turkish bath.

Permanente engineers T. A. Dungan [2] and F. Van Loenen were much interested in possible use of goop as an incendiary, and together we made crude burning tests. I decided to report the opinion that goop offered some

[2] Theodore A. Dungan, b. 1909 Berkeley, Calif.; A.B. California, 32; Gay Eng. Corp.; Permanente Metals Corp.; Food Machinery and Chem. Corp.; Bechtel Corp.

promise and to recommend that the Permanente group be provided with a supply of M-47 bomb cases and bursters. We agreed on a series of formulations of goop with which these would be loaded and readied for trials to be conducted on completion of my mission on the Adams Plan. These trials came off in the period May 30–June 3, 1943 in a quarry above San Jose and pointed the way to further development. Dungan and Van Loenen took over from that point and I was merely an interested observer of tests at Edgewood on June 28 with Permanente goop-filled M-47s and August 24 on goop-filled 500-lb. bombs. Permanente was one of the Kaiser enterprises, and Henry J. Kaiser attended the second tests. Eventually the 500-lb. bomb filled with goop and fired with a Hershberg-type phosphorus–TNT burster was accepted and standardized (M-76), and the entire plant production of 50–60 tons per day was diverted from ingot magnesium to goop filling for the 500-pounders. Ted Dungan served (1943–1945) first as consultant to CWS and then with the British Petroleum Warfare Division in England.

At one of the many conferences E. B. Hershberg suggested the possible use of cast iron for construction of the 500-lb. bomb. Why present the enemy with 270 lbs. of high grade steel on each drop? A cast iron bomb would require only a very light explosive charge to open the casing and hence a lighter, stickier gel could be used. Opinions varied as to whether or not such a bomb would penetrate a 6-in. factory roof and operate inside the structure. The only way to tell was by experiment, and everyone consulted in CWS and NDRC thought the experiment interesting and worth while trying. Our group was to procure 25 bombs and parts (on an order from Harvard University) and to make plans for assembly and filling of the bombs at Edgewood with Capt. Wiley W. Carr. The United States Pipe and Foundry Co. agreed to do the casting and Chief Engineer Edward Hering, of the general office in Burlington, N.J., sat down with Hershberg to study drawings of the steel 500-lb. bomb and work out a modified design applicable to cast iron. Thus the thickness of the casing was increased from 0.3 to 0.5 in., and that of the nose from 1¼ to 1½ in. A different scheme had to be used for suspension of the bomb in a bomb bay. U. S. Pipe worked fast and the bombs were soon cast in their Chattanooga, Tenn. plant (the cost, estimated on a production of 10,000 bombs, was $58 per unit). Mr. Hering made arrangements with the Grinnel Co. in Providence, R.I. to fabricate bursters and load them with phosphorus. Parts were shipped to Edgewood, where Capt. Carr had made arrangements for filling and mounting, and tests were carried out at Edgewood over a period of about a month. I had obtained invitations to the tests for Mr. Hering and two associates of U.S. Pipe and advised Mr. Hering as follows:

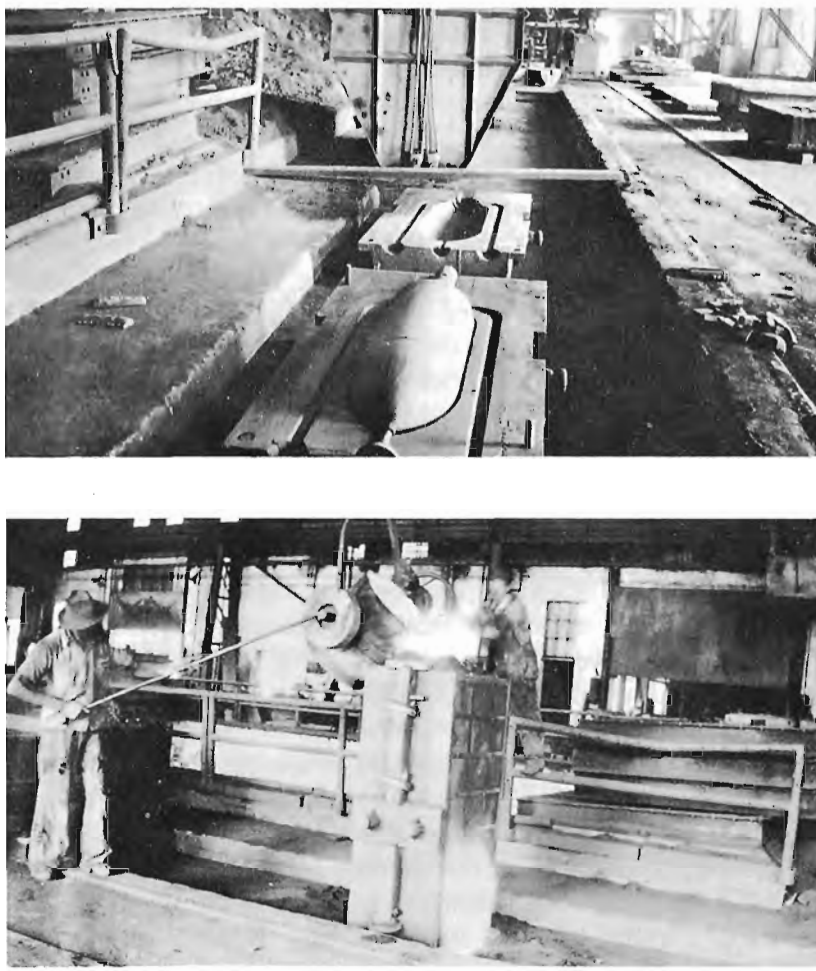


Fig. 7.2. — Cast Iron Bomb in Production

Oct. 8, 1943. "I usually take the Federal Express arriving in Baltimore at about 7:15 a.m., have breakfast at the station, and rent a Hertz car for the 20 mile drive to Edgewood. I would be glad to have you and your companions go along."

I might have added that I invariably stayed at the Hotel Lord Baltimore and dined in style, however late, at Miller Brothers. Baltimore proved to be a friendly stopover point. The Lord Baltimore bartender, who could perform neat tricks of magic, once offered me two tickets for the night

baseball game, received from a customer who had been called out of town, along with a blind date. After a trip on which I had spent a pleasant evening bowling with Art Cope,[3] I dictated the expenses and jokingly added "bowling, 90 cents." The German-born secretary took the remark seriously and the expense account was returned with a polite note disallowing the entertainment item.

Static tests were carried out first to determine the proper charge of TNT-tetryl. Hershberg had prepared pelleted charges of varying diameter and his estimates had been so accurate that a proper balance was found with expenditure of only a few bombs. The filling was a 13.5% Napalm gel. Then came the bombing tests on the H-field structure, which required several days. Only two of the four quarters of the structure were intact enough to present satisfactory, if small targets, and only one bomb could be fitted into the modified bomb bay at a time. However, enough satisfactory hits were obtained to show conclusively that the bomb did have the desired power of penetration and that it functioned satisfactorily. For example, one bomb dropped from 4,000 ft. crashed through an upper and lower concrete girder, severing all reinforcing rods, and fired on the main concrete floor. Distribution and ignition were satisfactory, with numerous good sized fires (the structure contained no combustibles).

After the last test we all went back to Building 330 to review the results. Everyone was happy about the performance and we started to discuss the next move. But one of the officers then recalled that the Navy had a special safety requirement on all munitions. Reference to the Navy manual indeed showed that a bomb must withstand the shock of impact on free fall from 20 ft. onto concrete. No one, least of all the U. S. Pipe engineers, thought there was much chance that the cast iron bomb would take this punishment, but in due course bombs filled with sand and water to the appropriate weight were dropped from heights of 20 ft. and then 10 ft. They smashed up. That is the end of the story.

[3] Arthur C. Cope, b. 1909 Dunreith, Ind.; B.S. Butler College, 29; D.Sc. Wisconsin (Adkins), 32; Bryn Mawr College; Columbia Univ.; Massachusetts Institute of Technology; President, American Chemical Society, 1961.

8. *The City Slicker and the Paul Revere*

NAVAL VESSELS and many other ships are fueled with a heavy, thick oil which is transported in tankers, stored in tanks close to the water edge, and carried in the bulkheads of ships. There was an occasion, early in the war, when a considerable part of the Italian fleet was caught in an Adriatic Harbor flooded with oil through bombing by British aircraft. Attempts to ignite the oil with all available bombs and flares were unsuccessful, and a rare opportunity was missed. Oil on water burns with a viscious flame to produce a wall of fire rising to a height of 40–50 ft. The Office of Strategic Services figured that partisan groups could arrange for release of oil around enemy ships in a harbor, and set up a requirement for a device with which to ignite oil slicks.

Our group had talked over this problem but had thought of no solution. On my first visit to the Permanente plant at San Jose, Dungan and Van Loenen had demonstrated ignition of the magnesium–oil goop in contact with water. In the warm atmosphere of California in the spring, and with partially dried-out goop, prompt ignition took place every time. A goop fire is very hot. Thus here was a clue to a possible solution of the oil slick problem. I explained the problem to Dungan and Van Loenen and they agreed to make further trials. On my return trip to California a few weeks later Dungan and Van Loenen came down to Los Angeles to show me color movies of igniters made up in tin cans, lily cups, and paper bags. The Permanente engineers did not know much about what constitutes a practical munition, but they were highly skilled in manipulating a hazardous material, and they developed and shipped to us by air a succession of batches of dried goop containing an amount of oil adjusted for suitable ignition. The code for identifying this material by telegram was "Slick Mix." For air express shipment in batches running to 100 lbs., Slick Mix was packaged in hermetically sealed tin cans. During the summer of 1943, Hershberg, Crenshaw, and I filled a variety of containers and tried them out on the Charles River (in between passage of shells and wherrys) but with little success. However, by September we had hit upon a model which offered some promise. A 1¼ x 12-inch celluloid tube was first fitted with

a liner of corrugated cardboard and then loaded with goop. Celluloid plates cemented on each end had holes which were closed with a sugar syrup to give a delay of 4–5 minutes. The ends were then sealed with a supposedly moisture-proof paper easily removed with a draw string. One idea was to make the cylinder of such a size that it would fit in the end of the bamboo pole used, we understood, by all Chinese stevedores in loading and unloading Japanese ships.

A little later we learned from work on the Pocket Incendiary that celluloid is not proof against moisture and became aware of the necessity for absolute protection of the highly sensitive filling against moisture. And we had learned something about boxes and about wax. So we removed the moisture-containing sugar syrup, replaced the paper seal with a metal slip cap, and dipped the whole unit in wax (Fig. 8.1). For construction of a second model, we called on Gair Box Company for the design and construction of a cardboard carton of suitable size (Fig. 8.2). The bottom of the box was glued and the unit dried thoroughly before insertion of a liner of corrugated cardboard and with a sheet of asbestos paper at top and bottom, as the box floats on water, to direct flames along the water surface. A layer of Permanente Slick Mix was introduced, followed by a filter-paper thimble containing absorbent cotton, which was slid in until the mouth was next to a wedge-shaped marking on one narrow side with the arrowed instructions to CUT and to PULL UP. With this scheme the operator could form a tear tab with a knife, pull it to open the water port, and throw the box into water. The box was then glued shut, let dry for maximum escape of water from the glue externally, rather than to have it sealed in with eventual deterioration of the magnesium, and wax dipped.

The two units were tried out on the Potomac on November 3, 1943 in tests conducted at the Navy Explosives Investigation Laboratory at Stump Neck, Md. Those present included Lt. L. E. Allison and other Navy personnel, Chadwell, Crenshaw, and myself. The directive called for use of Bunker C oil, the standard fuel of the U.S. Navy, but none was available. Lubricating oil (SAE 50) was tried, but at a water temperature of 11° C. proved to present too difficult a target. The only fuel available was Fuel Oil No. 2, which is a little too easily ignited for a satisfactory test. We decided to run the tests with the lighter oil and schedule a later test with Bunker C oil if the results were favorable. The results may be described with quotations from Lt. Allison's report. In a trial on a slick of about 80 sq. ft. formed by 10 gal. of No. 2 Fuel Oil, a box-type igniter

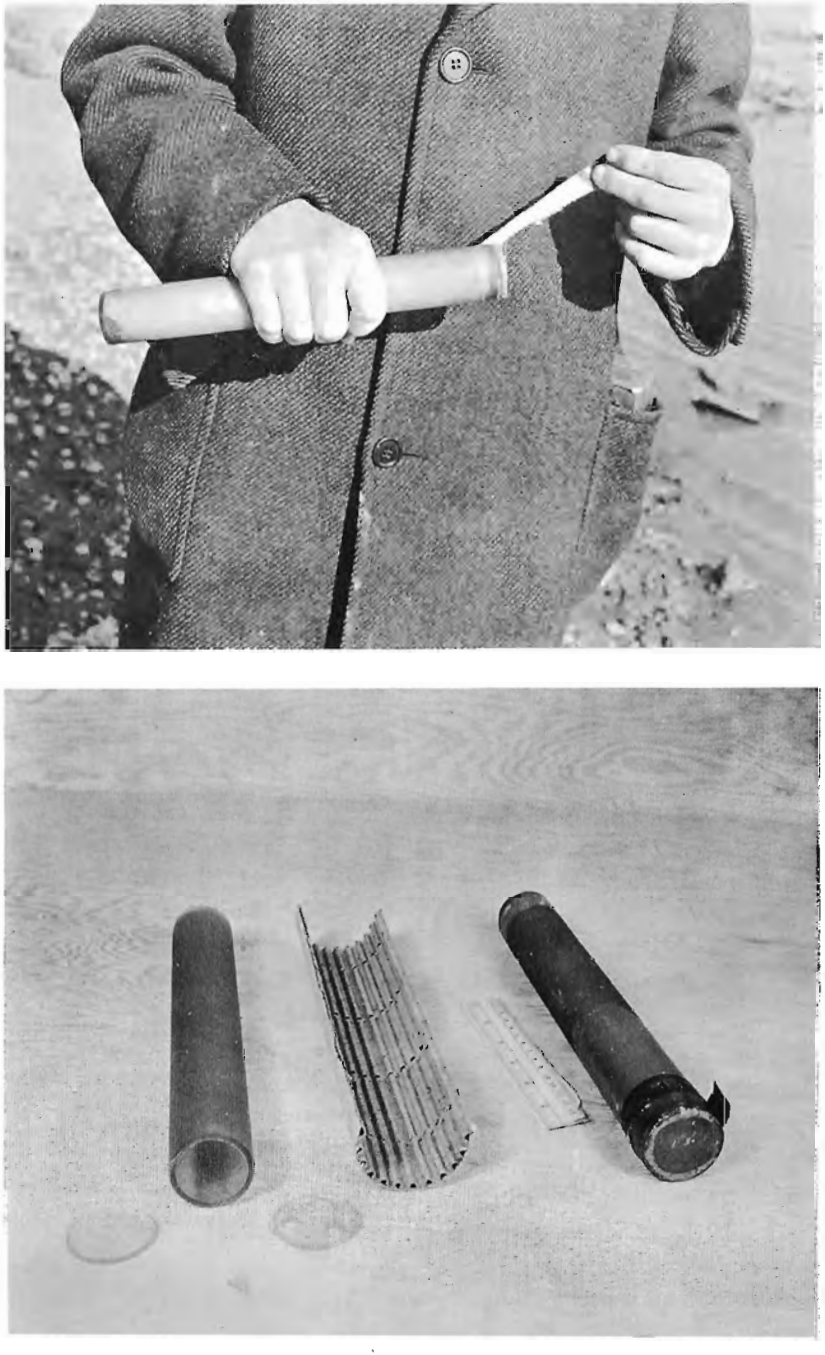


Fig. 8.1. — Candle Oil Slick Igniter

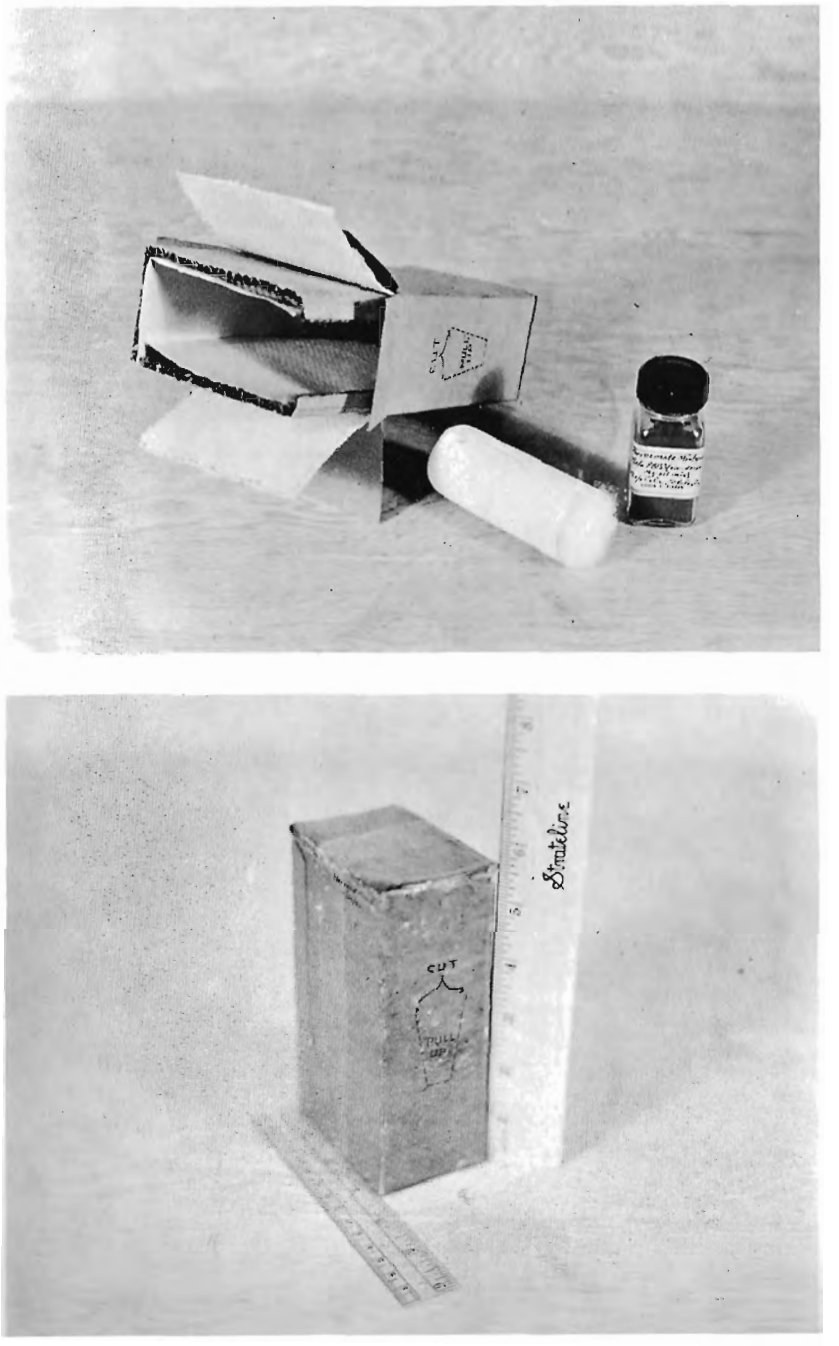


Fig. 8.2. — Box Oil Slick Igniter

"operated extremely well and ignited the oil very well. The flames attained a height of approximately 15 ft. and burned violently for 10 minutes. The water temperature before ignition was 11° C. and directly following the burn-out of the oil was 40° C."

The candle-type igniters proved unsatisfactory. They ignited properly but consistently failed to set fire to the oil. The cylinder floated at an angle of about 25° to the water surface and the flame was thus directed upwards. Furthermore the candle seemed inclined to scurry about in the oil, whereas the box stayed in place and produced a more concentrated attack. The candle was improved but eventually abandoned. Everyone liked the box-igniter, for which Stanley Lovell of OSS suggested the code name CITY SLICKER. Since the next test would be with the heavy Bunker C oil and in water colder than 11° C., we thought it well to anticipate trouble and try to improve the functioning of the gadget. What about warming up the water as it entered the box? Why not do this with calcium carbide? This chemical reacts with water at 0° with liberation of heat and generation of the combustible gas acetylene. We tried the scheme and it worked beautifully. Three City Slickers equipped with calcium carbide bags adjacent to the entry port were tried out with slicks made by pouring a 10-gal. can of Bunker C oil into a 10 x 10 ft. confining frame on a small flowing stream near Washington at a freezing temperature (January 11, 1944). Each one ignited promptly and set fire to the oil. There were many more tests and some further improvements. Most notable was a modification of the City Slicker which made it a two-purpose munition operable by land or by sea. For this I suggested the code name PAUL REVERE, which soon became official.

I will skip a whole succession of ups and downs in experimentation and in numerous NDRC and Service tests and conferences and turn to my 59-page final report of Sept. 27, 1944, for this describes the version of Paul Revere that was put into production. The construction and mode of action of the oil slick-igniting element will be evident from the drawing at the upper left in Fig. 8.3. Use of a brass tear tab facilitates exposure of the entry port when the unit is to be activated by contact with water. The carton is cut from the best quality jute paper board as used by the Quartermaster Corps for the K-ration, and it is double-coated inside and out with a Vinylite lacquer that adds to strength and moisture-resistance and effectively seals in oil in the fuel. After being filled, the carton is sealed with an oil- and moisture-proof adhesive and finally dipped in wax. The Paul Revere can be placed under a sprinkler for two weeks, or floated on water for the same period,

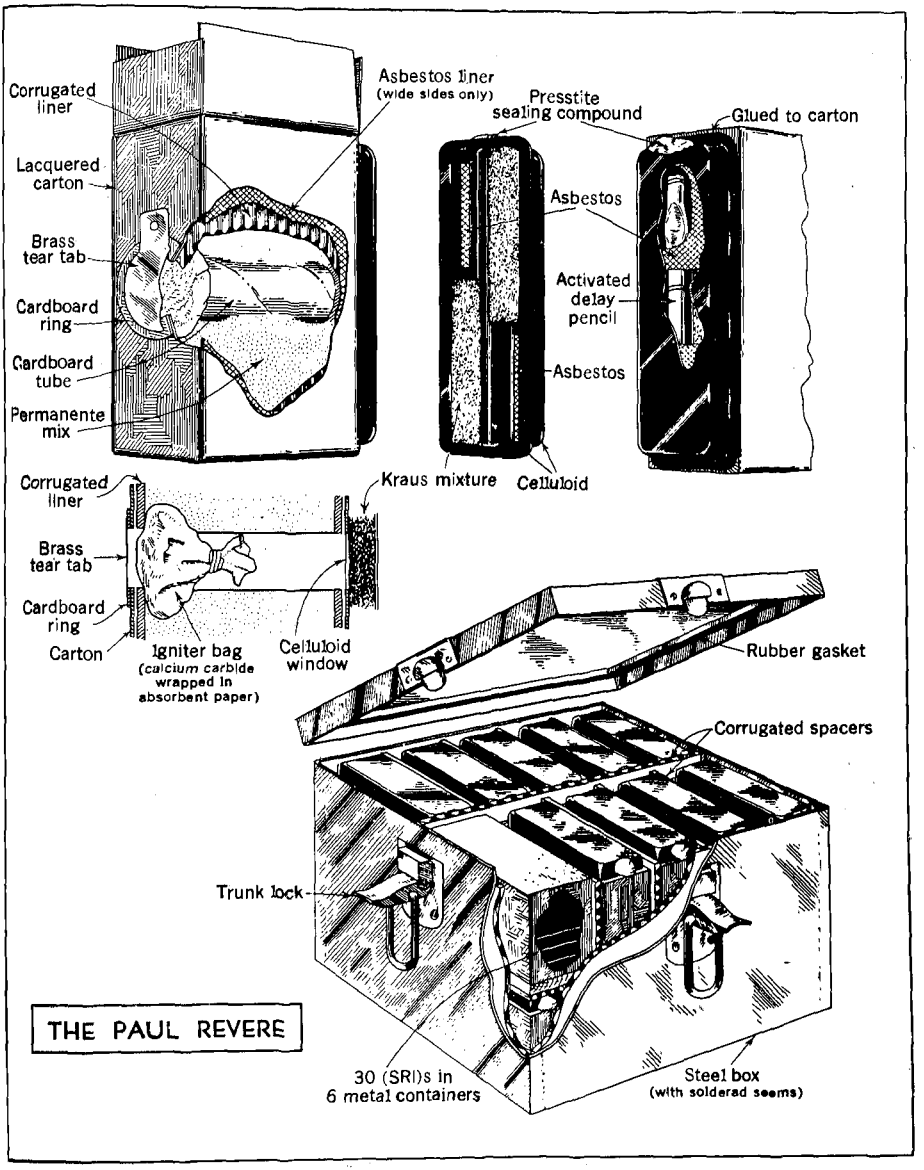


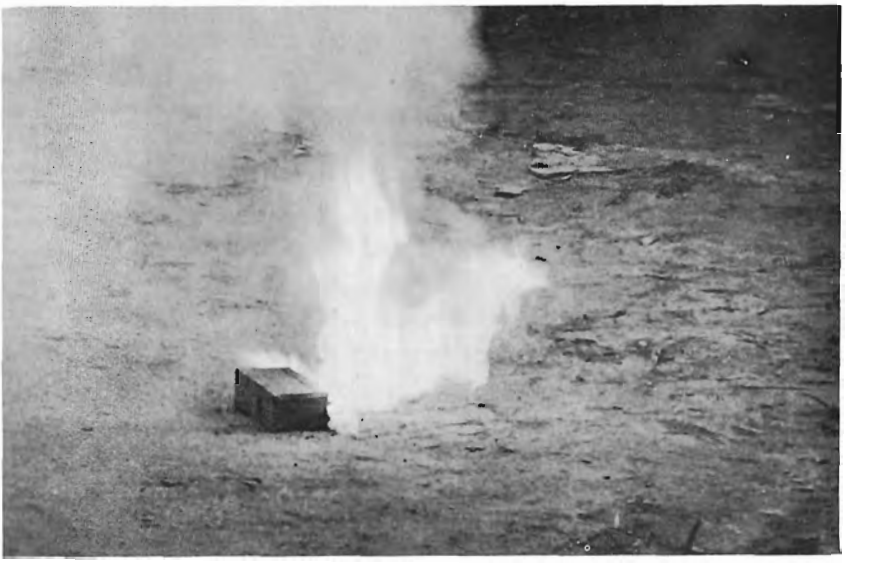
Fig. 8.3. — Construction and Packaging

and it will still ignite 15–30 seconds after ripping off the tear tab and dropping the unit into water. It also stands up on exposure to 100% humidity at 150° F. The bag containing ¼-in. pieces of calcium carbide is held in place against the potential water port by a cardboard tube through

which water can enter when the tear tab is removed. The water is warmed by flowing through the calcium carbide and easily ignites the Permanente mix of oil and magnesium at 0° C. One or the other of the asbestos liners, at the top and bottom sides as the box floats, directs the flames along the surface of the water. At the other narrow side of the box the cardboard tube abutts a 1-in. hole in the corrugated liner and so centers on a hole in the carton which is closed with a 1-in. glued-in celluloid window. The black member of the unit is a celluloid case made by Standard Pyroxoloid at Leominster. The molded material marked Kraus mixture was concocted by the NDRC group of Professor Charles A. Kraus at Brown University. A batch is made by stirring 10–11 parts of paraffin into a mixture of 88–89 parts of granular potassium perchlorate and 1 part of finely divided carbon. The mixture is heated with steam and worked mechanically until homogeneous, soft, and plastic, when it can be molded into two recesses in the plastic case, where it sets to a hard mass. The celluloid case is then glued onto the carton in the position shown. A central well in the plastic case is for insertion of an activated time-delay ignition pencil, which is the same as that cemented to the Pocket Incendiary; the Service designation is SRI. An opening at one end of the pencil well is initially sealed with Presstite, a soft, water-proof rubber gum. The Paul Revere is prepared for delayed operation by digging away enough of the rubber gum to expose the pencil well, inserting an activated and armed pencil, and sealing the hole again with the gum. The operator should be instructed to insert the igniter end first, as a precaution in case of premature firing, but the unit is made to operate if the pencil is inserted either way, since it is possible that neither instructor nor operator is able to read English. Thus, with the pencil in either position, the igniter sets fire to a block of Kraus mixture and the flame is restrained by a short asbestos liner and is caused to travel toward the middle, burn out the celluloid window, enter the cardboard cylinder, and ignite the Permanente mix. This construction is a good demonstration of Hershberg's inventiveness.

Fig. 8.3 shows a steel box designed for packaging 19 Paul Reveres and a supply of 30 ignition pencils (SRIs) covering a range of time-delay. The steel box, in itself completely moisture proof, provided extra protection to the already moisture-proof contents. I did not preserve any Paul Reveres which could be tested later, but one of the steel-box containers serves as an effective mouse-proof container for bird seed.

The time-delay pencil can be used for operation of the Paul Revere either by land or by sea. When the unit is operated with delayed ignition on water, the Kraus mixture occasionally burns out without direct ignition



Paul Revere by Land

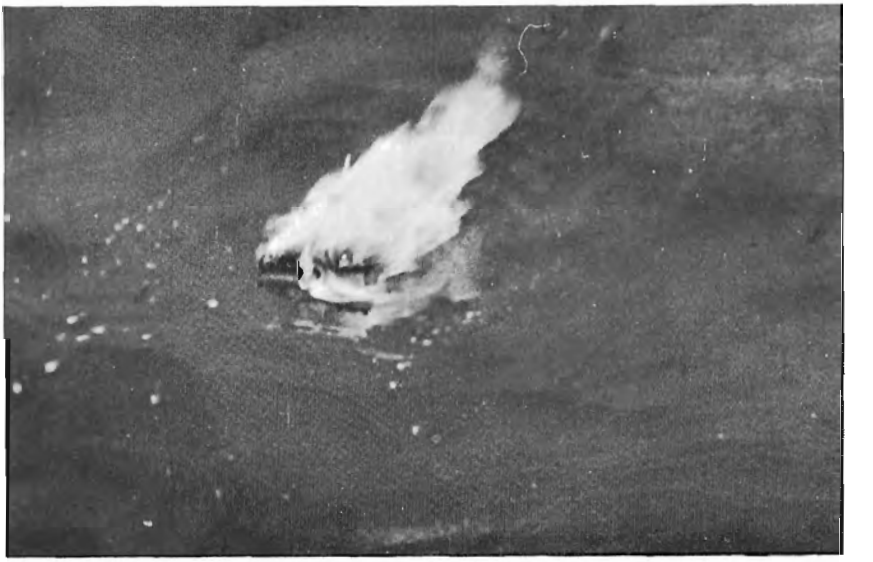


Fig. 8.4. — and by Sea

of the main fuel charge, but, even so, the celluloid window invariably is burned away with exposure of an opening for the entrance of water through the back end of the cardboard tube, with resultant ignition.

The Paul Revere (PR) and Pocket Incendiary (PI) supplement each other. The PI burns for 12–15 minutes and if properly placed will set fire to wet or painted wood or to other combustible materials which are difficult

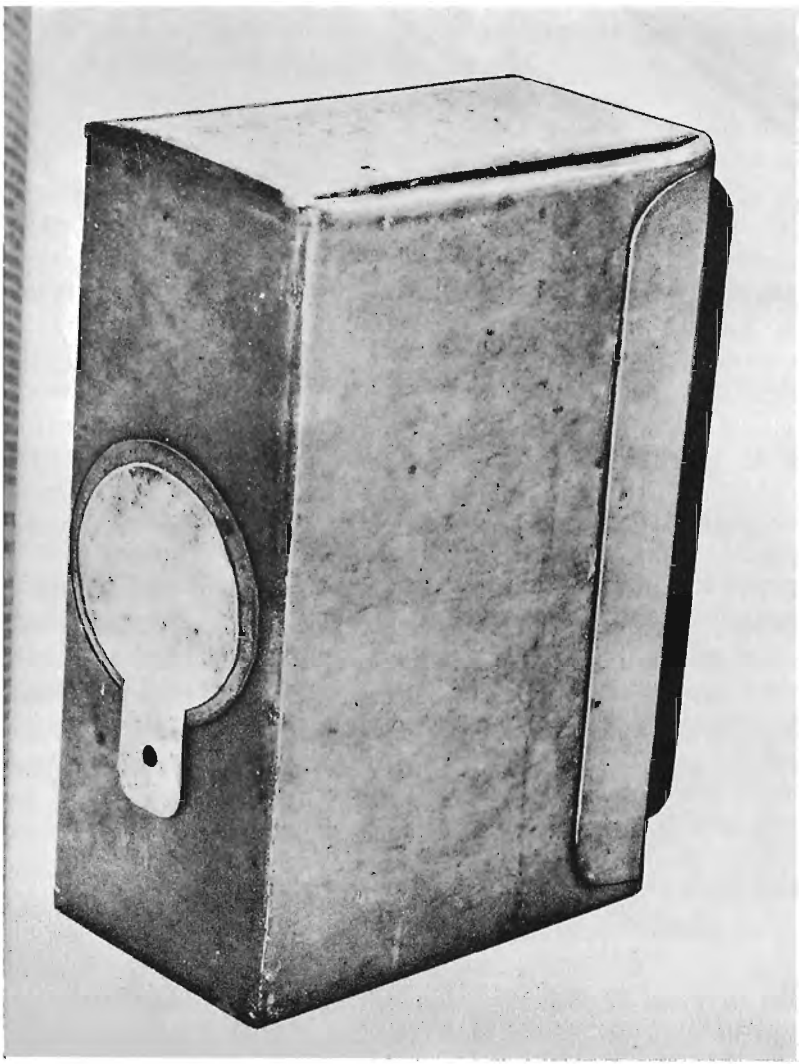


Fig. 8.5. — The Paul Revere (wax-dipped)

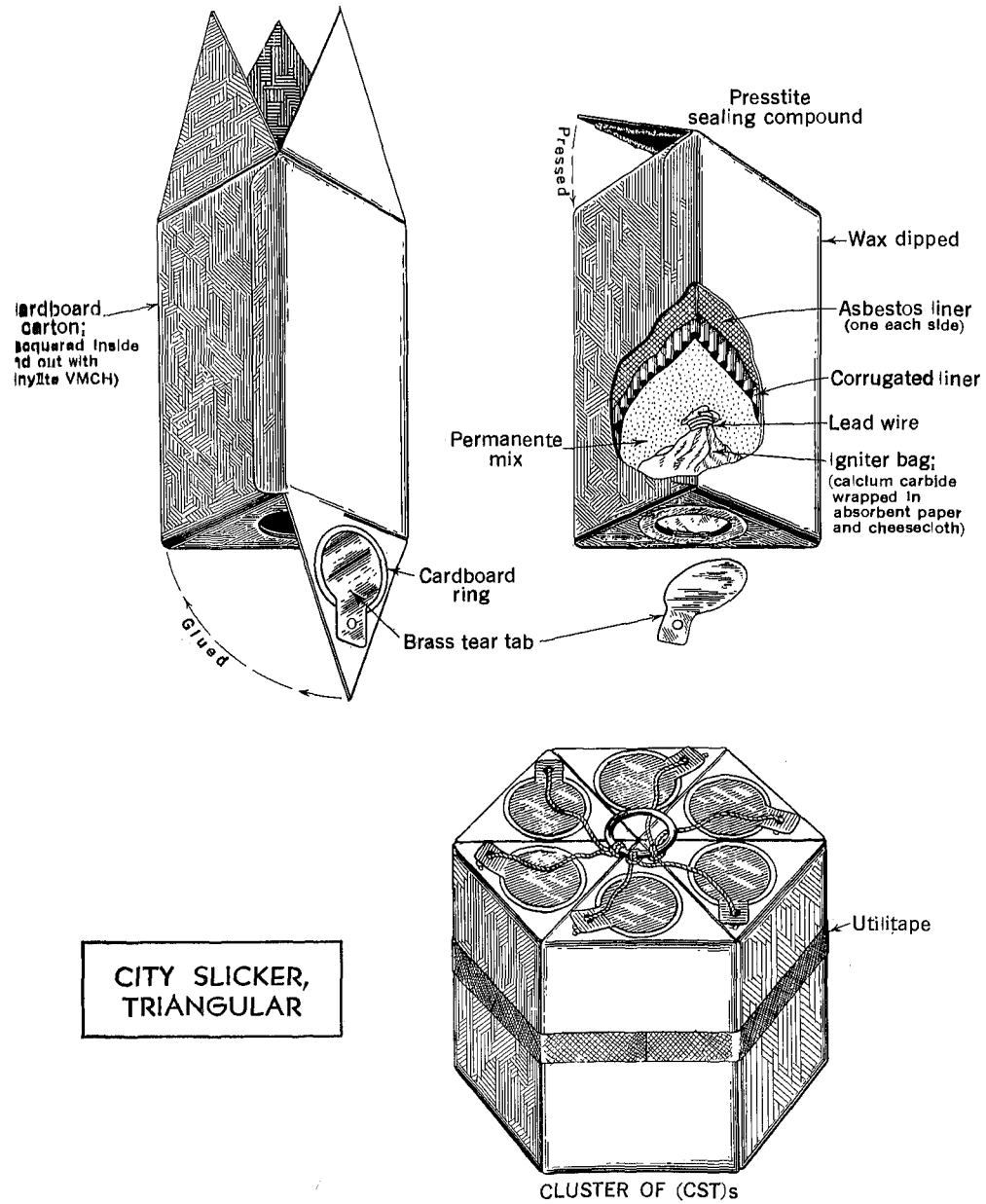
Fig. 8.6. — *Pocket Incendiary with Two Pencils of Different Time-Delay*

to ignite, but it is easily extinguishable and should be planted where it will escape early detection. Also, the PI is incapable of igniting oil slicks and it is smothered by oil. The PR burns with a directed flame of high intensity for about 1½ minutes. As a time-delay incendiary for land operations, the PR is particularly useful for setting fire to oil stores, since it carries its own oxygen and cannot be smothered. It will ignite very thick oils even when immersed in the oil or in a closed barrel or tank. Since the PR will operate in a closed container and produce enough gas pressure to blow the container open, it can be planted to function in a suitcase or shipping box. During the short period of highly intense burning, the fire cannot be put out with water, carbon dioxide, or carbon tetrachloride. The PR is thus valuable for an attack on a strategic and well-guarded target. The chance of success is greatly enhanced by planting two or three PRs in different locations, for any one of them will fully occupy the attention of a firefighter and thus give the others a chance to operate. The long-burning PI is preferable in operations where early detection is unlikely.

Interest by the Navy in an oil slick igniter was expressed in a directive of March 13, 1944 naming Commander John F. Gallagher as the chief liaison officer. As originally stated, the requirement was for an igniter which would function practically instantaneously when dropped onto water at a velocity of at least 500 ft. per second. In a first trial at Dahlgren Naval Proving Ground, Dahlgren, Va. on March 21, 1944, City Slickers of the best type then available were released in clusters from a Torpedo Bomber in a dive close to the water at 350 knots, a pretty sight. They worked reasonably well, but improvements were indicated. Two subsequent tests are described in the following diary entries:

> April 5, 1944. Dahlgren, Va. "Second test of slick igniters for the Navy. Tried triangular cartons (good) and plastic bottles (poor) loaded into sectioned M-47s."
>
> May 19–24, 1944. Eglin Field, Fla. AAF Service Test of the Navy igniters. "The tests were ill-conceived and poorly run. The attack bomber, flying at 200 ft. and 300 knots, scored no hits on the very small 200-gallon oil slick, partly because no practice shots were allowed and partly, I was convinced, because the safety wires had not been removed in loading the second lot of clusters into the plane. Although the official report was unfavorable, I was well satisfied with the performance of the igniters and the operation of the clusters. — Good swimming at the Officers Beach Club; did some surf casting."

The eventual final design of a fully satisfactory Navy igniter, designated CITY SLICKER, TRIANGULAR (CST), is shown in Fig. 8.7. The requirement that the unit be stronger and ignite faster than the original box model had led to improvements which were incorporated into the final construction of the Paul Revere. One seemingly small but important modification was use of Presstite for sealing the bottom of the loaded carton, for this eliminated internal moisture from glue. A quick-opening cluster conceived by Hershberg is shown in Fig. 8.8. The Morgan Construction Co. at Worcester, Mass. under the able leadership of Myles Morgan, fabricated cases for the first trials out of M-47 bomb cases, and then designed the case shown in the drawing, which calls for two identical stampings from 18-gauge sheet steel. The Navy plan was to mount wing-clusters of City Slickers on all aircraft going out on a mission. The cluster had to open quickly for release from a low-flying dive bomber. The cluster shown in the drawing can be mounted either onto a wing or onto the regular 100-lb. station of Army and Navy bombers. The two sections of the casing are hinged



CITY SLICKER, TRIANGULAR

Fig. 8.7. — Oil Slick Igniter for the Navy

securely together at the nose to prevent initial opening at this point when the cluster is released into the airstream. Assembly is done as follows. The tear tabs are removed from two CST units, a bead of Presstite Sealer is laid around the igniter hole of one unit, and the open end of the second unit is pressed firmly against that of the first one, with the holes abutting. The elastic seal so formed closes both members of the pair and provides protection against moisture in the course of assembly, as over a coffee break or a lunch period. Trials showed that igniters so sealed together break apart in the air. After the 12 CST pairs have been loaded, the semicircular wood spacer is put in place and fitted with guide rods and springs, the upper member of the casing is forced down with compression of the springs and secured with steel straps provided with quick-opening buckles which cannot operate until the safety wire has been withdrawn. The circular sections of wood held in place by Utilitape serve merely for proper positioning of the cluster for loading into a sheet metal container for shipping, and are removed before the cluster is mounted, by the suspension lugs, onto a shackle in the bomb bay. The safety wire passes through both buckles, as does the slightly longer arming wire, which when withdrawn allows the buckles to spring open. The loops in the two wires are secured to the bomb bay. The wires are of such length that, on release of the cluster from the bomb shackle, withdrawal of the safety wire for activation, followed quickly by withdrawal of the arming wire, opens the cluster at a safe distance below and behind the aircraft. The result is a shower of individual igniters forming an oblong pattern on water. On release from a Torpedo Bomber at altitudes of 150–600 ft. and speeds of 175–300 knots, the clusters opened an average of 40 ft. behind the plane and 25 ft. below it. As estimated from aerial photographs, the patterns were of the following areas:

Altitude of Release	*Diameter of Pattern*
600 ft.	150 ft.
450 ft.	80 ft.
175 ft.	55 ft.

The CSTs withstood impact on water at these high speeds and low altitudes very well, and the majority ignited and burned in the normal fashion.

Another method of employing the igniters that seemed even more attractive is as a munition of opportunity to be carried in aircraft operating over water and released by hand in a cluster of six units bound together by a band of tape. Such a cluster is shown in the drawing illustrating the CST igniter (Fig. 8.7). The assembly includes provision for quickly exposing

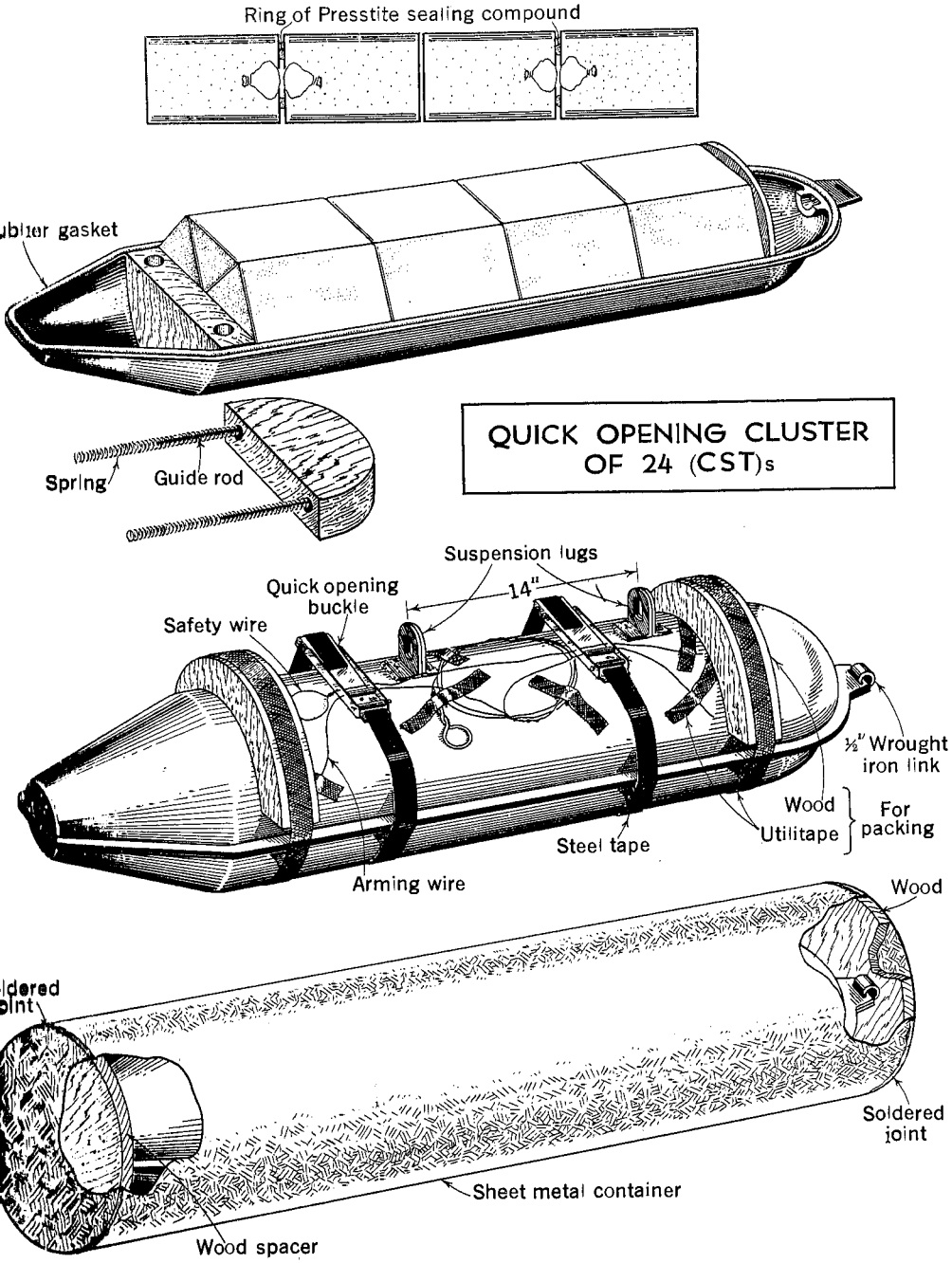


Fig. 8.8. — Wing-Mounted Cluster

all six igniter holes prior to release. This cluster falls through the air as a unit and on hitting the water breaks apart and scatters the igniters into an oblong pattern. The igniters skip along the water and come to rest some 25–30 yards ahead of the point of impact of the cluster.

Limited trials indicated that Paul Reveres also perform well on release from aircraft.

9. *Ignition of a 1,000-Gallon Oil Slick*

My copy of the directive for this trial is neither dated nor signed and bears no identification of the issuing office. It reads in part as follows:

> "This is an experiment on the
> City Slicker and not a demonstration.
> All observers will be prepared to leave the Acme Hotel at Beach Haven, N.J. at 0600 of the morning of the 10th. They will be conducted to the Coast Guard docks where they will board Coast Guard vessels and be transported to Little Beach Cove, the scene of the experiment. 1,000 Gallons of Navy Special Oil will be released at approximately 0750 to form an oil slick on the water."

The experiment was conducted on June 10, 1944. A report of the results, marked SECRET, was submitted by Capt. Floyd R. Frazee to Col. Carl F. Eifler, but I do not remember the Service group to which either officer was attached. The operation may have been a true experiment, but the following excerpts from the official report show that it had some flavor of a demonstration.

> "The entire observing party of approximately 35 persons from all armed services and various war boards left Beach Haven, N.J. at 0631, embarking for the point of the experiment in Coast Guard Boats 36428, 36004, 36425, 38625, 38025, and 40023 and arriving at 0728. The experiment area is known as Little Beach Cove, N.J."

LIST OF PERSONNEL ATTENDING EXPERIMENT ON CITY SLICKER

Brig. Gen. Lyle H. Miller, USMC	Captain Dale O. Allison
Cmdr. John F. Gallaher, USN	Captain McGowan
Lt. Cmdr. John M. Shaheen, USNR	Lieut. Pedro J. Aguirre
Lt. Cmdr. Carleton Kelsey, USCG	Lt. (jg) Theodore A. Morde, USNR
Lt. Cmdr. C. J. Sullivan	Lt. (jg) Beaumont Wright
Major John M. Jeffries	Lt. (jg) W. B. Simmons, USNR
Captain Floyd R. Frazee	Chief Spec. Phot. Edward Pyle

Chief Spec. Phot. Thomas Dowling
Chief Spec. Phot. Joe Lykins
Phot. 1c Gus Schenk
Phot. 1c Philip Bath
Lieut. Charles N. Fisher, USNR
Lieut. William E. Duggan, USMCR
Captain Emil V. Hegyi
Lieut. John W. Stansfield, USNR
Lieut. Lee E. Echols, USNR
Lieut. John G. Martin, USNR
Lieut. E. A. Scanlan, USNR
Phot. 2c Newt Jones
Phot. M. Toplewski
Dr. Louis F. Fieser
Dr. H. M. Chadwell
Mr. Edward Zeltner
Mr. C. E. Sullivan

Marine Corps General Miller, in company of an OSS observer and a photographer, made observations from a blimp flown in from Lakehurst, N.J., and I met him only at a jolly party at the hotel after the tests. Although only one member of the Harvard group is listed as having witnessed the test, there were two of us at the post-test party. Mary Fieser [1] had spent a day with me at National Fireworks Co. at North Hanover, Mass., where I supervised production of a batch of about 50 City Slickers for the test, and it seemed only proper for her to share a rare opportunity for an automobile trip. The necessary extra-ration gasoline was easily arranged for, because a car was needed for transportation of the City Slickers from the National Fireworks plant to Beach Haven. Since Mary was attached to the group informally and had no NDRC card, and since John Gallaher was the only one of the officers whom I knew well, I hesitated to ask that she be permitted to see the test. Had we met General Miller before rather than after the test, she surely would have had a seat in the blimp.

As noted above, the take off by boat was nearly on time (Fig. 9.1). A Coast Guard barge carrying a supply of 50-gallon barrels of Navy Special Fuel Oil was anchored at the south end of Little Beach Cove (Fig. 9.2). The blimp appeared overhead at 0800. Experimental dumps of 5 gallons each were made to determine the pattern the slick would take and the direction in which it would move. The finding that a heavy offshore wind had more effect on the slick than the tide, prompted the decision to change the original plans and move further up into the cove. Finally, at 0905, dumping began. 1,000 Gallons of oil was dumped, three barrels at a time, in a period of 11 minutes, and Flagship 40023 took a station at the upwind end of the oblong slick. A boat was stationed at either side, and the other boats of the party were in scattered positions for observation and safety. About nine minutes after the oil had been dumped a triangular City Slicker

[1] Mary Fieser, b. 1909 Atcheson, Kansas; A.B. Bryn Mawr, 30; M.S. Harvard (L. F. Fieser), 31; Research Fellow, Harvard University.

Fig. 9.1. — Coast Guard Flagship 40023

Fig. 9.2. — Coast Guard Barge

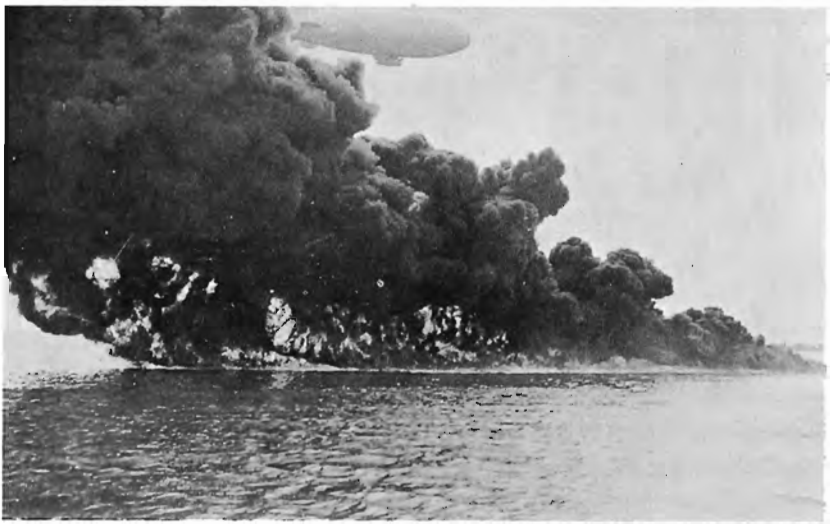


Fig. 9.3. — Oil Slick Afire

thrown into the slick from one of the boats at the side ignited the oil and started the raging fire shown in Fig. 9.3. The fire burned into the wind. Estimates based on photographs indicated that the flames were 40–50 feet in height and of a billowing type; the area of the flame was estimated as 17 x 117 yards. The total burning time was 7 minutes.

A Navy plane was scheduled to come in from Anacostia to drop a cluster of six City Slickers, Triangular, but failed to appear. However each boat had a supply of City Slickers of both the triangular and the box type, and enough oil was still available for a number of small slicks. Hence "experiments" went on for some time and a total of 30 igniters were used up. Everyone seemed happy and we had a gay party of celebration in the evening. My diary comment was:

> "Successfully ignited a 1,000-gallon oil slick and satisfied the Service crowd, but recognized that this batch from National Fireworks, driven down with Mary, was below par."

Production trouble!

10. *Production of the First Thousand Paul Reveres*

Tests of the PR and CST units by many of the Services, as well as a succession of NDRC humidity and firing tests, soon exceeded by far the production capacity of our small group, and E. B. Hershberg and the boys were sorely needed on other projects. The National Fireworks Co. at West Hanover, Mass. seemed ideally equipped for work of this type and presumably could handle mass production in case the devices should be accepted. The company was agreeable, and a contract for trial production of units was signed on May 18, 1944. E. B. and the boys must have had all the responsibility for indoctrinating the supervisors, foremen, and foreladies, since I was at Eglin Field, Florida from May 19 to May 24, conferred on June 6 with Drs. Shannon and Loeb in New York on the malaria problem, and returned to West Hanover only on June 7 and June 8. The assembly line of girl workers was not quite ready to function, and things had to be speeded up for production of enough units for the test at Beach Haven on June 10. The units were completed too late on June 8 for testing samples before taking off with Mary early on June 9. Most of the Service men attending the tests had not seen a really good City Slicker in operation and seemed well satisfied with the units tested, but the performance was far inferior to that of units made in the Gibbs Laboratory.

I made several trips to National Fireworks in June and July trying to straighten out the production, and Mary often went along. It was a hectic period. While trying to standardize production, we were experimenting with new batches of Permanente Mix supplied by Ted Dungan, for example, a lot designated Quicker Slicker. Every so often a request would come in for a supply of Slickers for a new test or special project. One, from the Navy, was for a couple of clusters of CST-units for Commander Mang to take along in a B-17 bomber on an exploratory mission in the Pacific. The request came in on a Friday afternoon, and shipment had to be made by nightfall on Saturday. No triangular units were on hand. By telephone I appealed for help to a National Fireworks foreman, and he, Mary, and I

worked all day Saturday and completed the assembly and filling of enough units to fill the bill and a few extras for surveillance tests. The assembly procedure used at the time called for forming the box by glueing bottom flaps in place with a water-containing glue and drying the box carefully at 60° C. to remove moisture. After lacquering, insertion of liners, calcium carbide bag, cardboard cylinder, and fuel, the flaps at the end were sealed with glue as before, and dried before the wax dipping. Since time was pressing, we shortened the drying time at each of the two steps. At day's end the igniters were loaded into my LaSalle and Mary and I set off for the Laboratory, where the Slickers could be loaded into modified M-47 cases. As we crossed Beacon St. I smelled smoke, and on touching the Slickers in back of the seat I found that they were hot. I swung into the first side street and quickly yanked the container out of the car and spread the igniters out in a row on the curbstone. Inhabitants of nearby houses were mystified but could be given no explanation. After a time the boxes cooled off and we loaded the cases, shipped them off by special Naval Transport plane, and so completed the mission. Next week I tested a sample batch of these igniters and found that they were dead; they had been ruined by the moisture in the glue. I called Washington in an effort to relay a message to Commander Mang, but was told that he was then beyond contact. I felt very badly. By chance, I ran into Commander Mang a few months later in the cafeteria at Edgewood Arsenal. Before I had a chance to apologize, he apologized. He had stored the clusters of igniters temporarily in a munitions pit on a conquered Japanese island, and on return had discovered that the igniters had been stolen by raiders.

Production at National Fireworks improved but never became satisfactory. Requests were coming in for batches of as many as 390 Slickers at a time. So, in July, the trial production at National Fireworks was closed out as a failure due to: poor Mix from Permanente, need for protection against high heat and humidity during assembly, need for improvements in the design and scheme of loading. These problems were all subsequently solved and a successful trial production run conducted in the Gibbs Laboratory by a group of Army–Navy wives working under Mary as forelady, with assistance from my able secretary Mrs. Ruth Dague. Even before that Mary had taken over liaison between Permanente and National Fireworks in my absence, as is evident from the following telegram to T. A. Dungan:

June 23, 1944. "NEW DEVELOPMENT REQUIRES 500 LBS. OF NEW 20 SECOND PM MIX AS SOON AS POSSIBLE. IF YOU HAVE NOT ALREADY SHIPPED SOME WOULD IT BE

POSSIBLE TO DO SO SOON. WE CAN ARRANGE FOR AIR TRANSPORT IF NECESSARY.

MARY FIESER"

In the period June 13–24, 1944, I first spent several days working with the Permanente group on the development of improved Slick Mixes. I next visited the Marine Barracks at Klamath Falls, Oregon, where Commander Coggeshall [1] had set up an effective organization for the experimental treatment of malaria; I hoped to interest him in running clinical tests on a compound synthesized by my antimalarial research group. The night train from Sacramento to Klamath Falls was held up for eight hours by a freight wreck ahead, and on awakening in the morning I found that we were stalled a little south of Dunsmuir. It was a beautiful spot, and alongside was an attractive stream, the Sacramento. I had worked my way about halfway up the breakfast line when the urge became so great that I pulled out of line, rigged up my rod, and went down for some fly casting. Alas, the train got under way just as I was starting and I had to catch it on the run. The lake at Klamath Falls was said to abound in rainbow and steelhead trout, but there was no opportunity for a try. However I put in a couple of days fishing for speckled trout on Lundy Lake in the High Sierras with Bill Young [2] and a group of other U.C.L.A. chemists. Train travel back to Sacramento was without incident, but at Sacramento an angry mob was waiting for the coach train to Reno, and it took some little strategy to get into the small group of selectees; the 150-mile trip took six hours. At Reno I sent Mary a wire stating that I had requested air transportation from Reno to Boston for arrival on Saturday night and asking her to secure the necessary priority. The telegram was phrased in such a way that it could be shown to a priority official. At midnight I took the Inland Stage bus for a stop called Mono Lake School. I was sound asleep on arrival at 4 a.m., but my friends were on hand to flash searchlights and stop the bus. On return to Reno after a relaxing day and a half at Lundy Lake, I was pleased to find at Reno a notification of priority, and I reached home as scheduled. The telegram from Reno to Mary had put her on the spot. She did not know about the fishing trip and could not imagine what kind of war work I could be doing in Reno. The priority officer knew of one mission that takes husbands to Reno and felt sorry enough for Mary to grant her request.

[1] Lowell T. Coggeshall, b. 1901 Saratoga, Ind.; A.B. Indiana, 22; M.D. Indiana, 28; Div. Biol. Sci., University of Chicago.

[2] William G. Young, b. 1902 Colorado Springs, Colo.; B.A. Colorado, 24; Ph.D. Calif. Inst. Techn. (Lucas), 29; University of California, Los Angeles.

Fig. 10.1. — At Lundy Lake; Debate with Bill Young on Fly vs. Spoon

When the Paul Revere was finally accepted, OSS asked me to write specifications and to arrange for and to supervise the production of a first thousand units. By then it was clear that assembly should not be done across the continent from the source of the temperamental key ingredient, but as close to the Permanente plant as possible. I hoped to find a company in or near San Jose capable of conducting a trial production run of 1,000 Paul Reveres on order from Harvard University and decided to have the supplies shipped to Permanente; perhaps this company would itself take the job. Lyon Southworth, Assistant Director of the Harvard Chemical Laboratories, was very helpful in arranging for the production and shipment to California of all the parts and materials required for making, finishing, and packaging the 1,000 PRs (and 2,000 to spare, in case of trouble).

Mary and I spent the period October 4 to November 6, 1942 in California directing the pilot production. Ted Dungan was en route to England to work with British Petroleum Warfare, his wife and children were visiting relatives, and they very kindly offered us the use of their house at 460 Churchill Road in Palo Alto. Priority for extra-ration gasoline enabled me to rent a car for daily trips to San Jose and return. For purposes of rationing a Hertz car is listed as a taxi, and I recall the concern of Technical

Fig. 10.2. — Permanente Office Building

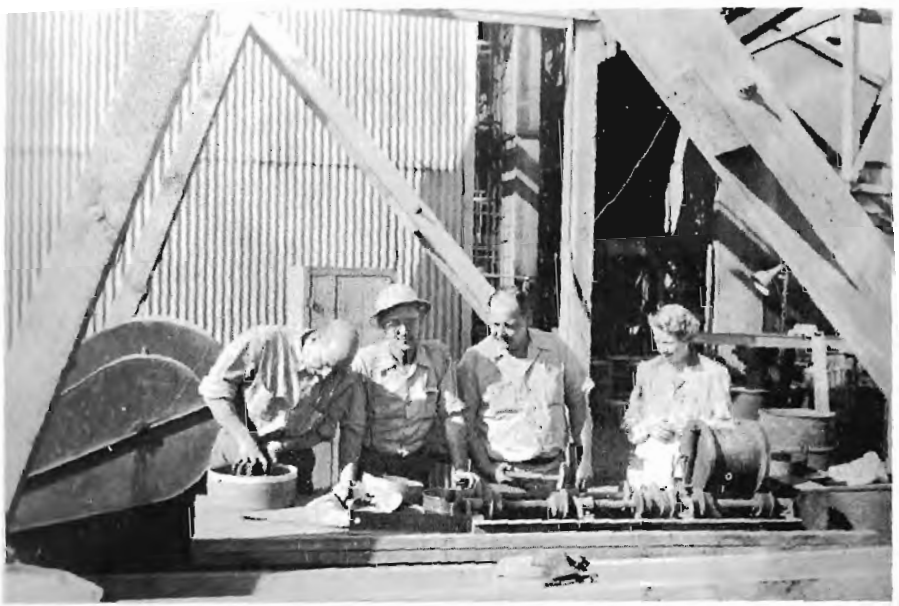


Fig. 10.3. — Al Peck (in Helmet)

Aides Chadwell and Lothrop over an item on the expense account of two or three hundred dollars for "taxis."

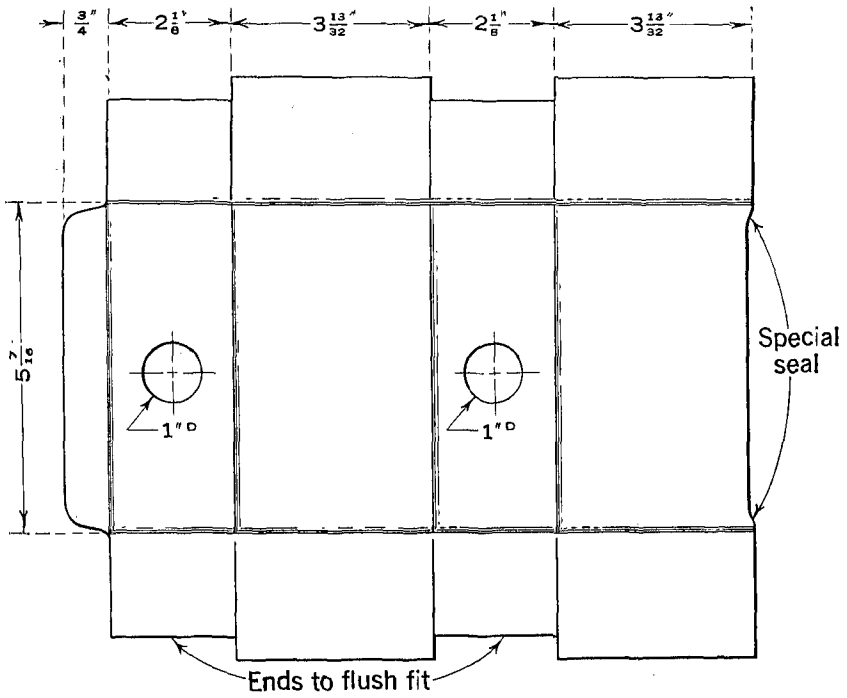
It was a pleasant surprise to learn that Permanente not only had decided to take the job but that Al Peck had been assigned to the project and had already taken several steps to put things in readiness. Capt. T. D. Alexander, appointed by OSS as observer, was already on hand.

A major problem of standardizing the Permanente Mix was solved by a retort process (Fig. 10.4) in which excess oil is removed from goop by vacuum distillation in a special still in which the charge of goop is dis-

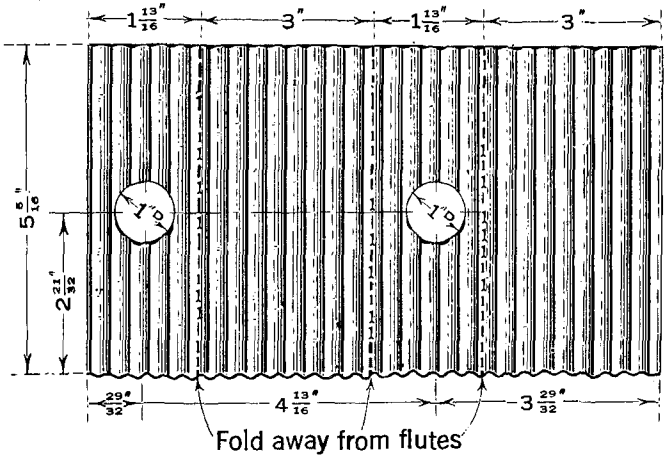
Fig. 10.4. — Retort Process

tributed in strands over a series of baffle discs on a rotating shaft for disintegrating and discharging the dried material.

The cartons are supplied as flat sheets; the specification drawing (Fig. 10.5) shows the method of folding carton and liner and the "special seal" required to make a tight fit with the side flap. In the first step a cardboard ring is glued around one of the holes and a brass tear tab is glued onto the



PR CARTON



PR CORRUGATED LINER

Fig. 10.5. — Carton and Liner

Fig. 10.6. — Glueing on the Tear Tab
(Depression of the lever raises a weight for insertion of the freshly glued cartons.)

ring, as shown in Fig. 10.6. Next a 1-inch celluloid disc is glued into the second hole. In the Harvard production this operation had given no trouble because disc and hole had been cut from the same, accurately machined punch (one I had had made earlier for cutting discs of filter paper). After insertion of a disc, application by brush of a narrow band of adhesive at the juncture gave the tight closure required to prevent later penetration of lacquer into the celluloid case when this had been cemented in place. The celluloid discs supplied by a manufacturer, however, were slightly out of round and irregular and they did not fit well. Furthermore, the California girls tended to apply glue much too liberally, and the heavily daubed seals remained soft in the interior. This fault was remedied by the simple expedient of discarding the 1-inch disc and using instead a 1¼-inch transparent celluloid disc and sealing it to the inside surface of the carton as follows. A gluer is made from a 1-inch diameter glass test tube by grinding the lip to a level surface about $\frac{1}{16}$ inch wide. The gluer is pressed lightly onto a glueing sponge and then onto the carton; application in the desired location is easy because of the transparency of the glass gluer. When the 1¼-inch transparent disc is put in place and pressed down, the glue is squeezed to the edge of the celluloid to form a uniform and secure bond.

The carton is then folded along the central seam and laid flat, with the side flap (on the left, Fig. 10.5) projecting beyond the "special seal" section on top. Adhesive is applied to the side flap, and it is folded and secured in place with a weight. A celluloid case loaded with Kraus mixture is cemented onto the narrow side of the box over the celluloid window; the operation is done by applying adhesive to the rim of the celluloid case, pressing the case down onto the flat cardboard carton, and securing it with a 4-lb. weight until the glue has set. The flat carton is then sprung open and slipped over a rectangular wooden forming block, and the bottom is sealed by folding over the two small flaps, applying glue to the two large flaps, sealing these down in turn, and placing a 5-lb. weight over the seal. The first of a series of 10 boxes will have set by the time the last one has been glued. The next operation is to roll a wad of Presstite gum into a ball, flatten it on one side, press it lightly over the opening of the pencil well of the celluloid case, and mold it down smoothly on the case. The partially formed boxes were then dried carefully. Lacquering of the box-casing assembly, inside and out, was done at Permanente by dipping in lacquer and drip-drying in an efficient rotating 4-unit machine. After insertion of the liner and loading of the box with Permanente Mix, calcium carbide bag, and cardboard cylinder, the top flaps were secured in place with Glyptal cement (Fig. 10.7), and the box was dipped in wax (Fig. 10.8).

Since the improved Permanente Mix was considerably more active than before, adjustments had to be made in the calcium carbide igniter system in order to achieve the desired times of ignition and burning. The hole in the liner was made larger (1¼ inch) than the hole in the carton (1 inch) so that the igniter bag would seat nicely into the 1¼-inch liner hole and fit flush against the 1-inch opening, as observable on removal of the tear tab for inspection. The igniter bag was made by wrapping the calcium carbide in a layer of absorbent paper and an external layer of cheesecloth. The cardboard cylinder was made longer so that, with the liner inserted only part way into the carton, the tube and igniter bag could be fitted into the two exposed holes and so adjusted with the fingers that when the liner is pushed in place a snug fit at both ends is secured. The Permanente Mix could then be loaded from a hopper in one simple operation. Results of water-ignition tests of 43 PRs made with modified igniters (Fig. 10.9) were entirely satisfactory: ignition time, 13.3 seconds (average); burning time, 1 minute and 9 seconds.

In working out various adjustments and in providing instruction to the six-girl assembly line, several hundred cartons were partly assembled and about two hundred were loaded and expended for performance, inspection,

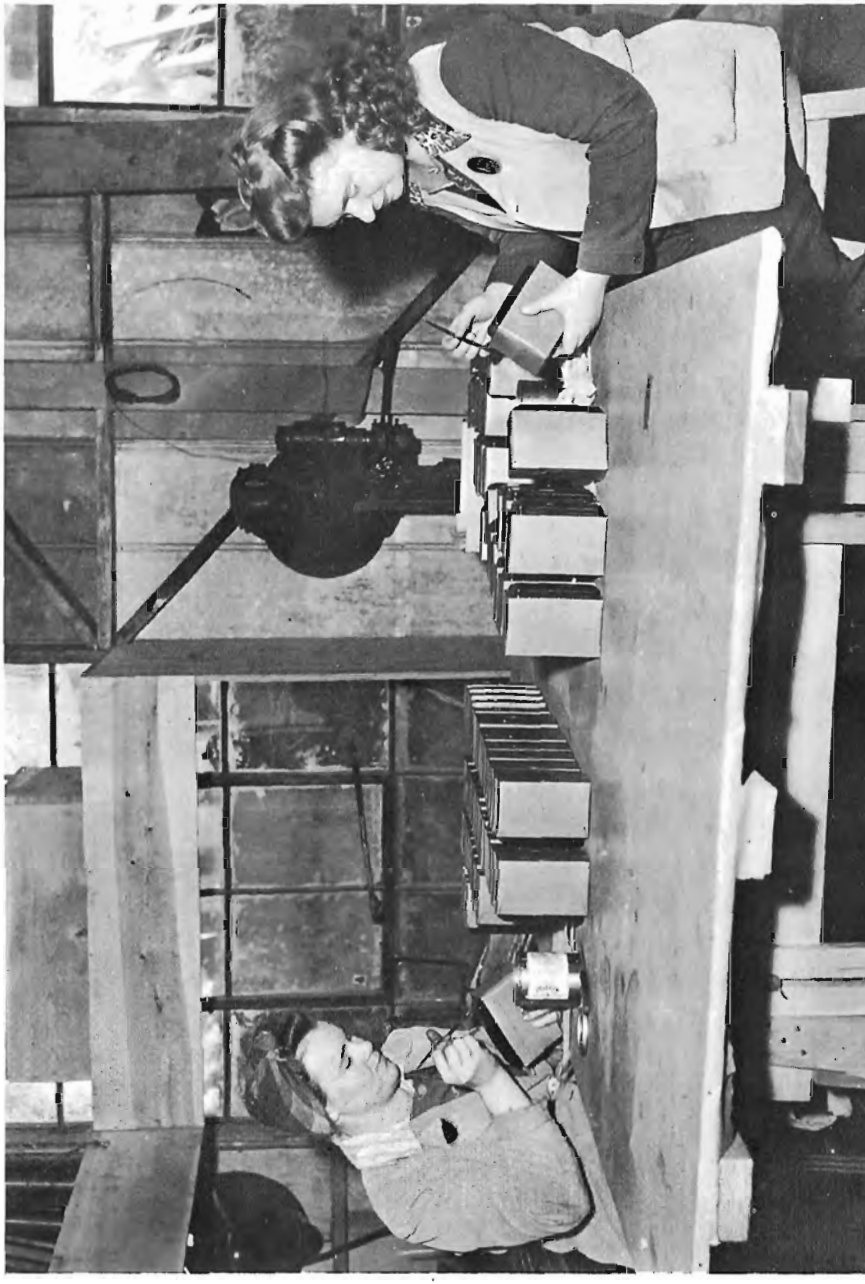


Fig. 10.7. — Top Flaps Sealed with Glyptal Cement

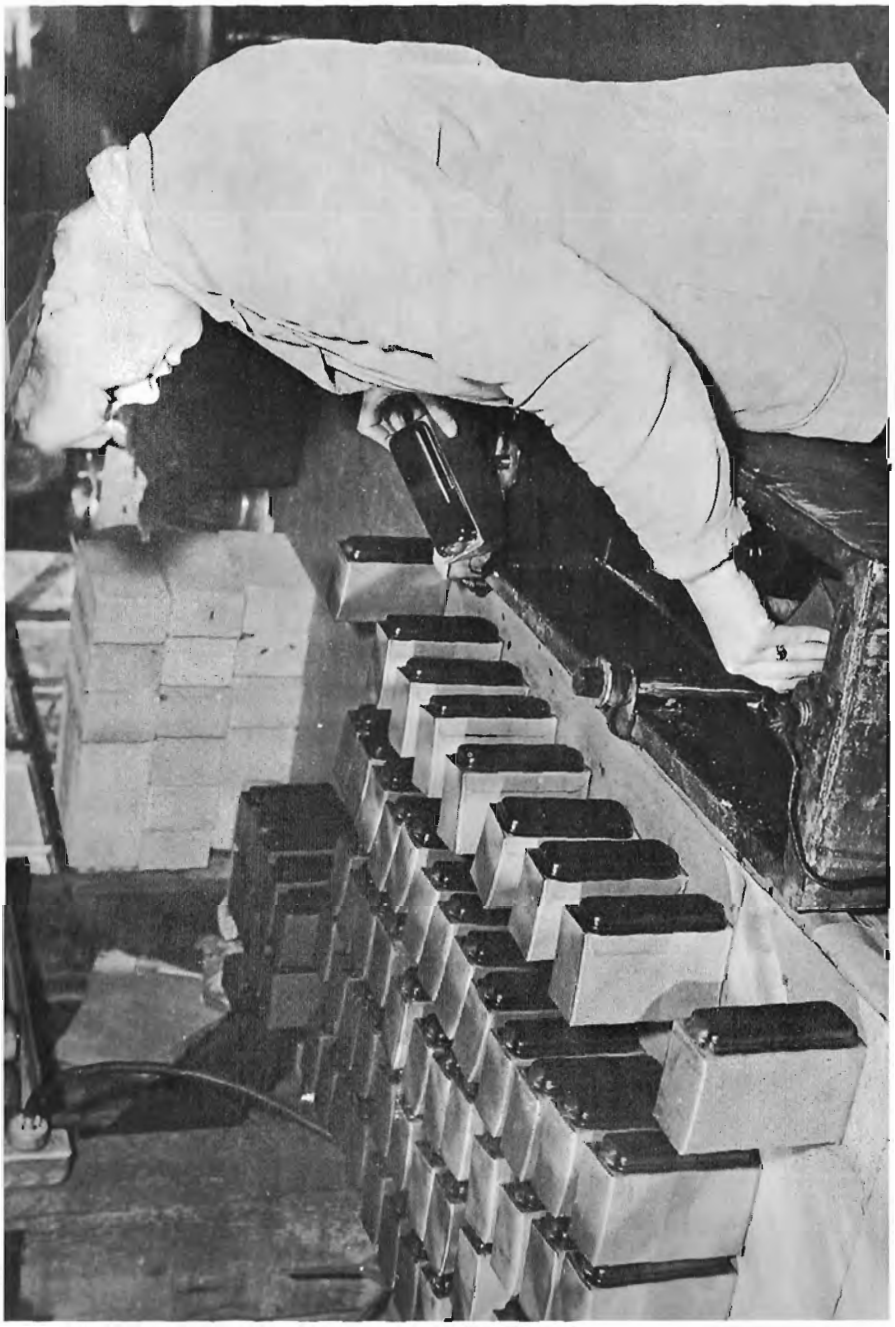


Fig. 10.8. — Dipping PRs in Wax

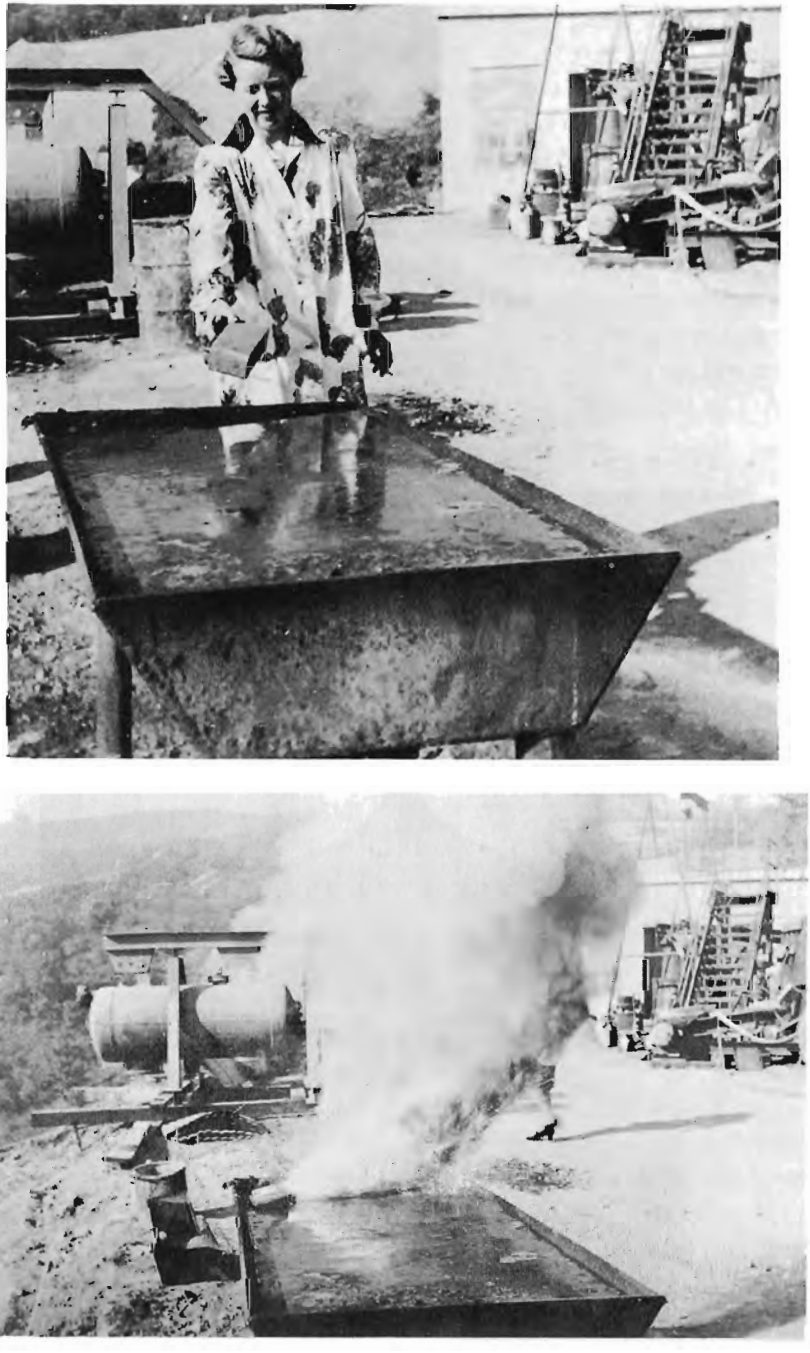


Fig. 10.9. — Mary at the Testing Tank

and surveillance tests, or to check changes in construction or in assembly procedures. Each carton was numbered consecutively, and a record was kept of the dates of lacquering and loading, the batches of materials employed, and any variation in the operations. Numbering also provided a record of each day's production. After PR No. 445 had come off the line, the procedures were all frozen and production was started on the 1,000 units. All earlier cartons on hand, whether loaded or partly assembled, were declared "experimental" and destroyed.

No further incidents need be recorded, except perhaps a pleasurable drive to Carmel in our "taxi," justified on the rather slim excuse of a visit on the way to a Kaiser plant for extraction of magnesium salts from sea water. The mission was completed with success.

11. Sabotage of Motor Vehicles

I HAVE NO record of this project, and assume that the directive from OSS came by word of mouth. The requirement was for some way in which an operator at a Polish filling station could sabotage German trucks and tanks en route to the Russian front. Our solution was a pellet of Napalm, made by compression of 14 g. of Napalm powder with a Carver press. In the course of filling a tank with gasoline, the operator drops a pellet into the

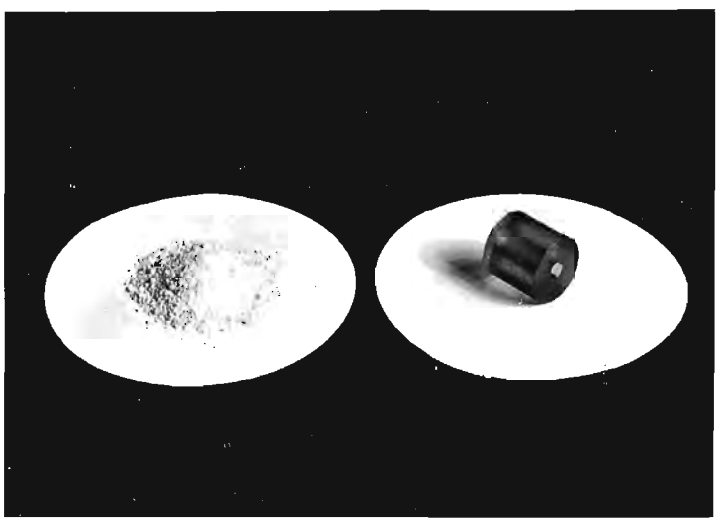


Fig. 11.1. — Napalm Powder (14 g.) and Napalm Pellet (14 g.)

tank. Solvation of the pelleted material is very slow, but after a period so long that one or more additional tankfuls will be required, the pellet swells to a sticky glob the size of a baseball, and is sucked into the feedpipe to the carburetor. That should do it.

12. *Antitank Grenade*

THE ONLY report on this project is in the form of a patent (Fig. 12.1), but this gives all pertinent details. The "Molotov cocktail," a glass bottle stuffed with cotton waste and filled with gasoline, was ignited with a match before being thrown. This crude device found some use in World War I, and I had heard of no later improvements. Why not a grenade filled with a gelled fuel and provided with an igniter? Such a weapon seemed impractical for most forms of combat, but possibly a tank has some point of vulnerability. On November 25, 1941 I visited Aberdeen Proving Ground, Maryland to inspect all types of American tanks then available, as well as drawings of German tanks. A turret presents one target for an incendiary grenade, but I was impressed particularly with the size and power of the air-intake. A hit by a grenade somewhere near the air-intake, which would splatter a sticky burning gel over the metal surfaces might be very destructive. Flames drawn into the tank might make the compartment uninhabitable, cause stoppage of the motor, or combustion of the lubricating oil in the revolving turret.

In discussing this possible problem with the boys, I noted that, unfortunately, American tanks are considerably more vulnerable than the German tanks; it might not be advisable to present the Germans with the idea. Possibly for this reason, but more probably because there were so many other more pressing problems, the grenade project was set aside for a time. After Napalm gels of satisfactory quality had been made available, we took up work on the problem and developed the grenade shown in Fig. 12.2. The container is an ordinary glass jar with a tin screwcap. An igniting fluid which remains fluid at $-40°$ C. is contained in a glass ampoule made from two concentric tubes joined by a ring seal (glass blowing by E. B.). The fluid is introduced through a narrow tube sealed to the eventual bottom and this tube is drawn out to a tip and sealed. The breaking of this ampoule on impact of the grenade on a target is ensured by inclusion of a steel ball in the central well. The ball is secured in a safe position by a cotter pin; until this cotter pin is withdrawn there is no danger of breaking the ampoule on accidental jarring, or on dropping the unit. Withdrawal of the pin allows

2501766

THE UNITED STATES OF AMERICA

TO ALL TO WHOM THESE PRESENTS SHALL COME:

Whereas Louis F. Fieser, of Belmont, Massachusetts, George C. Harris, of Wilmington, Delaware, Samuel S. Hershberg, of Stoughton, Massachusetts, Morley Morgan, of Grosse Pointe, Michigan, Frederick J. Novello, of Lansdowne, Pennsylvania, and Stearns T. Putnam, of Newark, Delaware, assignors to the United States of America as represented by the Secretary of War,

HAVE PRESENTED TO THE **Commissioner of Patents** A PETITION PRAYING FOR THE GRANT OF LETTERS PATENT FOR AN ALLEGED NEW AND USEFUL IMPROVEMENT IN

INCENDIARY GRENADES,

A DESCRIPTION OF WHICH INVENTION IS CONTAINED IN THE SPECIFICATION OF WHICH A COPY IS HEREUNTO ANNEXED AND MADE A PART HEREOF, AND COMPLIED WITH THE VARIOUS REQUIREMENTS OF LAW IN SUCH CASES MADE AND PROVIDED, AND

Whereas UPON DUE EXAMINATION MADE THE SAID CLAIMANT s are ADJUDGED TO BE JUSTLY ENTITLED TO A PATENT UNDER THE LAW.

NOW THEREFORE THESE **Letters Patent** ARE TO GRANT UNTO THE SAID

United States of America as represented by the Secretary of War

OR ASSIGNS

FOR THE TERM OF SEVENTEEN YEARS FROM THE DATE OF THIS GRANT

THE EXCLUSIVE RIGHT TO MAKE, USE AND VEND THE SAID INVENTION THROUGHOUT THE UNITED STATES AND THE TERRITORIES THEREOF.

In testimony whereof, I have hereunto set my hand and caused the seal of the Patent Office to be affixed at the City of Washington this twenty-eighth day of March, in the year of our Lord one thousand nine hundred and fifty, and of the Independence of the United States of America the one hundred and seventy-fourth.

Attest:

Law Examiner.

John A. Marzall

Commissioner of Patents.

Fig. 12.1

INCENDIARY GRENADE

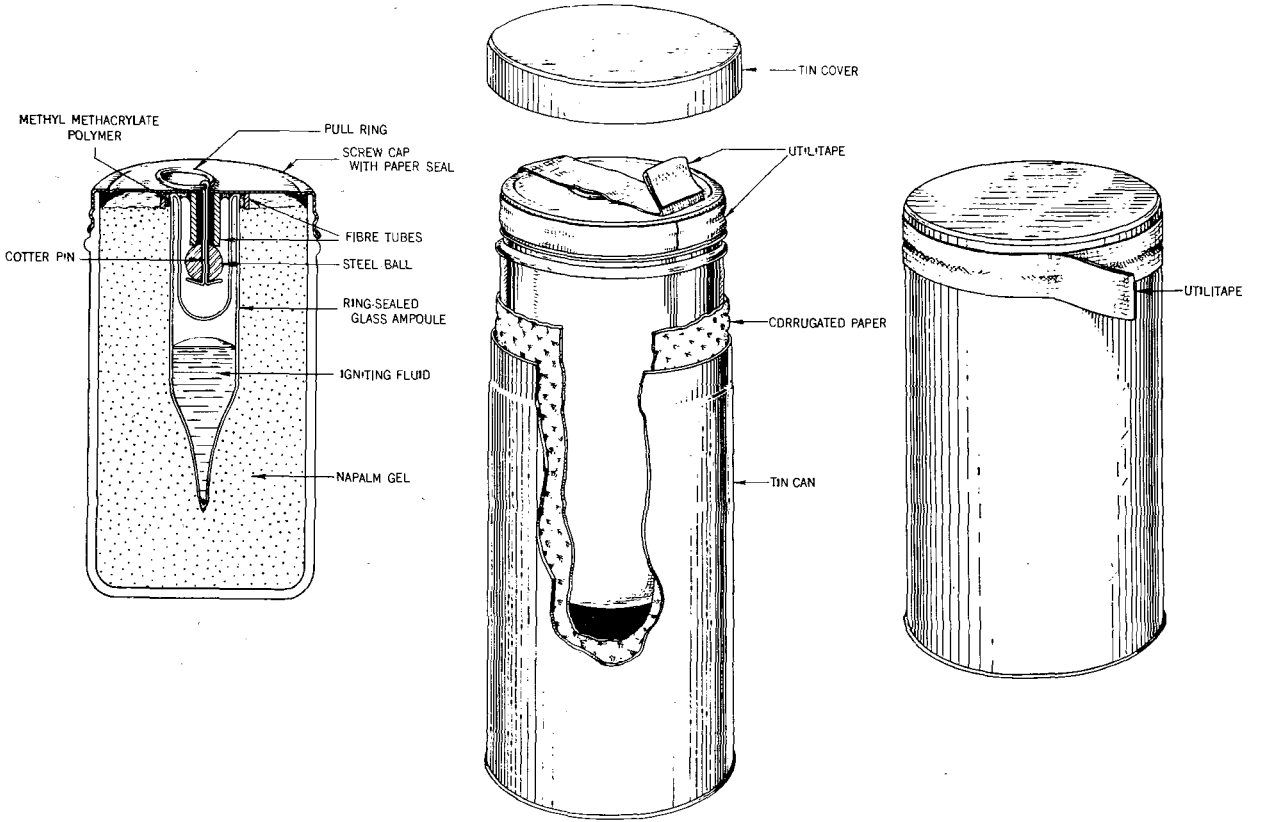
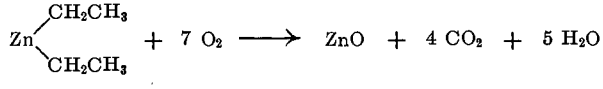


Fig. 12.2

the ball to fall only a short distance into the recess, and the impact is far short of that required to break the glass, but the ball is now readied for action. When the grenade is armed and hurled at a target, the impact invariably breaks the ampoule and the fluid ignites the Napalm gel. The ball is mounted at the end of a short section of fiber tube cemented to the screwcap. For assembly, the screwcap, with fiber tube and ball in place, is placed in an inverted position, the ampoule is inserted, and the annular space between the two members is filled with methyl methacrylate, which is then caused to polymerize.

As igniter fluid we used diethylzinc:



Diethylzinc

Since this liquid takes fire immediately on contact with air at all temperatures, the preparation of the chemical is an operation calling for rigid exclusion of air from all apparatus by use of an atmosphere of nitrogen, remote control, and extreme care. The assignment to prepare a large batch of diethylzinc for our experimental production of grenades was taken over and ably executed by an NDRC group at the University of Maryland under Nathan L. Drake. Mary met the Federal Express on which Nathan had personally transported a large iron pipe packed with sealed glass ampoules containing the igniting fluid, and she recalls that, on arrival, his face was white as a sheet.

For protection in carrying, the grenade is packed in a metal container with a corrugated paper liner. A piece of strong tape fitted across the screw cap of the grenade, and folded over at one end to form a tear tab, secures the ring joined to the cotter pin and prevents accidental withdrawal; it also facilitates withdrawal of the grenade from the container. The slipcap cover of the container is held in place by an easily removed seal of tape. The packaged grenade will stand considerable shock and rough usage. It is not set off by rifle fire, for when a bullet shatters the glass ampoule the igniting fluid is surrounded by gel and hence is protected from contact with air; in a short time the fluid either is destroyed by chemical reaction with the gel or is diluted to such a degree as to become ineffective. Even when removed from the tin container, the unarmed grenade is not hazardous. If it sustains a sufficiently severe impact by dropping onto a hard surface, the outer glass jar breaks first and takes up the bulk of the impact, and the ampoule is cushioned by the surrounding gel. The grenade functions satisfactorily at

all operational temperatures (−40 to +150° F.) and passes the British cyclic humidity test.

These admirable qualities, as well as the novelty of the device, were recognized by the patent office but not by the Armed Forces. Indeed, the fact that NDRC lawyer Capt. Benny Bull allowed listing of no less than six inventors is evidence that the patent was regarded as valueless. The most secure patent is one listing a sole inventor. In the case of the Napalm patent, Capt. Benny stood firm against vigorous pleas on my part to include the names of the entire group.

Officers of the U.S. Armed Forces regarded the grenade as too hazardous to the home team. But determined fighters who were not so particular turned up elsewhere.

I had known of the researches of organic chemist Dr. Chaim Weizmann since graduate school days, when I had repeated one of his published procedures with gratifyingly good results. Although many Jews, particularly rabbis, are under the mistaken impression that Weizmann discovered TNT, all chemists know that his great contribution to Britain in World War I which led directly to the Balfour Declaration of a homeland for Jews in Palestine was the discovery in 1911 of a process for the bacterial fermentation of cornstarch to acetone and *n*-butyl alcohol. Acetone was desperately needed by the British for compounding the shell propellant Cordite. Weizmann later developed a process for the production of the hydrocarbon isoprene, of which rubber is a polymer, and which is currently being used for production by synthesis of a polymer identical with natural rubber. During World War II Dr. Weizmann made a visit to this country as consultant on the synthetic rubber research program and while in Boston invited me to dinner at the Copley Plaza. We spent the evening talking about chemists and about chemistry, for I knew next to nothing about his activities in the Zionist movement, and I fell under the spell of his charm, wit, and wisdom. The ultimate upshot of this first meeting was that after the war I gladly accepted Dr. Weizmann's invitation to serve on a committee of six American scientists to lay plans for establishment in Palestine of the Weizmann Institute and lay the cornerstone for the first new laboratory (the existing Daniel Sieff Laboratory became part of the new Institute). On May 11, 1946 Mary and I, together with other committee members and wives, sailed on the troopship Vulcania bound for Naples and then Alexandria, Egypt. Among those who sent us off were E. B. and Charlotte Hershberg and Ph.D. students Armin and Evie Wilson of the antimalarial research group. On the same day Hans Heymann delivered, in my behalf, a lecture in Detroit on antimalarials. Concerning an extremely

Fig. 12.3. — Rehovoth, Palestine

interesting trip which extended to June 2, 1946, I shall mention just one incident. I was asked to give several lectures and soon found that a lecture delivered at Rehovoth should not be repeated later at Jerusalem, for a substantial part of the audience would be the same as before. When it came time to give a talk at the Haifa Technion, the supply of ready-to-hand lectures was running low. Since the Technion is an engineering school, a talk on our experiences with incendiaries seemed appropriate; and I told about some of the gadgets described in this book. Later, after the war with the Arabs had broken out, I received a letter from Dr. E. David Bergmann of the already operating Weizmann Institute. He described a recent research, and then stated that he had become interested in following up "the line of investigation, mentioned in your lecture at Haifa, on the immobilization of moving objects." I understood what was wanted and wrote a letter first describing recent work of my group and then, in suitably obscuring scientific terminology, supplied a formulation for preparing Napalm and a literature reference for identification of the igniting fluid as diethylzinc. As in the case of projects conducted for U.S. Services, particularly the OSS, I later heard very little about operational results. However, I had

learned something about Egyptian tanks when, in returning from Palestine, Mary and I had flown over the Negeb in a tiny Egyptian plane and reached Cairo in time for a close connection with a Pan American plane, only to be stalled in Cairo for eight days until one arrived. During this frustrating wait, we moved out to the Mena House near the pyramids where, at the swimming pool, we made friends with a couple of Egyptian army officers on leave. They had some interesting tales, for example of techniques they used for conforming to the letter of religious fasting periods without hunger or loss of weight. I was reminded of a story current at Oxford about an Egyptian prince who, to the surprise of fellow students, was not exactly a teetotaler. His Aide's explanation: the Prince was so holy, that everything he drank turned to water. Our officer friends had served a tour of duty together in a tank battalion, and they told us that the tanks were rickety old obsolete contraptions bought second-hand from the British.

13. *Bat Bombs*

EARLY IN THE war Lytle S. Adams, a Los Angeles dentist, proposed a plan for a surprise attack on Japan by release from bombers of bats put into hibernation by cooling, each carrying a small incendiary bomb. The bats would come out of hibernation on reaching warm air and carry the incendiaries into highly combustible Japanese houses at sites very favorable for starting fires. My first opinion of the plan was expressed in a letter to Earl Stevenson:

> July 10, 1942. "I am inclosing the very interesting proposal for a surprise attack on Japan. The idea is ingenious and certainly has attractive features. On looking at the proposal from the point of view of the incendiary problem involved, I see certain difficulties which, in my opinion, would just about block execution of the plan . . ."

Inventor Adams had a weird conception of munitions, particularly of incendiaries, and the plan hung fire for a year. I was called to a conference at Edgewood Arsenal on March 18, 1943, and told that CWS had received a directive to supply incendiary units to be carried by bat vectors (Diary: "The project seems silly, but . . ."). There were further conferences at Edgewood on April 1 and April 19, and on April 14 I conferred with Adams in Irwin, Pa. The upshot of the discussions was the decision to stage a first test at Muroc Army Air Base, not far from Los Angeles. I was to supply bat-bomb incendiaries and work on the project in cooperation with good Edgewood friends Col. R. Bruce Epler and Capt. Wiley W. Carr.

It was easy enough for our group to develop the micro version of the Pocket Incendiary shown in Fig. 13.2. A Napalm-filled celluloid case was fitted with an improvised time-delay pencil good enough for initial trials. A PI igniter is mounted in one end of a celluloid tube drilled with one hole for release of gas pressure and another for insertion of a cotter pin as arming member for safe transport. A copper-plated steel spring in the celluloid tube is compressed by a steel wire secured to a copper pin passing through the tube at the open end. Activation is accomplished by injecting, by hypodermic syringe, a solution of copper chloride of suitable formulation to

Fig. 13.1. — Mexican Free-Tailed Bat
(*Todarida mexicano*)

corrode the steel wire after an appropriate period. With a 30% solution in water, the pencil fires in 20 minutes. A solution of 8% copper chloride, 10% water, and 82% propyl alcohol gives a delay of 6 hours; solutions in glycerol-water mixtures cause firing of the pencil after 2–3 hours delay.

The first experiments would be to test the carrying capacity of bats and to determine at what altitude the animals would come out of hibernation. Dummy bombs of different weights would be attached to bats put into hibernation by refrigeration, and the bats would be released from a plane at different altitudes. Muroc Army Air Base was selected because of the availability of a large dry lake with a hard flat surface which was nearly white. On this surface it should be possible for parties in jeeps to patrol the area and spot non-flying free fallers. We were not too sure that this method of observation would be successful, and made a last-minute decision to supply equipment for operation of an alternative scheme. This involved replacing the Napalm gel in the bomb shown in Fig. 13.2 by the same weight of red phosphorus. This would catch fire and provide a dense smoke to serve for spotting the units at long range and determination of their distribution. An incendiarist, mammalogist, and photographer were to make

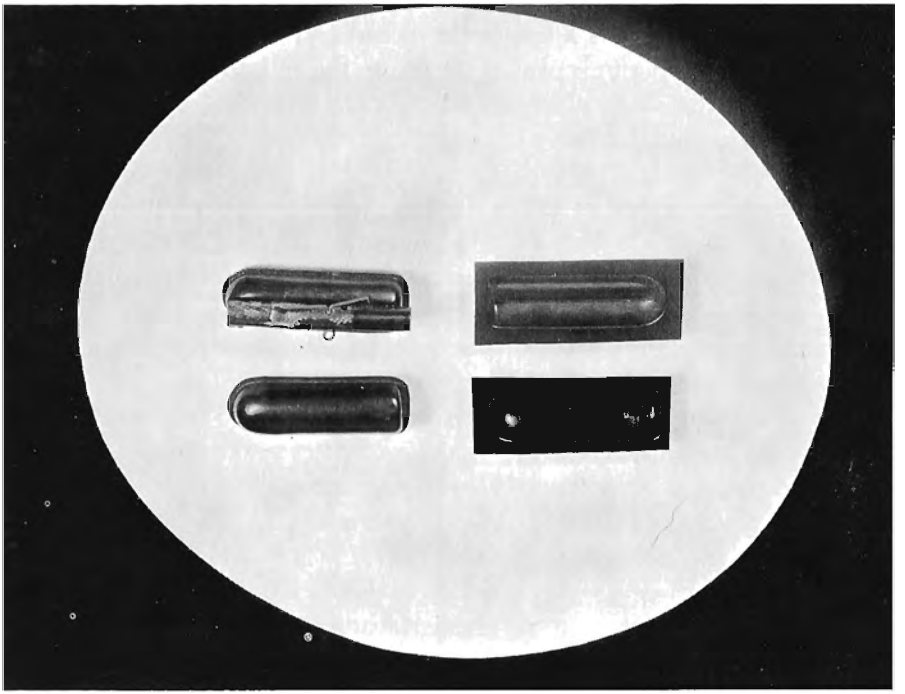


Fig. 13.2. — Bat-Borne Napalm Gel Incendiary with Time-Delay Pencil

each flight. This scheme also would permit a check on the operation of the igniter and time-delay mechanism. Red phosphorus, like the more active white variety, is of only low value as an incendiary, and hence the risk of destroying property was much less than if the bomb contained Napalm.

Standard Pyroxoloid made up about 3,000 bomb cases, filled some with Napalm, and sent a supply to us for loading with red phosphorus in the Gibbs Laboratory. Pencils were manufactured by A. C. Gilbert Co. Bill Young had been brought into the project because of his convenient location at the University of California at Los Angeles, and he arranged to have an assembly crew in readiness consisting in a group of graduate students and research assistants headed by Jack Roberts.[1] This group had advance instructions for making up four time-delay solutions and about the assembly. Pencils and Napalm-filled cases were shipped by the suppliers directly to U.C.L.A.

There remained the problem of transporting 800 bomb cases containing

[1] John D. Roberts, b. 1918 Los Angeles; B.A. U.C.L.A., 41; Ph.D. U.C.L.A. (W. G. Young), 44; Mass. Inst. Techn.; California Inst. Techn.

red phosphorus across the continent. Our own experience with the red variety was limited. It is a low-grade incendiary, to be sure, but it burns when ignited, and it surely would set fire to the celluloid case if it should combine with the oxygen present in the case with any speed. We were willing to take chances, but not to trust these bombs to unattended shipment by rail or to any form of air transportation. However, the problem was solved by a shoestring play. A refrigerated truck for bat hibernation had been built at Wright Field, Dayton, Ohio and was to be driven to California by a crew which included an Army Air Force photographer assigned to the project. By luck, the truck had not yet taken off at the time the bombs were loaded, and there was just time to reach Dayton by train. None of my regular group could be spared, so I called on Dick Turner,[2] who was working with me on the cortisone problem. Dick was given a large tin box, and instructed that if the box got warm or started to smoke he should throw it out of the window. Dick reached Wright Field without incident, delivered the box to the truck crew and passed on to them the instructions received. Box and truck reached Los Angeles intact, the box was delivered to Bill Young, and the truck placed at the disposal of Inventor Adams, who had agreed to have it loaded with 3,000 bats in time for the test. Pilots and crew flew a B-25 Bomber from Eglin Field, Florida out to California for use in the test.

The evening of May 17, 1943 was set for a conference to arrange plans for moving to Muroc the next day; the place was the home of Lytle S. Adams of Los Angeles. Col. Epler and I on arriving together noted that the truck from Wright field was parked outside. We were horrified to find that Adams had invited a large company, including ladies, to a dinner party in celebration of the initiation of field tests on the Adams Plan, supposedly a highly secret project. Eventually the Colonel and I took Adams aside and asked about the bats. We finally extracted the news that it was mating time for local bats and that Adams had been able to catch only 150. The nearest source of bats in quantity was Carlsbad, New Mexico, where huge labyrinthal caves are inhabited by bats by the millions; they fly out of these caves in the late afternoon in swarms that darken the sky.

Epler and I agreed on the following plan for the next day. He would fly in the B-25 to Carlsbad, load bats into eight large crates with which the truck was provided, and fly them over to Muroc. Carr, Adams, and I and a few others would meet at the U.C.L.A. Laboratory, pick up Bill Young and the incendiary supplies, and proceed to Muroc Army Base by com-

[2] Richard B. Turner, b. 1916 Minneapolis; A.B. Harvard, 38; Ph.D. Harvard (Fieser), 42; Rice University.

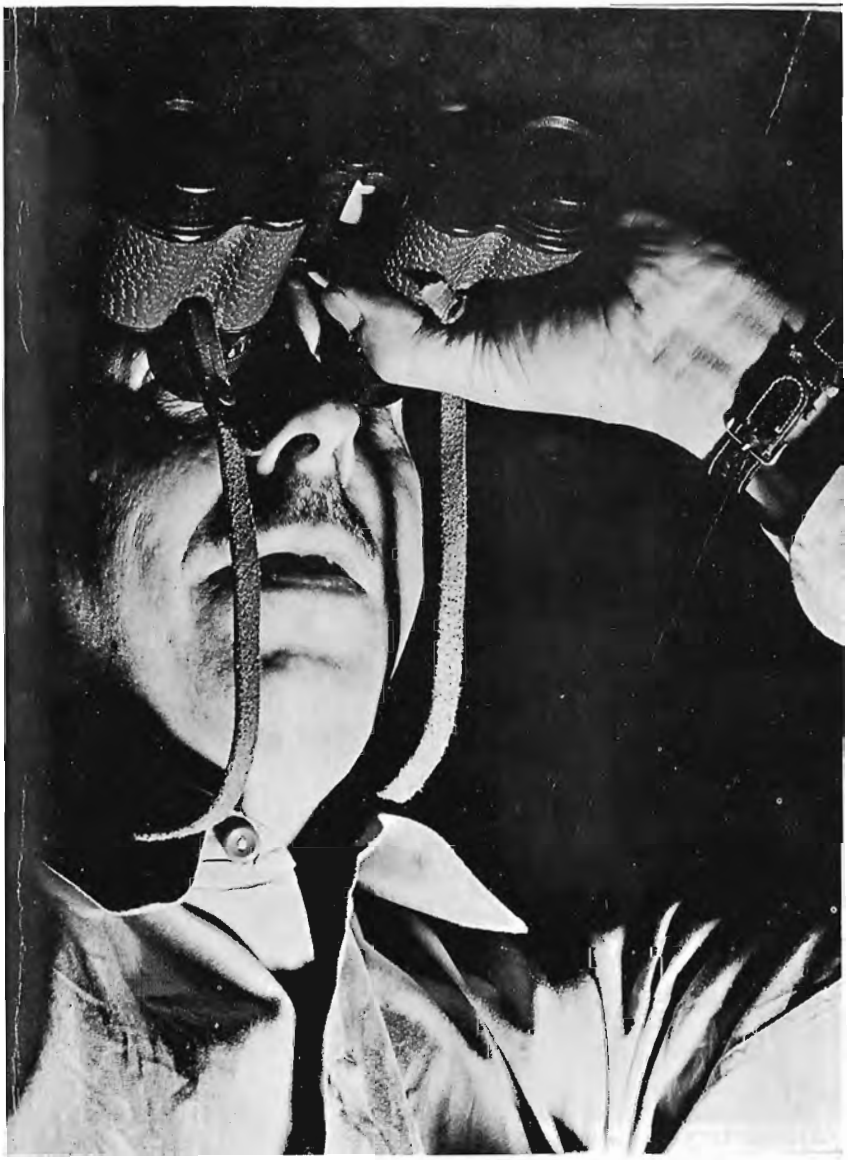


Fig. 13.3. — Muroc Army Air Base

Fig. 13.4. — Carlsbad Army Air Base

mand car and jeep. Everything went off on schedule, and shortly after dinner the bomber flew in loaded with shrieking, kicking bats. The airmen had taken delight in a form of hunting which consisted in swinging landing nets at the mouth of an inexhaustible cave, and the crates were all jam full. The crates were loaded into the truck and the refrigeration turned on full tilt. But the howling went on without abate for a couple of hours, and it became evident that the refrigerating unit was not adequate to cope with such a large amount of body heat all of a sudden. So we mounted a series of fans in positions to blow air in over cakes of ice. Finally, about midnight, the noise ceased; hibernation had been accomplished. A night watch of soldiers took over, and we turned in.

The next day the bats were still nicely quiet and we started a trial with the lightest of the dummy bombs. A first batch of bats in hibernation with weights attached was dumped out of the bomber at a low altitude, 2,000 ft. as I recall. The ground crew scurried around in jeeps and eventually located a group of free-fallers large enough to show that few if any of the bats had come out of hibernation. Other batches were released from higher and higher altitudes, which made reconnaissance increasingly difficult. Eventually it was clear that the bats were not in hibernation but dead. Our cooling had been too efficient, too sudden.

In flying to Carlsbad, the Colonel had landed at the Carlsbad Army Air Base, and had seen there a suitable test area. So we all flew over to Carlsbad in search of facilities for testing close to the source of testees. The Colonel and I called on the Commanding Officer, showed him that our orders were marked secret, and outlined the facilities required for a project we could not reveal but which, we could say, was then sponsored by the Air Corps. We were in luck. An auxiliary landing field had just been constructed in a remote area, and it was complete with observation tower and administration building. It was ours. We housed the incendiaries and other equipment in the newly built wooden office building, learned by experimentation the proper technique for putting bats into hibernation, and proceeded with the trials. The results were encouraging. The carrying power of a 10–11 gram bat is indeed amazing, some 15–18 grams; the incendiary bomb was in this range (17.5 grams). Bats can carry such loads for miles. And bats with dummy bombs released in housed areas dragged the loads into sites highly favorable for fire-starting. We released bats successfully at various altitudes both from the B-25 and from an open Attack Bomber, in which flying was great fun. The smoke bombs functioned satisfactorily and provided further information. Col. Epler and the other officers all favored a full-scale trial with live incendiaries to be injected for a 10-minute take-off just before

release. I considered a live test highly hazardous and likely to lead to disclosure of the project. I also thought it unnecessary. But the officers insisted that a report to the CWS and AAF chiefs would be incomplete without it and so, on a Saturday, a live test was scheduled for the following Monday.

On Sunday Bill Young and I persuaded AAF photographer Lt. H. L. Dean and a few others to go out to the private flying field to take pictures illustrating the various steps. One (Fig. 13.5) showed injection of corrosive fluid into the pencil, another removal of the safety band, another painting of the case with acetone to soften the plastic, another attachment of a bomb

Fig. 13.5. — Professor (later Vice Chancellor) W. G. Young injecting timing fluid into a bat bomb

to the breast of a bat by slight hand pressure. Each scene was shot two or three times with a different operator to provide a choice (and possible souvenirs). For the final scene, a bat equipped with a bomb was restrained from flight by an invisible thread and brought out of hibernation. The pencil was injected with 10-minute fluid and the bat placed on a piece of sagebrush. The photographer made ready to shoot and the rest of us were crouched around the sagebrush. Then someone glanced over his shoulder and saw smoke. The administration building was on fire. We saved the refrigerated truck parked alongside, but nothing else. The building, all our supplies, and some valuable photographic equipment, went up in smoke. The observation tower was a mesh of twisted steel girders. The test was over, and the Colonel and I had the unpleasant duty, in thanking the C.O. for his hospitality, of reporting the accident. The C.O. was not greatly concerned; after all, it was a secret project. Probably what had happened in the confusion was that one operator placed an activated and armed bomb on a rail outside the building and another, unknowingly, picked it up and placed it indoors.

The accident really did not change the status of the project, which now seemed to me very promising. Warren Lothrop felt the same way, and Leeds Northrup accepted a contract to develop an improved time-delay pencil, which was all that was needed to complete a satisfactory incendiary. Bill Young kept in touch with a group of mammalogists at the University of Southern California and became enthusiastic about the plan. Interest by Service Groups lapsed for a time, but eventually the project was taken over by an aggressive group of Marine Corps officers headed by Lt. Col. R. H. Rhoads, who was charged with developmental work on the project for the Navy.

Still lacking was information on the effectiveness of the tiny incendiary in starting fires, but by this time excellent facilities were available at Dugway Proving Ground, Toele, Utah. As a means of securing accurate data for comparison of the M-69 with other bombs, the Standard Oil Development group engaged architects who had practiced in Germany and in Japan and arranged for construction of two test-structure groups. One target consisted of 6 adjoining German houses; the second contained 12 two-family Japanese houses. The wood used in the structures was chosen to correspond to lumber used in German and Japanese construction. Adequate supplies of materials were on hand for repair of the structures between tests. Tests for comparison of the M-69 with the CWS thermite Bomb are on record.[1]

[1] W. A. Noyes, Jr., "Science in the War. Chemistry," pp. 392–394, Little, Brown and Co., Boston, 1948.

When a bomb scored a hit and started a fire, the fire was rated as of A grade if it could not be controlled by a well trained and equipped pair of fire guards in 6 minutes. A fire which became ultimately effective if left unattended was a B fire, and one judged nondestructive was a C fire. In view of earlier comments about the two bombs entered in these first tests, a summary of results is of interest. This comparison struck a long overdue coup

Percentage of Bombs Starting Fires

Fire Classification	German Houses		Japanese Houses	
	Thermite	M-69	Thermite	M-69
A	0	37	26	68
B	18	16	14	13
C	82	47	60	19

de grâce to the thermite bomb. These and other Dugway tests were of considerable value in drawing up plans for the bombing of Japan.

On December 6, 1943 Gen. Kabrich requested the Commanding Officer at Dugway to schedule a test of the small incendiary bat-bombs in the Japanese village. Those present would include Col. Rhoads, Officer in Charge of the Navy project, Capt. R. M. Smith, Executive Officer, Capt. S. R. Leigh, Flight Officer, Dr. Young and myself. The tests were run off on December 17–19, 1943 under very severe conditions of temperature (15–39° F.) and humidity of the wood. No bats were included this time, but bombs were planted at sites known from experiences at Carlsbad to be likely points of delivery. Only a limited number of tests were allowed and, since no bombing was involved, a fire engine was used to extinguish bad fires in order to save the test structures, and the classification of each fire was judged by CWS Major Levis from experience gained in tests on bombs. It was evident that the Napalm gel used was too thin, for when an incendiary rested on a slanting surface or over a crack the gel flowed down the surface or through the crack; this fault could be corrected easily. Even so, the performance of the 18-gram bomb was good. Each bomb planted was, of course, a hit, and 29% of the hits resulted in Class A or B fires.

As shown in the table above, the M-69 bomb gives 81 A-B fires per 100 hits. Since the roof coverage in the Japanese village is 60%, 60 bombs out of each 100 will hit and produce 60 x 0.81 = 48.6 fires. However a feature of the M-69 is that a bomb may fail to score a hit and still eject gel onto a target and start a fire. The records show that 100 ejection hits give rise to 47 A-B fires. Ideally, every miss could be an ejection hit. Then the 40

bombs out of each 100 that score no direct hits could give rise to as many as 40 x 0.47 = 18.8 fires. On this idealized basis, the cluster of 60 M-69 bombs that is suspended from one of six "500-lb."-bomb stations of a B-25 bomber could start 40.4 fires. A shell of the dimensions of this cluster of 60 M-69 bombs would hold approximately 1030 bat bombs. In a release of bat bombs over Tokyo the percentage delivery on target should be extremely high. However, for a conservative estimate, suppose that the actual effective delivery amounts to only 50% and hence, on the basis of the results of the preliminary tests, 14.5% of the incendiaries taken on a mission should start fires, and a shell equivalent to the cluster of M-69s should give 149 fires, 3.7 times the yield from the M-69 cluster. Furthermore, whereas the M-69 cluster weighs 424 lbs., the bat-bomb cluster occupying the same space in the bomb bay would weigh only about 100 lbs. Since the light clusters do not have to be aimed, additional clusters could be carried in the cabin of the aircraft. There is space in a B-25 for as many as 50–60 light clusters. Allowing an outage of 50% for extra fire power and auxiliary gasoline tanks, each B-25 could carry 25 light shells and hence assume responsibility for starting 3725 class A and B fires.

I wrote a report of the results and calculations in Los Angeles on December 20, 1943, after saying good-bye to Bill and Helen Young in Santa Monica. He and I had an adventure on the return trip. The Marine officers were returning to the Marine base at El Centro, California by a Navy plane and offered to drop us off at Santa Monica. Difficulty in starting the motors in the frigid Utah weather held up the take-off for two or three hours. During the wait a CWS officer who was working on vesicants learned that Young and I were heading for the Los Angeles area and asked us to deliver a wooden box marked "POISON GAS" to an NDRC group at the University of Southern California. We agreed to do so. At the end of a pleasant flight, Young and I were up front enjoying a beautiful view of the valley and then of the coast and after a time the pilot indicated the location of the landing field for which we were heading. We saw nothing but houses. It was the carefully camouflaged landing strip of the Douglas Aircraft Co. Afraid of trouble in restarting the motors, the pilot decided to merely slow down enough for us to hop out, rather than make a full stop. We waved good-bye and then realized that we were in a ticklish position. We had no papers authorizing our presence in a plant of the strictest security regulations; we had no military escorts, and we could hardly conceal a sizeable box and its identifying marking. Luckily a truck came along driven by an amiable sergeant who was headed for the gate, and he offered us a ride. Young's house was not far away and our obliging savior drove us

home. We put the box in the garage, greeted Helen, and went upstairs to wash. After a time we heard Helen speaking to someone at the door: "No, there are no doctors here. No box of poison gas." It was the Douglas security officer. Bill had an inspiration. He took out of his pocket a letter from me to him about the Dugway test, opened it only far enough to show the SECRET stamp, and said "I'm sorry, but we cannot show you our authorization and orders on *this* project."

The Marine group had become enthusiastic about the use of bat vectors. One useful improvement suggested by Col. Rhoads was to make the underside of the bomb casing concave to provide easier and more secure attachment to the bat. Since the departure of Adams from the work, NDRC had rechristened the Adams Plan and called it "X-ray." Dr. George A. Perley of Leeds and Northrup, devised for X-ray the time-delay pencil shown in Fig. 13.6, a little gem. Seventy-five percent of this 8-g. device is made of a combustible magnesium alloy. Withdrawal of the striker release pin drives a striker into a tiny glass ampoule and allows a solution of electrolyte to

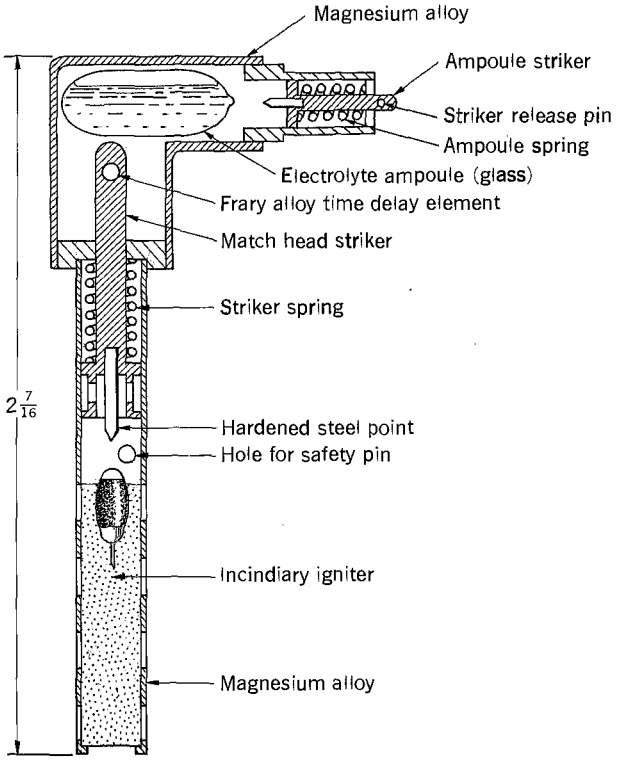


Fig. 13.6. — Perley Time-Delay Pencil

start corroding a bar of Frary alloy held in shear by compression of a spring. On release of this second spring, provided the safety pin has been withdrawn, a steel pinpoint is driven into the match head of an igniter of the type used in the Pocket Incendiary. By February 17, 1944, Leeds and Northrup was ready to place orders for the steel molds, dies, and drilling jigs required for large-scale production of the pencils. On March 3, 1944 Warren Lothrop stated that:

> "Perley estimated a six weeks' lapse between notification and production of 4,000 delays and a three months' lapse for production of 100,000 units."

On December 21, 1944, the day after Dr. Young and I had returned from the Dugway test, I went out to Hollywood for a conference with Larry Crosby of the Larry Crosby Research Co., an engineering firm set up jointly by Larry and Bing Crosby. The conference was about a container, or shell, or bomb, to be loaded with bats in hibernation, with incendiary bombs attached. My records are incomplete, but my recollection is that Dr. Young made the first contact with Larry Crosby Research Co., that Col. Rhoads then assumed responsibility for the design of the container, and that the Larry Crosby outfit came up with an acceptable design. The shell, which would accommodate approximately 1030 bomb-bat assemblies and would be mounted on the "500-lb."-station of a B-25 bomber, was a casing of thin steel with a rounded nose and an extendable parachute at the tail. On release from a plane, the parachute would open and allow the bomb to fall slowly into warmer air. The shell contained a series of circular trays, each about 1½ inches in height. The trays were divided into a series of rectangular compartments, each containing a bat in hibernation, with an attached incendiary. Each tray was connected to the trays immediately above and below it by cords allowing maximal separation of trays to a distance of about 2 inches. Wires which, on withdrawal, would activate the time-delay mechanisms of the bombs in one tray, were secured to the tray immediately above. On release of the bomb shell and opening of the parachute, the trays would be pulled 2 inches apart and all time-delay mechanisms activated. The safety pin of each bat-bomb assembly was secured to the bottom of the compartment in which it rested. As the opened bomb shell slowly descended into warm air, bats in succession would wriggle out of the compartments and fly away, and in doing so they would pull the safety wires and arm the bombs.

Imagine, then, a surprise attack on Tokyo in which a succession of bombers would operate at high altitude for about half an hour, say starting at midnight, each delivering a load of bat-bombs equivalent to some 3,700

fires. There would be no explosions or fire bursts to give warning, and the bombers would depart. With the activated mechanisms all set for a four-hour delay, bombs in strategic and not easily detectable locations would start popping all over the city at 4 a.m.

An attractive picture? All those working on the project thought so. Then, suddenly, X-ray was cancelled. I never learned the reason, but can make a guess. The bats would be vectors for bombs, but they would be vectors also for germs. Our side might be accused of initiating biological warfare. But the job was done very effectively by M-69s.

14. *The Book*

I HAD TAUGHT organic chemistry for five years at Bryn Mawr and for six years at Harvard before getting the first break of a half-year sabbatical leave. With our car aboard, Mary and I sailed on March 29, 1937 on the cruise ship Saturnia for Trieste. We had no set plan other than to start in Italy and move north as the season progressed, at a pace geared to the ripening of asparagus and strawberries. Decisions were made from one day to the next, for reservations were not needed. In the course of touring sixteen countries, we took in our share of museums and galleries and shops and had many pleasant holidays. There was sailing at Portofino, Italy; fine beaches at Pareggi and San Remo, Italy; Gairlock, Scotland; Saltsjöbaden, Sweden; Esjberg, Denmark. Fishing for trout was good at Titisee in the Schwarzwald, Glenurquhart, Scotland, and Skei, Norway. In Norway and Finland we fished for salmon, sea trout, brown trout, and grayling.

But the trip also had a professional flavor, for we visited many chemical laboratories, particularly those devoted to research on the newly discovered steroid sex hormones or on vitamin D, for a monograph I had written on steroids had been published just a year earlier. Seven of the chemists we met for the first time were, or were to become, Nobel Prize winners. Two of them, Hans Fischer and Heinrich Wieland, together with their wives, took Mary and me on a pleasant mountain-climbing excursion south of Munich. Furthermore, I kept in touch by letter and cable with my research group back in Cambridge throughout the trip.

The situation encountered during the war was quite different. I had to give up all teaching, terminate a 10-year program of research on carcinogenic hydrocarbons, and stop thinking and talking about chemistry. War projects took me on 197 out-of-town trips in this country and one abroad, and there were many delays in transportation, with waits in hotels or stations. Projects at arsenals and other military bases always involved delays; the bomber would be out of order or held up by weather; the bombardier could get no hits; or it would be Sunday and the military men would not work. I hate to be idle; I like to teach and I regretted being separated from teaching. So, on April 1, 1942, I started working on a textbook of organic

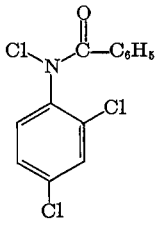
chemistry. Mary had agreed to keep me supplied with literature notes to take along on trips, or to work on in evenings at home. Her supply soon exceeded my rate of production and she took up authorship and wrote some of the best chapters in the book.

I made it a rule to turn out an average of at least four pages of manuscript per day, regardless of the day, the location, or the circumstances. The manuscript, annotated with dates and places of writing, looks like a travel diary. Typical locations are as follows:

Indianapolis RR station
Train to Columbus, Ind.
Palmer House, Chicago
Train to Hunstville Arsenal, Ala.
Barracks, Huntsville Arsenal
Hotel Lord Baltimore
Jefferson Proving Ground
Plane, Indianapolis to Boston
Coach from Albany to Boston
Back Bay RR station
Federal Express
Coach, New York to Boston (sitting on a suitcase in the vestibule)
Pittsburgh Airport
Planes to and from West Coast (flights of 31–52 hours)
Muroc AAF base (wrote on the desk in a reconnaissance car)
Carlsbad AAF base
Biltmore Hotel, Los Angeles
San Jose, California

The last of my chapters was one on steroids, and much of the material for this could be taken from my monograph, and so I saved this job for the first western trip of about two weeks on the Adams Plan. The trip offered many good opportunities for writing, and seven evenings at home sufficed for completion of the chapter. Thus my chapters of Fieser and Fieser were finished on June 12, 1943 and the book was published on July 21, 1944.

While the writing was in progress, I attended a meeting of NDRC Division 19, to which I was a consultant, and listened to reports on the impregnation of clothing with chemicals to provide protection against war gases. The British had developed a good reagent against mustard gas known as British Impregnite, and prospective new decontaminants were compared with this substance as standard. It was my understanding from the discussion that this British development had been made between wars and was open information, and so I made a note of the formula and put it



British Impregnite

in our book. Several weeks after the book appeared I had a call from Washington stating that the book had disclosed a military secret and asking that I consult with the publisher on the possibility of recovering all books that had been sold or given away to teachers. I made a trip to Washington to seek help from Capt. Benny Bull, able CWS attorney who had taken his Ph.D. degree at Illinois under Roger Adams and then studied law. Benny recalled seeing something pertinent in *Chemical and Engineering News*, and a brief search located the item. This magazine sometimes publishes a "Who Makes It" list of chemicals in demand and not offered by usual suppliers. The list in question carried the chemical name of British Impregnite and hence constituted a disclosure prior to mine. The request had been submitted by a junior CWS officer at Dugway Proving Ground; presumably he had been assigned the problem of preparing a batch of the material and was looking for an easy way out. In any case, I was off the hook.

The book has gone through several editions and has been translated into Chinese, German, Indonesian, Italian, Japanese, Polish, Russian and Spanish. It may be described as an implement for peace which arose as a by-product of war.

15. *Destruction of Documents in Danger of Confiscation*

OUR GROUP learned at one stage of the war of an OSS requirement for a device to be carried by an operator or agent in enemy territory for the rapid destruction of notes or incriminating documents in danger of confiscation by the enemy. The problem appealed to E. B. Hershberg, who followed up suggestions of CWS officers and developed MESSENGER POUCH DESTROYERS of two designs, one of which is shown in Fig. 15.1. The celluloid case is made by cementing together two identical top sections of the Pocket Incendiary. Kraus mixture, the incendiary, is made up in the form of two briquettes molded to fit into the left-hand half of the case. A charge of explosive and a time-delay element fit into a cylindrical insulator block which occupies the right-hand side of the case. This block is made of Marinite, a specially hardened preparation of diatomaceous earth, which is strong and easily machined. The design of the explosive train is shown in Fig. 15.2. Slow-burning Ensign-Bickford pellets are molded in such a shape as to make them interlock and ensure transmission of fire from successive units of the train. The explosive charge of pelleted tetryl weighs approximately 4 grams.

The destroyer is operated by a pull on the ring of the fuze lighter to withdraw the cotter pin, and the unit is placed in the messenger pouch containing a folio of documents. After a delay of about 5 seconds, ignition occurs and the Kraus mixture burns up in about 45 seconds. The conflagration proceeds vigorously for about 2 minutes and then, as the fire begins to die down (2½ minutes), a violent explosion blasts all parts of the original assembly beyond recovery or recognition.

The device was described in a report of December 21, 1943. The record of subsequent events is not clear, but apparently the explosive-type destroyer was abandoned as the result of tests conducted by OSS. Whether the destroyer tested was that designed by Hershberg or a similar destroyer of British design, I do not know. In any case determined OSS men, either

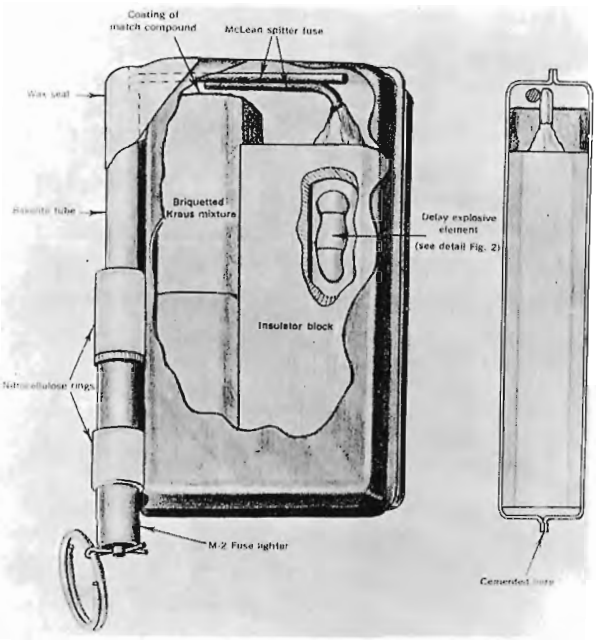


Fig. 15.1. — Messenger Pouch Destroyer

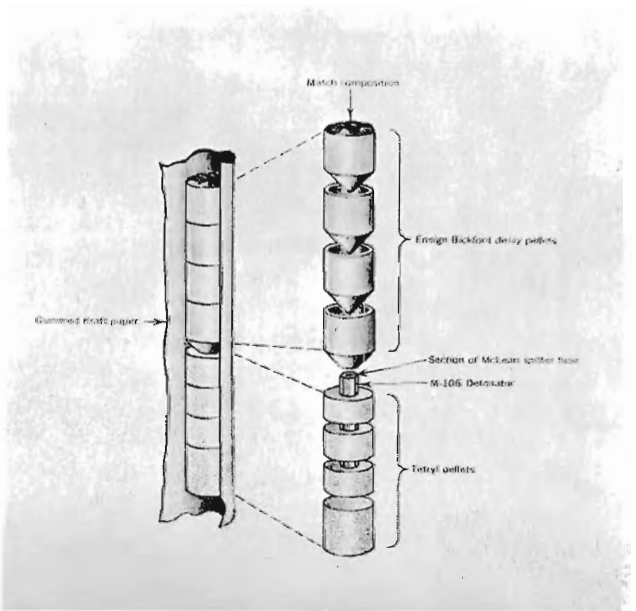


Fig. 15.2. — Delay Explosive Element

Fig. 15.3. — Destructible Notebooks

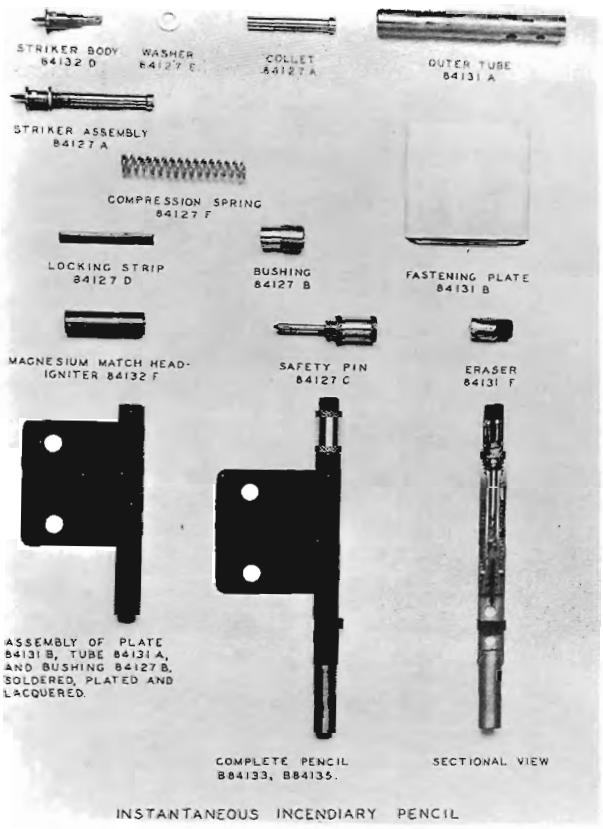


Fig. 15.4. — Igniter for Destructible Notebook

THE PYROFILM NOTEBOOK IN ACTION

FIGURE 2.

Fig. 15.5

Fig. 15.7

defying the light explosive charge or ignorant of its existence, were able to recover the ashes within the delay period and decipher the writing.

So E.B. went to work again and compounded the incendiary plastic Pyrofilm from equal parts by weight of celluloid and finely powdered sodium nitrate. Pyrofilm resembles ordinary celluloid in appearance. Ignited by a match in the open, it burns steadily but feebly, with liberation of droplets of sodium nitrate. When Pyrofilm burns in contact with paper, the seething molten sodium nitrate enters into reaction with the organic material, robs the paper of its structure, and produces an unidentifiable ash. The notebooks shown in Fig. 15.3 are loaded with Pyrofilm, 2–3 sheets of note paper, Pyrofilm, etc. The igniter pencil is built to look like an ordinary pencil and is equipped with an eraser. Details of construction are shown in Fig. 15.4. E.B.'s photographic sequence showing the Pyrofilm notebook in action (Fig. 15.5) requires no comment except that the "agent" of Scene 4 is Walter E. Colburn, handyman on the project. Instructions supplied for use of the gadget are shown in Fig. 15.6.

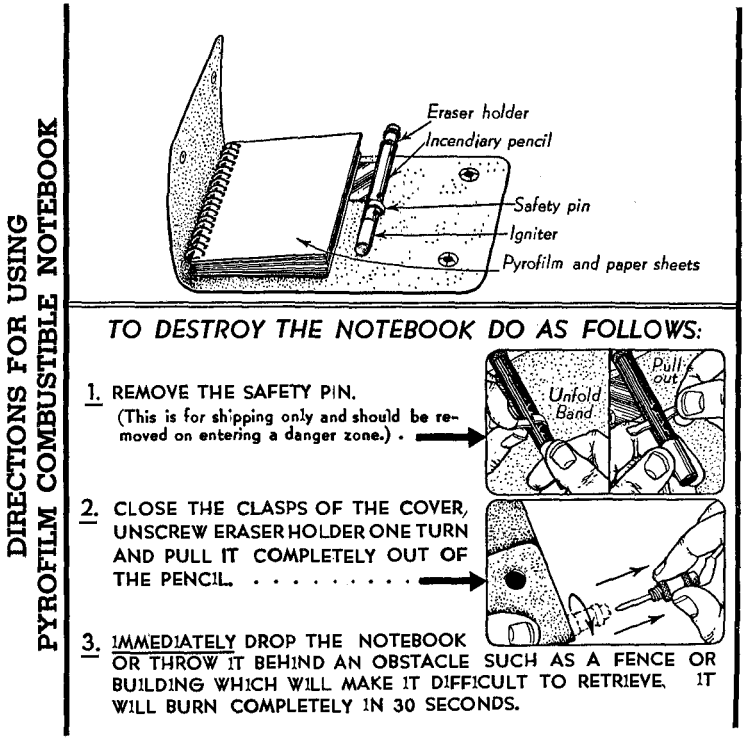


Fig. 15.6

Enthusiastic (and artistic) E.B. devised also the briefcase model shown in Fig. 15.7. The design and method of operation followed suggestions from DeWitt Clinton. The envelope, an accordian-type fiberboard folder (Expanding Wallet), carries an igniting pencil and contains five sheets of Pyrofilm, each surface-coated with celluloid lacquer. Four sets of secret documents of not more than 17 sheets each are inserted between the Pyrofilm sheets. The envelope is carried in a brief case and the safety strip is removed on entrance into a danger zone. For operation, the envelope is removed from the brief case, the eraser holder is unscrewed one turn and pulled out, and the envelope is thrown behind a fence or other obstacle which will impede retrieval. The igniter sets fire to the Pyrofilm sheets, and destruction is complete in 45–60 seconds.

16. *The E-19 Bomb*

AMUNITION ONCE accepted carries an M (for MARK) number (a son born to Warren and Margot Lothrop during the war was named Mark Lothrop). An E-number means that a munition either is in the experimental stage or is making a final bid for acceptance. The E-19 bomb was completed late in the war and was not tapped for the elite M-society. But it was a fine bomb.

Work on this development started in the summer of 1942, and a 70-page final report is dated June 15, 1945. This report was prepared by E. B. Hershberg and lists the participants as: Norman F. Thompson and Morrill Dakin, of the Factory Mutual Research Corporation, Norwood, Mass.; Myles Morgan and Strickland Kneass, Jr., of the Morgan Construction Co., Worcester, Mass.; Hershberg, and myself. Many other individuals and several other companies participated as well, but I am afraid that I cannot acknowledge all contributors in a brief account of the project.

In April, 1942 Hershberg discussed with our group the possibility of combining a tail filled with white phosphorus with a magnesium bomb body. His first idea was to utilize as a canister for phosphorus the empty tin tail of the CWS M-50 thermite bomb. Magnesium castings which Earl Stevenson had had made up with the idea of developing a larger and more effective thermite bomb were used in a first trial. The castings were hexagonal cylinders, open at both ends, about the size of the M-69 bomb. E.B. filled some of these with thermite and mounted at one end a tail-container of white phosphorus with a central blowout tube which would be burned open by fire from the bomb with liberation of the phosphorus as a screening smoke of phosphorus pentoxide. The performance was promising, and in August we put in an order to the American Aluminum Co. for magnesium bomb bodies of two types, one with a pointed nose and the other with a flat nose. The castings were stamped HU-1 and HU-2. A tail assembly was made up at Noblitt-Sparks according to a scheme originating with Hershberg and perfected by Edmund Ludlow in consultation with E.B. While the parts were being fabricated, we had many discussions about the ultimate filling and assembly with Norman Thompson and his Factory

Mutual group, and they demonstrated several burning tests in various containers simulating a bomb body. None of Thompson's proposed assemblies seemed to us practical, but we did like his idea of combining a magnesium casing with a charge of high boiling oil or grease. Thompson had various schemes for applying the idea, such as having a core of oil encased in celluloid, but none seemed adaptable to a manufacturing operation. I think I was the one to suggest using a hard material such as rosin which could be cast into a central core without requiring any container. This was tried out on November 3–9 and found successful, and Hershberg was able to prepare completed bombs for a demonstration at the Factory Mutual test station, then in Everett, on November 10, 1942 before an assembly of CWS and NDRC groups. This bomb, with a magnesium casing and a rosin core, was a complete, functioning assembly (Fig. 16.1). Some were fired by impact onto concrete, some were fired statically in FM test structures. Everyone was impressed with the vicious flame produced by the combination of magnesium and rosin. An objective appraisal is contained in a letter from E. P. Stevenson to R. P. Russell:

> November 16, 1942. — ". . . the bomb is more than twice as effective as the M-50 (thermite bomb). The test structures at Everett have been fired at a distance of 30 inches within a minute, while with the M-50 considerably longer time is required at 15 inches and even then the blaze is quite feeble.
>
> "Dr. Fieser and his group are also studying the problem of ballistics. He proposes to carry out tests in the Harvard wind tunnel and, as arranged with Slim (Myers), probably will ask for use of the mortar at Bayway."

Guidance was indeed obtained from tests in the Harvard wind tunnel (R. von Mises and G. Kuerti) and in the Bayway mortar. The E-19 had been made to the exact dimensions of the M-69 so that it could be fired in the same mortar and clustered with M-69 equipment. Hershberg had misgivings about the strength of the thin-walled magnesium casting and thought that a perforated steel sleeve insert might provide the best method of reinforcement if it did not decrease the burning power too much. Thompson presented this plan at a conference with several members of the division and there was general agreement that Thompson and Hershberg should go to Cleveland and arrange for production of a trial batch of reinforced castings. Hershberg alone was able to make the trip. On November 17 he arranged with American Aluminum Co. for the casting of magnesium over a perforated steel insert to be fabricated by Noblitt-Sparks.

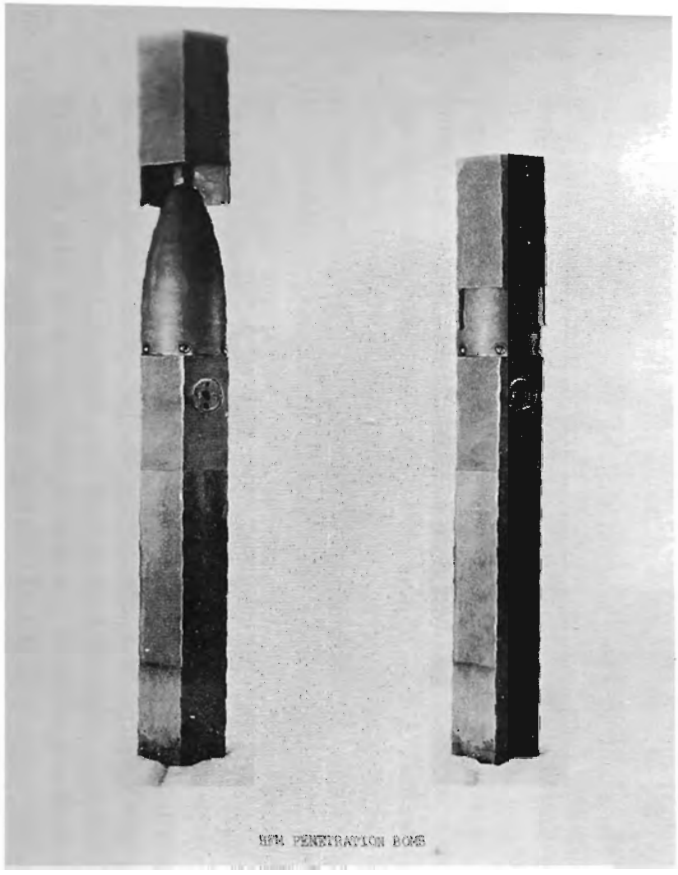


Fig. 16.1. — Early Version of E-19 Bomb
(The tail is held extended in flight by a spring: for assembly into a cluster, the spring is compressed. This scheme makes room for *more incendiary* material.)

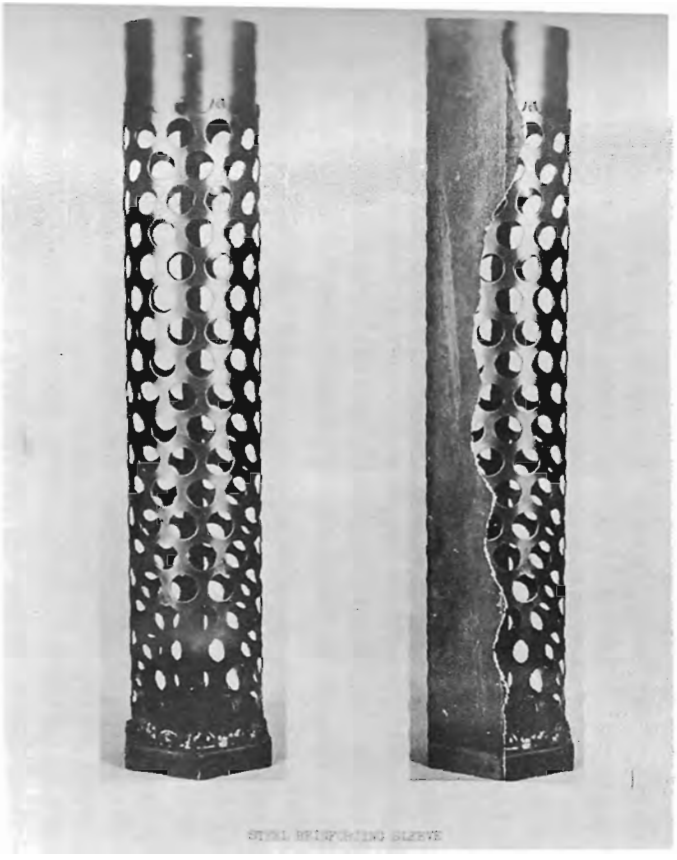


Fig. 16.2. — Steel Reinforcing Sleeve

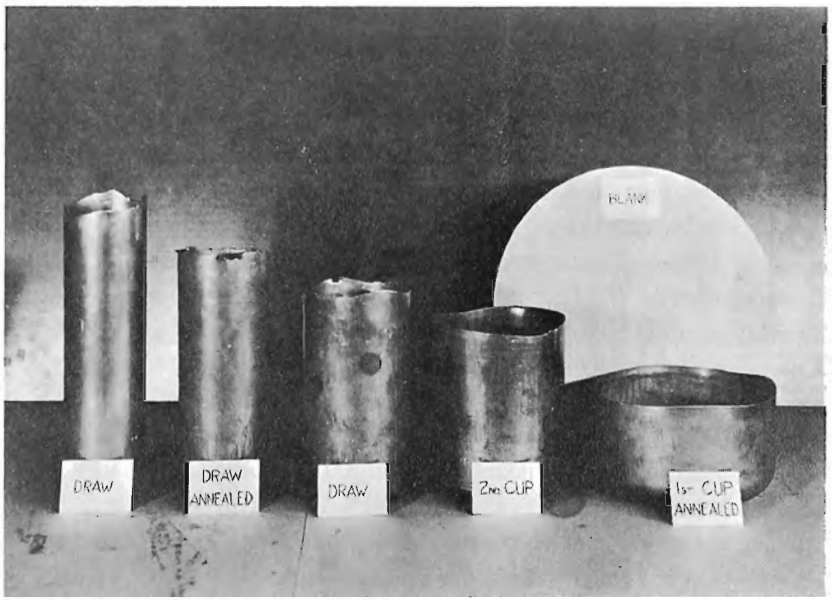


Fig. 16.3. — Forming the Steel Jacket (right to left)

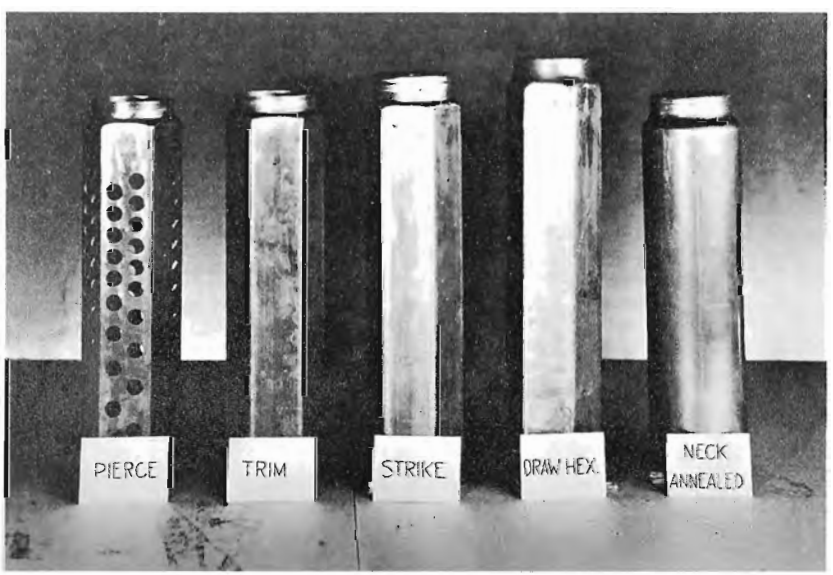


Fig. 16.4. — Next Steps in Forming the Jacket

The first castings were made on November 25 (Fig. 16.2). By this time Hershberg had located a better core material in the form of a black wax concentrate obtained from Kendall Refining Co.; this could be melted (m.p. over 300° F.) and cast by pouring. The first burning tests were very gratifying; the steel insert, far from interfering with the burning, had an unexpected beneficial effect. The magnesium oxide produced on combustion forms a plug at the ignited tail end of the bomb and pressure builds up sufficient to blow out through the perforation holes long flames consisting of burning particles of magnesium and burning gas formed by cracking the hydrocarbon core. We had not seen this effect in any previous assembly.

By May 8, 1943, when the rapidly improving bomb was demonstrated to American and British Service groups in the new Factory Mutual test station at Norwood, Mass., the name had changed several times: H-FM Bomb, E-1 Bomb, E-1 PERTA Bomb (a count showed the presence of 10 major chemical elements, nearly a whole PERiodic TAble). After the sudden ending of the Adams Plan tests at Carlsbad, I looked into facilities on the West Coast for experimental and production casting of magnesium and obtained good help and advice from an able scientist in uniform, Lt. Herbert Gutowsky [1] of the CWS Procurement Division. He advised me to go to San Francisco, briefed me on the CWS Procurement officers there, and paved the way for a cordial reception. Some of the officers went out with me to the Permanente plant to witness the tests on goop bombs, and Capt. Etzler took me to American Radiator and Standard Sanitary Corporation at Richmond for a talk with manager Howard I. Detro. Detro's outfit had done some very fast work in getting the M-50 into production, and we were looking for ideas on the production of the castings with inside reinforcing sleeves. The castings shown in Fig. 16.2 had been made with a temporary mold, but the American Aluminum people were afraid that permanent molding over the insert, necessary for production runs, might present difficulties. Mr. Detro agreed. One of the CWS officers suggested that the steel reinforcing sleeve be on the outside of the magnesium, and since this seemed to be an interesting possibility I left an order with Detro to make a mold and turn out a few trial castings. Although the job required fabrication of steel sleeves and nose pieces, as well as making the mold and doing the casting, Detro telephoned ten days later saying that he had made some initial castings and that the method seemed entirely feasible for production.

Tests on the California castings showed that the bomb burns satisfactorily

[1] Herbert S. Gutowsky, b. 1919 Bridgman, Mich.; A.B. Indiana, 40; Ph.D. Harvard (Kistiakowsky), 49; Univ. Illinois.

Fig. 16.5. — Assembly of Magnesium Casting, Jacket and Nose

Page No. 244 ~~239~~

Operators: Dahin, Hershberg, Scott — Test No. 145 — Date Jan. 27, 1944

Location Norwood

Observers: Preece, Fieser

Incendiary and Explosive Loading	Details of Bomb Construction
Tail: Live	Short - drawn cup
Diaphragm Chamber and Propellant:	
Black powder, g. 5 g	
Smokeless powder, g. 1 ring	
Fuze: Live	
Body: #24 mix Sealed SS-50	Casting:
Sleeve:	Gauge:
Nose: Forged Cr, Mo - not undercut	

Weight of Component Parts
UNITS:

Nose. - - - - - - - - - - - - - - - -
Body. - - - - - - - - - - - - - - - -
Filling (Mix #) - - - -
" (Mix #) - - - -
Hydrocarbon Core. - - - - - - - -
Fuze. - - - - - - - - - - - - - - - -
Diaphragm. - - - - - - - - - - - -
Propellant. - - - - - - - - - - - -
Tail. - - - - - - - - - - - - - - - -
TOTAL:

Mortar Firing Data

Charge: 35g + 5g squib - Smokeless

Depth of propellant chamber:

Angular displacement at ..... R.P.M. 31.3 degrees.

Velocity, 403 ft./sec.

Fig. 16.6

with an external reinforcing sleeve. However, the method of production ultimately found most satisfactory was simple insertion of a magnesium casting into an external steel sleeve. Figs. 16.3 and 16.4 show steps in the fabrication of the steel jacket, conducted by Noblitt-Sparks; assembly of the bomb parts is shown in Fig. 16.5.

Tests conducted at Bayway in a mortar with which bombs could be fired at known velocities onto concrete slabs showed that the E-19 bombs of early design needed improved impact strength. Bayway was too remote for any but a few initial tests, but Norman Thompson installed at Norwood a bomb-firing mortar patterned after the latest Standard Oil Development model. Mortar tests, recorded on data sheets illustrated in Fig. 16.6, soon showed that a bomb with an external reinforcing sleeve, whether cast integrally with magnesium or joined by slip fitting, is stronger than one with an internal sleeve. However, even with this improvement, the bomb did not withstand impact on a 10-inch concrete slab at velocities much over 60% of the terminal velocity of about 700 ft./sec. At this point engineers Myles Morgan and Strickland Kneass of the Morgan Construction Co. in Worcester designed and had fabricated a succession of devices to be fitted onto the nose of the bomb for improvement of impact strength. The most satisfactory design was the nose compression cup illustrated in Fig. 16.5. This nose cup adds little to the weight or length, but is capable of absorbing a large proportion of the impact energy. With this improvement, the bomb was found capable of functioning after impact on an imbedded concrete slab at terminal velocity. No significant change in ballistics was noted, and mortar tests indicated that the compression cup promotes penetration of concrete.

F. B. Hershberg encountered and solved a succession of problems in the design and assembly of the component parts of a highly intricate mechanism. He lined up one firm for the manufacture of this piece and another for that, and got the E-19 into trial production at the National Fireworks Co. for the supply of bombs required in increasing quantities for a succession of tests. I witnessed bombing tests at Edgewood and at Dugway and backed E.B. up when he needed it, but on the whole the problems in mechanics and technology were beyond my scope and he handled the whole job of standardization and production practically singlehanded.

So let us turn to Hershberg's report of the E-19 in its final form, Fig. 16.7. The drawing shows the assembly of the bomb body parts and nose cup already illustrated in the photographs of Fig. 16.5. Note the safety plunger on the horizontally mounted fuze (of modified M-69 design). When bombs are put up in a cluster, the plunger of one bomb is pressed

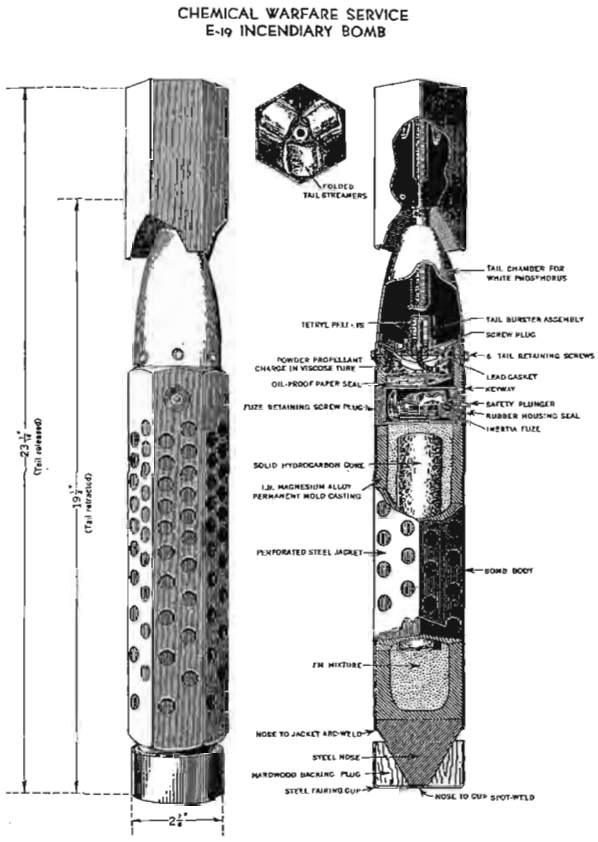


Fig. 16.7

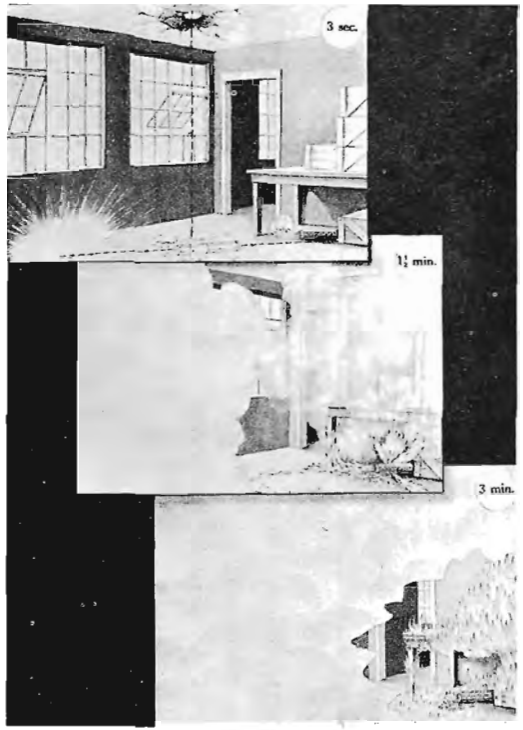


Fig. 16.8. — E-19 Bomb in Action

against the wall of a second bomb and so held in the safe position until the cluster is released. The filling, marked FM MIXTURE, which is loaded by compression around a central core of solid hydrocarbon wax, is a Factory Mutual concoction of flake aluminum, sodium nitrate, barium nitrate, sulfur, and oil. This is ignited satisfactorily by a flash of black powder from the fuze and burns with an intense heat sufficient to ignite the magnesium casing and crack the hydrocarbon wax to combustible gases. Internal pressure forces jets of intensely hot flame to issue from successive perforations in the steel sleeve for 3–4 minutes; residual flame and heat from the slower burning magnesium persists much longer.

The bomb is fired by an inertia fuze with a delay of about 3 seconds. A separating charge of powder propellant shears off the tail canister and ejects it in one direction and, at the same time, ignites the bomb and kicks it in the opposite direction with sufficient force to cause it to come to rest against a wall or other obstacle and thus assume a position favorable for starting a fire. The tail canister loaded with white phosphorus carries an explosive charge which operates a few seconds after the tail has been separated from the body, and produces an obscuring smoke screen that deters fire fighting. The explosion wave is sufficient to shatter windows and blow out doors and so provide a draft. Fig. 16.8 shows how the bomb operates.

Fig. 16.9 demonstrates the penetrating power of the bomb when shot from a mortar onto a slab of reinforced concrete. Shot No. 307 at 473 ft./sec. punched out a hole large enough for the bomb to go through, but the bomb bounced off. Shot No. 308 at 603 ft./sec. penetrated cleanly. Shot No. 309 at 570 ft./sec. struck too close to the edge and was deflected after causing considerable destruction.

These and similar mortar shots established that a speed of 500–550 ft./sec. is optimum for penetration of the heaviest type of industrial roof construction. Note, in Fig. 16.7, that the box tail is packed with three folded streamers. The bomb had good ballistics and flight stability without streamers, but the terminal velocity was too high and led to excessive penetration. The streamers were added to act as brakes and reduce the impact speed to about 500 ft./sec.

Penetration tests showed that in the majority of cases the tail stays in place even when a bomb penetrates a thick concrete roof or suffers abrupt setback on a concrete slab. However, in a glancing hit, the tail may be ripped off the body on impact, prior to operation of the separating charge. However, the firing system had been worked out in such a way that the bomb is ignited even though the tail is lost. Furthermore, the canister of white

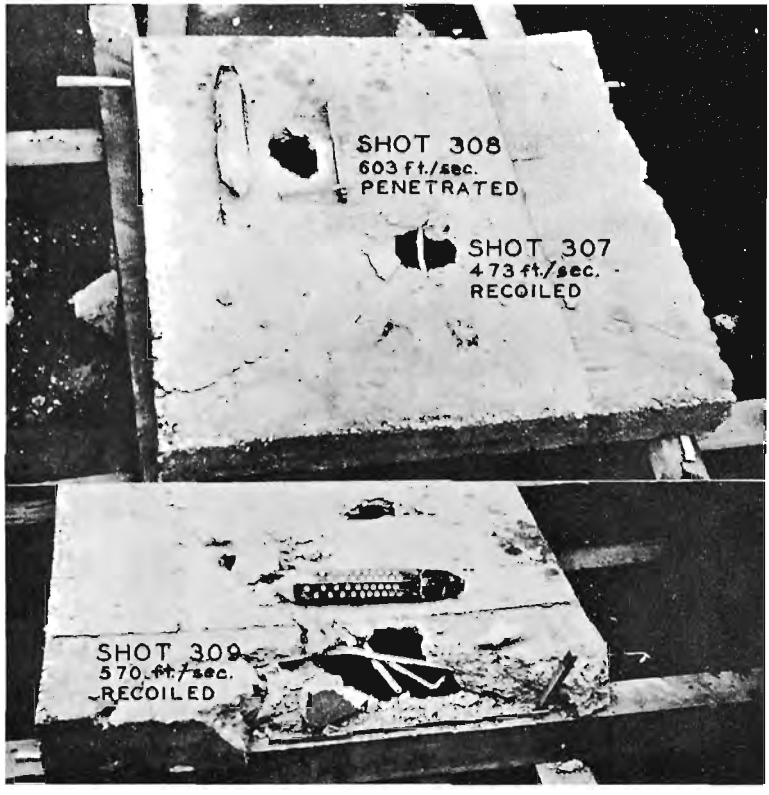


Fig. 16.9. — Mortar Shots into a 6-inch Reinforced Concrete Block

phosphorus is not necessarily wasted, for it retains its fuze and detonator and will operate if it is reached by the fire. Even if fuze or detonator is impaired, the tail is exploded by fire alone, with devastating effect. The sketch of Fig. 16.10 shows a tailless bomb functioning after penetration of a factory ceiling; the box tail was ripped off, but the canister containing phosphorus travelled through the large hole and presumably functioned later.

Figures 16.11–16.15 show component parts of the intricate mechanisms, and the assembly operations as carried out on the production line at National Fireworks. The layouts were all arranged by E. B. Hershberg, who also took the photographs. He had built a magnificent bomb.

The work had been done under Division 11 and, shortly after the end of the war, work on all projects pertaining to this division was declassified. The decision to throw open the data on the E-19 bomb and to scrap dies and

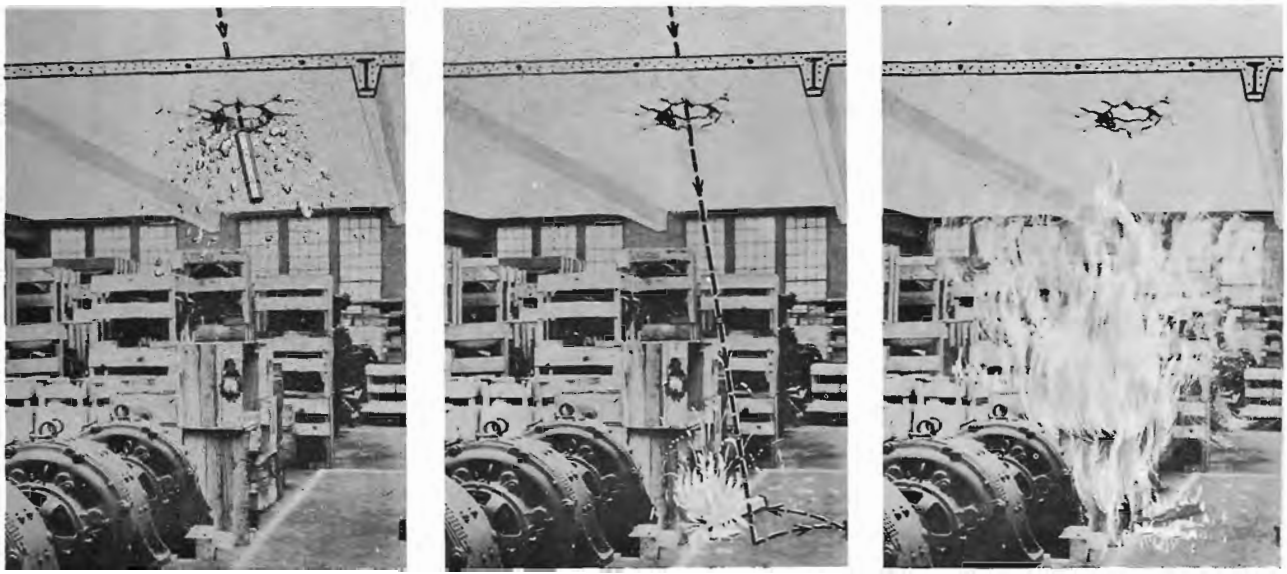
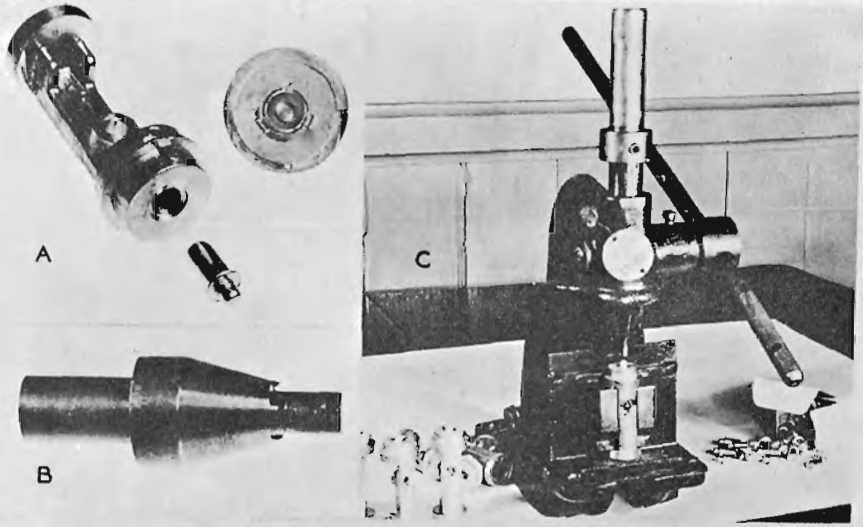
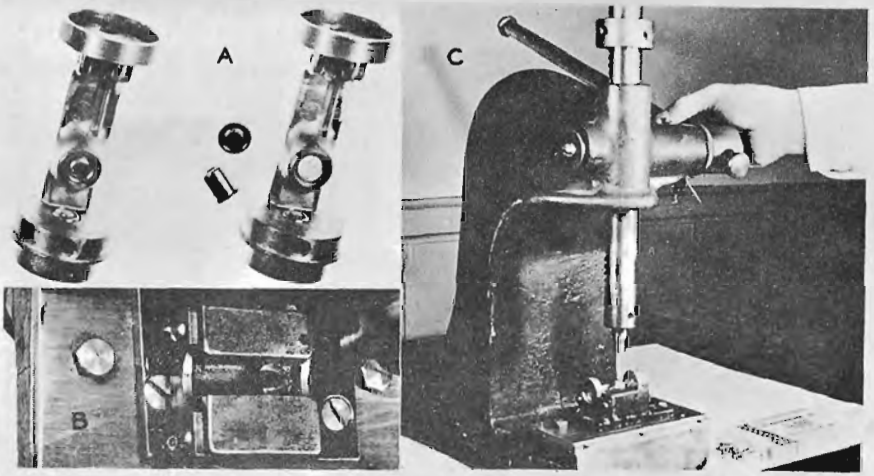


Fig. 16.10. — Tailless Bomb Preceded by Phosphorus Canister

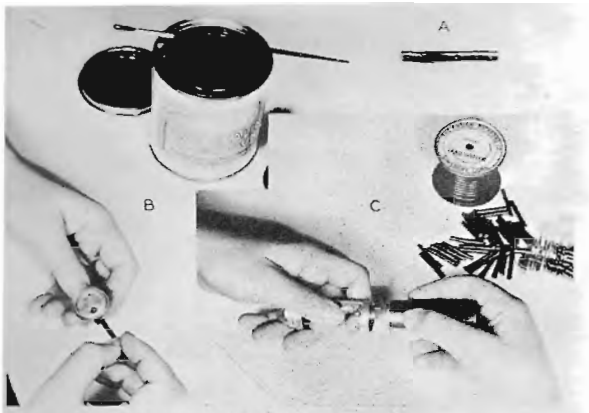


STAKING SAFETY PLUNGER INTO FUZE. A. COMPONENTS AND COMPLETED ASSEMBLY. B. STAKING TOOL. C. PRESS WITH FUZE HELD IN FIXTURE.

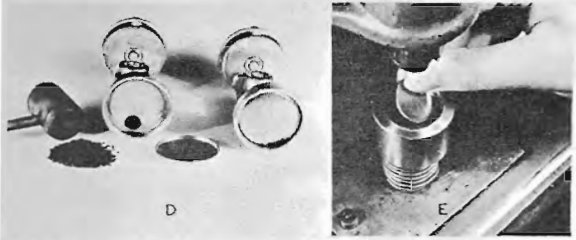


CRIMPING PRIMER INTO FUZE BODY. A. COMPONENTS AND COMPLETED ASSEMBLY. B. BASE HOLDING FIXTURE DESIGNED TO PREVENT DISTORTION OF FUZE AND WITH RELIEF VENT FOR ACCIDENTAL FIRING. C. CRIMPING OPERATION. NOTE THAT HAND IS PLACED TO GIVE LEVER A FLIP RATHER THAN SLOW PRESSURE.

Fig. 16.11

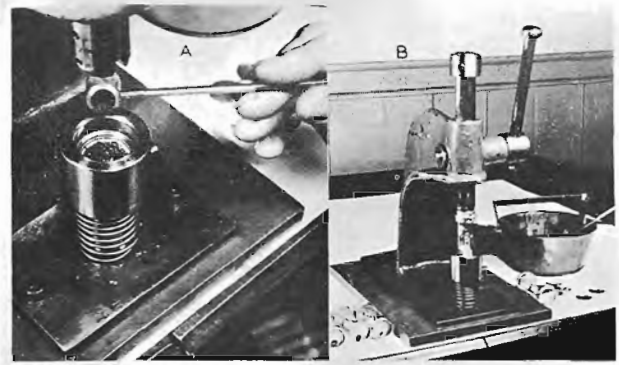


LEAD SPITTER FUSE ASSEMBLY.
A. FUSE CUT TO LENGTH AND PAINTED WITH SEALER.
B. INSERTING IN FUZE BODY.
C. POSITIONING.

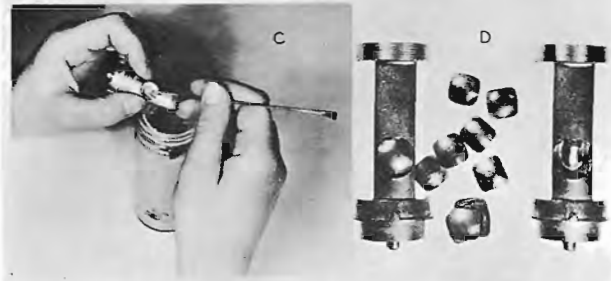


D. IGNITER CHARGE
E. INSERTING CHARGE CUP IN PRESS.

Fig. 16.12



A. ADDING MEASURED POWDER CHARGE.
B. PRESSING LOADED IGNITER CUP INTO FUZE BASE.



C. TIPPING END OF SPITTER FUSE.
D. ASSEMBLY OF PRIMER FLASH CHAMBER COVER.

Fig. 16.13

Fig. 16.14

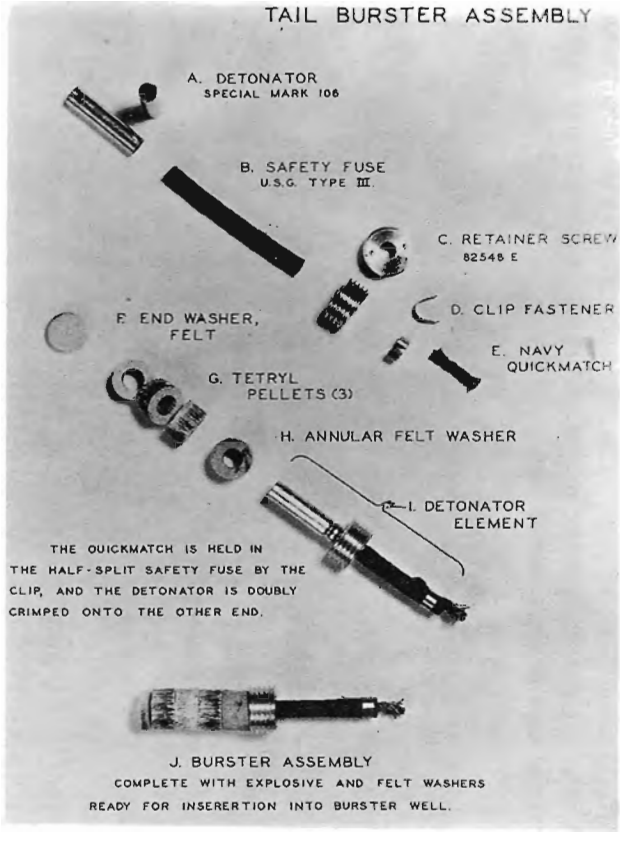


Fig. 16.15

molds worth many thousands of dollars probably eliminated any possible future utilization of a seemingly valuable advancement in incendiary munitions. War is gigantically wasteful, but that is only a minor charge that can be brought against the crime of war.

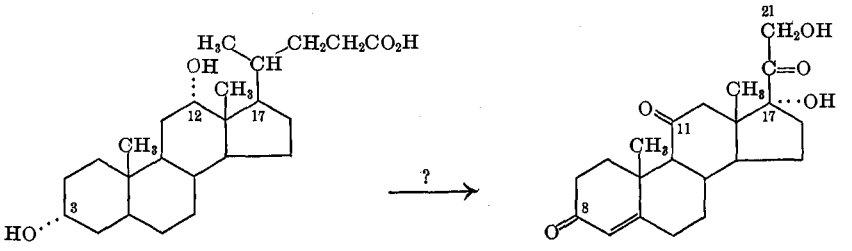
17. *Cortisone*

On removal of the adrenal glands of dogs by surgery, adrenalectomy, the animals die, on the average, after 7–10 days. If, immediately after adrenalectomy, the dogs are given an injection of a cell-free extract of beef adrenal glands the survival period is extended to 10–78 days. The response of cats is better. In experiments reported in 1930 by W. W. Swingle and J. J. Pfiffner, the average life span of 25 adrenalectomized control cats was 7 days, whereas animals treated with extract remained in perfectly normal condition for 40–80 days. "They eat, play, and fight with one another, and keep themselves sleek and clean." The cell-free extract must contain a hormone, that is, a chemical substance capable of being carried in the blood stream and necessary for normal functioning of the body. This one became known as the life-maintenance hormone.

Researches in 1936 by T. Reichstein in Switzerland, E. C. Kendall [1] at the Mayo Clinic, and Oskar Wintersteiner at Columbia University led to isolation from beef and hog adrenal glands of the hormone cortisone and a number of related steroids. Tested in animals, cortisone showed physiological activities of various sorts, but none suggested any use for the hormone in therapy. But a man cannot live without benefit of at least one of two small adrenal glands located just above each kidney; cortisone must have functions of importance. Clinical investigations might disclose uses for the hormone not evident from replacement experiments with animals, for not all afflictions of man occur in animals or can be induced in animals. For an adequate clinical study of cortisone for treatment of just one disorder a supply of about 100 grams of material would be required. In a typical isolation experiment, Reichstein's group processed 900 grams of crude concentrate from 1,000 pounds of adrenal glands derived from about 20,000 cattle and isolated 75 milligrams of cortisone. Hence the only way open for obtaining cortisone for clinical evaluation was by synthesis.

Reichstein had succeeded in elucidating the structure of cortisone in 1937–1948, and hence a target was available for application of methods of

[1] Edward C. Kendall, b. 1886 South Norwalk, Conn.; B.S. Columbia, 08; Ph.D. Columbia (H. C. Sherman); Mayo Foundation; Nobel Prize 1950.



Desoxycholic acid — Cortisone

synthesis. But this target was a particularly difficult one to hit. No other natural steroid available as a starting material for synthesis carries an oxygen function at position 11. Desoxycholic acid, isolable in quantity from ox bile was the most promising starting material. It has such a function at the 12-position; how transpose this function to position 11? How shorten the five-carbon side chain of the bile acid to the two-carbon side chain of the hormone? How introduce an OH group at position 17?

At the beginning of the war no answers were available to any of these questions. Hence a problem of extreme difficulty was involved when, in 1942, the Committee on Medical Research launched a program of co-operative research with the objective of finding a method for the synthesis of enough cortisone for exploration of its possible application to medicine. No wartime use for the hormone could be set as an objective other than one based on rumor. A German submarine returning from the Argentine had been captured and the cargo identified as adrenal glands; possibly the Germans were using extracts containing the life-maintenance principle for extending the endurance of airmen in sustained flights and in providing protection against blackout. After the war, Dr. Otto Bayer, whom I had known while at Frankfurt-am-Main on a travelling fellowship, told me that the submarine was carrying liver for work on vitamin B_{12} in his I. G. Farbenindustrie laboratory.

The principal participating groups were those headed by W. Bergmann at Yale, T. F. Gallagher at the University of Chicago, L. H. Sarett at Merck, E. Schwenk at Schering, E. S. Wallis at Princeton, and O. Wintersteiner at Squibb. The University groups operated under contract with and subsidy from the Committee on Medical Research. I had a small contract that took care of the salaries of Drs. Long and Turner; Dr. Blout had a research fellowship, and Fields and Ettlinger were graduate students. I

Anonymous Research No. 5; *OEMcmr–168* $26,025
July 1, 1942 — June 30, 1944
"Synthesis of Cortical Hormones"

Louis Long, Jr.
Richard B. Turner
Elkan R. Blout (National Research Fellow)
Melvin Fields (Harvard Fellowship)
Martin Ettlinger (Harvard Fellowship)

was appointed Consultant to the NRC Hormone Conference and served as adviser to the Conference Chairman, Hopkins' physical biochemist W. Mansfield Clark; my first advice was that he purchase a copy of my new monograph.

The cooperating groups exchanged information through periodic meetings in Washington. By far the most optimistic and enthusiastic member was E. C. Kendall, who had been the first to isolate cortisone. At each meeting he reported very promising progress with a big advance just around the corner. I was the least optimistic. Although I never expressed any doubt about ultimate success, I was so conditioned by experiences in the expedited production of munitions that success to me meant cortisone in clinical trial by VJ-day (Victory against Japan). I did all that was called for as consultant, such as visiting Kendall's laboratory in Rochester, Minnesota to check on the status of the work and the nature of his research group, but I did not participate in the research of my own group or spend much time on their problems. Elkan Blout remained only for the duration of his fellowship.

Kendall's enthusiasm and skillful experimentation eventually made the first breakthrough: a practical if lengthy method of transposing an oxygen function of desoxycholic acid from position 12 to position 11 (not the original oxygen atom; the one at 12 was taken out and a new one put in at 11). Kendall's route seemed to me very attractive. I had in Dick Turner an extremely capable experimentalist who was getting nowhere because I had given him a poor problem, and so I arranged to transfer Turner from my group to Kendall's group. Turner's contributions greatly speeded up the work at Rochester. A discovery by Schwenk provided a neat shortcut in the Kendall route. Sarett discovered a first method for introduction of the 17-OH group and, utilizing 11-oxygenated intermediates prepared by the Kendall method, achieved a first synthesis of cortisone. The method was not practicable, but a second method for introducing the 17-OH group developed by Sarett did provide a complete synthesis that appeared capable of application, at least on a laboratory scale. An efficient new method for shortening the bile acid side chain discovered in the Ciba laboratories in Switzerland eliminated several steps in a very long synthesis. The Ciba

method was discovered independently by Martin Ettlinger of my group before news of the prior Swiss work was available.

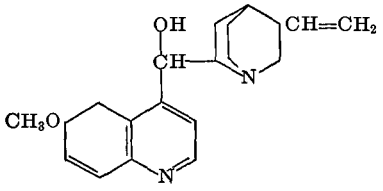
A first synthesis of a complicated compound by a research chemist or a research team affords only a few milligrams of the final product. Production of a batch of cortisone sufficient for a clinical trial called for careful study and improvement of each of the 32 steps worked out by the various research groups. Even if yields are good, the amount of material processed decreases rapidly with the number of steps. If the yield in each of two steps is 80%, the overall yield for the two steps is 64%. The overall yield in a synthesis of ten 80% reactions is 10.6%. Successful completion of a production run with a 32-step synthesis required use of a sufficiently large batch of starting material. A Merck developmental group initiated work on the laboratory synthesis of cortisone in March, 1946. Some of the early steps were done in the pilot plant. Kendall conducted some of the production work in his laboratory and sent material to Merck for further processing. A total of 1,270 lbs. of desoxycholic acid was processed. The first batch of cortisone was available for clinical trial in May, 1948.

Early clinicians had noticed that women suffering from rheumatoid arthritis frequently are relieved of symptoms during pregnancy. Rheumatologist Philip S. Hench of the Mayo Clinic had noted in 1929 that sometimes rheumatic patients are relieved also by an attack of jaundice. Since the steroid hormone progesterone exerts a favorable control over pregnancy but is without influence on arthritis, Hench speculated that during pregnancy some other steroid hormone capable of controlling arthritis is released into the blood stream in increased amounts; the same hormone might escape metabolic destruction in a liver impaired by jaundice. Cortisone, a steroid hormone of the adrenal gland first isolated at the Mayo Clinic and made available for clinical trial by cooperative work constantly spurred by Kendall's experimentation and enthusiasm, was an obvious choice for trial by Hench at the earliest possible occasion. The opportunity arose in May, 1948. In April, 1949 Hench and Kendall announced dramatic success in use of the synthetic hormone for treatment of rheumatoid arthritis. Other therapeutic uses developed later. A new and useful field of medicinal chemistry had been discovered. The Nobel Prize in Medicine for 1950 was awarded jointly to Hench, Kendall, and Reichstein.

18. *Antimalarials*

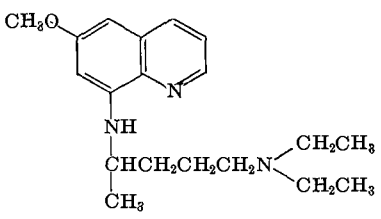
HUMAN MALARIA is caused by four species of the protozoan *Plasmodium*, which are transmitted by females of the *Anopheles* mosquito to man and back again. The parasites transmitted from the salivary gland of the mosquito are sporozoites; they undergo transformation in the human body and appear in red blood cells as trophozoites, which develop to schizonts. The regular bouts of chills and fever occur in an asexual cycle in which the schizonts divide into merozoites, a few of which develop into sexual forms, gametocytes, which, in a mosquito that acquires infected blood, undergo a cycle of sexual reproduction and produce sporozoites. No test animal is known that can be infected with the *Plasmodia* responsible for human malaria, but compounds can be screened for antimalarial activity by assays in certain animals infected with other species, particularly *P. lophurae* (duck), *P. gallinaceum* (chicken), *P. knowlesi* and *P. cynomolgi* (monkey). A particularly prevalent form of malaria in man caused by the species *P. vivax* is not fatal but involves regular relapses of fever that may continue for years. Between relapses the blood is completely free of trophozoites, and yet a tissue form of parasite appears to persist and lead to eventual relapse; in *P. gallinaceum* infection in the chicken a tissue form (exoerythrocytic) is easily demonstrated. In malarial therapy of paretics, the infection is transmitted by injection of blood, and termination of fever is not followed by relapse, because the tissue form is absent. The ideal drug for vivax malaria would be capable of destroying sporozoites (prophylaxis), trophozoites (suppression), and tissue parasites (cure).

Use of bark of a certain tree in treatment of malaria was introduced into Europe in 1869 shortly after the material had been employed successfully in treating the wife of Count Cinchon, Spanish viceroy of Peru. The tree was named cinchona, and when the active principle was isolated by French chemists in 1880 is was given the name quinine. The drug found wide use in malaria therapy as a suppressive; it controls bouts of fever but is not a curative drug. Cinchona plantations of the Dutch East Indies produced almost the entire world supply of quinine, and a shortage of quinine in Germany during World War I stimulated search for a synthetic substitute.

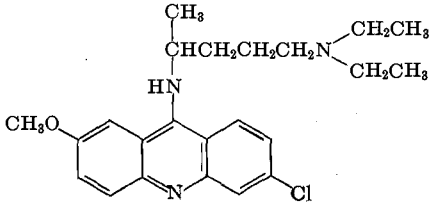


Quinine

Chemists of the I. G. Farbenindustrie found two promising candidate compounds, plasmochin and atabrine. Plasmochin is about 60 times as active as quinine in avian malaria but the therapeutic dose is too close to



Plasmochin



Atabrine

the toxic dose for practical use. Atabrine, which has the same side chain as plasmochin, is suppressive but not curative, and it was initially regarded as too toxic for general use.

The Japanese invasion of the East Indies in 1942 cut off the allied nations from about nine tenths of the world supply of quinine, and our troops were faced with the prospect of fighting Japanese in malaria-infested battlefields of the Pacific and of losing to malaria one man out of every three sent to the area. When the Office of Scientific Research and Development was organized in 1941 there was established within it a Committee on Medical Research.[1]

> "During its existence the Committee expended some $24,000,000 — a sum which would have supported our share in the war for only four hours — in approximately 600 contracts with 133 universities, foundations, and commercial firms."

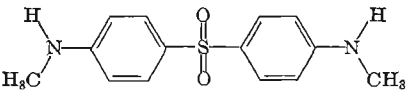
In 1941 Drs. L. T. Coggeshall and John Maier of the International Health Division of the Rockefeller Foundation found that certain sulfa

[1] J. P. Baxter, "Scientists Against Time," p. 300, Little, Brown and Company, Boston, 1947.

drugs effect complete eradication of parasites in some cases of avian and monkey malaria. Trials with humans suffering from naturally acquired malaria were somewhat inconclusive but encouraging. Maier found further that the action of sulfa drugs is inhibited by *p*-aminobenzoic acid, which does not inhibit quinine or atabrine. Therefore the sulfa drugs must act on plasmodia in a different manner than the older drugs. This clue suggested that a systematic investigation of the correlation between structure and activity in the new series might lead to a superior antimalarial drug. At the suggestion of Drs. Coggeshall and Maier, I initiated in the school year 1941–1942 a program of synthesis of new compounds for testing. A grant from the Rockefeller Foundation enabled me to engage Dr. Hans

Fig. 18.1. — Hans Heymann

Heymann as Rockefeller Foundation Fellow. A first publication reported the synthesis of 15 derivatives of *p*, *p*-diaminodiphenyl sulfone, which were assayed for antimalarial activity by Dr. Maier. Three showed some activity

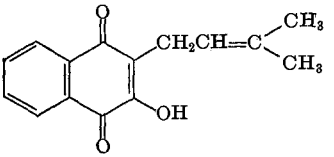


p, *p'*-Bismethylaminodiphenyl sulfone

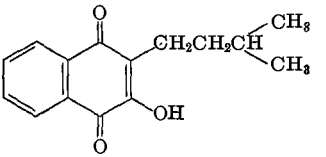
and one of these, the one of greatest potency and least toxicity, is that formulated.

Our antimalarial work soon took a new course. In the CMR program some thirty-nine chemical research groups operated under government contract in coordination with pharmacological and clinical research groups. One line of investigation was the synthesis and biological documentation

of all conceivable isomers, derivatives, and analogs of the known antimalarials. The second line of attack consisted in the biological screening of organic chemicals of a wide variety of types in the search for antimalarials of a new kind. In one such screening program, chemists of the Abbott Laboratories in North Chicago visited some of the larger universities to collect and code miscellaneous research samples and send them to the pharmacological group of Dr. Arthur P. Richardson at the University of Tennessee Medical School at Memphis for screening assays against *Plasmodium lophurae* in ducks. Of several hundred compounds assayed, all but three proved to be completely inactive. The three active compounds, which possessed definite if weak antimalarial activity, were related naphthoquinones that had come from my laboratory and that I had taken from a collection of lapachol samples bequeathed to me by Samuel C. Hooker on his death in 1935. The most interesting of the three was



Lapachol



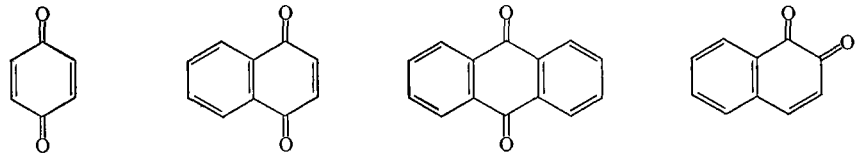
Hydrolapachol

hydrolapachol, the dihydro derivative of lapachol, a yellow pigment that occurs in the grain of certain tropical trees (Lapacho heartwood, Bethabarra wood, Surinam greenheart).

English-born Samuel C. Hooker was awarded the Ph.D. at Munich at the age of 21 after only one year of research. A romance with an American girl student at Munich led Hooker to come to the United States, where, finding no suitable academic posts open, he had accepted employment in a sugar refinery in Philadelphia. Hearing of the presence in the city of a young German-trained chemist, a Philadelphian manufacturer of fine fishing rods and bows and arrows from rare Bethabarra wood imported at considerable expense from South America, consulted Hooker about the possible utilization of the yellow pigment of the wood, and provided the young chemist with quantities of Bethabarra sawdust and waste cuttings. Hooker found the chemistry of lapachol a fascinating subject for spare-time research, and in the period 1889–1896 he published a series of 10 papers reporting the complete elucidation of the structure of lapachol and a description of an amazing array of novel reactions of the interesting substance. In 1896, although he had not completed the research and had stated in his last paper that "I shall hope to return to the consideration of

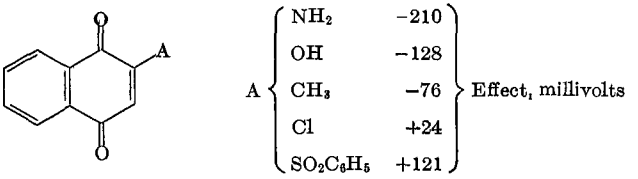
this problem in the future," he felt obliged to abandon this side activity and devote his whole energies to the professional field of sugar technology. His efforts were eminently successful; he introduced the beet sugar industry into American practice and achieved so many other successes that he eventually rose to the post of executive vice president of his company. But he had not forgotten lapachol, and he was ever conscious of the promise of further work stated in his last paper. So, as soon as he had acquired an adequate competency, Hooker retired from the sugar business in 1915, built a private laboratory in the former stable in back of his elegant residence in Brooklyn, and shortly resumed the lapachol research exactly where he had left off in 1896.

Lapachol is a quinone. My research for the doctorate under Professor James B. Conant had consisted in preparing a large number of quinones and measuring their oxidation-reduction potentials. I had studied the un-

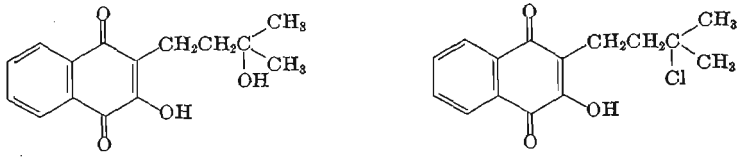


Potentials in Alcohol (volts)

substituted quinones formulated and some others and had made measurements on a number of substituted quinones in order to determine the effect on the potential of the substituent groups. Some groups lower the potential,



some raise it. The direction and magnitude of the shift provides an accurate measure of the inductive effect of the group. A year of postdoctoral work with von Braun at Frankfurt-am-Main and with Perkin at Oxford provided ample opportunity to plan independent research in my first job of teaching at Bryn Mawr College. One inviting problem was to determine if the inductive effect of a group can be transmitted down a chain of carbon atoms. A search through Beilstein showed that the only known compounds of the type required for the investigation had been prepared by Hooker from natural lapachol. These lapachol derivatives would serve my



Typical Lapachol Derivatives

purpose nicely. A comparison of the potentials of the two derivatives formulated with that of hydrolapachol would establish the inductive effects of the OH and Cl substituents.

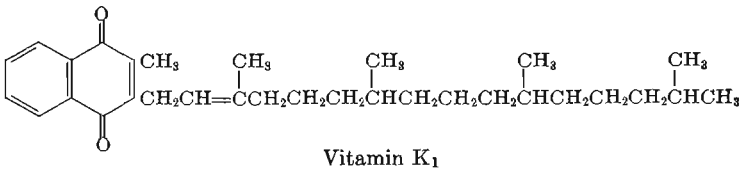
But Hooker's papers had stopped in 1896; what chance would there be of obtaining samples, or advice about Bethabarra wood, in the year 1926? However, I found Hooker listed as a member of the German Chemical Society at the address 82 Remsen St., Brooklyn, New York. Installed in the Bryn Mawr laboratory early in the summer, I wrote to Dr. Hooker to tell him about my problem. His prompt reply assured me of supplies of any of his compounds I might need and included an invitation to visit his laboratory. I lost no time before making a visit, which was the beginning of an intimate association that continued to the day of Hooker's death. I

Fig. 18.2. — Samuel Cox Hooker, 1864–1935

was fascinated by an elderly gentleman of rare personality and ability. He not only was one of the most gifted experimental chemists I have known, but also an accomplished magician. The second floor of the former stable had been made into a theatre where he performed tricks for members of the American Society of Magicians, of which Hooker was one of the few non-professional members. The building also housed part of a chemical library which was, and is, one of the finest in the world; it is now the Kresge-Hooker Library at Wayne State University. Having lost contact with

academic chemists of his own age, Hooker was glad to have a young friend who was enthusiastically interested in his beloved lapachol chemistry. On his death in 1935, I edited, or wrote, or completed by experimentation, eleven posthumous papers reporting his researches of 1915 to 1935. He bequeathed to me a small platinum spatula which he had made and his extensive collection of beautiful samples of compounds derived from or related to lapachol.

When Arthur Richardson told me by telephone that his assays had revealed antimalarial activity in hydrolapachol, I shouted with joy. I loved quinone chemistry. I had synthesized the natural quinones lapachol and vitamin K_1. There had been four papers on quinones with Conant, 19 solo papers, 7 papers with Mary, and 30 with other co-workers. I decided at once to switch our then small antimalarial program from sulfones to



Vitamin K_1

quinones and to expand the project. Charley Heidelberger had joined Hans Heymann in the sulfone work, and they were told to carry the work to the nearest respectable stopping point and change to the quinone program.

Fig. 18.3. — Charley Heidelberger

By the fall of 1942 most of the men and women who eventually joined

the antimalarial group were hard at work under a CMR contract. Members of the group are indicated in a listing for the Harvard record:

Anonymous Research No. 6; OEMcmr-242 $94,101

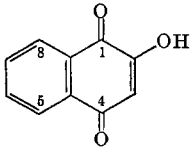
October 1, 1942–December 31, 1945

"Chemotherapy of Malaria"

Ernst Berliner
Frederic C. Chang
William G. Dauben
Martin Ettlinger
Melvin Fields
Charles Heidelberger
Hans Heymann
Wyman R. Vaughan
Mao-i Wu
Armin Wilson
Evelyn Wilson
Louise Wiarda (technician)
Sally S. Shy (technician)
Shirley R. Katz (technician)
Grace Nahm (technician)

Fig. 18.4. — Fred Chang

Drs. Chang[1] and Heymann served as my lieutenants on the project. Several of the others were graduate students at the time, took their degrees in whole or in part on the antimalarial work, and then stayed on with postdoctoral status. Fred Chang had taken his degree in 1941 for work on a brand new synthetic reaction, and guest worker Dr. Albert E. Oxford from England had made the synthesis still more practical. The papers had hardly been published when we started using the method for the synthesis

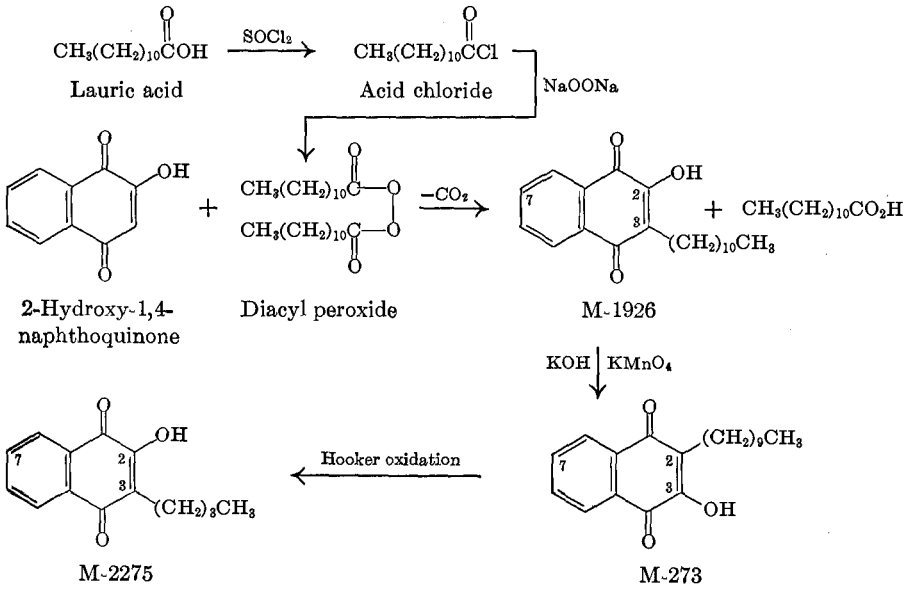


2-Hydroxy-1,4-naphthoquinone

of candidate antimalarials. A component required for all the snytheses was 2-hydroxy-1, 4-naphthoquinone, which acquired the name "Confidential Nucleus." This compound was readily available. I had already standardized one good method for "Organic Syntheses" and I developed a second one during the wartime research. The other component was a fatty acid, about any fatty acid. Each one gave a different compound, which was coded and sent down to Richardson for assay in ducks infected with *Plasmodium lophurae*. So my boys started running through the list of available acids, the way the Anonymous Research 3 boys had run through less pure samples in the Napalm push. Dr. Marlin T. Leffler set up a parallel program on naphthoquinone antimalarials at the Abbott Laboratories and this group also used chiefly the new method of synthesis. The two groups avoided duplication and identified the source by M (for Malaria) numbers; compounds supplied by the Abbott and Harvard groups were identified by numbers in three and in four digits, respectively.

Suppose, for illustration, that the acid component is lauric acid (C_{12}, cf. Napalm). This is converted by a standard reaction into its acid chloride, which in turn is brought into reaction with sodium peroxide at 0° to produce the diacyl peroxide. When this peroxide is allowed to react with 2-hydroxy-1, 4-naphthoquinone in acetic acid, carbon dioxide is evolved, the hydrocarbon part of lauric acid is slapped onto the Confidential Nucleus, and we have the new compound M-1926 for assay. A second reaction which proved to be extremely useful is a remarkable one discovered by Dr. Hooker in retirement and now known as the Hooker oxidation. On reaction of M-1926

[1] Frederic C. Chang, b. 1905 San Francisco; A.B. Columbia, 27; Department of Chemistry, Lingnan Univ., China, 30–38, 46–51; Ph.D. Harvard (Fieser) 41; Univ. Tennessee Medical School, Memphis, Tenn.

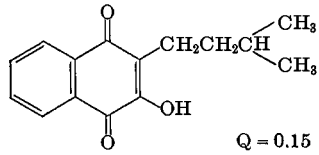


with alkaline potassium permanganate in the cold, a CH_2 unit mysteriously disappears from the side chain and the product is M-273. Hooker oxidation of M-273 affords M-2275, again with loss of a CH_2 unit. Dr. Hooker himself had synthesized the compound analogous to M-2275 but with the side chain $-(CH_2)_6CH_3$, and he converted it by 7 consecutive oxidations into hydroxynaphthoquinone. Dr. Hooker suspected that the quinone ring must open in some way and then close again, and was pleased when my students Arnold Seligman and Jonathan Hartwell obtained results which supported the idea. They marked the second ring with a substituent at position 7 and found that the hydroxyl group originally at the 2-position appears in the oxidation product at the 3-position. The hydroxyl and hydrocarbon groups change place at each step.

In working on vitamin K_1 in 1940, I had found that this naphthoquinone reacts with hydrogen peroxide in an alkaline medium to give an oxide. Whereas K_1 is a yellow oil highly sensitive to light, K_1-oxide is colorless and light-stable. K_1-oxide is the biological equivalent of the vitamin, and Arnold Seligman, at the Beth Israel hospital, introduced its use in clinical practice. I tried the reaction on lapachol in the hope of isolating an intermediate in the Hooker oxidation and obtained a colorless reaction product. Mary took over the characterization of the product, but structural elucidation was difficult and progress was slow. However, she established that the white compound is indeed an intermediate in the Hooker oxidation and

succeeded in isolating and characterizing eight derivatives from the lapachol intermediate and nine derivatives from corresponding intermediates from hydrolapachol and a simpler member of the series with a methyl side chain. The Hooker oxidation proved useful in the antimalarial work when carried out on a 1–2 g. scale, but with larger amounts the results were disappointing because a second Hooker oxidation was then superimposed over the first. But reaction with hydrogen peroxide completes a first step with no trace of overoxidation. So, while Mary was completing her work on the structure, I worked out a procedure for effecting the second step by oxidation with a copper salt, and developed a two-step process which affords products of high purity in yields of 85–90% and which is not limited with respect to the size of sample oxidized. Thus once a long side chain has been introduced by the diacyl peroxide method, all the lower members of the series can be prepared very easily by successive two-step Hooker oxidations.

Assayed in ducks, hydrolapachol is only feebly active; the quinine equivalent (Q), that is, the potency relative to the activity of quinine as



Q = 0.15

Hydrolapachol (M-1523)

standard, is only 0.15. That activity of this low order was picked up at all attests to the perfection of the Richardson assay procedure. Our problem was to vary the structure in all conceivable ways in the search for compounds of increased potency. Hydrolapachol has an isopropyl group separated from the quinone ring by two methylene groups. The Hooker collection contained samples of the Hooker oxidation products with one and with two methylene groups removed, and these showed no detectable activity. As expected from this finding, lengthening of the side chain was attended with enhanced activity. Each succeeding member of the iso series was synthesized through M-2287, with the chain $-(CH_2)_8CH(CH_3)_2$, prepared by Charley Heidelberger. As the hydrocarbon side chain increases in size, antimalarial activity rises steadily, reaches a maximum with a chain of 9 carbon atoms, and then falls off. The same relationship was noted in the straight-chain series, in which a succession of 17 compounds was topped by M-2256, $-(CH_2)_{16}CH_3$, synthesized by Martin Ettlinger with the peroxide from stearic acid.

Fig. 18.5. — Martin Ettlinger

For analysis of data, we plotted dose-response curves reported by the pharmacologists, that is, dose in milligrams per day against reduction in parasitemia, and by reading off from the curve the dosage level corresponding to 95% reduction in the count and determining ED_{95}, the effective dose of drug at a standard level. Data for a given series can then be analyzed

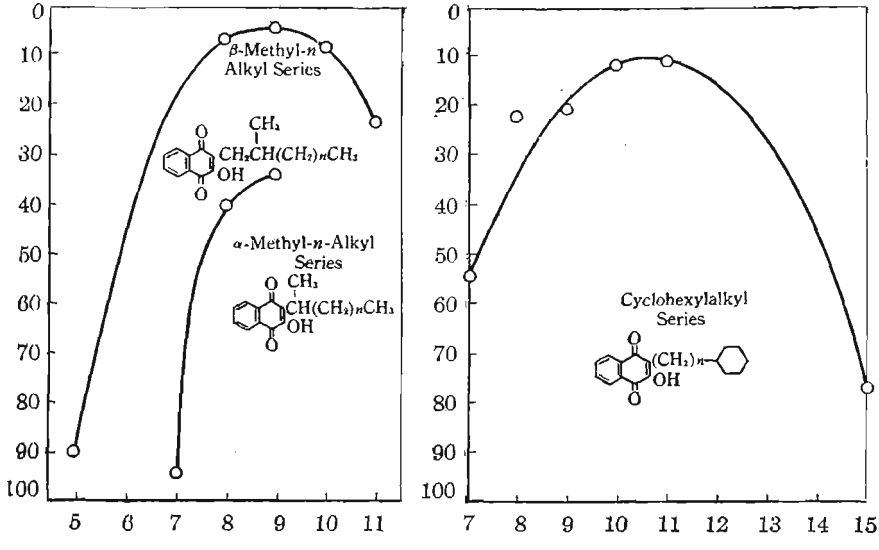
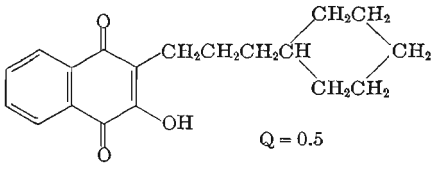


Fig. 18.6. — Effect of Lengthening the Side Chain

by plotting ED_{95} (abscissa) against number of carbon atoms in the side chain (Fig. 18.6). Curves for different series all show the characteristic rise to a maximum and then a fall, but the position of the maximum shifts from series to series. In the two series shown in the plot on the left maximal potency is reached with a side chain of 9 carbon atoms, whereas in the cyclohexylalkyl series best activity is attained with a chain of 10–11 carbon atoms.

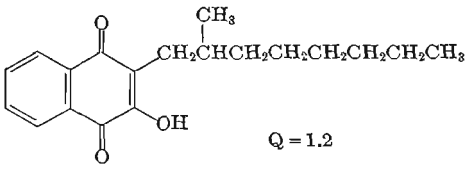
Excitement rose high when Harvard entries started falling at or near peak-points. Bill Dauben's M-1916 is five times as active as hydrolapachol



M-1916

Fig. 18.7. — Bill Dauben's M-1916

and has half the activity of quinine; the activity of this member of the cyclohexylalkyl series is represented by the dot over the 9-carbon mark. At about the same time Abbott hit the jackpot with their M-285; this member



M-285

of the β-methyl-*n*-alkyl series with a 9-carbon chain falls at the exact peak of the curve (Fig. 18.6).

The naphthoquinones had begun to interest the Big Brass of the anti-malarial program, all medical men: E. Kenneth Marshall, James A. Shannon, and Robert F. Loeb. At first our M.D. friends, dubbed by Hans and Fred "The Croakers" (cf. Damon Runyon), took a dim view of our

compounds. They were unorthodox. They bore no structural resemblance to quinine, atabrine, or plasmochin. They did not contain nitrogen. In our view, the fact that compounds containing only carbon, hydrogen, and oxygen showed therapeutic activity, at least in animals, was very exciting. Standard antimalarial, arsenical, and sulfa drugs all contain nitrogen. So do penicillin, streptomycin, tetracycline, chloramphenicol, and erythromycin. The naphthoquinones are still unique. Then, in 1943 J. H. Bauer, W. G. Downes, Delphine H. Clarke, and Max Theiler of the Rockefeller Foundation established that naphthoquinone drugs are not only suppressive but curative. They effectively destroy the exoerythrocytic forms of malaria parasites of chickens infected with *Plasmodium gallinaceum*. If active in man, they should destroy not only trophozoites, and suppress fever, but also destroy tissue forms of parasites postulated to be responsible for relapses. Tissue forms, or exoerythrocytic forms, are easily demonstrated in infected chickens, but remain hypothetical in man, in ducks, and in monkeys.

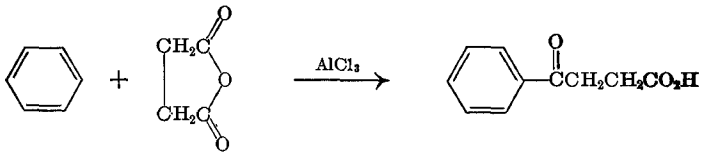
> "Shannon, Marshall — Hallelujah! Have hot drugs,
> but what's it to you?
> We have drugs of action fair
> against the bug that isn't there."

"If active in man." I spent the day October 26, 1943 in Washington conferring on the Adams Plan, and on flare markers for guidance of an airborne invasion (a project later dropped). I then went to Baltimore for dinner and a conference at the Johns Hopkins Club with E. K. Marshall, W. M. Clark, K. Blanchard, A. P. Richardson, and M. Leffler. We were informed that all existing drugs, for one reason or another, were inadequate and that in the opinion of the Review Panel the naphthoquinones offered the most promising of all leads to an effective antimalarial and should be submitted at once to pharmacological processing, followed by a clinical trial by Shannon at Welfare Island. The compounds to be tested were Abbott's M-285 and our M-1916, and a 250-g. lot of each drug was requested. Marshall was certain that the clinical value of the drugs could be assessed in a period of 30 days after receipt of the drugs. "I'll give you seven days, agreed?" For the synthesis of M-285, the acid required for the side chain could be prepared in two easy steps from a bromide available as an intermediate to a standard Abbott product, and so Leffler agreed to attempt production of M-285 in the time specified. He was aware of the time-consuming nature of the three steps needed to make the acid intermediate to M-1916 and felt that Abbott would be unable to make this

compound as well. I was all too well aware of the difficulties but, in the belief that the Harvard group had the stuff to work wonders, I accepted the assignment.

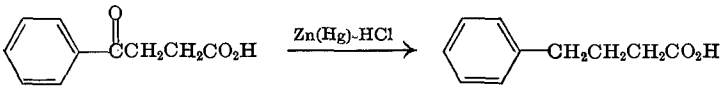
The next morning I raced from the Federal Express to the laboratory, rounded up all available organic research men, whether they were in my group or not, outlined the problem, and asked for volunteers for both day and night duty. Several volunteered at once; others heard about the project later and promptly joined. Heidelberger was recalled by telegram from Washington, D.C. and Dauben from Ohio. Even undergraduates joined in the project. Work started shortly after 10 a.m. on DAY 1, a Wednesday.

By evening 30 men were at work and 5 kg. of benzoylpropionic acid was in the making. The three steps in the synthesis of the M-1916 acid are shown in the formulation. Large-scale equipment was lacking and the



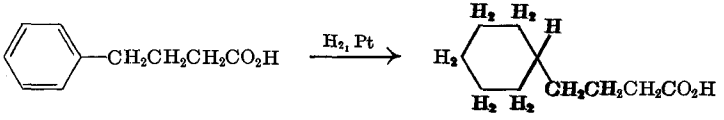
Benzene Succinic anhydride β-Benzoylpropionic acid

2. Clemmensen Reduction:



β-Benzoylpropionic acid **γ-Phenylbutyric acid**

3. Hydrogenation:



γ-Phenylbutyric acid **M-1916 acid**

Friedel-Crafts and Clemmensen reactions had to be done in a large number of runs in small flasks. The first Clemmensens were started Wednesday night and kept going continuously until Saturday in various parts of two buildings in batteries of up to 10 flasks and condensers. A Dartmouth group organized regular day and night shifts. Other people stayed during the night without much organization or sleep. General Expediters Heymann and Chang estimated the total amount of sleep lost as 300 man hours. The hydrogenation step caused the most trouble because it is fussy and hard to

make go to completion. The operation usually is done in a bottle fitted into a motor-driven shaker connected to a tank of hydrogen. The bottle is charged with phenylbutyric acid, a solvent, and the solid platinum catalyst. Efficient shaking is required to bring the three phases — gas, liquid, and solid — into intimate contact. Only a few commercial shakers were available and these were small. We first brought in from my house one of the old-style washing machines that swishes clothing to and fro in semicircular thrusts. The boys mounted two five-gallon flasks into the washer and improvised a hydrogenator which worked reasonably well except that it frequently sheared a bolt. A second hydrogenator was fashioned from a foot-driven sewing machine by strapping a bottle on the pedal and driving the machine by motor. Like the washing machine, the sewing machine required constant watching, for every so often it would pick up speed and threaten to jump off the table.

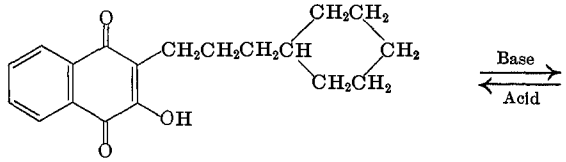
Well, the improvised hydrogenators worked and the boys made it. Asked for 250 grams of M-1916 in seven days, they had, in the BIG PUSH, turned out 350 grams in just six days, one of them a Sunday. And the clinical trial at Welfare Island? The drugs were to be tried out for suppression of malarial fever induced (by blood) in paretics for treatment of congenital syphylis. The test did not start on time and was still in progress at the time of the following commentary:

> December 8, 1943. Goldwater Hospital, N.Y. — "Sought information and urines from clinical trials; obtained little of either."

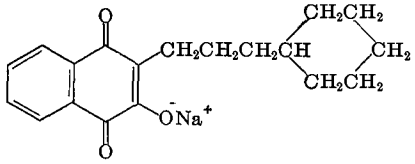
The result was clear-cut enough, but curious. Our entry into the competition was a boy, whereas Abbott's was a man. M-285 is four times as active in ducks as M-1916. Tested in man, M-285 proved to be completely inactive; M-1916 showed low-order but unmistakable activity. In the view of the medicos the naphthoquinones had failed in a fair trial; we were advised to switch back to the sulfones. But why the reversal of order of activity in birds and in man? Oh, "biological variation is common. Many facts of life are not yet understood."

But I was not in uniform, not under obligation to take orders. The boys all agreed that we should work on the metabolism of the drugs and try to clear up the mystery. On the trip to Goldwater Hospital I had taken along two wooden cases made by Walter Coburn for carrying four large glass bottles for urines which were to have been collected from the test patients, but the increase in weight on the return trip was not noticeable. However, inspection of the small samples proved interesting. The yellow naphthoquinone drugs have a hydroxyl group which is strongly acidic and they

react with even weak bases to form salts, which are bright red. Urine from patients given M-1916 by mouth was bright red; it must contain a salt of either M-1916 or a metabolite in which the hydroxynaphthoquinone group



M-1916 (yellow)



Salt (red)

was still intact. The fact that the urine from ineffective M-285 was also red strongly suggested that the red pigments were both metabolites. A simple method was at hand, then, for investigation of metabolism: administration of drug to normal subjects, collection of urine as long as it was red, acidification of the urine to liberate the free metabolite from its salt, and extraction with ether. We made up pills by putting M-1916 and M-285 into capsules and inquired of CMR about the availability of subjects. Dr. Allan M. Butler at the Massachusetts General Hospital had a set-up ideal for the purpose: a group of conscientious objectors who had volunteered for experiments in medical research. I told Allan over the phone that I had a bottle of pills in my hand which I wished to bring over to him. He checked his calendar and said that he had a date to be in Washington two weeks hence and would try at that time to obtain official sanction for this use of the objectors. I hung up and swallowed a first pill of M-1916.

December 10, 1943. Cambridge — "Case 1: M-1916, normal male (L.F.F.) — 12/10/43; 2 g. per day for 4 days; symptoms, none."

December 17–19, 1943. Dugway Proving Ground, Utah. — "Adams Plan, etc. Collected and extracted urines during this period."

The work on drug metabolism eventually included 75 trials in human subjects and provided data on metabolic oxidation products from 19 naphthoquinones. The work was done by chemists, but it involved so much experimental therapeutics, pharmacology, and biochemistry that five papers published after the war appeared in the *Journal of Pharmacology and Experimental Therapeutics* or the *Journal of Biological Chemistry* (17

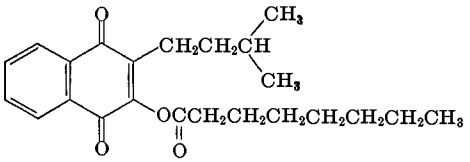
other papers appeared in the *Journal of the American Chemical Society*). The co-authors of the first paper on metabolism were Chang, Dauben, Heidelberger, Heymann, and Seligman. Arnold Seligman,[1] chemist, sur-

Fig. 18.8. — Discussion with Arnold Seligman and E. B. Hershberg (about 1937)

geon, and researcher on histochemistry and cancer, performed invaluable services at the Beth Israel Hospital. For one thing, he lined up as subjects a number of polycythemic patients who reported regularly to the hospital

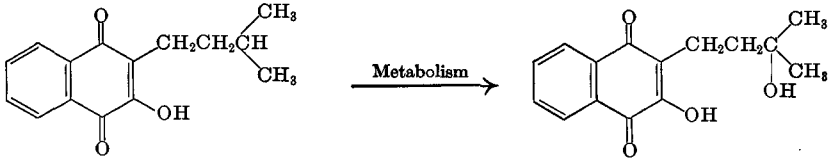
[1] Arnold M. Seligman, b. 1912 St. Johnsbury, Vt.; A.B. Harvard, 34; M.D. Harvard, 39; research on carcinogenic hydrocarbons, Harvard (L. F. F.), 34–39; Beth Israel Hospital, Boston, Departments of Surgery, Sinai Hospital of Baltimore and Johns Hopkins Univ.

to be bled for relief from excess red blood cells; in our records, these subjects were designated as polycythemic by an asterisk. In Case 19, R. S.* was given 4 grams of hydrolapachol caprylate during 6½ hours, and blood was drawn 13 hours later. This blood afforded 215 milliliters of plasma



Hydrolapachol caprylate
(molecular weight 370)

(very red), several times the volume obtainable from a normal subject. Urine is the best source for isolation of metabolites because large amounts can be obtained for processing. The Case 19 urines of days 2, 3, and 4 were all red and so were pooled, acidified, and extracted with ether. The caprylate ester administered had all been hydrolyzed in the body and fractionation of the extracted pigment afforded 34.7 miligrams of unchanged hydrolapachol and 44.0 milligrams of a metabolite easily identified as hydroxyhydrolapachol by comparison with a sample from the Hooker collection. The 215 milliliters of plasma from the same patient yielded 3.5



Hydrolapachol
(molecular weight 244)

Hydroxyhydrolapachol

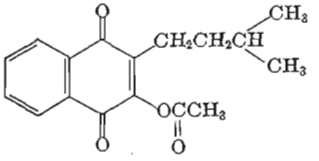
milligrams of hydrolapachol and about 1 milligram of hydroxyhydrolapachol. The results, checked with one other naphthoquinone, showed that metabolites found in urine are identical with those present in the much less accessible blood and hence that naphthoquinone pigments isolated from urine provide an accurate pattern of the naphthoquinone drugs and metabolites in blood.

The metabolism of hydrolapachol involves introduction of a hydroxyl group into the hydrocarbon side chain. The metabolite, hydroxyhydrolapachol, had already been assayed in ducks and found inactive, whereas the drug itself possesses weak but definite activity. Metabolic oxidation thus is attended with loss of therapeutic activity. However, the amount of hydrolapachol recovered unchanged in the 2 to 4-day urines was surprisingly

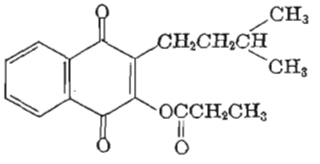
large. With all other drugs metabolism was so rapid that no unchanged drug could be isolated, even from day 1–2 urines. Other cases confirmed the conclusion that hydrolapachol is more resistant to metabolic deactivation than any other member of the series. A request for a clinical trial in paretics was granted. Prior to the metabolism studies, hydrolapachol had been cleared for administration to human subjects by a short-term chronic toxicity test in mice conducted at the Harvard Medical School first by Professor Otto Krayer and then by Dr. T. B. Astwood. When given at maximum level of food intake, hydrolapachol had killed 30% of the mice tested but still was rated safe enough for human consumption. With seventeen less toxic compounds the death rates were in the range 0–27%; seven that were more toxic but still safe led to death in as many as 90% of the mice. Free hydrolapachol, a solid, had been given orally to several subjects without evoking symptoms of gastrointestinal upset, diarrhea, heartburn, or malaise, but the blood levels were disappointingly low, about 4–5 milligrams per 100 milliliters of plasma. The caprylate had seemed promising because it is a liquid; a liquid should be absorbed from the intestines more efficiently than a solid. Trial showed that this ester does give a slightly higher blood level. Consideration of the molecular weights shows that the result was encouraging, for 1 gram of caprylate (370) absorbed through the intestines and then hydrolyzed by enzymes can give rise to only the following amount of hydrolapachol (244):

$$1 \times \frac{244}{370} = 0.66 \text{ gram}$$

A liquid ester of lower molecular weight might be absorbed even better and would give rise to a higher proportion of active drug. The acetate is



Hydrolapachol acetate
(molecular weight 286)



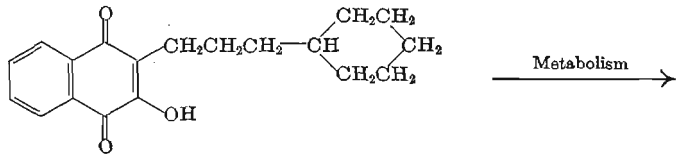
Hydrolapachol propionate
(molecular weight 300)

ideal with respect to molecular weight, but it is a solid. The propionate, being a liquid of much lower molecular weight than the caprylate, seemed the obvious choice for use in the clinical trial.

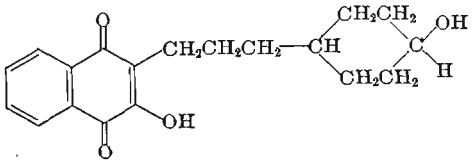
I made the decision to use the propionate with the idea that supporting data could be filled in as the clinical experiment proceeded. Both free hydrolapachol and the caprylate had been given to several subjects without

evoking substantial symptoms; the propionate of intermediate structure should cause no trouble. The first subject given the propionate was a hypochondriac patient under malaria therapy and not suitable for observation of toxic effects of the drug. Furthermore the blood level produced in the miserable paretic was only 5 milligram percent, and I felt sure that a much higher level could be achieved in a more robust person. Having already ingested 33 grams of assorted naphthoquinones without discomfort, I became the second subject (Case 29). At the same time that I went on drug regime Dr. Astwood started administering the propionate to mice. We joked about the concurrent tests that evening at a conference of cooperating groups at the Boston Psychopathic Hospital, after which I boarded the Federal Express bound for Edgewood Arsenal. My boys did not know where to reach me the next morning to warn me of a call from Astwood stating that his mice were dying off rapidly. I needed no warning. Never in my life have I felt so wretched as I did on day 2. I concluded my business at the Arsenal as soon as possible and spent the rest of the day stretched out on a cot in the officers' quarters. Drug regime for just one day had brought on a condition of malaise which lasted for about 36 hours. Edgewood doctors persuaded me to stop taking propionate pills and drew blood samples which were subsequently analyzed for the record. The blood levels achieved (13%) were twice those previously attained, but the price paid was excessive. The clinical trial was stopped.

Two crystalline metabolites of M-1916 were isolated. One, melting at 112°, was obtained from L.F.F. urine and plasma, and from plasmas of polycythenic patients F.* and C.* The other, melting at 155°, was isolated from C.* and D.* plasmas and from L.F.F. urine. Both had the same analysis and were characterized as resulting again from hydroxylation of

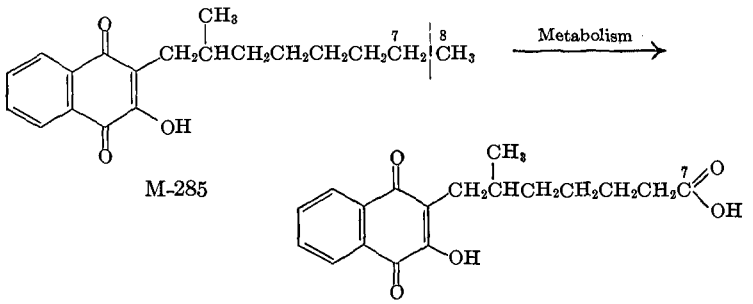


M-1916



155°-Metabolite

the hydrocarbon side chain, and the higher melting isomer was shown to be hydroxylated in the terminal position of the ring. The position of the hydroxyl group of the 112°-metabolite was not established. For a time it appeared that M-285 gives rise to several metabolites. Thus four crystalline products isolated from urines of L.F.F., H.H., and patient S.* (2 samples) melted at temperatures ranging from 154° to 174°. However, all had the same analysis, and eventually techniques of crystallization were developed by which all of the samples could be transformed into one of two identical products, either beautiful prismatic needles or leaflets, which are interconvertible polymorphic forms of a single substance. This metabolite was shown to be an acid of the structure formulated. Biological oxidation in this case eliminates the terminal carbon atom of the chain and oxidizes the



M-285

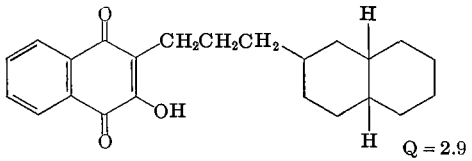
Metabolite

next position to a carboxyl group (CO_2H). Two atoms of oxygen are introduced rather than one, as in the case of M-1916.

An attractive postulate to account for the clinical results was that introduction of one oxygen into M-1916 decreases, but does not abolish antimalarial activity, whereas introduction of two oxygen atoms into M-285 completely destroys activity. Assays of several synthetic model compounds containing a carboxyl group showed them all to be inactive and the results strengthened our faith in the hypothesis. But for complete proof we needed to know the antimalarial activity, or lack of it, of the metabolites. An assay in ducks requires several grams of material, and such amounts could hardly be produced by metabolism in the livers of the only available test animal, man. Synthesis of one of the M-1916 metabolites eventually was completed but the process was too difficult and lengthy for laboratory-scale production.

Thus evidence for interpretation of the Goldwater results remained incomplete for a full two years. The Abbott group closed out their synthetic work, but my group was determined to complete the job undertaken and was confident that a thorough systematic study would, in the end, supply

all the information needed for development of a satisfactory drug. By synthesis and by Hooker oxidation, they completed each of the series first started and several new ones, and the work was rewarded by discovery of compounds of very high potency. Armin Wilson's M-2279 has over twice



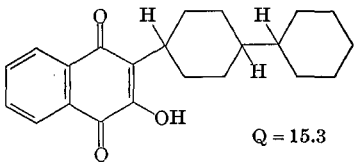
M-2279

Fig. 18.9. — Armin and Evie Wilson

the activity of M-285 and is nearly three times as potent as quinine. Melvin Fields' M-2293 topped them all with a quinine equivalent of 15.3. The weak activity of hydrolapachol (Q = 0.15) had been boosted by a factor of 100. These two compounds contain cyclohexane rings as does M-1916, and hence there was every reason to suppose that metabolism would result in hydroxylation of a ring in the same way and give metabolites of activity greatly surpassing that of the M-1916 metabolites. But the CMR bosses did not share our enthusiasm, and we remained beyond the pale:

June 6, 1944, New York. — "Conferred with (and fought with) Shannon and Loeb on malaria."

September 16, 1944, New York. — "Malaria Conference; a fight with the medicos. Afterwards at the Biltmore men's bar: disgust session with the boys (cf. Napalm)."



M-2293

Fig. 18.10. — Melvin Fields [1]

VJ Day on May 8, 1945 marked the start of termination of work under the CMR contract. Some of the boys suggested a celebration dinner at Locke-Obers to be charged to the contract. Lyon Southworth would not approve such a charge but suggested submission of a bill of adequate proportions for overtime work. Authentic overtime, particularly during the BIG PUSH, would have paid for many parties, and so the date was set for a dinner at which the sky was the limit and everyone could have whatever he pleased. Those torn for a choice between oysters winterplace and clear turtle soup had both. Martin Ettlinger put away three desserts.

After the war Drs. Heymann and Chang carried on the work without a break under a grant from the Rockefeller Foundation. Another effective participant was Dr. George Fawaz,[2] an experienced Lebanese chemist on sabbatical leave from the American University at Beirut who joined our group in January, 1946; his fiancee arrived in August and George and Eva were married in Boston.

Still lacking was information about the biological activities of the M-1916 and M-285 metabolites. Then W. B. Wendell of the Memphis group discovered that naphthoquinone antimalarials are potent inhibitors of respira-

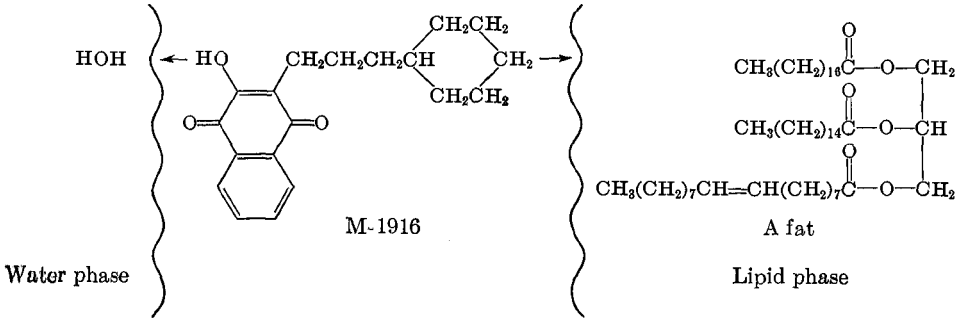
[1] The snapshots of members of the group were taken at a time late in the war when Ernst Berliner had left to take a teaching post at Bryn Mawr College. The move made little change in his contribution to the project, for at Bryn Mawr both Berliner and his Ph.D. student Frances Bondhus, later Mrs. Berliner, synthesized numerous additional naphthoquinones.

[2] George Fawaz, b. 1913 Deirnimas, Lebanon; M.S. Amer. Univ. Beirut, 35; Ph.D. (Organic Biochemistry) Graz, Austria, 37; M.D. Heidelberg, Germany, 55; Dept. Biochem., Amer. Univ. Beirut, 39–52; Dept. Pharmacology, Amer. Univ. Beirut, 53–.

tory systems. Red blood cells containing malaria parasites respire, that is absorb oxygen, and respiration is inhibited by a drug such as M-1916 at very high dilution. Rate of respiration can be measured by drawing blood from a duck infected with *P. lophurae*, centrifuging the blood to separate red blood cells from plasma, suspending washed red cells in a standard duck serum, and shaking the suspension with air in a closed apparatus in which the oxygen uptake can be measured with accuracy at successive intervals of time (Warburg apparatus). Runs are then made with addition of varying small amounts of drug and the extent of inhibition at each concentration determined. A plot of the results then establishes the concentration of drug required to produce 50% inhibition. For M-1916 the level of 50% inhibition of respiration is reached in a solution containing 0.3 milligrams per liter. Wendell examined several compounds of known antimalarial activity in ducks and found that relative antirespiratory activity follows closely the order of antimalarial activity. Since antirespiratory activity can be determined rapidly on a micro scale, the method seemed ideal for investigation of metabolites isolable in only very minute quantity from urines and plasmas. Hans Heymann and Fred Chang, with advice from Wendell at Memphis and from Eric G. Ball of the Harvard Medical School, installed a Warburg machine in our laboratory and a colony of infected ducks in the Biological Laboratory. Hans mastered the techniques of biology and biochemistry required and with the assistance of a succession of girl technicians completed several significant antirespiratory studies. Determinations of the relative antirespiratory activities of 76 naphthoquinones in Memphis and of 82 in Cambridge showed conclusively that the rapid test, which can be done with submilligram quantities in a Warburg machine, is a safe measure of antimalarial activity. Tests of metabolites established the early postulate as correct. The M-285 metabolite is devoid of antirespiratory activity; the two M-1916 metabolites have one-tenth the activity of M-1916. These weakly active hydroxylated metabolites are not oxidized further and persist unchanged for long periods in the blood stream. If a drug could be provided in the course of synthesis with a preformed hydroxyl group, it should be resistant to metabolic oxidation and should be as active in man as it is in ducks. The hydroxyl group would lead to decreased activity, but perhaps the effect could be counteracted by some other structural change.

The type of structural change required to provide counterbalancing adjustment for the hydroxyl group emerged from a study of the distribution characteristics of naphthoquinone drugs. A drug carried in the blood stream is distributed between the water phase and the water-insoluble lipid,

or fat-like phase. The hydroxyl group, being like that of water and hence hydrophilic or water-seeking, tends to draw M-1916 into the aqueous phase,



but the lipophilic hydrocarbon side chain merges with the hydrocarbon part of a fat and tends to draw the molecule into the lipid phase. Perhaps the antimalarial activity of a drug is dependent upon the balance between its hydrophilic and lipophilic character. A scheme for the experimental determination of this balance, for each drug, is as follows. A sample is dissolved in ether, $CH_3CH_2OCH_2CH_3$, a lighter-than-water and water-insoluble solvent with a high solvent power for lipids, and the solution is shaken vigorously with a water solution of known acidity, expressed as the logarithmic function pH. Standard buffer solutions are available of controlled pH ranging from strong acidity (pH 1) to strong alkalinity (pH 12). A part of the drug becomes distributed into the aqueous phase and the rest is retained in the ether. The layers are separated, the amount in each layer is determined colorimetrically, and the distribution ratio at the pH in question so determined. A second determination is made with an aqueous solution of higher pH; this time the proportion passing into the aqueous phase is increased, because more of the drug is present as its water-soluble salt. Determinations are made easily, but full interpretation of the results requires a mathematical analysis. Martin Ettlinger was ideally suited for work on this problem because when he entered the graduate school at the age of 17 he was already sufficiently qualified in mathematics to have published a paper in a journal of mathematics. Martin defined the logarithmic constant *p*E by the following equation:

$$p\mathrm{E} = \log \frac{\text{Concentration of quinone in ether layer}}{\text{Concentration of quinone in water layer}} + \mathrm{pH} - 2$$

The constant *p*E, the logarithm of the critical extraction value, is the pH

of a solution capable of extracting just $\frac{1}{101}$-part of quinone pigment from an equal volume of ether.

Determinations of *p*E by Ettlinger were supplemented by others carried out by George Fawaz, with the technical assistance of Eva Fawaz, until data were accumulated for several of some sixteen series of naphthoquinones that had been synthesized and assayed in ducks. Analysis of the results proved most interesting, as can be seen from the plot (Fig. 18.11) of *p*E

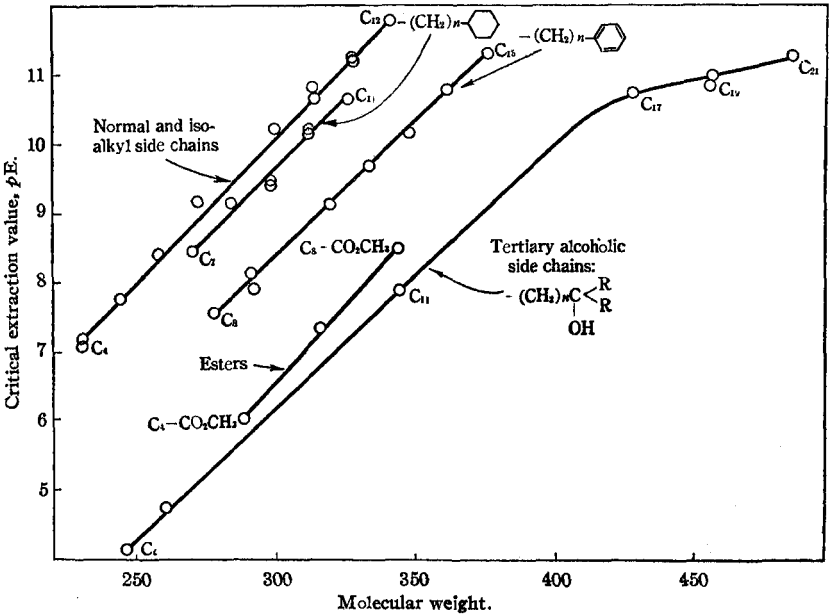
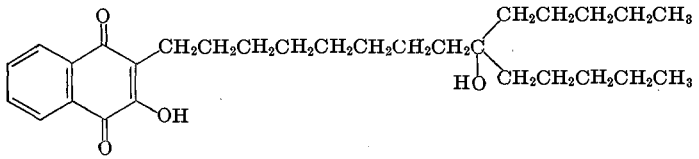


Fig. 18.11. — Variation of *p*E with Molecular Weight

against molecular weight. The points at the upper right marked C_{17}, C_{19}, and C_{21} can be ignored for the moment since these compounds were synthesized later. In all other instances the *p*E values for a given series fall on a straight line and the series lines are nearly parallel. The linear relationship makes sense; the lengthening of the hydrocarbon side chain by successive CH_2 units increases the molecular weight by increments of 14 and is attended with successive, equal increments in lipophilic character, as expressed by *p*E. That the series lines do not all coincide but are shifted at various distances to the right accounts for the relationship noted above that maximal activity in one series may be reached in a side chain of 8 carbon atoms but in a chain of 13 carbon atoms in another. If each line is marked

with a cross at the point of maximal activity in the series, the crosses all fall close to the best horizontal line drawn through them. The relationship can be expressed also by citing the pE of the most active member in each series: 10.2, 10.4, 10.9, 11.3, 11.8, 9.8, 10.9, 11.0. Thus the distribution work had revealed the important relationship that for maximal antimalarial activity a naphthoquinone must have a hydrophilic-lipophilic balance such that the pE value falls in the range 10–12. The very low position of the curve for the C_4–C_{11} hydroxylated side chain shows that introduction of a hydroxyl group markedly increases the hydrophilic character and lowers pE by about 4 units. This large drop in pE accounts for the tenfold reduction in the activity of M-1916 as the result of metabolic hydroxylation. Introduction of a hydroxyl group by synthesis would produce a similar effect, but a now obvious measure for compensation would be to increase the size of the hydrocarbon part of the side chain.

The information provided by this systematic study made it possible to draw up specifications for a tailor-made drug that would carry a hydroxyl group for protection against metabolic deactivation and have a pE value suitable for maximal antimalarial activity. George Fawaz undertook the synthesis of the four compounds with tertiary alcoholic side chains represented in the chart by the dots for C_{17}, C_{19} (two compounds), and C_{21}. The C_{17} compound was obtained from one of the C_{19} isomers by two Hooker oxidations. The bend in the curve was unexpected but immaterial, for the pE values all fell in the desired range. Of these compounds the best was M-2350, and we named it lapinone in honor of Dr. Hooker. Because of the large size of the molecule, lapinone is not absorbed efficiently when



Lapinone (M-2350)

given by mouth and initial assays by the usual technique of oral administration indicated only feeble activity. Reassays, conducted on the strength of high antirespiratory activity, disclosed that intramuscularly administered lapinone possesses high potency.

The question of whether or not lapinone is resistant to metabolic deactivation was answered as follows. Arnold Seligman administered the compound intravenously to patients at the Beth Israel Hospital and Hans Heymann determined the residual antirespiratory activity of naphthoquinone pigment extracted from plasma samples. The results were very

encouraging. Unlike M-1916, M-285 and all other naphthoquinones studies, lapinone retains a large measure of its original activity for periods of 20–40 hours after administration. Experiments with animals of seven species gave similar results. Mice metabolize M-1916 more nearly like humans than do any of the other six test animals, and lapinone administered to mice showed good retention of antirespiratory activity.

By the criteria of the laboratory tests, it appeared that the goal of developing a naphthoquinone antimalarial of high potency and of adequate resistance to metabolic deactivation in man had been achieved, and there was every reason to expect that lapinone would be effective for both the suppression and cure of malaria in man. But the war was over, the CMR committee, testing groups, and facilities for clinical evaluation had been disbanded, and the only clinical test conducted was a small-scale one carried out at the American University of Beirut, Lebanon by George Fawaz, who was in the process of transfer from organic chemistry to pharmacology. His report:

> September 23, 1948. Beirut, Lebanon. — "Nine patients with primary vivax infection were given 2 grams of lapinone per day for four days by intravenous injection in gelatin solution. The results were dramatic. The fever stopped a few hours after the first injection and the patients left the hospital in perfect condition with no parasites in the blood. Six of the patients reported back for examination and had gone without relapses when last checked after periods of from 13 to 15 months after termination of treatment."

A victory? Well, not from the point of view of practical therapy. Lapinone has one shortcoming: it is effective only by intravenous injection; the medicos want a drug that can be administered by the easier oral route. But those of us who participated in a long and exciting research regarded the ultimate outcome as a true victory. By following the scientific method, we had exploited to the limit an initial chance clue.

19. Training Manual for OSS Operators

IN PREPARATION for the trip west with Mary to work on the trial production of Paul Reveres, I addressed the following letter to good friend Professor Carl R. Noller of Stanford University:

> October 5, 1944. — "One member of the party has a future assignment calling for use of spoken German. He reads German reasonably well and has had practice in speaking during periods of study and travel in Germany. While on this trip he would like to brush up on the language. Perhaps you can help me locate a tutor. There would be no objection to one of those attractive coeds."

The interested party was one L. F. Fieser; I was slated for service as a member of the ALSOS mission with the armies of invasion expected soon to be pouring across the Rhine into Germany. I was inoculated in California, indoctrinated in Washington, and told to be alert for immediate call.

Fig. 19.1

November 30, 1944. Washington. — "OFS (Office of Field Services): further indoctrination for the ALSOS mission."

December 11, 1944. Washington. — "OFS; secured second papers, visas, etc.; was instructed to collect my uniform and gear at once from Boston and to be ready for immediate flight overseas."

December 13–22, 1944. Washington. — "Was processed for overseas service and put on four-hour call for flight to Paris by Air Transport Command. I was working furiously on the malaria Interim Report No. 10 and was glad to be delayed for several days; Mary was there to see me off and we had many farewell parties in the gay capital. After one dry run to the air terminal and then a further period of waiting, it became clear that von Rundstedt's Ardennes offensive would preclude any intelligence work in Germany for some time and, hence, I requested that my trip be deferred. The two-month wait gave me a chance to do some profitable experimental work on the malaria problem (sulfate esters) and to conduct and photograph a number of burns and so to collect material for the training manual Arson for the instruction of OSS operators."

The title (Fig. 19.1) was not of my choosing nor to my taste and girl editors messed up some of the writing, but the manual probably would serve the purpose of indoctrinating OSS operators in the science and art of effective fire starting. On September 25–26, 1944 I was taken to an OSS training camp of secret location in the Washington area to size up instructors and trainees for whom the manual was intended. Considerable material of use in the manual had been assembled in an OSS-NDRC project working out of Bryson City, North Carolina on August 26–31, 1944. The waterway of the Tennessee Valley Authority was to be extended by the flooding of a further large valley, and the houses, buildings, and railroad tracks in the area had been acquired for use in testing various OSS devices for sabotage. One group investigated techniques for blasting railroad tracks beyond possible use or quick repair; another studied the destruction of bridges. I headed an incendiary section and was allotted a dozen houses and one store for destruction by fire. Each structure could be used for several initial tests by extinguishing the fires at an early stage, before a climactic trial of the most attractive scheme for burning the structure down in the shortest possible time. Comparisons were made between the M-1 Fire Starter, the Pocket Incendiary, and the Paul Revere. Tests were made to determine the most effective ways of planting the incendiaries, of opening specific windows and doors for provision of an effective draft, and of arranging furniture for rapid progression of fire. An expert photographic crew took movies and stills that provided material for a training film and for the training manual.

The INTRODUCTION reads as follows:

"Fire is one of the most powerful weapons of modern warfare. In this war devastating blows have been struck at the enemy with incendiary

bombings from the air, flamethrowers in battle, and direct attacks on selected targets by special operators.

"In bombing operations explosives are indispensable for knocking out power stations, bridges, dams, water systems, and comparable targets, but the destruction is strictly localized. Incendiaries sometimes pay off more heavily because of the rapid spreading of fire from the area of impact of the bombs to neighboring areas. A few initially small fires can lead to a conflagration that engulfs and wholly destroys everything in its path.

"Fire is also lethal and terrifying to both military and civilian personnel. Thus the Isle de Cezembre, an island fortress off the coast of France, which was able to withstand prolonged bombardment with explosives by virtue of deep-set concrete fortifications, eventually capitulated to an attack with incendiaries on August 31, 1944.

"The operator is in a particularly favorable position to utilize to the full the tremendously effective power of fire. Armed with time-delay incendiary devices, he can take advantage of the all important element of surprise and still protect himself with an alibi for the time of the operation. He can use a very small incendiary, for the device need serve only as the igniter of combustible material in the target. He can place the incendiary where he chooses and can rearrange combustible objects to optimum advantage.

"Compare the bombardier and the operator. The former knows that if he gets through the flak, and if he gets on course, and if his aim is perfect, the percentage of hits will be no greater than the percentage of roof coverage — at best some 20-40 percent. He also knows that only a fraction of the bombs that hit buildings will land where they have a chance of setting fires, that there is always a lot of floor space, and that a bomb in the middle of a floor is next to useless. Finally, the bombing operation is well advertised to the enemy and the bombs will be attacked immediately by alert and trained citizenry by the thousand. That great damage can be done in the face of these very adverse odds is an indication of the intrinsic value of attack by fire.

"The operator also has to get through his particular kind of flak and get on course, but beyond that there is hardly an odd against him. His every bomb can be a direct hit and his every hit can represent a highly favorable location. He can time his incendiaries to go off when there is least chance of countermeasures and he can plant the devices where the fires will not be detected until they are at or near a point beyond control.

"Incendiary devices when cleverly used can be ideal weapons. For specific

missions the operator can select from a number of scientifically designed American and British stores.

"This manual is designed to illustrate the principles of fire starting which are applicable to the field and to describe incendiary devices that are available or easily improvised. Methods of starting fires are illustrated for the most part with the use of the time-delay Pocket Incendiary, containing gelled kerosene, or with the Fire Starter, which is a small incendiary of the same type.

"The prime objective in any incendiary operation is to get a raging fire going quickly. The time element is extremely important. A fire started in the corner of a room may still be extinguishable by a stirrup pump after a critical period, for example of 5 or of 15 minutes, but wholly beyond such control if allowed to continue any longer, say for 6 or for 16 minutes. Once a critical point has been reached fire travels with extreme rapidity. Anything that can be done to increase the speed of reaching the critical point will contribute greatly to the success of the operation."

Some of the principles of fire starting are illustrated by photographic records of experiments with the M-1 Fire Starter. With such a small device

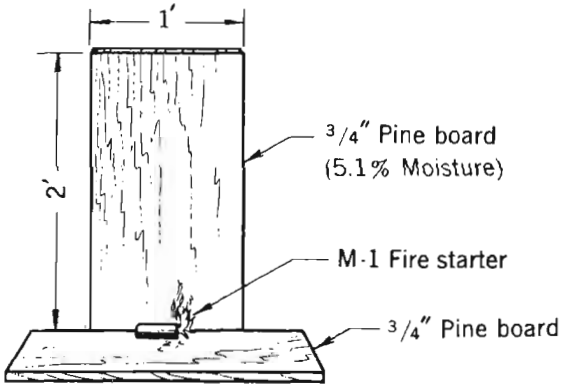


Fig. 19.2

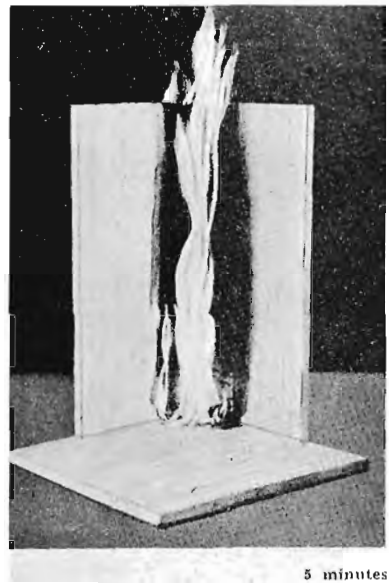


Fig. 19.3

a location against a single vertical surface (Fig. 19.2) is not very effective. A corner location (Fig. 19.3) permits radiation between two vertical sur-

faces. An incendiary planted at an eaves line (Fig. 19.4) is very effective, for radiation of heat from one wall surface to another one at close distance sets a permanent fire which travels by convection along the upper surface and radiates heat to the lower surface so that the two fires reinforce one another. A still more rapid result is obtained (Fig. 19.5) by placing the Fire Starter under a small board resting at an angle against one wall of a corner

Fig. 19.4

Fig. 19.5

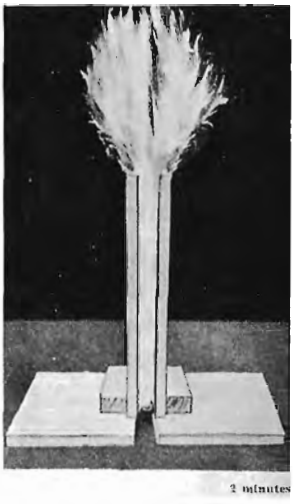
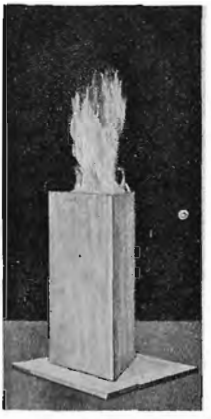


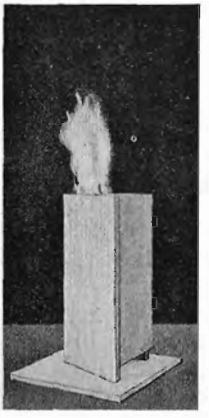
Fig. 19.6

2 minutes



5 minutes

Fig. 19.7. — No draft and fire is choked out from lack of air, despite excellent radiation



2 minutes

5 minutes

Fig. 19.8. — Draft by means of influx of air at the base of the structure promotes a rapid conflagration

and separated from the other wall by a ¾-inch vent. The board does much more than provide added fuel. It acts as a heat baffle to confine the heat and radiate it downwards without obstructing the draft. The space between two vertical surfaces (Fig. 19.6) is an ideal location for an incendiary. Heat from one surface is radiated to the other, and the channel between the boards forms a chimney that creates a draft and promotes combustion. Figs. 19.7 and 19.8 illustrate the importance of arranging for adequate draft.

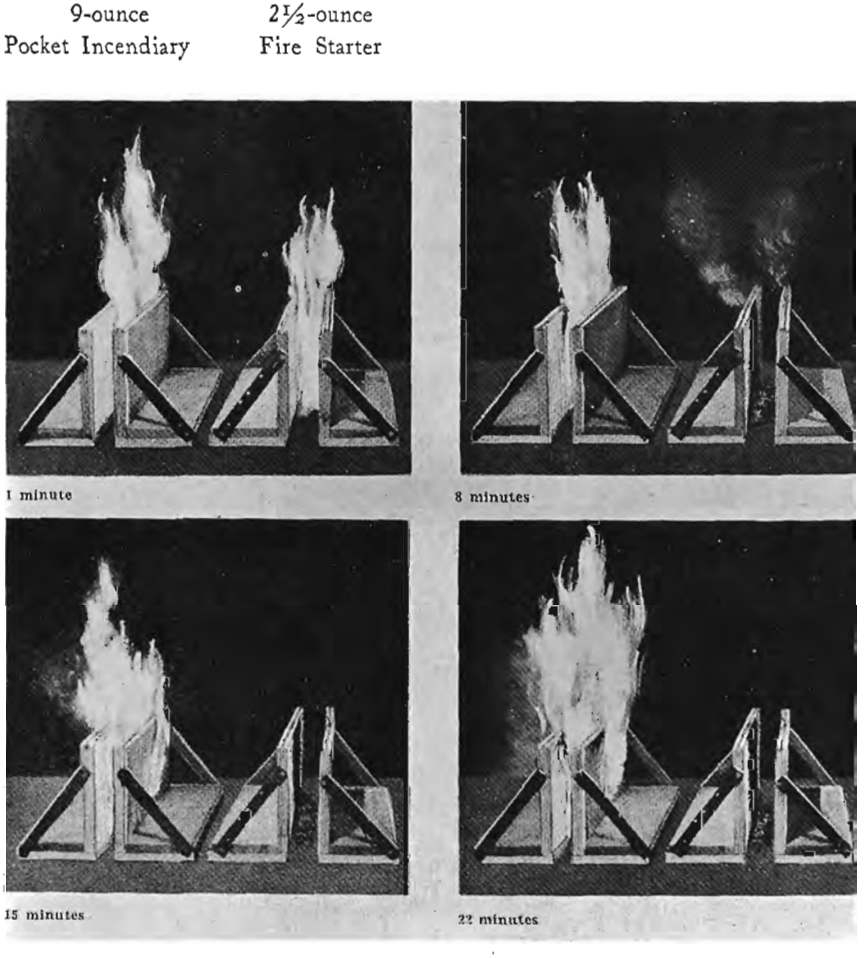


Fig. 19.9. — Comparative tests with spruce "packing case" structures immersed in water for 24 hours

Further illustrations of the same type demonstrate the insulating effect of paint and of charred surfaces. Another (Fig. 19.9) shows that the small Fire Starter does not burn long enough to set permanent fire to abutting packing-box structures after the wood has been immersed in water for 24 hours, but that the larger Pocket Incendiary will do the job.

The manual then suggests ways in which an operator can make practical application of these principles of fire starting. Fig. 19.10 shows scenes from the training film. The "operator," in this case a Naval Lieutenant assigned to the TVA project, planted a PI in a corner and placed an upturned chair over it, and opened an upstairs window and a downstairs door; the house collapsed shortly after the 15-minute scene. The sequence shown earlier in

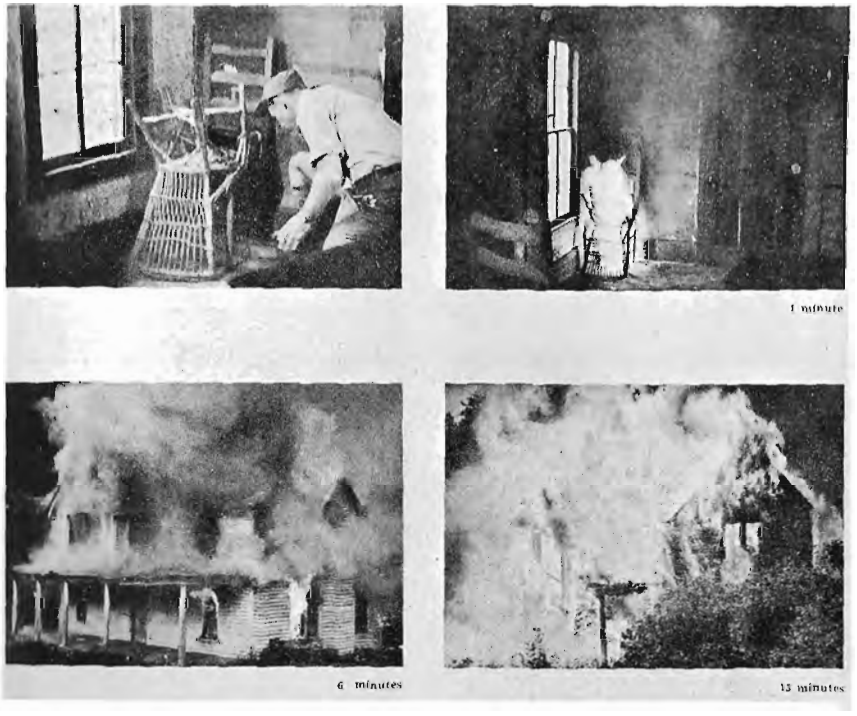


Fig. 19.10. — Rapid spread of fire under favorable conditions. Chair used as heat baffle

Fig. 6.15 demonstrates application of the principle of closely abutting vertical surfaces. These photos were taken at the Factory Mutual test station at Norwood. The dramatic sequence of Fig. 19.11 was also staged at Norwood. I recall that the pretty-girl picture caught on fire by mistake and

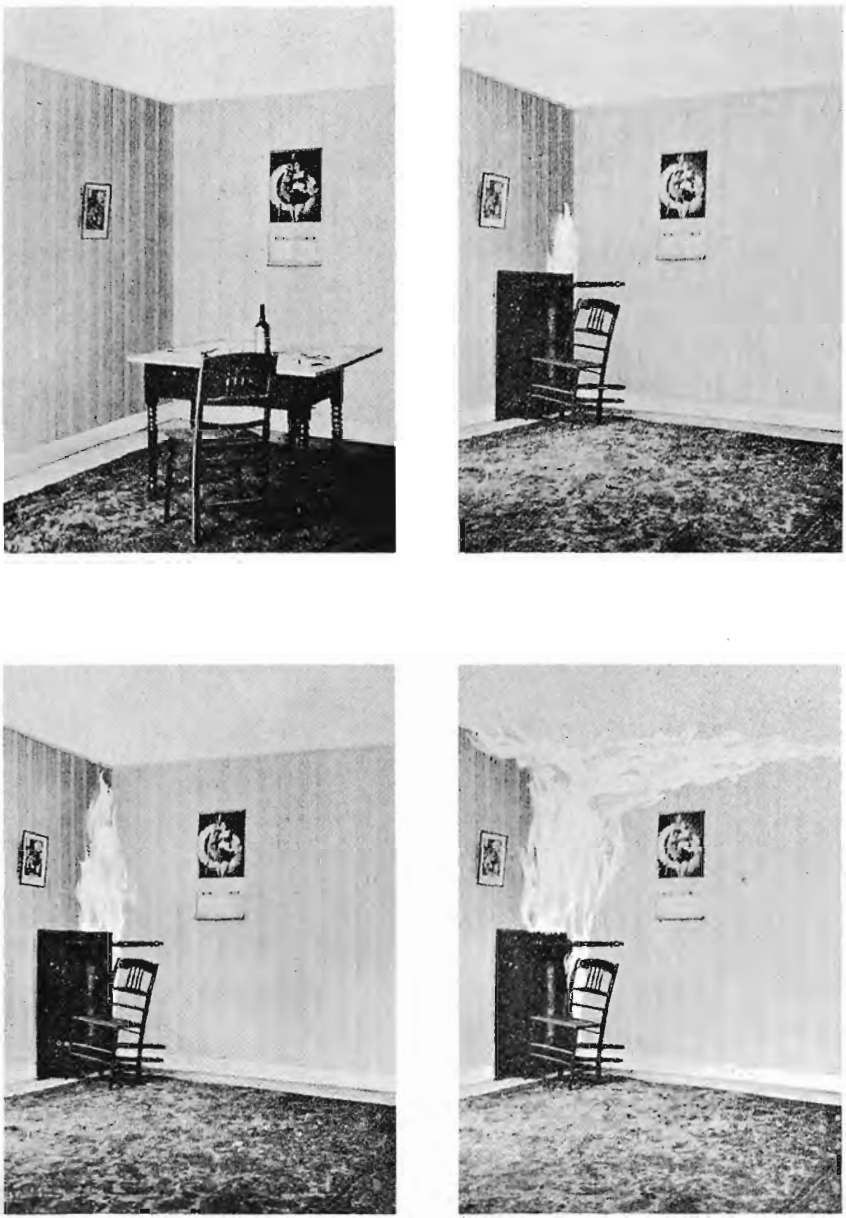


Fig. 19.11. — Pocket Incendiary operated in the space between a wall and the top of a turned-over table

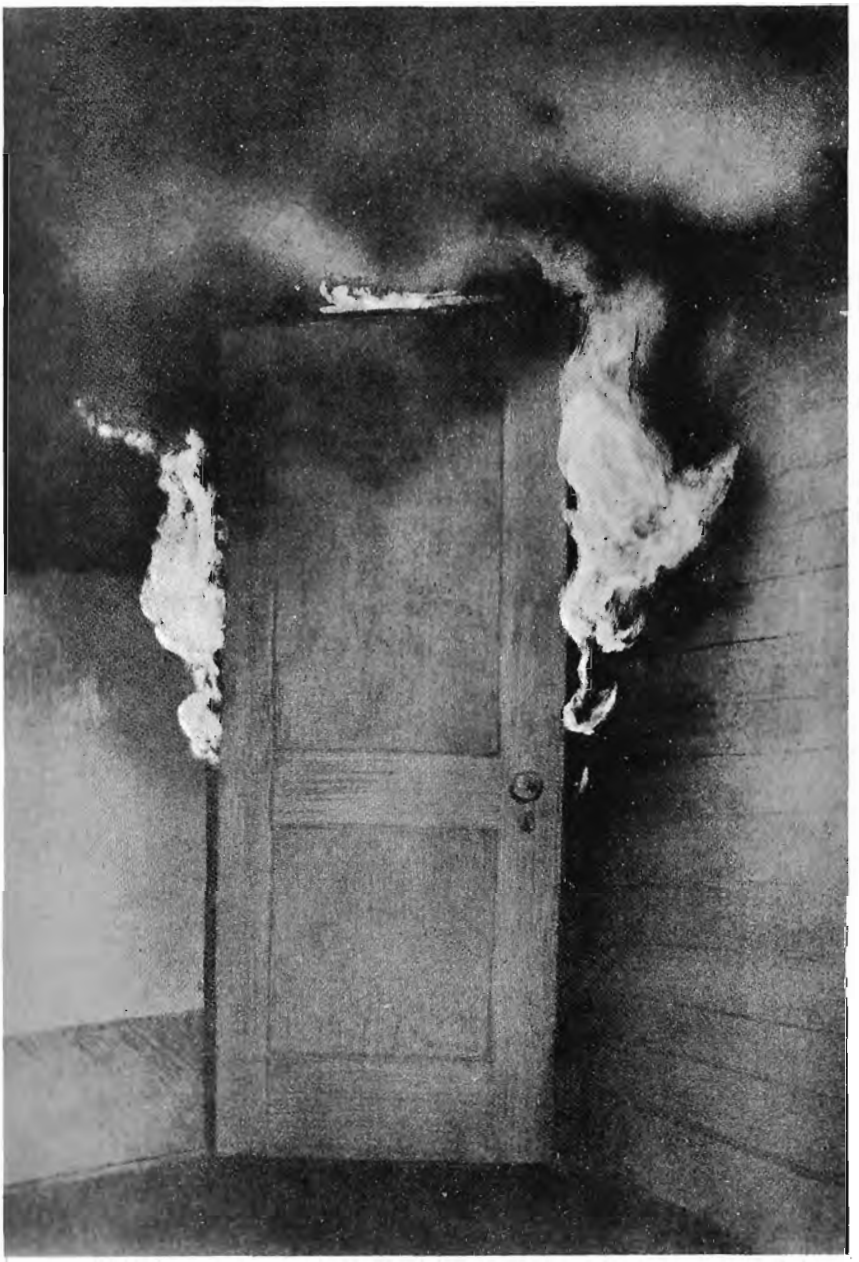


Fig. 19.12. — Pocket Incendiary operated behind a door opened to form a triangular chimney with corner. Photographed after 2½ minutes.

was restored by a retouch artist, who also supplied wallpaper and eliminated smoke. The application of theory to practice shown in Fig. 19.12 is a product of the TVA tests, retouched only for removal of an idling sergeant.

Fire can be a good friend as well as a deadly foe. In a protected burning area in back of a brick garage, I make use of the principles of starting and propagating fire in the disposal of limbs and trees and hurricane litter. Napalm gel has a peacetime use: eradication of crabgrass. In late August, when crabgrass begins to bear seeds, mowing is suspended for a few weeks to let the seeds accumulate. A thin Napalm gel is prepared in a glass jar with a screw cap punched with holes. Strands of gel are then laid over the grass, a small section being treated at a time. With an extinguisher in one hand, the operator tosses in a match with the other hand. The ugly seeds are completely destroyed. So is the grass; the lawn is initially black. But roots of the grass are not damaged and the first rain brings out a green, crabgrass-free lawn.

20. *Alsos Mission*

When the invasion of Germany again began to appear imminent, I was called to Washington for further processing and final take-off. Warned by the first experience that there might be some delay, I persuaded Mary to join me while waiting for my call from the Air Transport Command. So, in the period February 27–March 5, 1945, she and I had a pleasant sojourn in the nation's capital and again made the rounds of the elegant restaurants, particularly our favorite, Harvey's. Each dinner might be the last one for a time, and so was the occasion for a celebration. I felt a little silly in uniform

Fig. 20.1. — Washington, 1945

as an unarmed consultant to the army with assimilated rank of Colonel, which meant that I had this rank at Officers Clubs and in case of capture by the enemy. The period of waiting just sufficed for completion of the write-up of the Arson manual and for making the rounds to NDRC, CMR, OSS, British Air Ministry, British Petroleum Warfare, etc., to arrange for things to do in London in case of a dull period of waiting in Paris. An OSS officer,

noting the swagger stick given to me by Mary, had it modified so that pull on the handle withdrew a triangular dagger from a scabbard drille into the wooden stock. Finally I took off in a DC-54 plane operated by TW for the Air Transport Command in the first direct flight to Paris:

Lv. Washington 5 March 1945 at 14:05
Av. Stephenville, N.F. 19:40
Lv. Stephenville, N.F. 20:40
Av. Azores 6 March 1945 . . . 6:00
Lv. Azores 7:30
Av. Paris, Orly Airport 16:30

The ensuing two-month period included exciting adventures, unusua experiences, unbelievable sights and spectacles, and both amusing and try ing incidents. But any contribution an organic chemist might make to th secret mission was necessarily a minor one; I regard this trip as an activit of minor consequence and will confine a description to the terse diar comment:

March 6–May 8, 1945. — European Theater of Operations, U.S. Army, Expert Consultant with assimilated rank of Colonel, member of ALSOS Mission, Military Intelligence Service, attached to G-2 Headquarters Etousa.

"Reported at Paris but soon tired of loafing around offices and so flew to London (March 16, ATC from Orly) to promote our naphthoquinones, the E-19, etc. The break across the Rhine came sooner than expected and I was hastily recalled to Paris to go into the IG Farbenindustrie plant as soon as Ludwigshaven fell (delayed one day by weather, flew from Higgins Hill by ATG to Villa Courblay on March 26th). The main party had left and I proceeded in a jeep with Naval Lt. Hofer and Sgt. Pfunder to try to locate a G-2 Major Bullock in contact with a 6th Army Task Force either at Luneville or somewhere in Germany. We made our way almost into the IG plant but fortunately stopped to consult the commander of a tank battalion and found that he was awaiting orders to attack the still untaken plant. We finally found the T-force and the major, visited the IG when it was taken, interrogated IG directors and chemists, experienced 48 hours of an artillery barrage lobbing shells over our heads in preparation for Patch's 6th Army Rhine crossing close by our billets at Frankenthal, and so became initiated to an exciting series of adventures with the lashing drives of the victorious American armies.

"A diary of the activities in conquered Germany is included in an ALSOS report. As has been revealed in a report by our scientific chief Dr. Sam Goudsmit to a Senate Committee, the carefully concealed secret purpose

Fig. 20.2. — Braunschweig, Germany

of the ALSOS Mission was to find out just what the Germans were doing on nuclear fission and the development of an atomic bomb. When it became apparent that the Germans were years behind us and that we had more scientists on the intelligence mission than they had on the problem, I decided to concentrate on diversionary activities and in particular to sound out the temper of my German colleagues. It soon became apparent that these men had not been sacrificing their scientific activities for the war effort, as we had, and I decided that it was time for American chemists to get back to research and scholarship. Therefore, I cut the corners, passed up the opportunity to linger, visit and sightsee, and came home at the earliest opportunity (left Orly by ATC the night of VE day; flew by way of the Azores and Newfoundland to Washington; dinner at Harvey's with Mary on VE + 2 day!)."

21. *Experiments for Students*

It is my firm conviction that teaching is materially improved by participation of the teacher in research. Personal examples for illustration are on record in our books, particularly in a laboratory manual of student experiments first published in 1935. I had learned to determine the percentages of carbon and hydrogen by the old macrocombustion method requiring a sample of 100–120 milligrams. I learned enough about the new semimicro method requiring only 50 milligrams from my research students to include a description in the 1935 manual. A full-page engraving of an improved assembly that appeared in the textbook of 1944 was drawn directly from Hans Heymann's apparatus. An interesting entry in the 1935 manual was illustrated with the drawing shown in Fig. 21.1. Parallel cuts

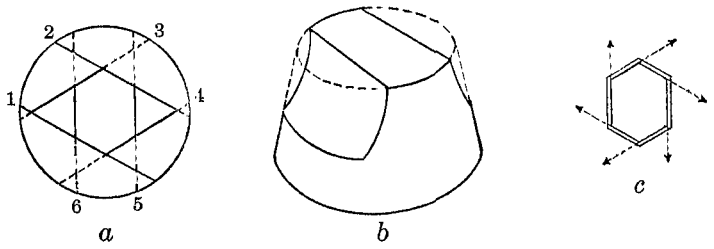


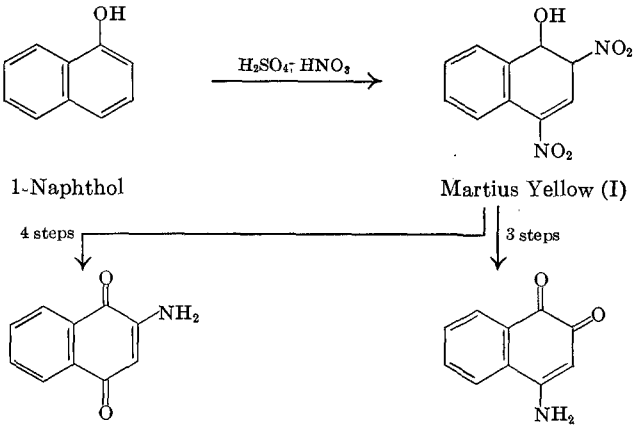
Fig. 21.1. — Cutting a Benzene-ring Stamp from an Aged Stopper

1 and 2 (*a*) are made by slowly drawing the blade of a razor back and forth with steady pressure. After each line has been cut to a depth of 2–3 millimeters, the stopper is cut away roughly to give the tapered slice *b*. Cuts 3 and 4 are made in the same way, and then cuts 5 and 6. The stopper is then cut away on all sides. To clear out the inside of the hexagon and leave an outline, cuts about 1 millimeter deep are made as indicated in *c* by careful working with a sharp blade. The cut in each corner of the ring does not show in the completed stamp. The rubber in the center is carefully cut away by folding back each edge, laying the razor flat, and cutting horizontally at a depth of 1 millimeter. The inventor of this gadget is one of the

research students mentioned in this book. Perhaps the reader can guess his identity when I say that it takes considerable dexterity to cut out perfectly proportioned stamps measuring as little as 3 millimeters across. The inventive and skilled carver is now Surgeon-in-Chief of the Sinai Hospital, Baltimore.

Lest I give the impression that the entire book was made up of contributions by my research men, I will cite the history of the Martius Yellow experiment. Karl Alexander von Martius discovered in 1868 that nitration of 1-naphthol gives a bright yellow compound which dyes wool and silk. It was soon introduced as a dye for wool under the name Martius Yellow. The dye has the unique property of rendering dyed cloth mothproof. A possible analogy to the mothproofing effect occurred to me when a gray squirrel was trapped in our empty house for three days. In repeated efforts to escape, the squirrel chewed seven window sashes beyond repair. The sashes attacked were in different rooms, but all of them were stained; painted sashes were left untouched.

Martius Yellow is easy to prepare and the early literature reports its conversion, in a series of steps, to compounds which I shall designate Red Quinone and Orange Quinone. I became interested in securing samples of



these quinones during a half-year period of postdoctoral research with Professor Conant in 1925. I thought it of interest to measure the oxidation-reduction potentials of these two quinones over a range of pH from strong acidity to strong alkalinity. If I only had a small supply of each compound I could make the potentiometric measurements on my own time in evenings

and on Sundays. However, the literature directions were scanty and unclear and I had no time to work on the preparations. So I engaged graduate student George H. Reid to work as my private assistant and prepare the samples. George worked for two weeks, which was all my research budget would stand, and got exactly nowhere. It was nearly ten years until Mary and I did a joint piece of work on the problem with results more exciting than anticipated. I prepared the red and the orange quinone, and a number

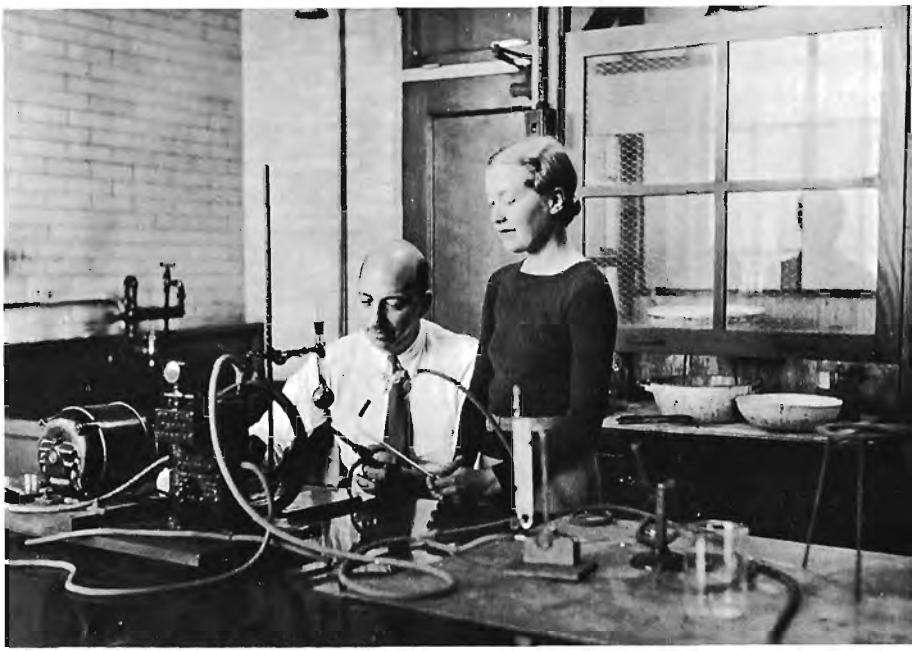


Fig. 21.2. — Joint Work of 1934 (Photo by E. B. Hershberg)

of related compounds, and Mary made the measurements (Fig. 21.3). The compounds in the series derived from Martius Yellow are sensitive and easily destroyed, but once suitable tricks for handling them had been discovered I found that all the reactions can be conducted very rapidly and afford products of high purity in excellent yield. There are four isolable intermediates between Martius Yellow, Compound I, and the final quinones VI and VII. The entire series thus includes seven compounds. These all form beautiful crystals and include a range of color: yellow, bright red, white, dark red, orange.

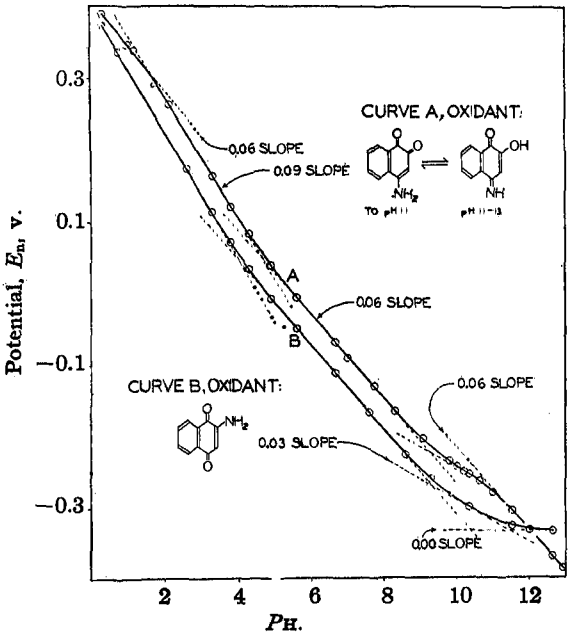


Fig. 21.3. — Relationship between pH and the potential of half-oxidation for the aminonaphthoquinones

With a little further work I adapted the reactions to student experimentation and included the Martius Yellow sequence in the 1935 manual. The directions call for starting with 5 grams of 1-naphthol and saving small samples of each recrystallized intermediate. Students in my course seemed to enjoy this synthetic sequence and before long it became the basis for an annual Martius Yellow Prize Competition. From one to three students from each of 10–12 sections were selected for entry by the section supervisors. The competition involved extra work and was voluntary, but no one ever failed to exercise the option. A new book of ours or new edition often served as a suitable first prize, and gratis copies were obtainable for the purpose from the publishers. Each year three postdoctoral members of my group served as judges and rated the contestants on technique and neatness, but the prime test was in the number of samples submitted and the total working time. Solutions could be set aside for crystallization over

Friday night without this counting as working time. The next day each contestant would check in again, and finally check out when the samples had been bottled and the bench cleaned up. There was little room for bungling, for the loss of Compound II eliminated the remaining compounds in the subseries. The fiery red Compound II crystallizes beautifully, but supersaturates to a marked degree; unless the inner walls of the containing vessel are scratched by an exactly correct technique, no product is obtained at this point. Students who, all year, had ignored the admonition to study the manual before reporting for laboratory work, checked into the Martius Yellow competition with the procedures either memorized or outlined in charts. For once they worked with full efficiency. When the student time for preparation of satisfactory samples of all seven compounds hovered around the four-hour mark, I entered the competition as a sort of pacemaker. The judges invariably ruled me disqualified on the ground of whistling in the laboratory, smoking, or getting advice from Mary Fieser, and so I did not interfere with any prize awards. But with the practice gained each year I made the following times: 1950, 3:16; 1952, 2:54; 1954, 2:26; 1956, 1:59. But the class included some extremely able students who were glad to accept the challenge. Louis Harris beat me in 1954 with a time of 2:19. I topped this record two years later with a time of 1:59, but Howard R. Sloan lopped 9 minutes off of this time and holds the record at 1 hour and 50 minutes. In little more time than some students spend over lunch or at the movies, this keen operator had prepared seven beautiful compounds and cleaned up all the flasks and apparatus used.

The second edition of the laboratory manual, published in 1941, included new student experiments based on local researches on vitamin K_1 and on resin acids. Another feature is evident from the following passage of the preface: "I take pleasure in acknowledging particularly the invaluable advice and assistance of Dr. E. B. Hershberg, who kindly contributed the section on glass blowing and who is responsible for the design of the majority of the pieces of apparatus illustrated in the engravings."

When the time came for a third edition (1955) I decided that the whole book needed revision and modernization. More synthetic sequences of the Martius Yellow type should be included. New techniques of separation and isolation should be demonstrated. At least one example of the use of an enzyme in preparative chemistry seemed desirable. I spent a full year and a half in the laboratory or shop working out new experiments and devising apparatus with which new and old experiments could be carried out by an able student with the finesse of a research chemist. Nearly all the experi-

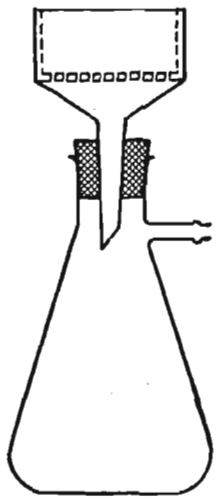


Fig. 21.4. — Büchner Funnel and Filter Flask

ments were to be done on a small scale (1–2 grams), for this shortens the time, cuts down the expense, and makes the operations neater. But small-scale experimentation required low-cost equipment not then available. One operation, suction filtration of a precipitate or a crystallizate, can be done with a porcelain funnel with a perforated plate (Fig. 21.4) which is covered by a circle of filter paper. The sidetube is connected to a water suction pump by a piece of rubber tubing of such a wall thickness that the tube will not collapse under vacuum. In the improved assembly shown in Fig. 21.5 the rubber stopper is replaced by a neoprene filter adapter of appropriate size. The assembly on the left for large-scale work has a wide enough base to stand up by itself when the heavy rubber suction tubing is attached, but the two smaller units are liable to topple over unless supported in some way. My solution was to provide a filter block (Fig. 21.6) having a groove into which

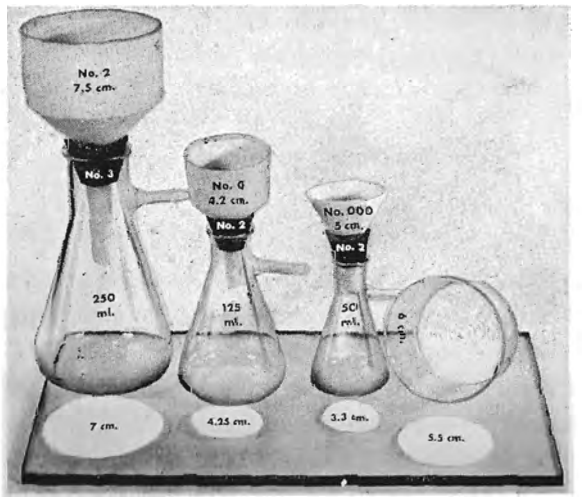


Fig. 21.5. — Matching Filter Assemblies

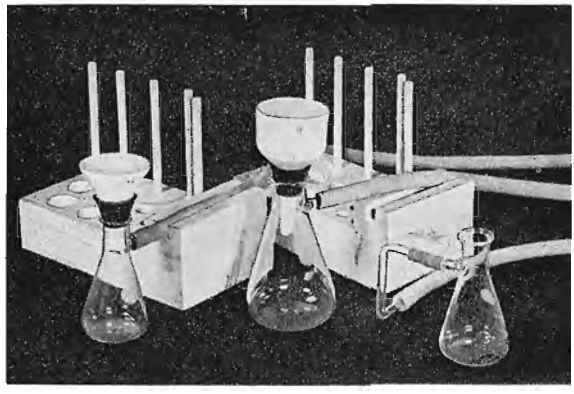


Fig. 21.6. — Filter Blocks and a Filter Flask Adapter

the suction tubing fits snugly to form a support. The neat glass adapter was designed by William J. Leanza of the Merck Laboratories. The filter block (Fig. 21.7) serves also for the support of test tubes of four sizes, flasks, graduates, and stirring rods (one flattened on the end for crushing crystals to facilitate solution). A filter flask is not connected to the water pump di-

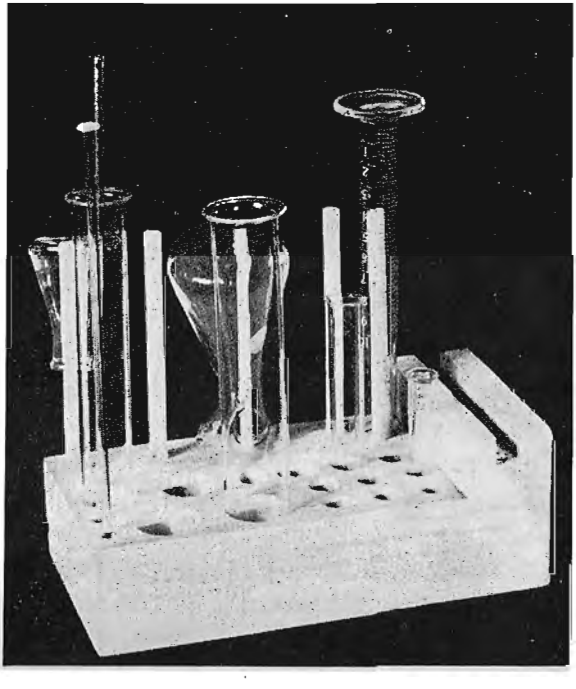
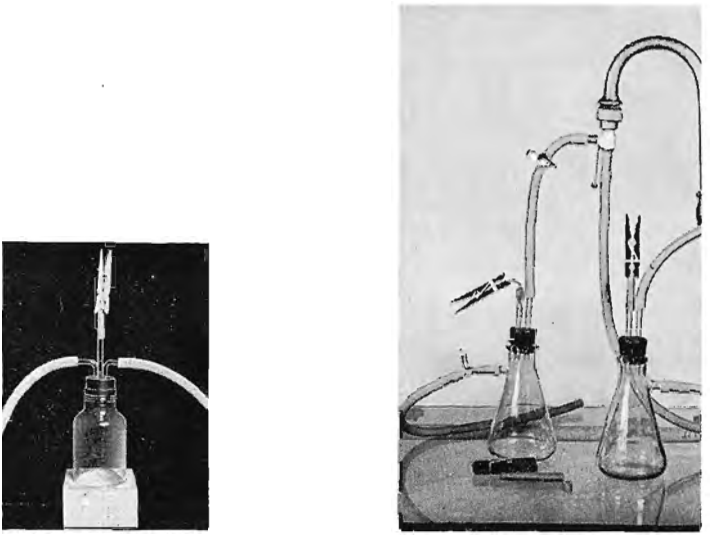


Fig. 21.7. — Filter Block

rectly but through a second vessel that serves as a reservoir for equalization of pressure and as a trap for filtrate that may be carried over in a foam from the filter flask. This reservoir-trap must be provided with a valve for ready release of pressure in case the filtrate starts to foam. A researcher can use a filter flask with a 2-hole rubber stopper; a glass tube inserted in one hole makes connection to the filter flask and a glass stopcock inserted in the second hole serves as pressure-release valve. But a stopcock costs $3.40 and the Pyrex filter flask fitted with a rubber stopper costs $1.90. A supply of such traps for my class of some 250 students would run into money. A trap of my design (Fig. 21.8) now supplied by Wilkens-Anderson Co. costs



a b

Fig. 21.8. — Filter Traps

$1.25 each. The vessel is an inexpensive soft-glass bottle mounted for stability in a wooden support block. The valve for release of pressure is a 1-cent spring clothes pin from Woolworth's which closes a short section of thin-walled rubber tubing. Use of the device soon disclosed another valuable function, shown in the photograph (b) of earlier and more costly models. Before suction is applied the spring clothes pin is upright (right), but as suction is applied the thin-walled tubing collapses and the clothes pin bends down as shown on the left. The operator crimps the delivery tube shut and notes the angle of dip attainable with his pump turned on full force. When connection is made to a flask or other system to be evacuated,

he can note at once whether or not the system is tight. If, for example, he uses a cork stopper instead of a rubber one, he may be warned from the "pressure gauge" that air is leaking in through worm holes.

A useful accessory gadget is a steel spring clip screwed onto a hardwood clothes pin (Fig. 21.9). Rounded sections of two sizes support test tubes

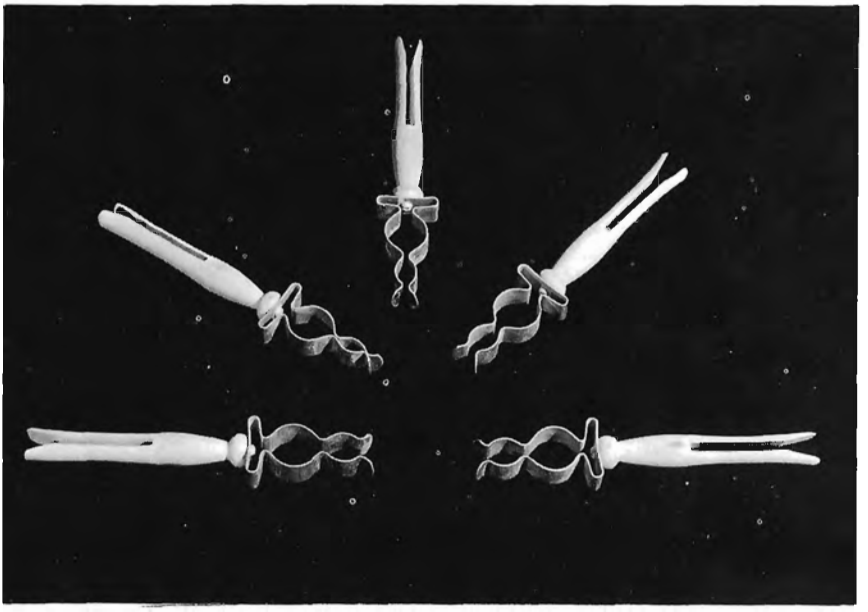


Fig. 21.9. — Spring Clips

and flasks of a wide range of diameter. The double-grip clip was patterned after a commercial model designed to hold heavy tools and with a spring so stiff that it breaks glass. I took a model fashioned out of copper to a spring maker in New York and he turned out a trial lot of steel clips by hand at a cost of $1.67 each. The clips proved satisfactory, and clips made by tool machinery are now listed by Wilkens-Anderson at 6 cents each. A clothes pin with clip attached serves as a convenient holder for manipulating flasks and test tubes too hot for the fingers. In combination with a dowel stand (Fig. 21.10), it can be used also for the support of tubes and flasks. The clothes pin fits snugly at different depths in holes drilled into the vertical dowel. Use of this equipment for the convenient support of separatory funnels is shown in Fig. 21.11. This photograph, which appeared in the manual in color, demonstrates techniques learned in the antimalarial research. Each funnel contained a solution of 100 milligrams

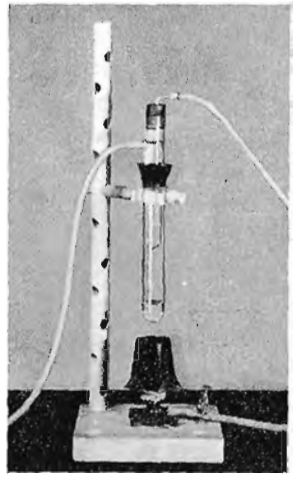


Fig. 21.10. — Cold-finger Reflux Condenser Mounted on a Dowel Stand

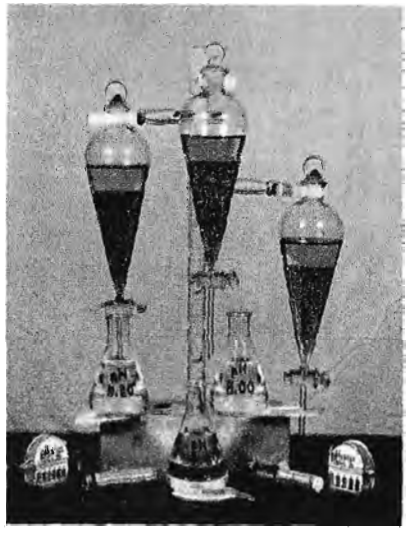


Fig. 21.11. — Determination of the Extraction Coefficient (pE) of Lapachol

of lapachol in 100 milliliters of ether, which had been equilibrated with 100 milliliters of buffers of pH 8.00 (right), pH 8.60, and pH 9.20. The pH values were marked on the Erlenmeyer flasks and visualized with strips of Hydrion pH Paper taken from the convenient dispensers shown to right and left. In the color photograph one could easily see a deepening in color with increasing pH. The amounts extracted into the aqueous

buffer as the red salt were: pH 8.00, 4.7 milligrams; pH 8.60, 16.6 milligrams; pH 9.20, 43.7 milligrams. The data for each solution permit calculation of pE; the three results were 7.31, 7.30, 7.31.

Inclusion in the manual of an 8-page folio of color photographs alarmed some purchasers who assumed that this had increased the cost of the book. Such was not the case. The reproduction cost of about $4,000 came out of my royalty. The extraction picture of Fig. 21.11 later brought an unexpected return. The Hydrion pH Paper contains a series of dyes which cause a progression of color changes from red at pH 1, through brown at pH 6, green at pH 8, to blue at pH 11. The cleverly designed dispenser (Fig. 21.12) contains two rolls of paper. Color charts at front and back are

Fig. 21.12

for matching the color of a strip of test paper after it has been dipped in a buffer solution and so determining the pH to 0.25 unit. This useful device is manufactured by Micro Essential Laboratory, Inc., Brooklyn, N.Y. The color photograph evidently made a hit with the company, for I received a beautiful extra-thin gold Movado watch and an accompanying letter from Mr. Walter Florin:

> June 7, 1962. — "Life is generally full of surprises and we hope the following is one that pleases you.
>
> We are celebrating the 25th anniversary of Hydrion pH Paper production. In honor of this milestone we are sending to you a HYDRION 25th ANNIVERSARY WATCH.

We thank you for your selection of Hydrion in presenting certain experiments in your Organic Chemistry Text."

I can give Hydrion paper a further plug in the present book without embarrassment, for I do not expect to be around for the 50th anniversary.

An electric hot plate for use in crystallization is an indispensable item of equipment in research, but the best model at the time available cost about $25. A project to develop a hot plate for students interested Dr. Irving A. Kaye, who was spending a sabbatical year working with Mary on a sapogenin problem, and he joined me in the search. An idea suggested by Kaye got the project moving: use of the standard 3-inch ceramic element of the ordinary electric coffee maker. This ceramic piece costs about 8 cents each. We devised a simple technique for winding a coil of resistance wire of the proper wattage and inserting it into the grooves of the ceramic piece. An assembled heating element is shown in Fig. 21.13 resting on top of a tin can. The empty can, cut away as shown in the middle, forms a stand for support

Fig. 21.13. — Homemade Hotplate

of the heating element and handles (left). Experience with these early heaters dictated the specifications for the ship-shape model developed by Wilkens-Anderson Co. and marketed for $2.95 (Fig. 21.14).

Sorely needed was something better than the fractionating column described in the first two editions of my book and shown in Fig. 21.15. Actually I inherited the Hempel column packed with pieces of glass tube when I took over Chemistry 2 from Dr. Conant, and I believe it was used in most schools. A fractionating column for the separation of two liquids of different boiling point is supposed to operate as follows. Once the column has been heated throughout its length, vapor rich in the more volatile component comes off at the top into the condenser, and condensate rich in

Fig. 21.14. — Wilkens-Anderson Hot Plate

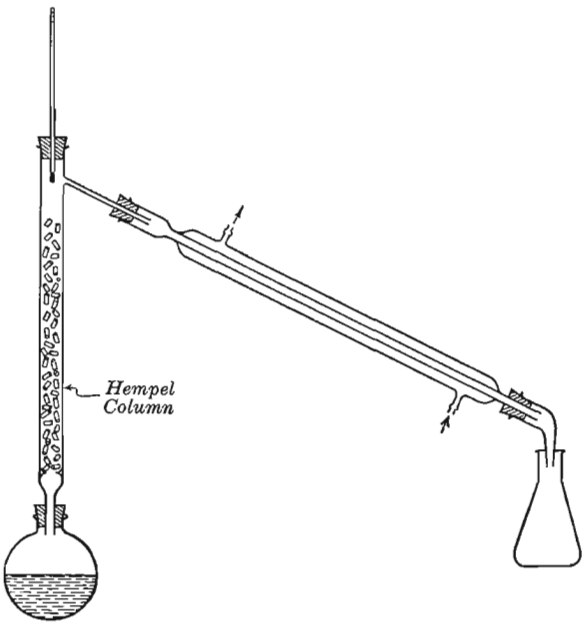


Fig. 21.15. — Distillation with a Fractionating Column

the less volatile component drips back into the flask at the bottom. Throughout the column, rising vapor pushes its way through descending condensate and volatilizes the more volatile part of it while giving up to the liquid phase its least volatile part. For efficient operation the porous packing must

present just the right balance between obstruction and free flow. Glass helices (Fig. 21.16) form the preferred packing in research work, and a trial in the simple student column of Fig. 21.15 for the separation of ben-

Fig. 21.16. — Glass Helices

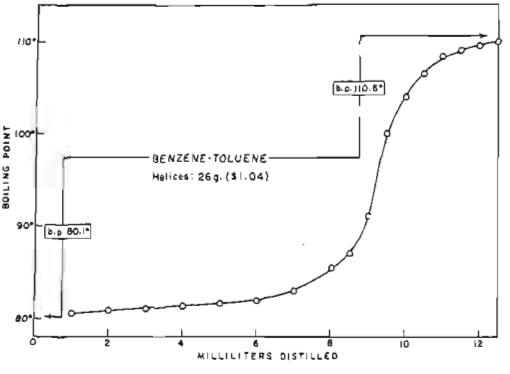


Fig. 21.17. — Distillation Curve (Helices)

zene and toluene gave the very satisfactory distillation curve shown in Fig. 21.17. But helices are very expensive, very fragile and easily spilled, and some little time is required for loading a column, if it is to be done properly. A column of glass beads (Fig. 21.18) performed satisfactorily, but beads,

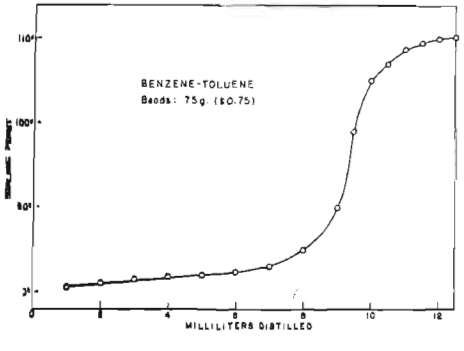


Fig. 21.18 — Column of Glass Beads

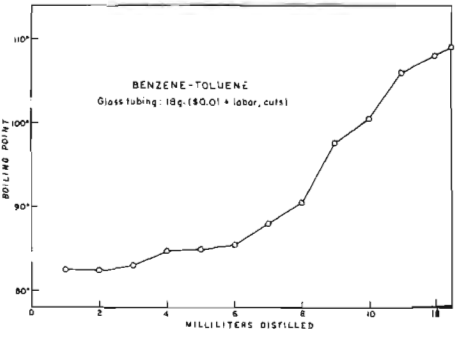


Fig. 21.19. — Column of Glass Tubes

surprisingly, are almost as costly as helices. The only excuse for using cut up sections of glass tube was that the material is cheap, that is if the labor and hazard of cutting and breaking is ignored. The performance certainly is miserable (Fig. 21.19).

In the hope of getting an idea of some other material that might serve as packing, I made the rounds at Woolworth's. The aquarium department offered a special form of charcoal for water purification; this looked inviting, although a little expensive, and I purchased a box. But I saw something better at the kitchen department: a stainless steel scouring sponge. Back at the laboratory I found that I could pull out a sponge to a length of about 20 inches, cut it in half, catch the end of one piece onto a wooden dowel, and thrust it into the column (Fig. 21.20). The performance? A distilla-

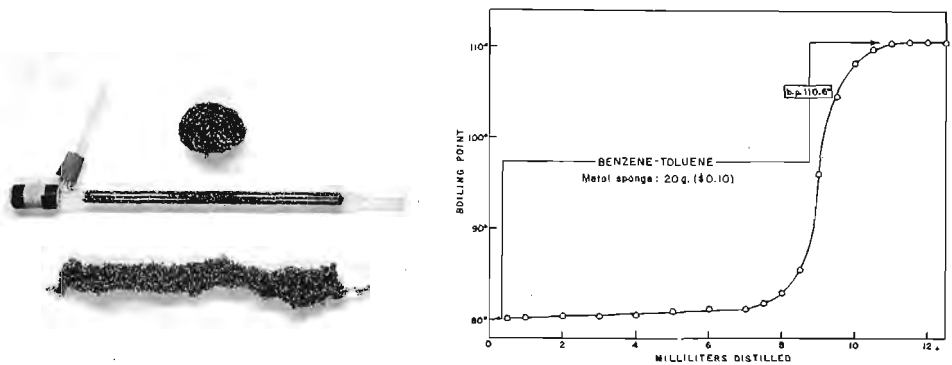


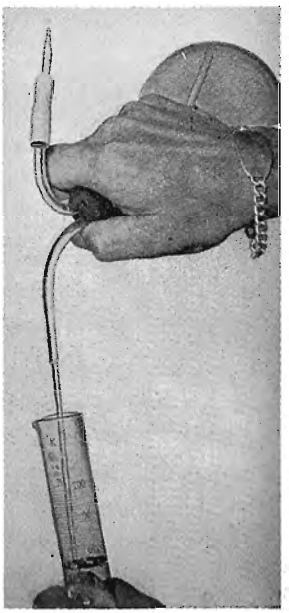
Fig. 21.20. — Stainless Steel Packing and Distillation Curve

tion curve obtained by the same operator in the same apparatus is a little sharper and better than that obtained with glass helices. Nowadays the student is given an unknown containing a mixture of any two liquids of a list of ten, and in any proportion. The problem is to identify each liquid from the initial and terminal boiling points, and to establish the composition from the point of inflection of the distillation curve. With a sponge-packed column the experiment is really fun. The packing is unspillable and unbreakable and the cost is one-tenth that of helices.

As the book progressed I included more and more photographs, for they seem to me more realistic than sketches. Action shots (Fig. 21.21) demonstrate techniques of manipulation and perhaps stimulate the student to





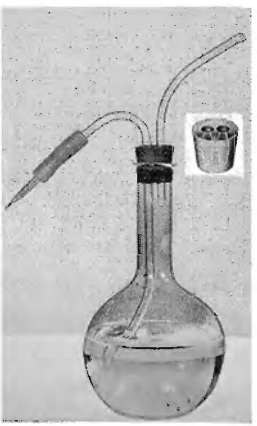
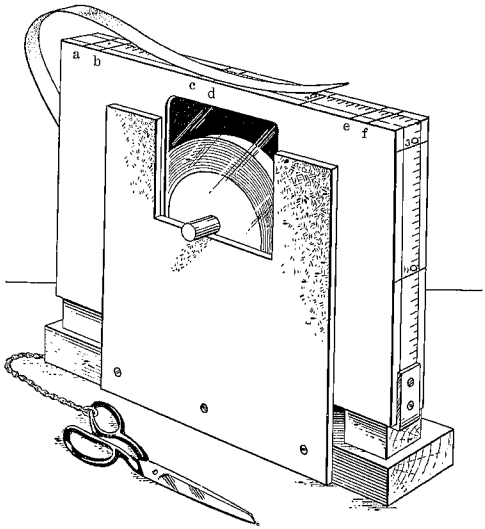


Fig. 21.21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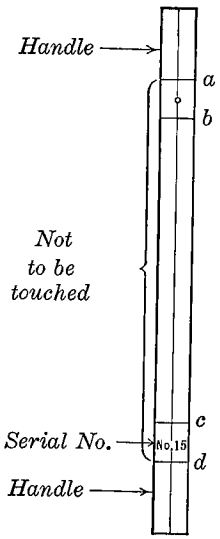
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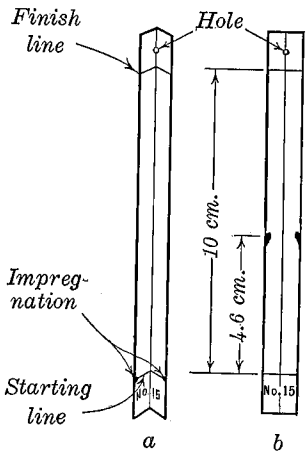
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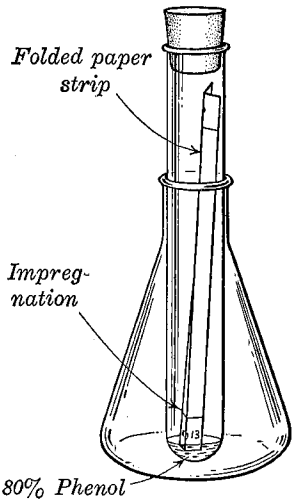
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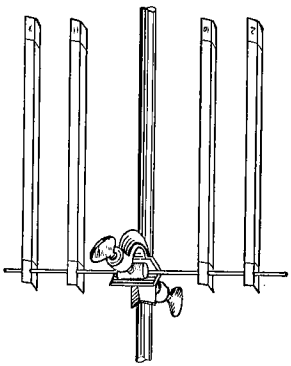


Fig. 21.22

think about development of dexterity. Sketches often are invaluable, for example for description of a technique for carrying out paper strip chromatography (Fig. 21.22), but a summarizing photograph (Fig. 21.23) is attractive, particularly when reproduced in color.

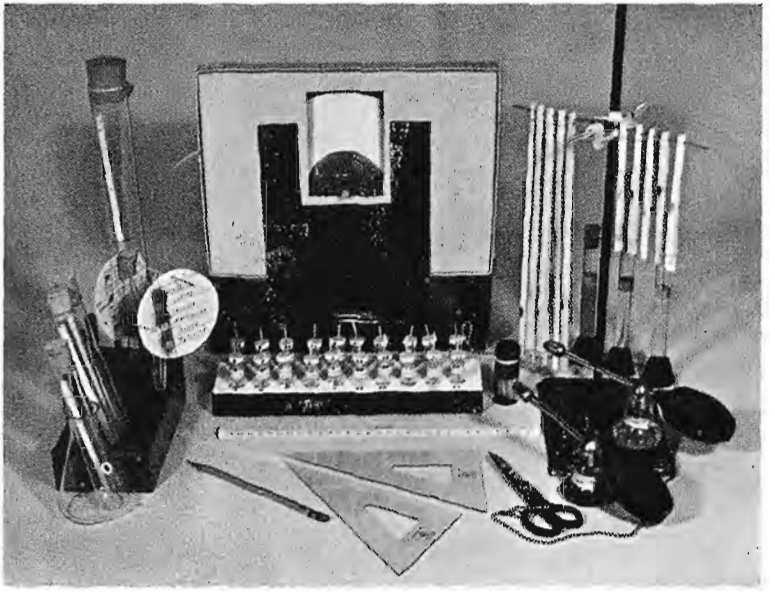


Fig. 21.23

22. *The Movie*

Introduction of the new experiments and new equipment described in the revised manual for the laboratory work of 250 students presented more of a problem than I had anticipated. Neither the instructor in charge of the laboratory work nor the 16 graduate student teaching fellows had had any experience with the equipment or the experiments. I decided to put on demonstrations, as I had done with success some years earlier when the class was smaller (a good one: How NOT to crystallize). But in two groups of 125 students each anything much smaller than a 1-liter flask could not be seen by those in back. The lecture table had no steam bath or proper kind of sink and suction pump. Innumerable items of equipment and chemicals had to be assembled in my laboratory on the second floor of one building and transported to the basement of a second building. What was done for the Tuesday sections had to be repeated for the Wednesday section. I then thought of a movie, and the idea became more and more appealing as I looked into this possibility. Photography could ensure perfect visibility to a student group of any size, even of phenomena on a micro scale. For example, a sample melting in a capillary tube could be shown in a close-up. Striking phenomena could be demonstrated ideally by color photography. An experiment lasting many hours could be shown in a few minutes of film. The demonstrations could be done with all the facilities of one's own laboratory.

A photographer friend went over a tentative script estimated to cover about fifty minutes of film and figured that he could make the movie at a total cost of $3,000. I applied for and received a grant in this amount from the Charles H. Tozier Fund for Visual Education in Science. On a speaking trip to the University of Kansas, I heard of a local producer, the Centron Corporation, with considerable experience in instructional films, and a visit to the studio soon convinced me that my film, to be any good, would have to be done professionally at a cost estimated at $30,000. In seeking further funds, I addressed a letter to an official of the Ford Foundation who turned out to have been a student in my course twenty years earlier, in the period when I conducted demonstrations. He told me

of an appropriation about to be announced to a Committee on Utilization of College Teaching Resources. My project seemed directly in line with the objectives of this committee and I was awarded a grant in the maximal sum of $25,000. As producer I selected Centron Corporation of Lawrence, Kansas, and they agreed to do the $30,000 job for $28,000 and absorb the difference.

I will not record here the trials and tribulations of a first try at making a movie except to note that on the first few days of production in my private laboratory in Cambridge we were confronted with an outside temperature of 96°. Four 2,000-watt spot lights and a couple of 1,000-watt flood lights raised the temperature and made a mess of an otherwise neat laboratory. We were able to operate at all, and to keep perspiration from showing on hands and face, only by rigging up a couple of ash barrels of ice with fans blowing over them (the experience with bats at Carlsbad thus proved useful).

Laboratory scenes done in Cambridge ran to a total take of 6,000 feet of film. Processing, marking, and preliminary editing was done while I was abroad and I went out to Kansas in August to edit, make adjustments and fill-ins, plan the narration, coach the narrator in pronunciation of chemical terms, and for synchronous sound photography. It was necessary to take along the four neckties worn in the four parts of the film. In initial and terminal scenes I was to sit at the desk in my office and address the audience under a microphone. The synthetic office set up with props borrowed from the local university seemed nice enough, but I felt obliged to ask for removal of a periodic table, since I am a one-element chemist. The resulting bare spot on the wall unduly focused attention on a framed diploma, and we were afraid the audience might be able to read the legend: University of Kansas. The situation was saved by substitution for the diploma of a photograph that Mary and I had persuaded the cameraman to take for use in the preface of a forthcoming book (Fig. 22.2) and in this way our usual cat trademark (see later) became inserted in the film.

The studio scene of Fig. 22.1 shows me pointing to a blackboard containing formulas which will be recognized as the Red Quinone and the Orange Quinone of the Martius Yellow sequence. In Part IV of the film I explained the nature of the Martius Yellow competition and then, in a total of 18 minutes of film, ran through the major operations involved. The scene shown in Fig. 22.3 demonstrates the technique of countercurrent distribution for separation of a mixture of lapachol and the natural pigment lomatiol (structure established by Hooker). A solution of the mixture in ether is shaken in a separatory funnel with an equal volume of a buffer of

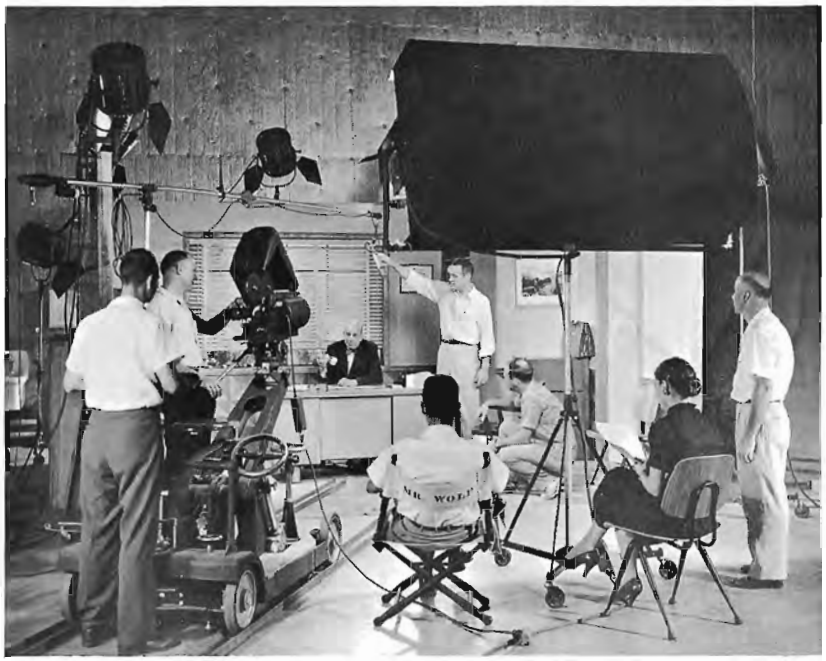


Fig. 22.1. — A Studio office: Harvard in Kansas

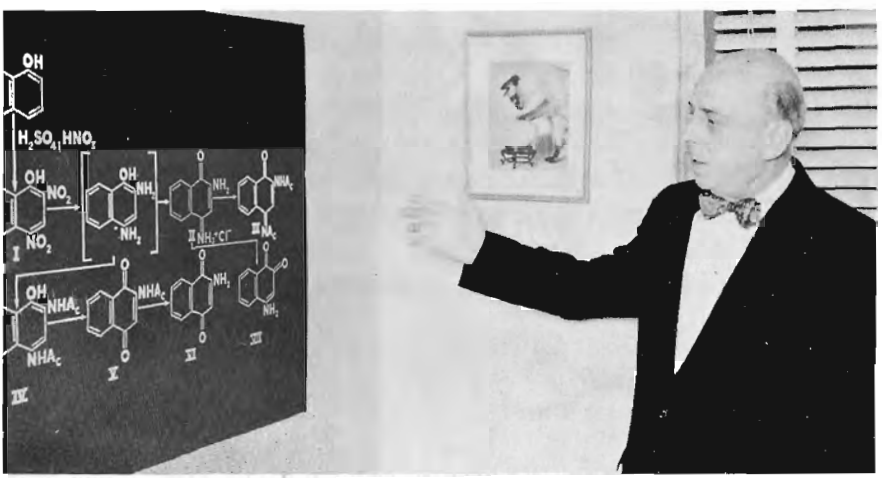
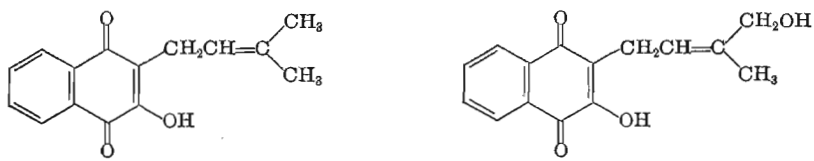


Fig. 22.2. — Studio Scene

Fig. 22.3. — Countercurrent Distribution

pH 8. The lower aqueous layer (red) is run into a second funnel and the first ether layer (yellow) is treated with fresh buffer. Fresh ether is added to funnel No. 2. The process is repeated until separation is achieved. Lomatiol carries an extra hydroxyl group and hence is more hydrophilic than lapachol and it moves along faster with the aqueous phase and concentrates in the funnel on the left. Lapachol turns up in highest amount in the funnel on the right. After the beginning operations had been photographed, I carried the distribution to the 9-funnel stage while Mary took the camera crew out to lunch and brought me back a sandwich. The last scene shows pure crystals of lomatiol and lapachol recovered from the two end funnels.



pK 5.0 (weakly acidic)

Another sequence of the film demonstrating the technique for isolation of cholesterol from gall stones provided an opportunity for making a photographic record of a rare collection of stones contributed by hospitals in the Boston area (Fig. 22.4) before they had been ground up and used for student experimentation. The prize stone of them all (still intact) weighs no less than 78 grams. From a horse, perhaps? No, horses are not admitted to Boston hospitals, and anyway a horse has no gall bladder.

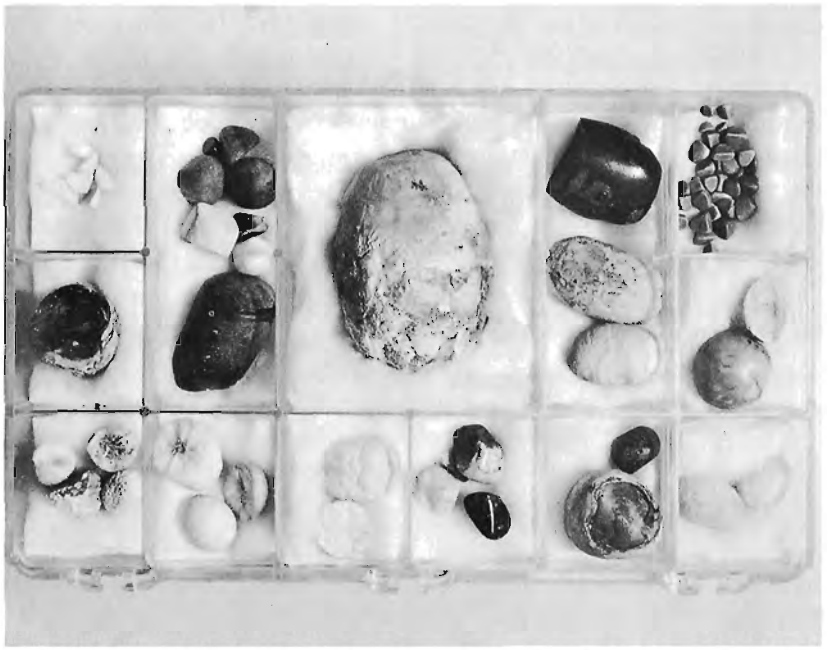
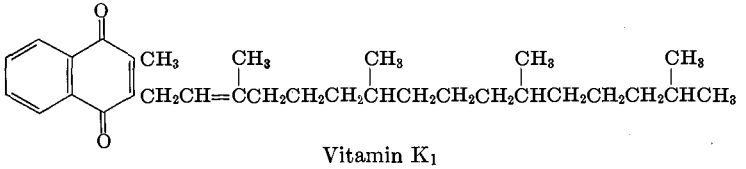


Fig. 22.4 — Gall Stones from Boston Hospitals

23. *Of Cats and Birds*

THE BOOK which Mary and I wrote during the war was published a few years after we had acquired our first Siamese cat, a magnificent seal-point male with deep blue eyes. Sired by Oriental Nanki Pooh, his family name was fixed, but he needed an appropriate baptismal name. The cat was born at the time of an exciting episode in research which culminated in the discovery of a method for the synthesis of vitamin K_1 which had been isolated from alfalfa.

The large hydrocarbon side chain is made up of four 5-carbon isoprene units; the lapachol side chain consists of one such unit; rubber is an



Vitamin K_1

isoprenoid polymer. The side chain of K_1 is identical with the hydrocarbon part of the alcohol phytol, which is a constituent of chlorophyll, and my synthesis involved reaction of phytol with methylnaphthoquinone in its reduced form.

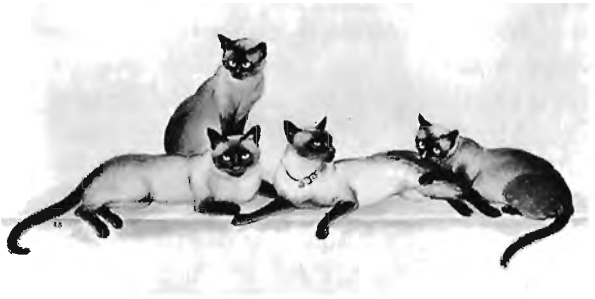
The process was protected by a patent in order to encourage one of the pharmaceutical firms to undertake the costly developmental chemical work, pharmacological processing, and clinical testing required for introduction of a new therapeutic agent. In accordance with the policy of my university on patents pertaining to public health, the patent was assigned for administration to Research Corporation, a nonprofit organization that applies royalties from patents to the support of university research; the contract specified that there be no benefits to the university or to me. Merck and Co. had assisted my research by carrying out bioassays on model compounds and then on the synthetic vitamin and hence was licensed by Research Corporation to manufacture the vitamin by my method.

Vitamin K_1 soon found uses in medicine. The normal function of the vitamin is to cause blood from a cut to clot and so stop flowing. The normal diet contains an adequate amount of the vitamin or of precursors from which it is synthesized by intestinal bacteria. A newborn baby does not derive vitamin K_1 from milk, and, in the first few days after birth, does not possess the intestinal bacteria capable of effecting the synthesis, and hence in this period a tiny pinprick may cause a hemorrhage leading to death. This once frequent cause of infant mortality now can be eliminated by administration of synthetic vitamin K_1 either to the mother in the last few days of pregnancy or to the infant. Vitamin K_1 is a yellow liquid insoluble in water, and it is absorbed properly through the intestinal wall only if it has been emulsified by the action of chemicals present in bile, a fluid stored in the gall bladder and delivered as required into the intestine. Sometimes a tumor or other growth impedes the flow of bile and the amount of bile salt reaching the intestine is not sufficient to promote adequate absorption of vitamin K_1. Surgery to remove the obstructing growth formerly was attended with the danger of excessive loss of blood, but now the patient can be given an intravenous injection of a water-soluble derivative of vitamin K_1 prior to operation.

And a name for the cat? We named him Syn Kai Pooh. In introducing a lecture on the synthesis of vitamin K_1, I said "I have a story to tell

Syn Kai Pooh

tonight about the naming of a cat." Since the lecture subsequently was published in *Science*, Syn Kai Pooh has a status of sorts in the scientific literature. By the time our first book was completed, Syn Kai had three companions, all Poohs, and we had become very much attached to them. On a whim, we decided to include a picture of the cats in the preface of *Organic Chemistry*. Supplied with individual photographs and descriptions,



Syn Kai, Pixie (rear), Cat-a-mouse, and Fluffy

artist Eleanor Blaisdell composed the drawing here reproduced. This unusual feature of a scientific book pleased some readers more than others, but inclusion of a cat picture in our next book, an advanced monograph, showed that we were committed to this form of trademark. The cat included, another great cat comparable to Syn Kai Pooh, arrived during World War II, when my research group developed Napalm. We wanted to associate the new Siamese with this discovery, but the word Napalm was a classified military secret. So the pedigree name chosen was J. G. Pooh, which to us meant Jellied Gasoline Pooh. Before long J. G. became modified to Georgie. Again a Blaisdell drawing from a photograph captured the spirit of this fine animal.

Georgie Pooh

We expected the German publisher of the first translation to consider our trademarks too frivolous for their public, but Verlag Chemie not only reproduced the preface picture but used the cat episode in advertising. Publishers of Italian, Japanese, Spanish, and Polish translations followed suit, but Russian editions of three of our books have remained catless. The Japanese firm Maruzen publishes not only translations of our books but also low-cost, offset-printed copies in English for sale in the Orient, in India, Pakistan, etc. The Japanese reception of our books was so flattering that when we acquired two new potential preface cats, in the form of a pair of twin male Poohs, we determined to find names having some Japanese connotation. A search of the dictionary suggested the names Shio Pooh and Tio Pooh. Shio comes from the word Kurushio, meaning the blue Japanese gulf stream (as blue as Shio's eyes). Tio is taken, with a slight liberalization, from the botanical name of Japanese bittersweet, *Meratia praecox*. Tio has the trick of leaping up onto doors and posing there and purring until I invite him to jump off onto my shoulder. The picture of Tio astride the

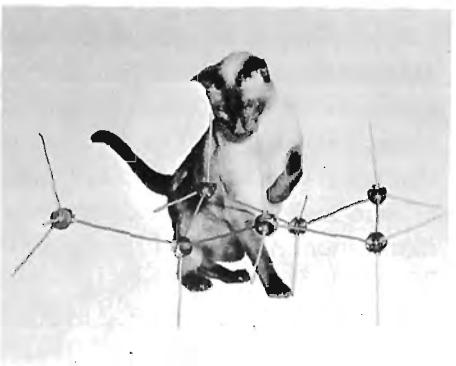
Tio

Shio

door of my study seemed appropriate for the preface of a book on laboratory experiments for students. Twin-brother Shio fancies himself as a beauty and likes to leap up onto the mantelpiece and gaze admiringly at himself in the mirror. Hence it was not difficult to get him to pose for a preface photograph for a book including the chemistry of the active principle of catnip. Tio and Shio also obliged us, after several hours of trial and use of

pieces of beef as bait, with a joint pose with a molecular model of cyclohexane which was used in our book of 1961. The photograph showing Tio dancing around a chemical model was used in a book of 1963.

The Twins



Tio

A small book designed for use in high schools in conjunction with plastic Dreiding models entitled "Chemistry in Three Dimensions" ended with the story of the isolation and characterization of civetone, a valued perfume base present in civet, a glandular secretion of the African civet cat. The obvious choice for a preface picture was one showing a model of civetone and a cat; a cat from Siam surely could dub in for one from Africa. David Sachs, a senior who had been doing research with me since his sophomore year and who was working on carbene chemistry during the summer, came out to our house on a Sunday morning and ran through three rolls of film. Shio, the handsome, obliged only when provided with shrimp trimmings as bait, but Tio posed willingly and well. For background, we used a 4′ x 5′ sheet of plywood painted white and resting on an oriental rug, but development showed that a number of the shots had to be eliminated because a section of tail was out of the field. The one we all liked best was of Tio in a thoughtful mood but lacking the tip of one of his ears (see photo), since the photograph had to be trimmed at the line dividing the white background from the rug. The ear tip showed in the negative, but was on the rug rather than on the white background. Resourceful David then made an untrimmed print, cut out a profile of the head with a razor blade, and so produced the photograph shown on the right. It seems to me that this art work improves the expression on Tio's face; he evidently has thought of something new of interest about civetone.

A book addressed to chemists on English usage stemmed from Mary's

Tio ex-ear

Tio intact

expertness in the field, and the preface picture shows the desk in her home study loaded with the Fowler books, the Oxford dictionary, and a photograph of our only female cat, Fluffy Pooh.

Our one cat who was not a Pooh was a chocolate brown Burmese with yellow eyes. Mary and I had gone in for dinner at the Amalfi Cafe to celebrate completion of the manuscript for the third edition of *Experiments* and we then stopped in at the cat show. The little Burmese had won the prize for best kitten and so was very costly, but we took to him and he to us and so we had another problem in nomenclature. One of our postdoctoral

men was working on the chemistry of ursolic acid, extracted from a plant collected on Cape Cod. The common name of the plant is bearberry, but Mary, a keen horticulturist and a stickler for the proprieties, insisted that we call it *Arctostaphylos uva ursi.* So the obvious name for a cat who is as brown as a bear was *Arctostaphylos uva ursi* (for Mary) or Ursi (for the rest of us). Not long after selection of this name, an editor and photographer for the *Scientific American* came to Cambridge to get photographic illustrations for an article of mine on *Steroids*, which appeared in the January issue for 1955. One illustration for the article was the assembly of extractors shown in Fig. 24.4; one extractor was charged with cranberry skins and the other with *Arctostaphylos uva ursi* (the plant). After all the careful adjustments had been made and the scene photographed, I had an inspiration. Mary went out to the house to get Ursi while we removed

Fig. 24.4

Fig. 24.5

the extractor on the left and substituted a large Büchner funnel and filter flask (Fig. 24.5). Ursi posed nicely for just a split second and then leaped out of the funnel. This picture appeared in the third edition of *Organic Chemistry*. One reader, noting the large volume of liquid in the flask (water introduced for ballast), remarked "What a cat."

We live in a wooded area and we started putting up bird houses and feeders long before we had cats. Actually the cats in no way influence our birding activities because they are house cats. Since Mary and I are both at the laboratory most days except Sundays, we do not let the Siamese roam about. They play with each other, and they have a gadget to play with which may be the only one of its kind in existence: a cat tree (Fig. 24.6). The nicely upholstered post with radial landing stages saves considerable

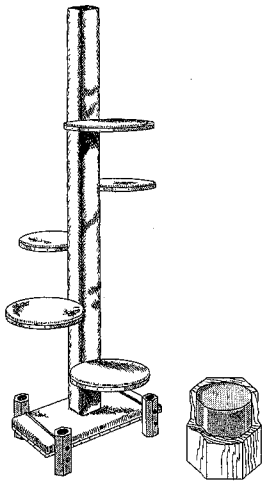


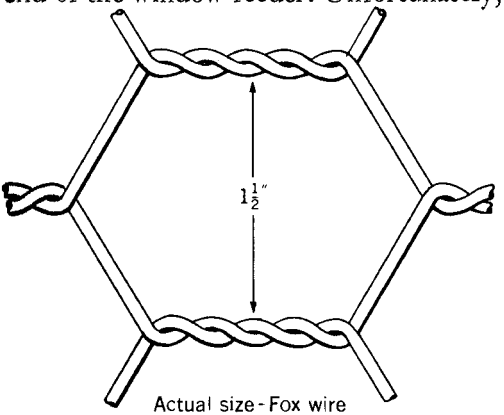
Fig. 24.6. — Cat Tree

wear and tear to furniture; the upholstery has to be replaced at fairly frequent intervals. The cats scratch away at the post, climb up and sit on the top, and in the evening each one takes his particular post on the tree for night feeding: for each, four small pieces of choice beef, chicken, duck, scallops, etc. The tree was made to my specifications by Walter Colburn of the incendiaries group. It is extremely steady, for the four hexagonal posts are sections of dummy M-19 bombs and each has a central core of lead. The dummies were used in ballistic studies by aerial photography.

Syn Kai liked walking on a leash, and Georgie loved automobile rides. To provide pleasurable outdoor life for all the cats, I built a 12 x 16 ft. cat garden 8 ft. high which is joined to a screened porch. It incloses the trunk of a large maple and is equipped with a cedar post for scratching and climbing, with boxes and pedestals for perching, with a sand box, and with runways around the sides, each equipped with a wide section for passing. The garden is screened with heavy turkey wire, which provides complete protection both to the cats and to visiting birds. When a small door leading to the porch is left open, the cats can go and come as they please. They love the garden.

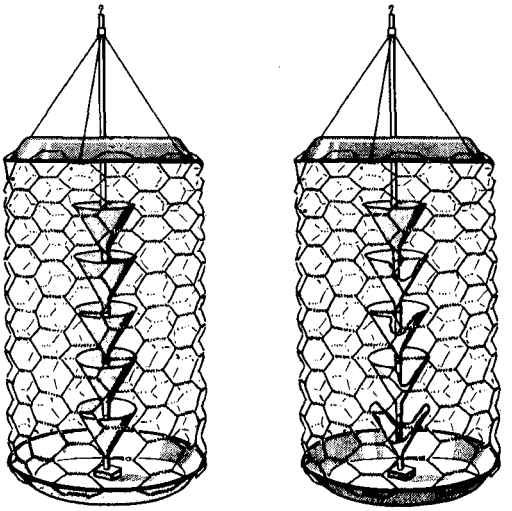
The cats took a lively interest in a bird feeder which I attached to the sill of a pantry window. Crowded up close to the window pane, they growled and gesticulated, but to no avail, for the chickadees and nuthatches seemed to know that they were safe and went about their feeding undisturbed. Excitement reigned particularly high in the cat world when a squirrel broke the code of the supposedly squirrel-proof feeder. This

window feeder was of the standard commercial type, but the front opening of the flat box was provided with a strip of glass which left only a narrow slit for entrance of birds. Our squirrel was a baby and could enter with ease. It was fun to watch the young squirrel, and although it interrupted bird feeding at periodic intervals and consumed an enormous number of sunflower seeds we continued to fill the feeder daily. The visitor eventually would be as big as numerous adult squirrels who had been attracted to the feeder only to find that their heads were too large. Our squirrel did grow, and soon began to make its entrance and exit with increasing difficulty. When, finally, it could not climb in, it became mad enough to break the glass and so to open the feeder to the entire squirrel population. A new piece of glass was promptly broken; a panel of wood was chewed away. I then devised a hinged door held open by a lead weight and so arranged that a squirrel walking across the top of the feeder would overbalance the weight and close the door. I thought that a squirrel could outwit me only by getting another squirrel to sit on the lead weight. Two or more squirrels evidently did cooperate, but what they did was to chew away the lead and close the door. Then I was told that fox wire is proof against squirrels but allows small birds to enter, and so I put a screen of fox wire on the open end of the window feeder. Unfortunately, the invulnerability of fox wire is a myth. I have a snapshot showing two large squirrels in the feeder at the same time. The hexagonal mesh of the wire seems to accommodate the cranium of most adult squirrels and, once the head is through, a hungry squirrel or a scared one has no difficulty squeezing its body through, in or out.



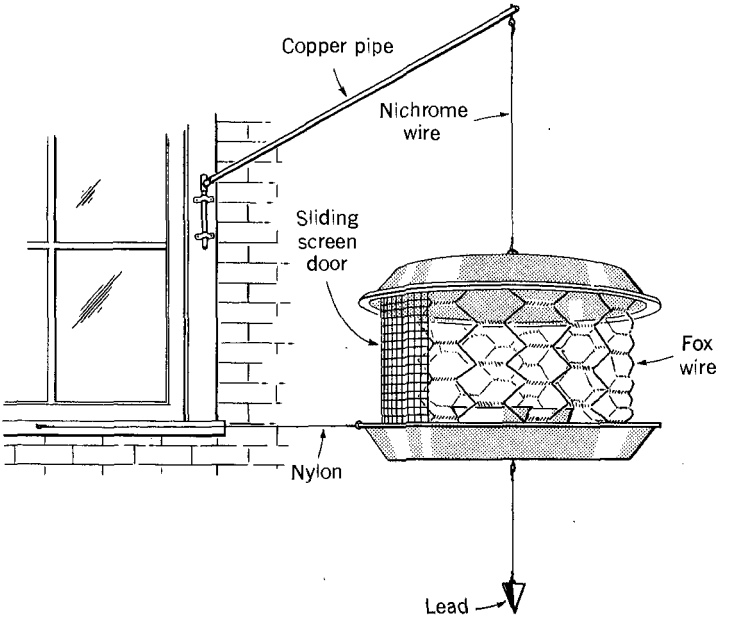
Actual size - Fox wire

So I conceded victory to the squirrels, abandoned the idea of a window feeder, and experimented with suspended feeders made from fox wire and aluminum pie plates. A feeder of one type, in which the containers for seeds and peanut butter were polyethylene funnels mounted on a dowel, was suspended from a branch by a fine wire which I thought would provide squirrel protection. The feeder was popular with small birds and excluded undesired large ones. However, it was not long before squirrels learned to leap an incredible distance from another tree, land on the feeder, and make their way in. They not only



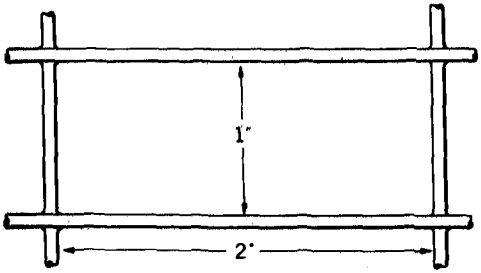
Funnel leeder - before and after

devoured the food, but they chewed up the plastic funnels, as can be seen from the drawing on the right. I then tried suspending a feeder from a copper pipe outside a window and over a driveway. The nearest tree was much too distant to serve as a take-off point, the feeder was about ten feet above the drive, and the only thing to climb on was a brick wall. All went



well for a few months, but my resourceful adversaries eventually located the feeder and learned how to climb up the bricks to the window sill and leap onto the feeder.

Thus my Sunday research extending over a period of years had been fruitless, and I was forced to suspend feeders on long wires in unattractive locations remote from trees and house. Then one Sunday when it was too cold for the cats to be outdoors I noticed four chickadees in the cat garden. Had they been trapped? No, they soon flew out of the garden and then flew in again. They either flew in as though the turkey wire were not there, or poised on the wire for an instant and then went in. Turkey wire is made of heavy 14-gauge wire welded together in a one-by-two-inch mesh. To get through turkey wire, an animal would have to have a skull measuring less than one inch in diameter, and I was certain that squirrels would be barred. To test further the reaction of birds, I mounted a tin saucer on a dowel stuck into the ground inside the garden and filled it with seeds. Within half an hour chickadees in rapid succession were flying into the garden for seeds and flying out to trees to crack and eat them. Satisfied that turkey wire is ideal for construction of feeders, I promptly reconverted the garden into a place for cats.



Actual size - Turkey wire

An inquiry about the source and standard use of turkey wire addressed to Haydn Pearson, genial country correspondent of the Boston Herald, was entered in his column and the question was answered both by Lyon Southworth and by Charles E. Monahan. A pen for raising turkeys has a flooring of turkey wire mounted a few feet above ground for protection against the dread disease Blackhead, which is spread through droppings. The one-by-two-inch mesh allows the droppings to fall through to the ground but supports the feet of small turkeys.

Not all hardware stores carry turkey wire, but it is easy enough to locate a source by telephone once you know the name. This wire lends itself nicely to the construction of feeders, which can be large or small, round or rectangular. A roof is needed for protection against rain and snow, but a piece of the same wire serves adequately as a floor. The style that appeals most to me is shown in the photograph. The roof is an 18-inch spun aluminum baffle made by the Hyde Bird Feeder Co., Waltham, Mass. Before assembling the feeder, I sprayed the wire with aluminum paint from a

Turkey-wire Feeder

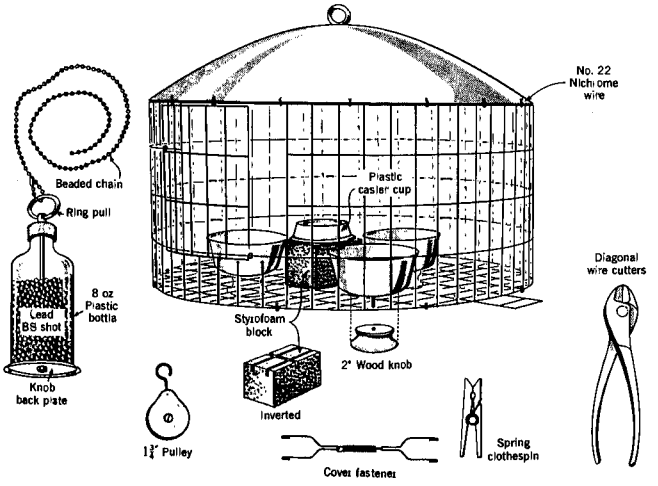
spray can. The plastic pans (F. W. Woolworth Co.) are conveniently filled from quart jars with metal spouts designed for lubricating oil. The seeds shown are black sunflower seeds, gray sunflower seeds, and hemp seeds. The pulley system with a counterbalance permits lowering the feeder for easy filling and also permits experimenting with the feeder at different heights. The beaded chain passes through a section of wire projecting from the bottom of the cage (at the left) to prevent swinging in the wind. The feeder is hung from a piece of turkey wire supported by two No. 20 nichrome wires attached with screw eyes to second-story window frames and by two wires attached to trees, even distant ones. With this arrangement the feeder can be located at any position desired, for example within view from a particular window. A spot at the edge of a walk or a drive permits easy access when there is snow on the ground. Nearby trees are desirable as take-off points where a bird can wait its turn.

In constructing the first turkey-wire feeders I used $\frac{3}{16}$-inch Nylon rope in the pulley system. I knew from an experience off Plum Island with Arnold Seligman that a 660-lb. tuna can be taken on a Manila hand line with a leader-section of the same Nylon rope and hence that Nylon is very strong. Possibly a squirrel could climb up the rope, but to what avail? For about five months two feeders did good business with the proper customers without incident, except that one morning we discovered a squirrel perched on the feeder. It climbed over and around and under the cage and spilled

some seeds, but on failing to gain entry it eventually jumped to the ground and did not return. But then the day came when Mary reported that in the time between my departure from the house and hers one feeder had fallen to the ground with the rope chewed in two. She had not seen the squirrel, but the position of the chewed part of the rope indicated that the marauder had clung to the turkey wire while performing its act of sabotage. I replaced the rope with a beaded chain and had no further squirrel trouble. This chain, made of brass on the ball-and-socket principle, is commonly used as an extension cord for an electrical pull switch. The friction is less than with a rope, and the weight of a squirrel lowers the feeder gently until it touches the counterbalance and stops.

If you care to construct a feeder, or to have someone construct one for you, you can choose between the horizontal style shown in the photograph and the vertical style shown in the drawing. I tried both styles, side by side, and then with the positions interchanged, and could detect no preference by the birds. The drawing shows one way of constructing a sliding door, which can be locked with the spring clothes pin or with an ironing board

cover fastener. A plastic caster cup cemented onto a block of styrofoam (Woolworth) is a convenient container for peanut butter. In cold weather peanut butter should be mixed with corn meal, for otherwise a piece may lodge in a bird's throat.



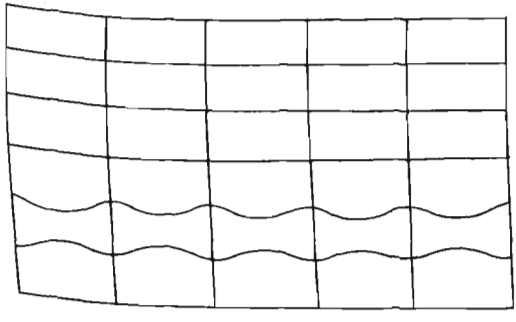
My birds, largely chickadees and goldfinches, prefer gray sunflower seeds to black ones. In tests conducted over one- to two-week periods in December and January, I found the average consumption of gray seeds to be 1.4 times that of black seeds. It was necessary to take account of a direction preference by exchanging pans, for a pan facing East was favored over one facing West by a factor of 1.9, probably because the arrangement of trees was more suitable. The color of the pan is immaterial. In the favored location the consumption of favored gray seeds averaged 396 seeds per day in one period and 556 seeds per day in another. Peanut butter consumed at the same time averaged 12.2 g. (0.43 oz.) per day. The birds are also very fond of hemp seeds, which are smaller and can be opened and eaten in about one third the time required for sunflower seeds. Thus the birds make three times as many flights for the small seeds as for the large ones. Approximate data for the seeds are given in the table. The kernel content of all the seeds is 54–57% of the total. A chickadee making a twenty-foot flight to a feeder travels at a speed of twenty miles per hour.

I do not yet know what desirable small birds other than chickadees, goldfinches, and purple finches will use turkey-wire feeders. Fortunately starlings and grackles are excluded, and sparrows do not seem enterprising enough to try. The likeable bluejays are left seedless, but Mary puts out plenty of bread for them. In case you are lucky enough to have nuthatches,

Seeds Contained in a Quart Bottle

Seeds	Weight, lbs.	Number
Black sunflower	0.73	3,700
Gray sunflower	0.82	3,800
Hemp	1.18	24,000

you can put a pan for them close to the edge and they will cling to a wire and take seeds from without. Should you wish to admit birds larger than the one-inch space will allow, you can enlarge some of the openings by squeezing appropriate wires together with a pair of pliers as shown in the drawing.



Nichrome wire, which is strong and rustproof, is useful also for the almost invisible hanging of tree branches that bend down overly far, and for straightening or supporting saplings and shrubs. A strip of turkey wire serves nicely for training a clematis or other twining vine. The feeders described merely illustrate some of many possible designs. If you acquire a feeder of any sort made of turkey wire and like it, you can thank our science-oriented cats for the suggestion.