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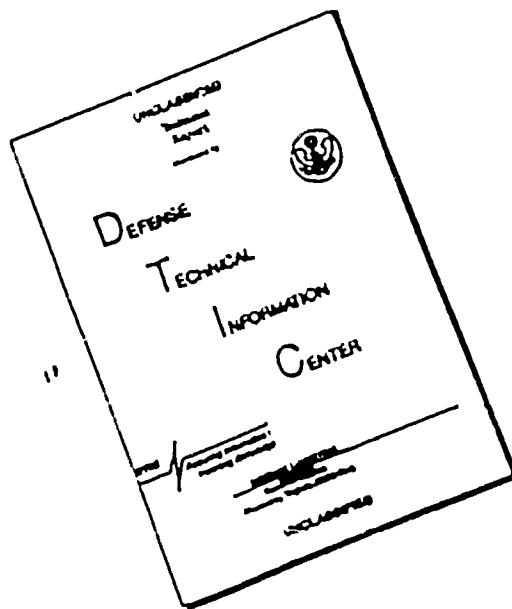
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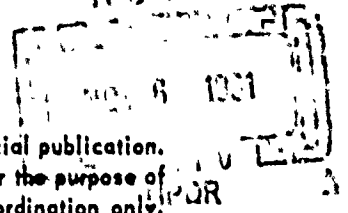
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RESEARCH ON THE FIRE AND EXPLOSION HAZARDS ASSOCIATED WITH NEW LIQUID PROPELLANTS

Progress Report No. 9
(Final Report)
May 1 to August 31, 1961

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INTRODUCTION

This is the ninth in a series of quarterly progress reports by the Bureau of Mines on studies of fire and explosion hazards associated with new liquid propellants. Under normal procedure this would have been the final report summarizing all work since April 1959. However, it proved awkward to combine a detailed reporting of new experimental results obtained during the present report period with a summary of old material. Therefore a separate summary report is being prepared and this will be processed for publication as a Bureau of Mines Report of Investigations.

The objectives of the research are outlined in Government Order NAonr-8-61. Briefly, the work includes three phases:

- Phase I: Ignition.
- Phase II: Flame Propagation, Pressure Rise Rates,
and Detonability.
- Phase III: Liquid Burning and Extinguishment.

The following fuels and oxidants have been used in this program: hydrazine, monomethyl hydrazine (MMH), unsymmetrical dimethyl hydrazine (UDMH), the mixed amine fuels MAF-1 and MAF-3, red fuming nitric acid (RFNA), nitrogen tetroxide, and air with various diluents.

RESULTS AND DISCUSSION

Phase I: Ignition - H. E. Perlee

Spontaneous Ignition

The spontaneous ignition temperatures (S.I.T.) of monomethyl hydrazine (MMH) vapor-air mixtures and unsymmetrical dimethyl hydrazine (UDMH) vapor-air mixtures in contact with 100 percent $\text{NO}_2^{*1/}$ were determined in the I-8 apparatus. These tests were conducted in the same manner as described previously.^{2/} The results of these tests are shown in figure 1. These show that for the same combustible concentrations the MMH has lower S.I.T.'s than does UDMH up to a concentration of

^{1/} NO_2^{*} represents the equilibrium mixture of NO_2 and N_2O_4 .

^{2/} This research, Progress Report No. 7, November 1, 1960, to January 31, 1961.

6.8 percent combustible; above this concentration the reverse is true. A similar phenomenon was observed^{2/} for the S.I.T.'s of the liquids when they were injected into NO_2^* -air atmospheres. Further S.I.T. tests were conducted with these combustibles using mixtures of NO_2^* and air as the oxidizing atmosphere in place of the pure NO_2^* . The results of these tests using two NO_2^* -air mixtures (50 percent NO_2^* and 10 percent NO_2^*) are shown in figures 2 and 3 for MMH and UDMH, respectively. As can be seen from the figures, the S.I.T.'s appear to approach a minimum in each case, with the exception of UDMH in 100 percent NO_2^* .

During the course of these and previous experiments, we had observed considerable scatter in our results (± 1 percent fuel). For the tests involving the vaporization of the liquid amines in an air atmosphere, this scatter was attributed to the heterogeneous oxidation of the combustibles. However, for the tests where liquid combustible was injected into an atmosphere of NO_2^* and air, an explanation for this scatter was not so apparent. Early investigations had indicated that this scatter could not be attributed to any of the more readily controllable (extensive) parameters, e.g., concentration fluctuations, temperature fluctuations, or mechanism of fuel injection. Therefore, we surmised that this scatter was due to more subtle factors which are probably intrinsic properties of the combustion process and not readily controlled by the experimenter. In order to justify this conclusion, a series of 600 tests was conducted to determine the probability of an ignition when liquid UDMH is injected into a known mixture of NO_2^* and air at 25° C. These tests were conducted as carefully as possible in order to minimize the error due to the extensive parameters. The results of these tests are shown in figure 4 where the probability of an ignition of liquid UDMH is plotted against the concentration of NO_2^* in the NO_2^* -air mixture at 25° C. The size of the circles in the figure represents the probable error due to the extensive properties. These results show that there is a gradual transition from a region of ignition to one of no ignition. The width of this transition zone (in this case 2 percent NO_2^*) is probably temperature-dependent for any specific fuel.

Phase II: Flame Propagation, Pressure Rise Rates, and Detonability - E. Litchfield

This phase of the program involves a study of the detonabilities of the hydrazoid and mixed amine fuels as pure vapors and in mixtures with air and with oxygen. It is not possible to premix these fuel vapors with NO_2^* and to hold such mixtures unreacted for testing; indeed, there are difficulties brought about by the reactivity of UDMH with air or oxygen as will be developed below.

Figure 5 is a plot of requisite energies for initiation of detonation of UDMH-oxygen-nitrogen mixtures. The data representing initial pressures of 1/4-atmosphere were taken from the previous report for comparative purposes. Measurements at 1/2-atmosphere initial pressure are new. These new data show that the pressure scaling relationship in the neighborhood of the energy minimum is approximately inverse cubic. Stoichiometric UDMH-oxygen mixtures at 1/2-atmosphere were initiated to detonation by a very weak exploding wire source having a stored energy of only 1.2 joules. Tests on the fuel-lean side of stoichiometric were conducted with both UDMH-oxygen mixtures and with mixtures in which the excess oxygen was replaced by nitrogen, no gross effect on energy requirement being observed. Only rough values were obtained in these tests which were regarded as preliminary to tests at one atmosphere.

The most peculiar feature of the half-atmosphere tests was that detonations could be obtained with relatively small energies in mixtures containing down to 12 percent UDMH, while no detonations were achieved with less than 10 percent UDMH. The peak reflection pressure observed from the detonation of 12 percent UDMH was 700 p.s.i. From considerations of the available bombs, it was decided to limit the tests at 1 atmosphere initial pressure to mixtures containing 12 percent UDMH or less. However, detonation was not obtained with such mixtures at one atmosphere even though a 12 percent mixture detonated readily at 1/2-atmosphere.

To this point, all detonation testing had been accomplished with mixtures made by the method of partial pressures; and all test results had been consistent and reproducible. The trouble was first assumed to have developed from condensation of fuel vapor and on this basis a flow system was then assembled for use in preparing mixtures at atmospheric pressure. The results obtained in this portion of the investigation were not too satisfactory; however, mixtures of UDMH and oxygen, indicated by sampling to be about 5.5 percent UDMH, were successfully initiated to detonation. When these mixtures were prepared in the same way with air rather than with oxygen, the tests gave no indication of detonation under comparable conditions. Despite uncertainties arising from decomposition of the mixtures on handling, the difference between mixtures with air and with oxygen, for UDMH concentrations approximating stoichiometric, is quite real and is in agreement with previously reported test results.

Additional UDMH-air mixtures were then prepared by the method of partial pressures and subjected to tests with stored energies up to about 580 joules. Again, no detonations were obtained although certain of the results are worthy of further consideration. Figure 6A may be taken as a qualitative illustration of these results. There is the

large pressure step on the rear of the record. Since the large step occurs at late times and not at early times, it clearly does not represent a shock formed in the initial discharge of the stored energy. If it be assumed that the wave is formed at the center of the bomb, it then follows that the mean velocity of such wave is about one-third of sonic velocity and is considerably in excess of anticipated normal flame velocities. For comparison, figure 6B is a pressure-time record on different scales of the events following discharge of the same 580 joules stored energy into air at ambient pressure.

There would seem to be little question but that mixtures of UDMH with air can be detonated at one atmosphere. Direct initiation of such mixtures was not accomplished by the energies used here but records such as figure 6A give a rather clear indication that deflagration to detonation transitions can occur. Larger stored energies were not utilized as no comparison data exist as yet for larger energies with conventional fuels. For similar reasons explosive initiation was not used.

Phase III: Liquid Burning and Extinguishment

Extinguishment of UDMH Fires by High Expansion Foams - E. Grumer and A. Bruszak

It was decided at an earlier stage in this project that the extinguishment of UDMH and MAF fires by foams would be outside the scope of our work. However, on watching a demonstration of fire fighting with high expansion foam,^{3/} we were persuaded that such a foam constitutes an attractive research tool; since the individual bubbles are very nearly of one size, one can administer water quite evenly to a small fire without much question as to the total quantity of water which is utilized. For this reason we resurrected a small foam-forming apparatus which has been used here in the development of suitable foams for combatting coal mine fires. This apparatus, along with a one-foot diameter tray in which fuel was burned, constituted a standard setup for testing the relative extinguishability of various flames by addition of water.

A 0.7 percent solution of Maprofix (ammonium lauryl sulfate) in water was sprayed into a 1/8-inch mesh cotton net which sloped away from the spray nozzle at a 45° angle. Air was passed through the continuously wetted net to yield 1/2- to 1-1/2-inch diameter bubbles which moved as a honeycomb through a short length of one-foot diameter stovepipe to the edge of the fire. The rate of air flow and of foam production was controlled by use of interchangeable orifices at the intake of a blower (see table 1). Measurements were made by stopwatch of the times required for extinction of one-foot diameter flames.

^{3/} This research, Progress Report No. 8, page 3.

The immediate finding was that fires supported by UDMH and other slow-burning fuels were remarkably difficult to extinguish as compared with fires of the faster burning hydrocarbons. This is shown by the data given in table 2 and by figure 7, which also includes reported addition rates of dry powders to accomplish extinguishment of hydrocarbon fires. Table 3 summarizes information which we consider pertinent to an interpretation of the results.

Flames of the three hydrocarbons, benzene, hexane, and xylene, are characterized by small values of the fraction, $\Delta H_v/\Delta H_c$, of their heat of combustion fed back to the liquid surface, which is given in column 2 of table 3. Therefore the flame zones may position themselves far away from the liquids and transmit heat to the liquid almost exclusively by radiation. As foam approaches the edge of such a flame, bubbles break and water is entrained in the radial draft of the fire, causing a decrease in flame temperature and a momentary lengthening of the flame, both of which factors decrease the radiation to the liquid. Foam almost immediately floats out onto the liquid surface providing a radiation shield. Thus all factors contribute to quick and easy extinguishment. As shown by the last two columns of table 3, the amount of water required for extinguishment is less than one would calculate to bring all parts of a steady-burning flame to a nonflammable concentration.

The three slow-burning fuels of table 3 are characterized by higher values of $\Delta H_v/\Delta H_c$ and by flames positioned closer to the liquid so as to transmit back a larger fraction of their evolved heat. We have estimated that convection accounts for about 75 percent of the total heat transfer with methanol at one foot diameter^{4/} and the flame sweeps back and forth within a few millimeters of the fuel surface. As foam approaches such fires the water is entrained in the burned gases above the flame zone but with little discernible effect on the flame zone itself. In several instances the fire continued to burn when completely covered with foam; eventually, extinguishment must occur through sheer dilution of the fuel by droplets of water. The fourth column of table 3 is meaningless as applied to these fuels.

Since high expansion foam would not be considered as an extinguishing agent for MAF flames, the significance of the above result lies in the direct parallel with extinguishment of fires by water sprays. Rasbash^{5/} speaks of the critical importance of a spray's

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- ^{4/} Zabetakis, M. G., and Burgess, D. S., Research on the Hazards Associated with the Production and Handling of Liquid Hydrogen. Bur. Mines Rept. of Investigations 5707, 1961.
- ^{5/} Rasbash, D. J., The Extinction of Fires by Water Sprays. Paper presented at Symposium on Fire Control Research, Division of Fuel Chemistry, meeting of ACS, Chicago, September 1961. Publication probable in Fire Research Abstracts and Reviews.

penetrating to the "heart of the fire" and calculates that heat transfer requirements for extinction are much lower if one cools the fuel than if one cools the flame. In discussion following the referenced paper, Rasbash stated that flame extinguishment by sprays is mostly a matter of combatting the convective heat transfer process, a conclusion with which our results with small fires are in complete accord.

The conclusion from the above results and discussion is that fires supported by UDMH vapors will be much more difficult to control by water sprays or by inert gases than would appear from their burning rates and thermal output rates. However the possibility of extinguishment by flooding the fuel surface with several volumes of water per volume of fuel is in no way affected.

Burning Rates of MAF-1 and MAF-3 in Wind - A. Strasser and
T. A. Kubala

The burning rates of MAF-1 and MAF-3 were determined on a windy day, with average wind speed of 10 m.p.h. and gusts ranging to 20 m.p.h. Under these conditions the overall burning rate of MAF-1 was 0.20 cm./min. and that of MAF-3, 0.14 cm./min. in a 122 cm. diameter tray. We can compare this with values of 0.18 cm./min. for MAF-1 and 0.10 cm./min. for MAF-3 burning in still air in a 76 cm. tray. It may thus be observed that the burning rates of MAF-1 and MAF-3 are but little affected by ambient winds since the increase observed may be attributed in great part to increased tray diameter. DETA alone increases in burning rate by 35 percent when tray diameter is increased from 76 cm. to 122 cm. although the burning rate of pure UDMH is relatively insensitive to the same change of tray diameter. It is thus not surprising that MAF-3, which contains more DETA, showed a larger relative increase in rate than MAF-1.

The effect of the wind on radiation from the flames is indicated by the fact that in the run with MAF-3, for example, the percentage of total power which was radiated during the run was down to an apparent value of 11 percent while previous data for this fuel indicate a value of the order of 40 percent of total power radiated. The random blowing about of the flame by ambient wind was observed to change the area of the flame by factors which would greatly affect total radiation. The photographs in figure 8 indicate the differences in the flame of MAF-1 at two different times, 10 minutes apart in the midst of a 30 minute run--one in nearly quiescent air, the other during a gust of wind. Although similar effects were observed with MAF-3, the flame was not bright enough to photograph well in daylight.

Comparison of Convective and Radiative Heat Transfer - E. Cook

Hottel^{6/} postulated an equation which describes the contributions of the various modes of heat transfer from flame to liquid as a function of burning rate. This equation and observed burning rates in large trays (more than 30 cm. in diameter) were used to determine the relative significance of convection and radiation terms. Values were calculated for the coefficient of convection, the shape factor for the radiation, and the extinction coefficient in Hottel's equation. Conditions assumed for the calculation were an initial temperature of 338° K. (near the boiling points of the fuels) and a flame temperature of 1600° K. Table 4 indicates the results which, in general, show that the heat transfer by radiation is far more effective than that by convection. In even the most favorable case for convection, that of methanol, radiation accounts for about three times as much heat transfer as convection in large fires, although the reverse is true at small diameters as noted on page 5.

^{6/} Hottel, H. C., Review of "Certain Laws Governing the Diffusive Burning of Liquids" (Blinov, V. I., and Khudiakov, G. N., Acad. Nauk, USSR, Doklady vol. 113, 1957, pp. 1094-98), Fire Research Abstracts and Reviews, vol. 1, 1958, pp. 41-44.

Table 1.--Control of foam flow by orifices.

Orifice diameter, inches	Foam flow rate, gals./sec.	Expansion ratio	Water output, lbs./sec.
2	4.7	650	0.060
1-1/2	2.3	625	.031
1	1.4	825	.014

Table 2.--Average elapsed times for extinguishing flames above
one-foot diameter tray using three flow rates of foam.

Fuel	0.060 lbs./sec.	0.031 lbs./sec.	0.014 lbs./sec.
	<u>sec.</u>	<u>sec.</u>	<u>sec.</u>
Benzene	9.8	11.3	28.9
Xylene	8.0	15.2	36.3
Hexane	16.4	50.3	--
Acetone	125	--	--
UDMH	80	--	--
Methanol	113	--	--

Table J.--Water requirements for extinguishing flames
with high expansion foam.

Fuel	$\Delta H_v / \Delta H_c$ ^{1/}	$\frac{\text{Moles water}^{2/}}{\text{Moles fuel}}$	Limiting oxygen index ^{3/}	
			From column 3 (stoich.)	From flammability limits
Benzene	0.0122	5.0	18.0	<u>4/</u> 13.9
Xylene	.0142	11.1	16.9	--
Hexane	.0106	6.9	17.9	<u>4/</u> 14.5
Acetone	.020	33	(7.5)	<u>4/</u> 15.6
UDMH	.022	42	(6.2)	<u>5/</u> 6.3
Methanol	.061	47	(2.7)	<u>6/</u> 14.1

1/ See table 1, Progress Report No. 5, this research.

2/ Minimum ratio for extinguishment as judged from figure 7 and from known burning rates.

3/ Minimum oxygen percentage for flame propagation.

4/ From Coward, H. F., and Jones, G. W., Limits of Flammability of Gases and Vapors (Bureau of Mines Bulletin 503), judged from data with CO₂ as diluent.

5/ From figure 1, Annual Report, April 1959 - April 1960, this research, applying to measurements at 150° C.

6/ From data given in Progress Report No. 7, this research.

Table 4.--Relative Significance of Convective and Radiative Heat Transfer from Flame to Liquid.

Fuel	Largest tray diameter used, cm.	Apparent coefficient of convection joules/cm. ² sec. ^{°K.}	Shape factor	κ , cm. ⁻¹	Convective heat transfer joules/cm. ² sec.	Radiative heat transfer joules/cm. ² sec.
Methanol	180	5.7×10^{-4}	0.036	0.16	0.73	2.32
Diethylene triamine	122	3.6×10^{-5}	.017	.0091	.941	.91
Xylene	91.5	0	.089	.038	0	3.17
Unsymmetrical dimethyl hydrazine	91.5	0	.18	.064	0	2.76
Hexane	91.5	0	.1	.021	0	3.03

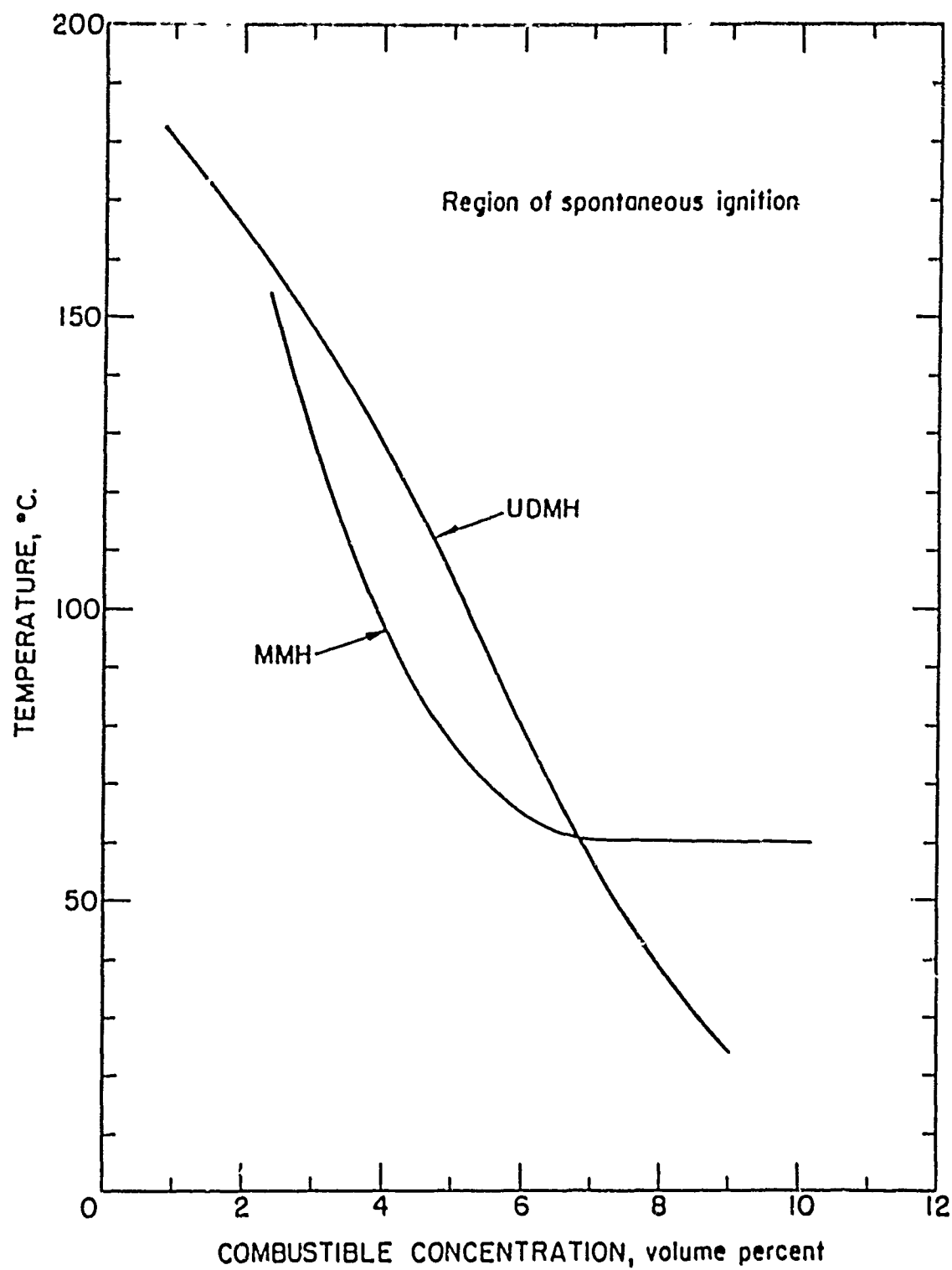


Figure 1.—Spontaneous ignition temperature of MMH-air and UDMH-air mixtures in NO_2^* .

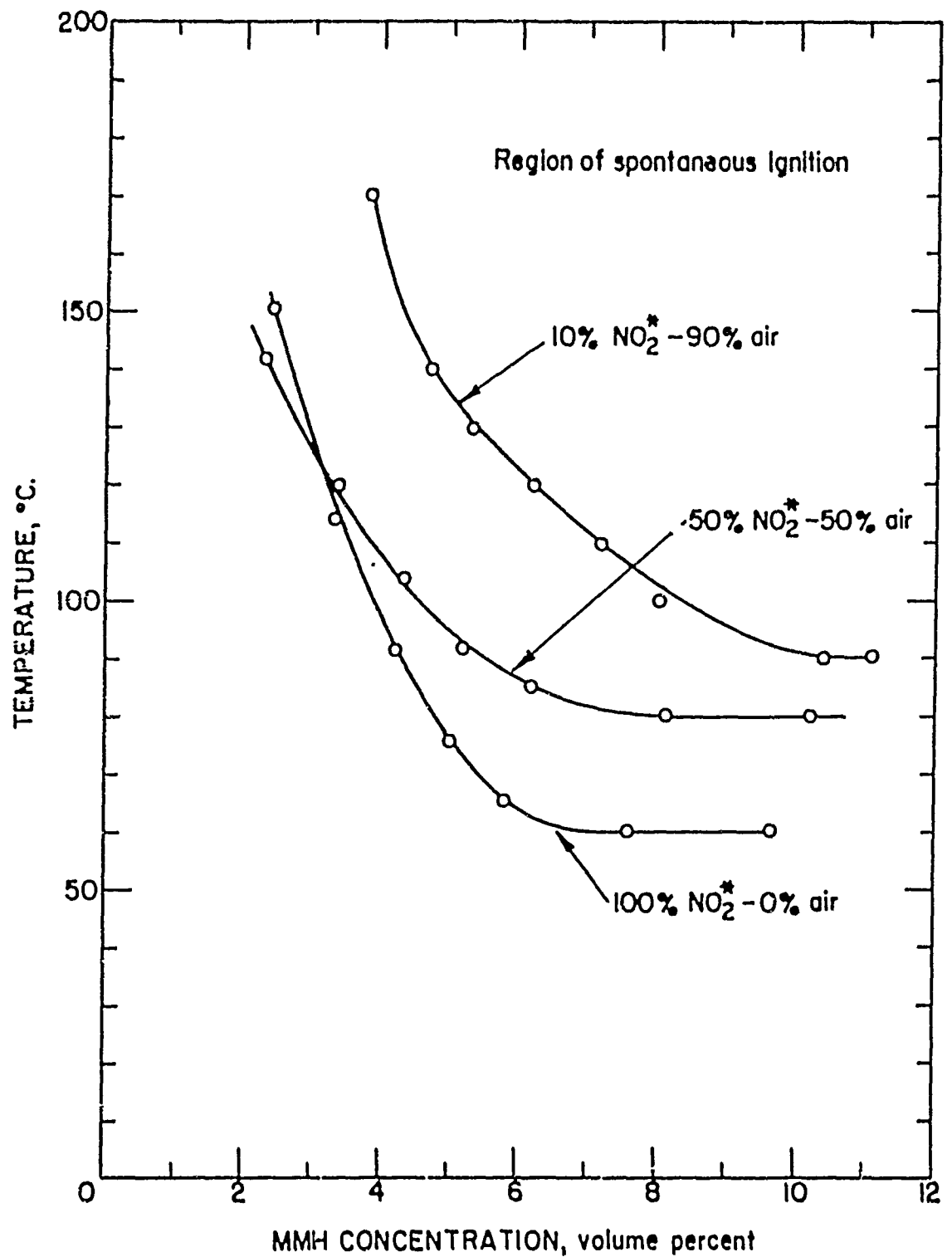


Figure 2.-Spontaneous Ignition temperatures of MMH-air mixtures in various NO_2^* -air mixtures at 25° C. as a function of MMH concentration.

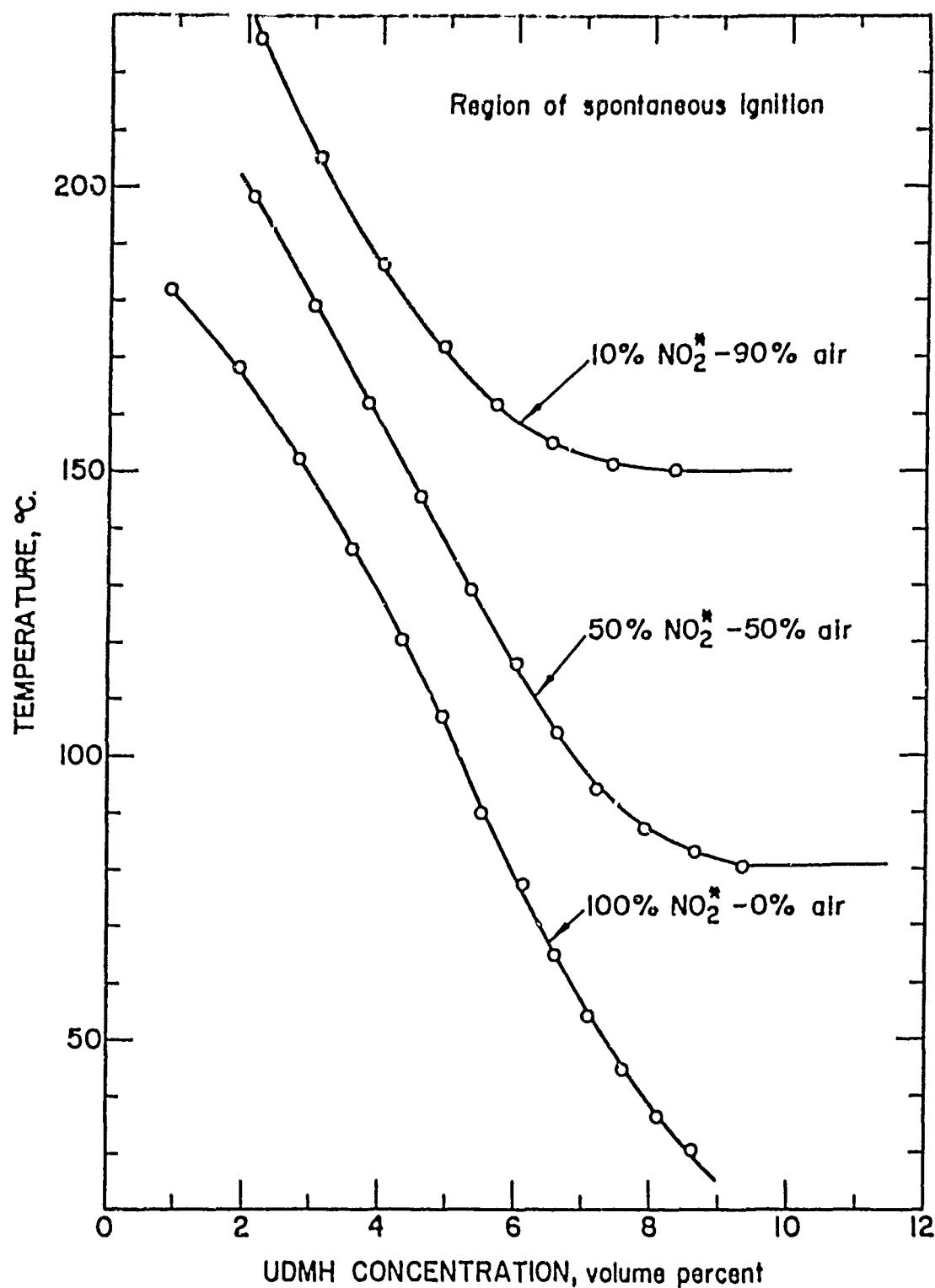


Figure 3.-Spontaneous ignition temperatures of UDMH-air mixtures in various NO_2^* -air mixtures at 25° C. as a function of UDMH concentration.

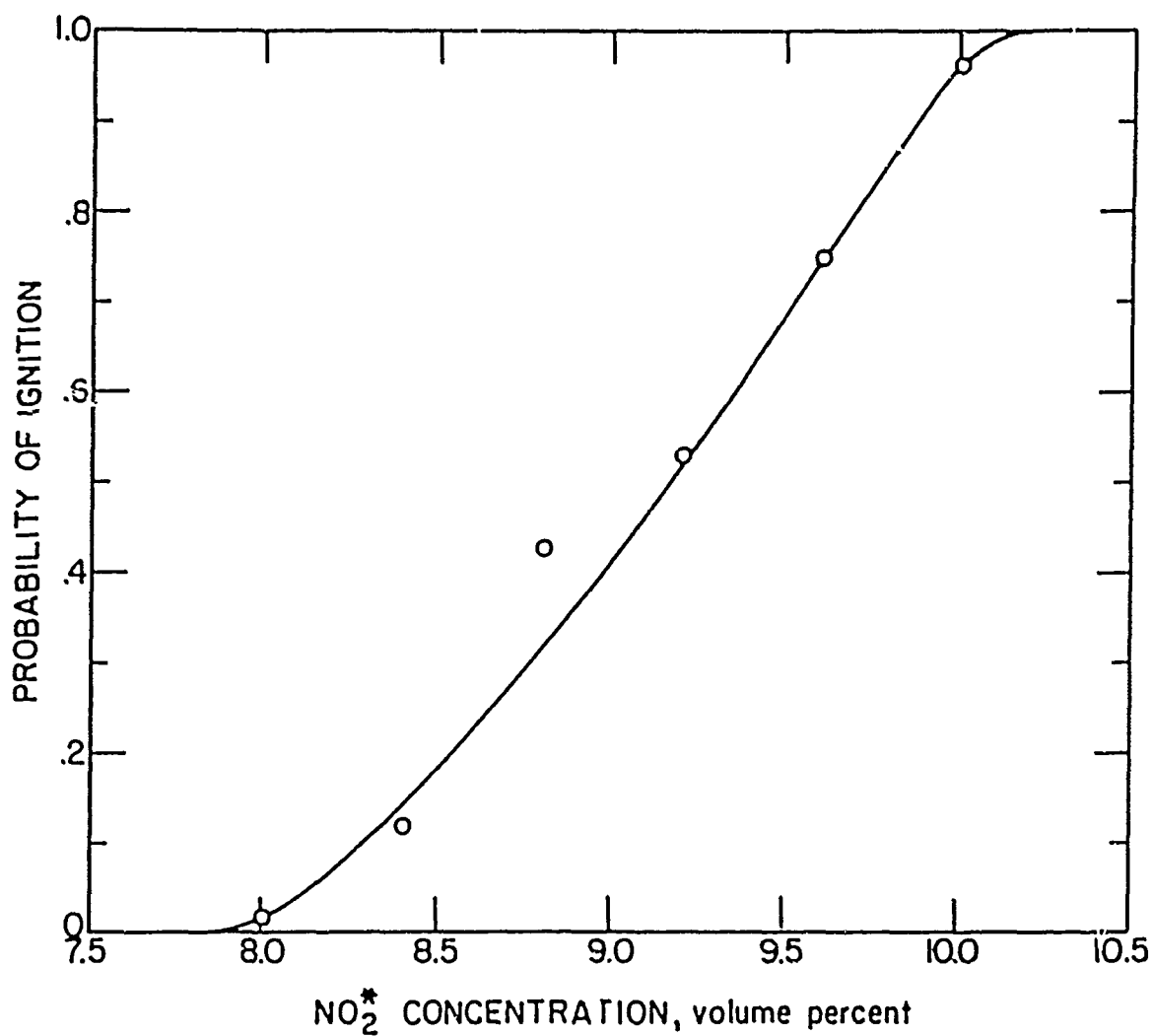


Figure 4.- The probability of ignition of liquid UDMH on contact with NO_2^* -air mixtures at 25° C. as a function of NO_2^* concentration.

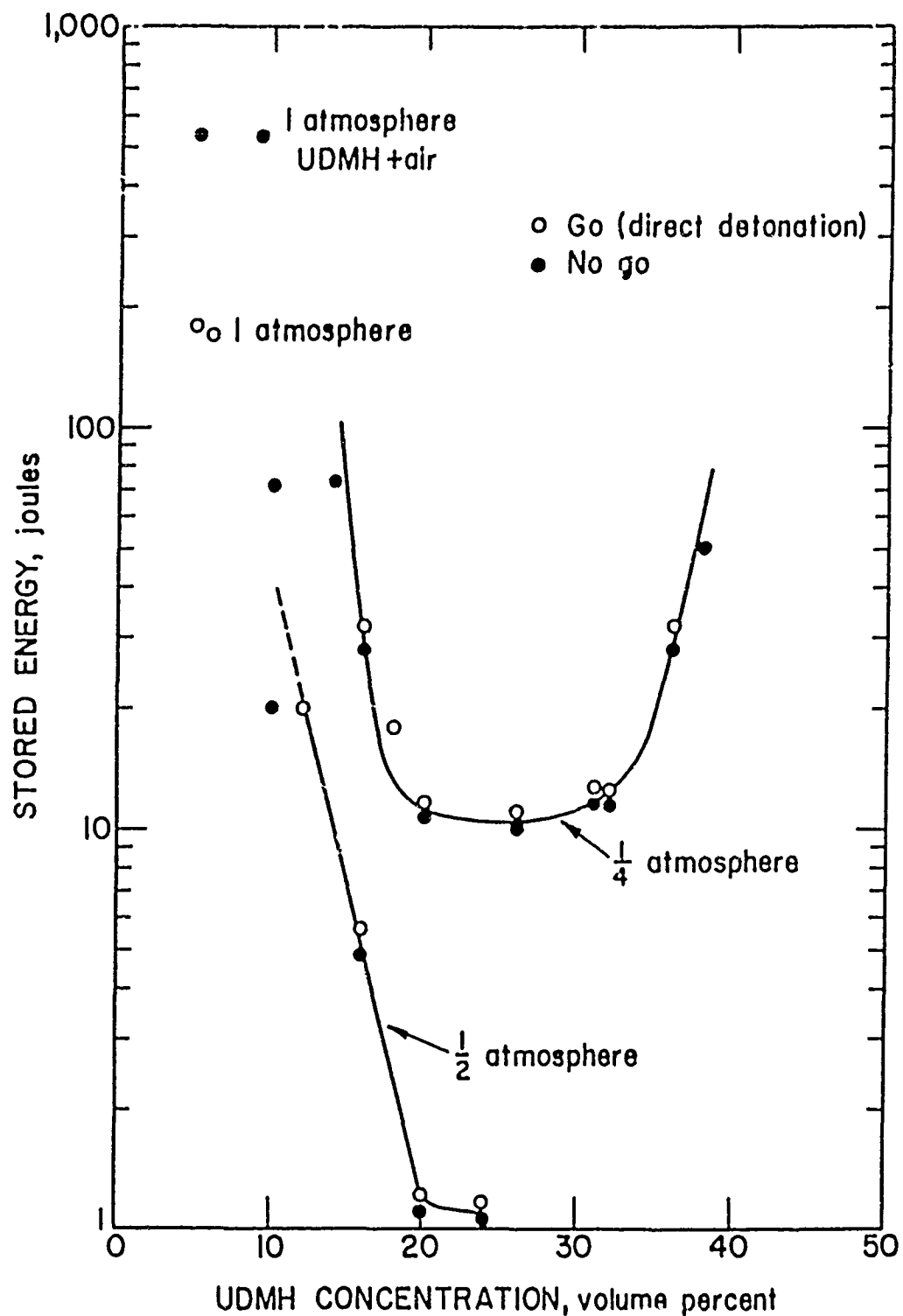


Figure 5.-Stored energy required for direct detonation of UDMH-oxygen mixtures. (Exploding wire in 1 liter bomb).

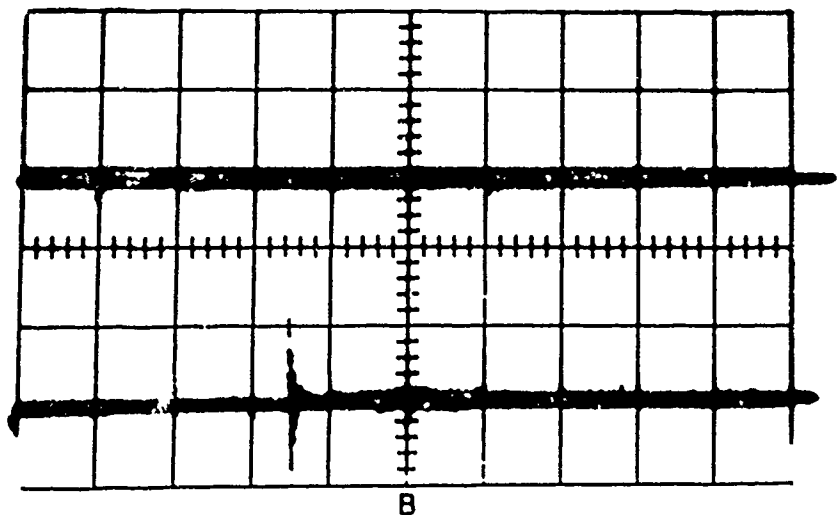
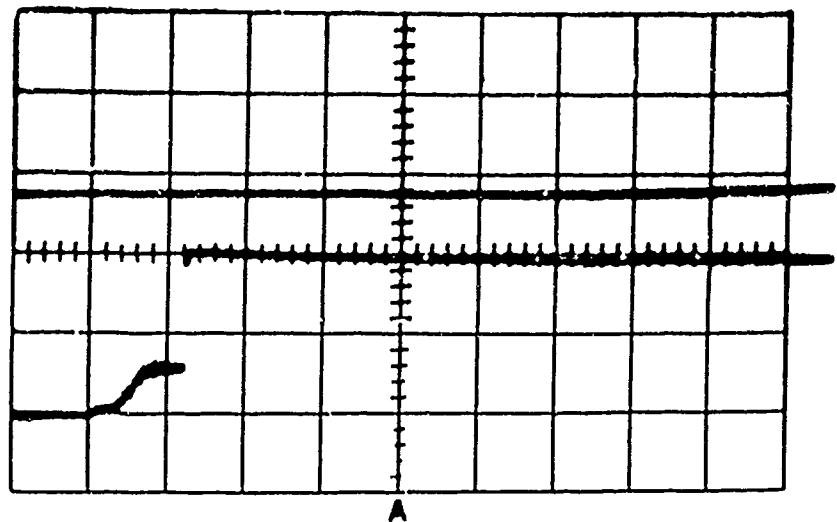


Figure 6.--Pressure-time records of events at the wall of the one liter bomb. Auxiliary sweep indicates a deflection resulting from a 200 p.s.i. pressure increase. One atm. pressure.

A. Discharge of 170 joules stored energy into mixture of 7 percent UDMH + 93 percent air. Sweep rate 2 milliseconds per major division.

B. Discharge of 580 joules stored energy into air. Sweep rate 1/2-millisecond per major division.

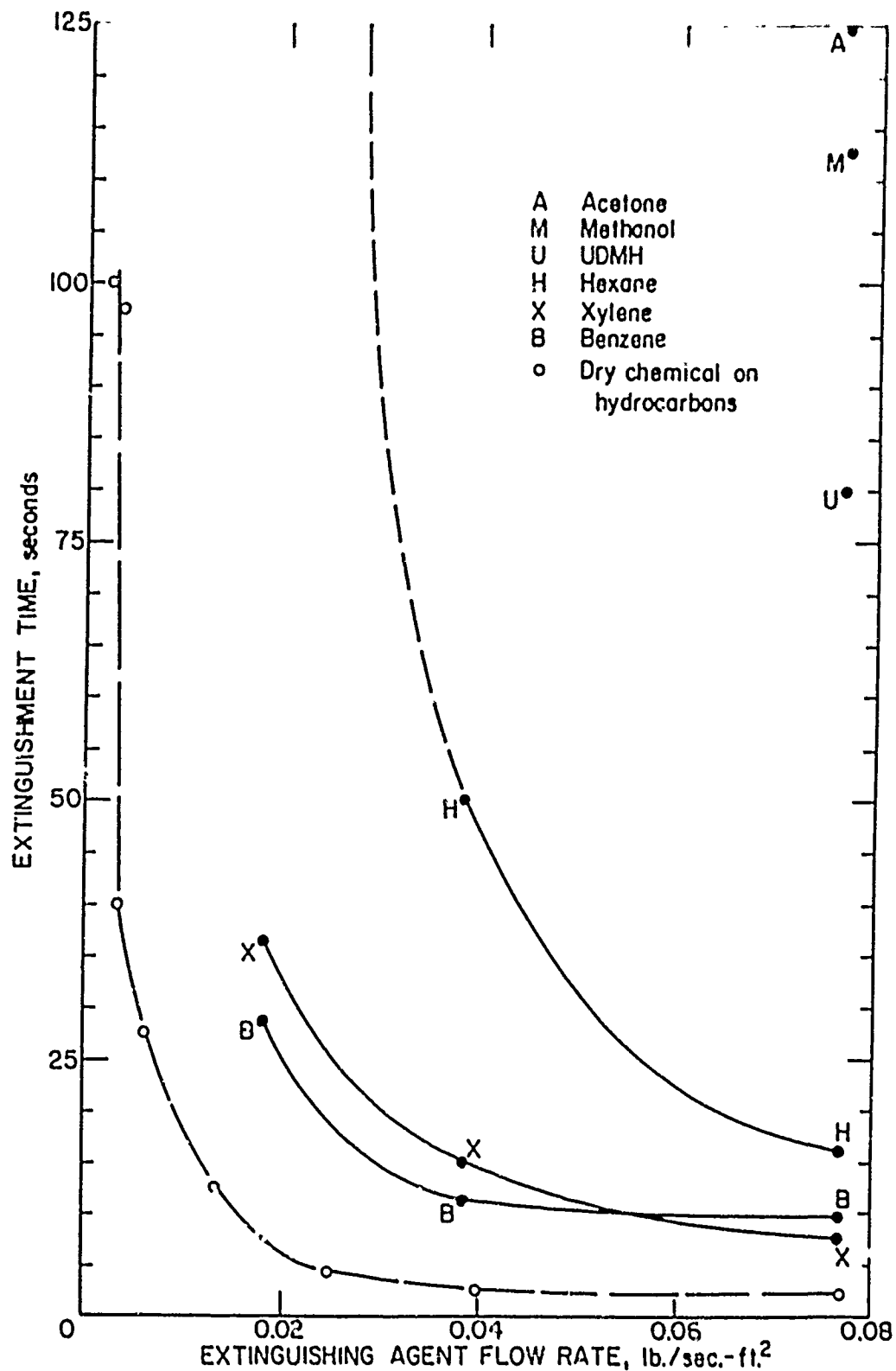


Figure 7.-Water and dry chemical flow rate requirements for extinguishment of liquid fuel fires.



Figure 8.--Burning of MAF-1 in 122 cm. diameter tray.
Photographs taken 10 minutes apart.

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