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Compression-Ignition Studies of a Liquid Monopropellant

A. H. MITCHELL* AND H. A. KIRSHNER*
Frankford Arsenal, Philadelphia, Pa.

Studies were conducted of the compression-ignition of a 60/40 mixture of ethyl and normal propyl nitrate, where the compression piston was hydraulically driven, enabling control of compression ratio and piston travel and velocity. The phenomena occurring during piston travel were followed by means of high-speed photography and measurement of pressure vs time. Compressions at three piston velocities were investigated at various chamber attitudes. Contrary to expectations, increases in piston velocity did not always lead to lower compression ratios for ignition. Minimum compression ratios for ignition were found to be sensitive to chamber attitude. Ignition was observed at the lowest compression ratio for the vertical downward compressions at the slowest of the three piston velocities tested. The effect of liquid turbulence during compression is related to the findings just described.

SEVERAL hypotheses have been set forth to explain the mechanism of initiation of explosions in liquids by compression. One proposal is that rapid compression of a liquid during impact can produce a temperature elevation if a portion of the liquid should crystallize.[1] Another investigator concluded from experimental data that shocks or impacts of the elastic type will cause decomposition reactions, and that initiation does not occur during the compression phase but rather during the expansion phase.[2, 3] The most generally accepted theory of the ignition process, however, is that gas or vapor bubbles are compressed adiabatically, undergoing a sudden increase in temperature.

There have been many studies conducted on the compression sensitivity of explosives, where ignition was explained on the basis of adiabatic compression of vapor bubbles. One such study demonstrated this when it was shown that the impact sensitivity of solid and liquid explosives is increased when minute gas or vapor pockets are entrapped in the material.[4, 5] Films resting on an anvil were ignited by dropping strikers on them. When air bubbles were trapped under the striker, the impact energy required for ignition was several hundredfold less than in the absence of bubbles. Liquid propellants have been ignited using a device called a locked-stroke compressor.[6–8] This device had been previously designed to study compression ignition of solid propellants. In the early work with solids it was observed that, whereas the solid was ignited on compression of gas surrounding it, quenching often occurred when the piston rebounded. This led to the development of a compressor that could be locked in the forward position. In the liquid experiments, a U-tube closed at one end and containing liquid propellant was introduced into the working chamber of the locked-stroke compressor. An air bubble of controlled size was contained in the closed end of the U-tube. A layer of water was placed over the propellant in the open end of the U-tube to prevent possible ignition at the surface. One important parameter that was not controlled, however, was piston bounce, where the piston is now the liquid column. Similar work was carried out, using high-pressure nitrogen in place of the locked-stroke compressor.[9] In another compression-ignition study, the liquid and vapor were contained in a chamber, where a metal piston was driven by high-pressure nitrogen.[10]

Whereas studies with solid propellants have shown that piston bounce can play an important role in compression ignition, no investigations of liquids, to date, have attempted to eliminate this parameter. The experiment described in this paper represents such an attempt. At the same time, it should be recognized that the conditions set up by the compression device used in this study represent an extreme test of sensitivity, since a "one-way" piston does not normally occur in practice.

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* Chemist, Pitman-Dunn Institute for Research.

Instrumentation

The equipment, which will be referred to as the "hydraulic actuated ignition system" enabled control of compression ratio and piston travel, velocity, and bounce. The apparatus was designed and constructed with a view toward measuring the pressure produced in a chamber (containing liquid monopropellant) as a function of compression ratio. In addition, provisions were made to use a glass chamber, so that high-speed photographs could be taken of the entire process.

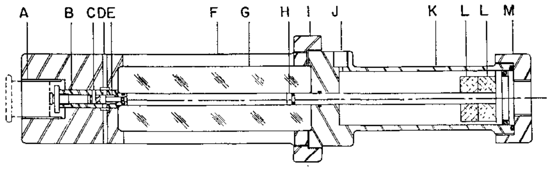


Fig. 1 Liquid propellant window chamber; the glass chamber (G) has the following dimensions: length $5\frac{1}{2}$ in., o.d. $1\frac{13}{16}$ in., i.d. $\frac{5}{16}$ in. Overall length of the system pictured here is $14\frac{1}{2}$ in.

A) Piezoelectric gage
B) Gage piston
C) Shear pin
D) Vent
E) Safety mechanism
F) Metal sleeve
G) Glass chamber
H) Piston
I) Connector
J) Air escape vent
K) Piston drive chamber
L) Spacers
M) Cap

The hydraulic actuated ignition system consists of two sections, the compression chamber section containing the reaction chamber and piston, and the hydraulic system that serves as the energy source to drive the piston. The assembly drawing of the compression chamber section is given in Fig. 1. Starting from left to right, the pressure transducer is a piezoelectric gage, whose output is fed through an appropriate electronic network to a Tektronix Model 535 Oscilloscope, where the pressure is recorded as a function of time. The gage is followed by the gage piston, which serves a dual purpose. It transmits the pressure from the reaction chamber to the gage, while at the same time serves as a safety valve. If the chamber pressure exceeds a predetermined level, a shear pin ruptures, enabling the center portion of the piston to back up. This permits gas from the chamber to travel into the center of the piston and out through the radial vent in the piston and the metal sleeve. The metal sleeve supports the glass window. A slot enables high-speed photographs to be taken of the glass reaction chamber and the piston as it travels through the chamber. The glass window is a laminated borosilicate cylinder having accurately machined dimensions. Three concentric glass tubes were fused together to produce the final product. This was done in an effort to improve the dynamic strength of the chamber. A steel chamber, having the same dimensions as the glass window, was also made up in order to reduce the number of windows required in the testing program. The connector serves to join the metal sleeve and piston drive chamber. The latter is the chamber that the hydraulic oil fills as it moves the piston. At the connector end of this chamber there is an escape vent. This opening permits escape of the air in the chamber as it is replaced by hydraulic fluid.

The small diameter of the compression piston is slightly less than the i.d. of the chamber wall, with an "O" ring affording the seal between the piston shaft and the wall. The larger diameter of the piston has an area twenty times that of the smaller end. This differential area piston prevents piston rebound up to chamber pressures of twenty times that of the driving pressure. Spacers, placed on the shaft of the piston, enable control of the final position of the piston, while its initial position is fixed by the operator prior to the test. This is done by starting out with a fully extended piston, and then pushing it back to its starting point with a metal rod, the rod being inserted where the gage piston is located. Piston travel is measured by inserting a depth vernier gage in place of the metal rod. The propellant volumes are measured by means of a Gilmont μ-pipette. The cap, which seals the piston drive chamber, connects to the hydraulic unit.

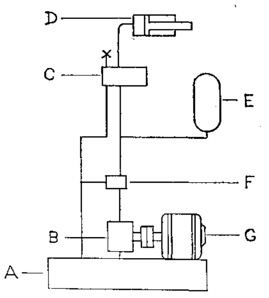


Fig. 2 Hydraulic system.

A) 20-gal reservoir
B) 1.9 gal/min, 3000-psi pump
C) Three-way solenoid valve
D) Compression chamber
E) 1-gal accumulator
F) Unloading valve
G) 3-hp motor

A block diagram of the hydraulic unit is presented in Fig. 2. Oil from the reservoir is pumped up to a predetermined pressure (maximum of 3000 psi), which is controlled by an adjustment on the accumulator unloading valve. When the required pressure is attained, the unloading valve dumps oil back into the reservoir until the pressure drops by about 5%. At that time the valve closes, permitting the pressure to build up again. This allows the pump to run continually, maintaining the approximate required hydraulic pressure. The accumulator is a piston-type device, with oil on the line side of the unit, and high-pressure nitrogen on the other side. It serves as a ballast, minimizing pressure drops while the compression piston is moving. The three-way solenoid valve which is d.c.-actuated, has a $\frac{3}{4}$-in. port, with an opening time of 2 to 3 msec. A 1-in.-i.d. high-pressure flexible hose connects the valve to the compression unit. When the valve is not actuated, the oil in the piston drive chamber is connected, through the valve, to a bypass line leading back to the reservoir. This makes it possible to push the piston back by hand at the completion of a test firing. The oil used was aircraft petroleum-base hydraulic oil, military specification MIL-0-5606.

The photographs were taken by a 16-mm Fastax camera at speeds of approximately 10,000 frames/sec. Back lighting was used, and a relay system synchronized the opening of the solenoid valve, the triggering of the oscilloscope, and the operation of the camera.

Results and Discussion

High-Speed Photography

Figures 3 and 4 present high-speed photographs taken of the rapid compression of a 60/40 mixture by weight of ethyl and n-propyl nitrates (hereafter called alkyl nitrate) in the glass-windowed chamber at three different attitudes and two different compression piston velocities. In these photographs the column of liquid appears as the light area and the piston as the dark area, which elongates as the piston completes its compression stroke. Disturbances in the liquid are indicated by a darkening of its area because the light transmitting properties of the disturbed liquid are much poorer than those of the liquid at rest. Total disappearance of the light area would then denote that the whole mass of liquid is very much agitated. The streak of light down the middle of the chamber ahead of the liquid column in the vertical upward compressions and ahead of the piston in the vertical downward compressions is light refraction due to the curvature of the glass windows (Fig. 3). Optical flats ground on the external surface of the glass windows did not alleviate this condition. It is apparent in the vertical upward and horizontal compressions that the front half of the liquid column becomes agitated while the portion nearest the piston remains relatively calm. Horizontal compressions apparently cause the greatest liquid agitation whereas vertical downward compression arouses the least agitation.

Figure 4 shows frames representing the results of vertical upward and horizontal compressions at two piston velocities, 5.7 and 17.3 fps, respectively. Piston velocities were obtained from the photographs by accurately measuring piston travel as a function of time. Average velocities of 5.7, 13.4, and 17.3 fps, respectively, over $\frac{1}{2}$ in. of travel were determined at driving pressures that could be reproduced to allow selection of any one of the three velocities for particular occasions. It is evident from the photographs that compression at the rate of 17.3 fps causes more liquid agitation than compression at the lower rate.

Photographs of the ignition process are not available. The shock of alkyl nitrate ignition caused the glass to become opaque during the interval of ignition and immediately afterward. This effect has been noted in the case of some glasses and plastics under shock attack.[11] Although the glass windows broke upon ignition in every case, it is believed that the windows did withstand the high pressures long enough to allow recording of the event if the opaque condition had not resulted. This blanking out effect occurred between 2 frames representing a time interval of less than 0.2 msec. Highest pressures encountered during compressions where ignition did not occur, 1300 to 1500 psi, were contained by the glass windows. Attempts to ascertain at what pressures the windows failed after ignition occurred were not successful. The observations made from the photographs of alkyl nitrate behavior under compression were useful in interpreting some of the data collected in the comparison of experimental and calculated adiabatic pressures and determination of minimum compression ratios for ignition. The compression ratio is the ratio of initial to final vapor volume.

Adiabatic Nature of the Compression Process

Steel inserts were used in place of the glass chambers for studies of deviation of pressures from the adiabatic and of

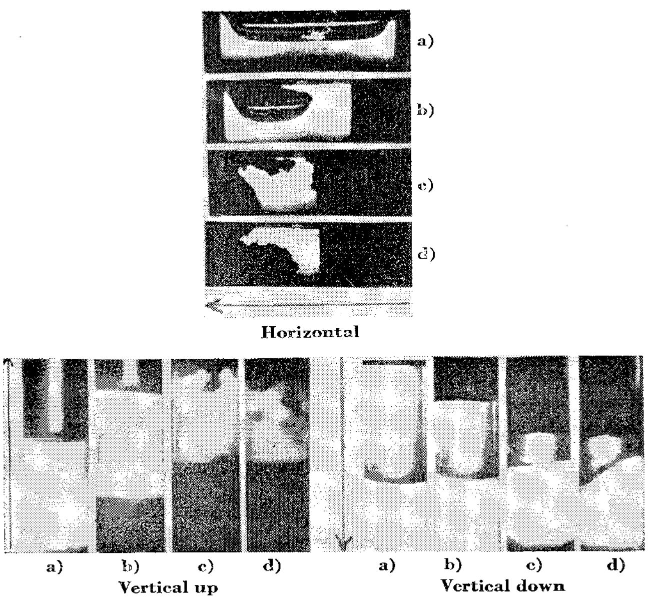
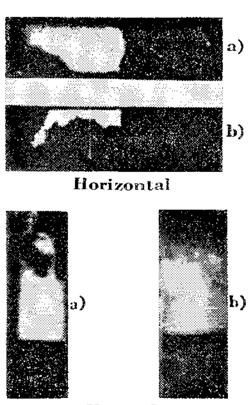


Fig. 3 Representative frames taken from high-speed photographs of alkyl nitrate undergoing compression at different chamber attitudes. The liquid is shown a) prior to piston movement, b) piston one-half way through stroke, c) at piston stop, and d) 2 msec after piston stop. The arrow denotes the direction of piston motion.

Fig. 4 Frames showing alkyl nitrate agitation as a function of piston velocity in the horizontal and vertical upward chamber positions. Pictures show liquid 4 msec after completion of piston travel at two different velocities: a) 5.7 fps, b) 17.3 fps. Extreme agitation is denoted by complete disappearance of the light-colored liquid mass.



a) 17:1 volume compression of water

b) 15:1 volume compression of alkyl nitrate with decomposition but no ignition

c) 17:1 volume compression of alkyl nitrate with ignition

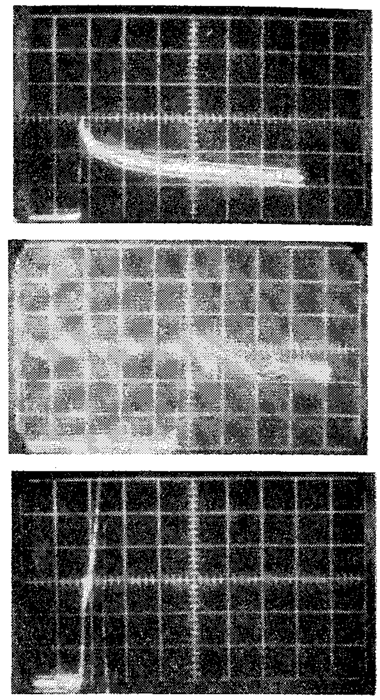


Fig. 5 Typical pressure-time curves of vertical upward compressions. Vertical scale: 1 division = 160 psi; horizontal scale: 1 division = 20 msec.

minimum compression ratios for ignition in the air-alkyl nitrate system in this apparatus. In these cases the pressure-time data obtained from the pressure station at the front of the apparatus (Fig. 1) provided the main clues as to what was happening in the chamber. From the pressure-time data it was possible to measure the peak pressure obtained upon compression, estimate the presence of any significant amount of alkyl nitrate decomposition without ignition, and to verify the occurrence of ignition. Typical pressure-time curves are shown in Fig. 5. Curves indicating probable decomposition of alkyl nitrate exhibit a more gradual drop-off of the peak pressure and higher final pressures than do curves obtained where no decomposition is possible. Ignition is marked by a rapid increase in pressure. Upon ignition, the system vented at approximately 50,000 psi. No attempts were made to measure the peak pressure after ignition, since interest was centered on the initial portions of the curves.

Rapid compressions of various air-liquid systems were carried out in the steel chambers. The pressures were measured and compared with the resultant pressures calculated for these systems, assuming compression of an ideal gas under adiabatic conditions. The end pressures were compared with pressures calculated assuming isothermal conditions. The water-air and the alkyl nitrate-air systems were studied at different compression ratios and at different chamber attitudes. In order to maintain a constant piston stroke (viz., $\frac{1}{2}$ in.), the compression ratios were adjusted by addition of small amounts of liquid. These volumes could be measured with a precision of $\pm$ 0.001 cm^3.

Table 1 shows the deviation from adiabatic conditions for compression of a water-air system in the vertical and horizontal attitudes. The percent of calculated adiabatic pressure actually recorded falls off at the higher compression ratios in the case of the vertical upward compressions. Increased cooling of the compressed gas phase with increasing compression ratio due to higher temperature gradients at the turbulent vapor-liquid interface is believed to be responsible for this increased deviation from the adiabatic condition. The horizontal compressions show a slight overall increase in percent of calculated adiabatic pressure with increasing com-

Table 1 Comparison of experimental pressures recorded for water-air compressions with pressures calculated assuming adiabatic and isothermal conditions[a]

Volume compression ratio	Calculated isothermal press., psi	Calculated adiabatic press., psi	Vertical upward attitude: Recorded press., psi	Vertical upward attitude: Percent of calculated	Horizontal attitude: Recorded press., psi	Horizontal attitude: Percent of calculated
5:1	74	140	135	96.4	75	53.6
10:1	147	371	279	75.6	175	47.2
15:1	221	652	481	73.8	368	56.7
20:1	294	966	599	62.0	568	58.8
25:1	368	1335	800	60.0	...	...

[a] Piston velocity and travel 5.7 fps over 0.5 in.

pression ratio. This was also noted for the horizontal alkyl nitrate compressions up to a compression ratio of 25:1, after which a definite drop is evidenced. As can be expected in view of the greater liquid turbulence and larger air-liquid interface with attendant greater heat loss, the overall percent of calculated adiabatic pressure obtained for the horizontal compressions is low. Turbulence and interfacial heat loss appear to be so great over the whole range of compression ratios that increased cooling at higher compression ratios due to higher temperature gradients is not evident here. A factor tending to offset heat loss at the higher compression ratios for the horizontal compressions is a slight decrease in turbulence at high compression ratios. This decrease in turbulence results from the method used to adjust compression ratios. Compression ratios are adjusted upward by adding small increments of liquid, so that at high compression ratios there is slightly more liquid and slightly less initial vapor volume in the chamber than at low compression ratios. Since there is less room for more liquid to be disturbed, a slight decrease in turbulence, interface, and heat loss might be expected. This factor plays a relatively minor role in the vertical compressions, where turbulence is not greatly affected by changes in the initial vapor volume.

The results of alkyl nitrate-air compressions in the vertical and horizontal attitudes appear in Table 2. Deviation from the adiabatic condition increases with increasing compression ratio for the vertical compressions. The horizontal compression results appear to vary randomly, but the application of correlation techniques shows that the compression becomes less adiabatic with increasing compression ratio for the horizontal compressions also. For the alkyl nitrate-air compressions the picture is also somewhat complicated by the additional factor of alkyl nitrate decomposition. In the horizontal attitude at compression ratios of about 20 to 1, the final pressure begins to exceed the isothermal pressure calculated for that ratio (see the last column in Table 2). Thus decomposition of alkyl nitrate is beginning at about the same compression ratio at which ignition would occur in the vertical attitude. Evidence of decomposition is given by the shape of the pressure-time curve (Fig. 5) and by the appearance of the alkyl nitrate after compression, which in these instances exhibits a yellow-brown color very likely due to NO_2, an early decomposition product. Partial decomposition rather than ignition occurs because of the extreme liquid breakup and high rate of heat exchange encountered in the horizontal compressions. Distribution of energy over a large area probably leaves no point source with a high enough temperature to initiate ignition, but the conditions are extreme enough to cause partial decomposition. It should be noted that NO_2 has been found to inhibit alkyl nitrate decomposition.[12] It would have been interesting to investigate the effect of piston velocity upon the adiabatic nature of the compression process, but this was not possible because the piston, at high velocities (13.4 and 17.3 fps) and when suddenly stopped, transmitted mechanical shock to the pressure-sensing devices. This caused pressure readings to be unreliable. Efforts to eliminate this shock with cushioning techniques or to cull it out with electronic filters were not successful.

Minimum Compression Ratios for Ignition

Minimum compression ratios for ignition of alkyl nitrate in this particular apparatus were studied as functions of chamber attitude and compression piston velocity. The results are indicated in Table 3. Contrary to expectations, increased piston velocities did not generally lead to ignition at lower compression ratios. The vertical upward and downward compressions at increased piston velocities required higher compression ratios to ignite, probably due to greater liquid agitation at higher piston speeds. At all piston velocities vertical downward compressions afforded ignition at the lowest compression ratios. Horizontal compressions appear to give ignition at lower compression ratios for high piston velocities, but in view of the fall off of percent calculated adiabatic pressure with increasing compression ratio (Table 2) and the complicating factor of alkyl nitrate decomposition

Table 2 Summary of vertical upward and horizontal alkyl nitrate-air compression results[a]

Vol. compression ratio	% loading density	Propellant vol., cm³	Calc. adiabatic press., psi	Calc. adiabatic temp., °C	Recorded press. vertical, psi	% of calc. press. vertical	Recorded press. horizontal, psi	% of calc. press. horizontal	Calc. isothermal vertical, psi	End press. vertical, psi	End press. horizontal, psi
5:1	40.5	0.546	140	267	96	68.6	...	...	74	32	...
10:1	47.1	0.635	371	448	224	60.4	160	43.1	147	96	32
13:1	48.5	0.653	539	532	274	50.8	243	45.1	191	140	108
15:1	48.9	0.660	649	577	277	42.7	256	39.4	221	160	147
17:1	49.4	0.666	776	646	306	39.4	320	41.2	250	160	173
20:1	49.9	0.672	966	683	Ignition	Ignition	430	44.5	294	Ignition	288
22:1	50.2	0.676	1115	753			470	42.2	323		365
25:1	50.4	0.679	1335	807			660	49.4	367		435
30:1	50.8	0.684	1720	888			530	30.8	441		270
35:1	51.0	0.687	2134	963			830	38.9	515		435
50:1	51.4	0.693	3510	1152			1150	32.8	735		634

[a] Piston velocity and travel 5.7 fps over 0.5 in.

Table 3 Compression ratios for ignition of alkyl nitrate showing effect of attitude and piston velocity

Attitude	Piston Velocity, fps 5.7	13.4	17.3
Vertical upward	20:1	40:1	45:1
Horizontal	>100:1[a]	100:1[a]	80:1
Vertical downward	<17:1[b]	20:1	27:1

[a] Limit of reliability.
[b] No data for lower compression ratios.

at the high compression ratios, the observed trend is difficult to explain. Ignitions obtained in horizontal compressions at high piston velocities were more violent than those observed at any other attitude, in several cases damaging the apparatus. This is no doubt a result of the extensive liquid-gas interface obtainable in the horizontal compressions.

Factors Involved in Heat Loss

Changes in vapor-liquid interfacial area due to various degrees of liquid agitation during compression have been the main explanation proposed to account for heat losses causing deviations of experimentally determined pressures from the adiabatic. It is recognized, however, that other factors might also be considered. Adiabatic pressures were calculated on the premise of a perfect adiabatic compression of air. Such a premise is far removed from the experimental situation where it must be assumed that other factors arising from the presence of liquid in the system may have a considerable, though undetermined, role in compounding or alleviating heat losses. Among these factors are liquid compressibility, vapor pressure, heat capacity, reaction kinetics, and absorption of the gas phase.

Conclusions

Studies of the effect of piston velocity and chamber attitude upon minimum compression ratio for ignition of a 60/40 mixture by weight of ethyl and n-propyl nitrate in the "hydraulic actuated ignition system" indicate the following: 1) of the three velocities investigated, the low velocity of 5.7 fps with the piston compressing air over the liquid (vertical downward attitude) affords ignition at the lowest compression ratio; 2) horizontal attitude compressions require the highest compression ratios for ignition at each piston velocity investigated; and 3) increase in piston velocity with subsequent greater agitation of the liquid does not, except perhaps for horizontal compressions, lead to ignition at lower compression ratios.

In spite of the unfortunate blanking out of the ignition process, use of the glass-windowed chamber has yielded high-speed photographs of compressions of alkyl nitrate during which ignition does not occur. Observation of these films has led to the conclusion that sudden compression of alkyl nitrate in the horizontal attitude at low loading densities (up to 50%) causes a variable high degree of liquid agitation, exposing areas of liquid-vapor interface in what appears to be random fashion not capable of duplication from compression to compression. Thus ignition site, delay, and probability of propagation are unpredictable from compression to compression.

At marginal compression ratios in the vertical compressions and in horizontal compressions where high end pressures were obtained, propellant partial decomposition occurred with probable evolution of NO_2. Since NO_2 has been found to inhibit alkyl nitrate decomposition, its presence most likely acts as a desensitizer, especially in cases where ignition by recompression of a system is attempted.

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