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The Combustion of Hydrocarbons With Nitric Acid at High Pressure

by

James T. Bryant
and
Stanley E. Wood
Propulsion Development Department
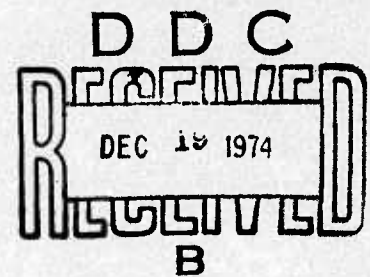
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R. G. Freeman, III, RAdm., USN . . . . Commander
G. L. Hollingsworth . . . . Technical Director

FOREWORD

This report presents the results of a small-scale laboratory investigation of the combustion of hydrocarbons with nitric acid at high pressure. This study was performed during the first half of fiscal year 1974 under the Navy Liquid Propellant Gun Technology Program, AirTask A350-350B/008B/3F32-363-502.

This report was reviewed for technical accuracy by Dean H. Couch.

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(U) *The Combustion of Hydrocarbons With Nitric Acid at High Pressure,* by James T. Bryant and Stanley E. Wood. China Lake, Calif., NWC, November 1974, 28 pp. (NWC TP 5588, publication UNCLASSIFIED.)

(U) A slightly modified Technoproducts instrumented Olin-Mathieson drop-weight tester has been used to study the combustion of liquid hydrocarbon fuels with nitric acid at high pressure. The effects of the following variables on ignition and the magnitude and reproducibility of the burning rate are discussed: (1) impact energy, (2) carbon number of n-alkanes, (3) oxidizer/fuel ratio, (4) hydrocarbon structure, (5) binary fuel mixture composition, and (6) acid concentration.

CONTENTS

INTRODUCTION

The successful development of a bipropellant liquid propellant gun (LPG) will offer a number of advantages over its existing solid propellant competitors, the most obvious being the elimination of bulky cartridge cases and the safety inherent in the separation of the fuel and oxidizer during storage. Moreover, actual experimental firings indicate significant improvements in ballistic efficiency. A wide range of oxidizer/fuel (O/F) ratios are possible. Also, a stratified-charge configuration is conceivable, whereby the O/F ratio in the main charge is optimized for ballistic efficiency and combustion products and the O/F ratio in the preignition chamber is optimized for ease of ignition.

One undesirable aspect of the propellant combination currently under evaluation in Navy bipropellant LPGs (n-octane + 90% nitric acid) is an initial pressure spike (about 75,000 psi). This is much higher than the base pressure (40,000-50,000 psi) which exists for most of the internal ballistic cycle. A rapid rise to a pressure plateau of 50,000-60,000 psi would be desirable. Shot-to-shot reproducibility has traditionally been a problem with LPGs and, while recent firings have demonstrated marked progress in this area, additional improvements are needed.

Very little LPG propellant evaluation has been done up until now; tentative propellant combinations have been chosen because of their physical and chemical properties during storage and handling and their availability. The time and expense involved in experimental gun firings has precluded a comprehensive evaluation of LPG propellant combustion at the high pressures obtained in a gun. A technique has now been developed whereby non-hypergolic bipropellant combinations can be conveniently studied in the laboratory at gun-chamber pressures (up to about 50,000 psi). The combustion of nitric acid with a large number of saturated hydrocarbons over a range of O/F ratios is reported below.

EXPERIMENTAL

The apparatus (Figure 1) used in these experiments was a Technoproducts Olin-Mathieson drop-weight tester incorporating a special pressure cell (Figure 2) originally designed for monitoring the pressure-time history during the combustion of liquid explosives and monopropellants.[1] The principle of its operation is as follows. Two[2] Viton o-rings are placed in the bottom sample cup; then small quantities (normally 30 microliters total) of the fuel and oxidizer are carefully syringe-injected into the space in the bottom of the cup (encircled by the o-rings). A stainless-steel diaphragm and vented piston are inserted onto the o-rings and the sample cup assembly is placed into the pressure-cell body. The retainer ball and cap are added and the cap is torqued to 7 in-lb. The assembled pressure cell is then placed in the

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[1] Griffin, D. N. *Initiation of Liquid Propellants and Explosives by Impact.* American Rocket Society Technical Paper No. 1706-71.

[2] While the standard procedure calls for only one Buna o-ring, Viton was used because of its greater compatibility with nitric acid; two were required to give reproducible ignition.

drop-weight tester. Ignition energy is supplied by the impact on the retainer ball by a known weight dropped from a known height above the ball. The sample pressure is imparted to the force transducer by means of the pistons and hydraulic fluid. The transducer output is displayed on a triggered oscilloscope and photographed. Because of the elasticity of the fluid coupling, up to 50% of the impact energy is absorbed by the cell, and the minimum ignition energies obtained with this device will be significantly higher than those obtained with the conventional rigid cell. The disassembled pressure cell is shown in Figure 3.

A typical pressure trace is shown in Figure 4. Time measurement was arbitrarily begun when the impact pressure spike would have returned to zero pressure if no combustion had occurred. A blank (nonreactive) experiment was always run for baseline calibration.

RESULTS AND DISCUSSION

IMPACT ENERGY

Preliminary tests were run with several hydrocarbons and nitric acid/water (90/10% by weight) to determine the impact energies required to produce ignition 50% of the time. These experiments were performed in the standard apparatus without the pressure cell so results could more easily be compared with those for other materials. The only modification of the standard procedure was substitution of the two Viton o-rings for the single Buna o-ring. The total propellant volume was 30 microliters at an O/F volume ratio of 2:1. The results are given in Table 1 along with a value for n-propyl nitrate reported in the Technoproducts manual. All the hydrocarbon/acid systems were relatively insensitive, and ignition could not be obtained using n-pentane.

EFFECT OF CARBON NUMBER OF N-ALKANES

The pressure cell was used to examine the reactions of a series of normal alkanes (C_5 to C_{16}) with nitric acid/water (90/10) at three different O/F ratios. Table 2 lists some pertinent properties of the hydrocarbon fuels. Volume ratios of 4:1, 2:1 and 1:1 were employed for the following n-alkanes.

1. n-pentane, C_5H_{12}
2. n-hexane, C_6H_{14}
3. n-heptane, C_7H_{16}
4. n-octane, C_8H_{18}
5. n-decane, $C_{10}H_{22}$
6. n-dodecane, $C_{12}H_{26}$
7. n-hexadecane, $C_{16}H_{34}$

The results are summarized in Table 3. The pressure 2 msec after the impact-pressure pulse is used as a measure of the pressurization rate; the maximum pressure reached in 18 msec (total sweep time of the scope) is also tabulated.

Figures 5-7 present pressure (at 2 msec) versus carbon number plots for the three O/F ratios tested. All the data points in Table 3 and Figures 5-7 represent the mean of at least three measurements except the point for hexane at an O/F volume ratio of 1:1; hexane reacted only once out of many attempts. It appears that the n-alkanes can be divided into three categories of reactivity:

1. Class I — high-volatility fuels (pentane, hexane) which are unreactive because they present an overly fuel-rich vapor phase.

2. Class II — intermediate-volatility fuels (heptane, octane) which produce a combustible vapor phase prior to or shortly after impact.

3. Class III — low-volatility fuels which require a significant fraction of the impact energy for vaporization to produce a combustible vapor mixture.

The category to which a compound belongs may depend upon the experimental conditions; apparently, n-decane may be in either Class II or III, depending on the laboratory temperature. On any given day the results with n-decane were reproducible, but the initial series of runs at an O/F volume ratio of 2:1 were completely different from a series conducted about 2 weeks later. This was probably a vapor-pressure-dependent phenomenon encountered because of poor laboratory temperature control. Hexadecane ($C_{16}H_{34}$) and dodecane ($C_{12}H_{26}$) would not ignite (250 kg-cm of impact energy) at an O/F volume ratio of 1:1.

EFFECT OF OXIDIZER/FUEL RATIO

Figures 8-11 are pressure (at 2 msec) versus O/F volume ratio plots for the hydrocarbons that exhibited reproducible rates over the extreme range of O/F ratios investigated. Included with the curves for the n-alkanes (Figures 8 and 9) are curves for TH Dimer (tetrahydromethylcyclopentadiene dimer) (Figure 10) and Decalin (decahydronaphthalene) (Figure 11). These compounds were included in the study because of their good chemical and physical properties, ready availability, and reasonable cost. TH Dimer is used in the Talos missile, while Decalin is being evaluated as a fuel for future Navy airbreathing missiles. Both displayed pressurization rate versus O/F curves similar in *shape* to those of the reactive n-alkanes, but at about 25-50% lower pressures. These fuels, which may be classed as reactive/nonvolatile, generally gave more reproducible pressure traces than the n-alkanes.

Two conclusions may be drawn directly by inspection of Figures 8 through 11.

1. Pressurization rates are greater at an O/F volume ratio of 2:1 than at 4:1 or 1:1 ratios.

2. Pressurization rate reproducibility is better at the intermediate (2:1) O/F ratio.

An additional, very pronounced, effect of O/F ratio was observed in these experiments. At an O/F volume ratio of 4:1 (oxidizer rich), the 17-4 PH stainless steel reaction cup was severely eroded in as few as 2-3 shots, while the cups lasted indefinitely with no evidence of erosion at volume ratios of 1:1 and 2:1 (fuel rich). However, erosion occurred only in localized regions around the upper o-ring, indicating that leakage along the metal surface and a high gas flow rate were required to produce the erosion. The erosion was caused by the combination of high temperature and the highly corrosive nature of the gas produced by incompletely burned nitric acid.

Some light is shed on these pressure rate and erosion observations by the theoretically calculated data (Figure 12) for n-octane and nitric acid/water (93/7). These data, obtained via the Navy's Propellant Evaluation Program, assume thermodynamic equilibrium and adiabatic reaction. As the O/F ratio is increased from a volume ratio of 1:1, the temperature increases to a maximum at about 2.7:1 and decreases slowly thereafter. At the same time, there is a compensating decrease in the volume of gas produced. Since these calculations were done at constant pressure (33,000 psi), the work done on a projectile is approximately proportional to the product of the moles of gas and the absolute temperature.

$$Work = P\Delta V \simeq n_g RT$$

where

P = pressure

ΔV = volume of products minus volume of reactants

n_g = moles of gas

R = gas constant

T = absolute temperature

A ($T \times n_g$) versus O/F ratio plot (calculated) is shown in Figure 13. Clearly, a greater overall efficiency is predicted for an O/F volume ratio of 2:1 than for either 1:1 or 4:1 since more work can be done on the projectile.

HYDROCARBON STRUCTURE

Two conclusions can be drawn with respect to the effect of molecular structure on the burning rates of hydrocarbons with nitric acid:

1. Chain branching promotes increased reaction, since trimethyldodecane ($C_{15}H_{32}$) burned faster than either n-dodecane ($C_{12}H_{26}$) or n-hexadecane ($C_{16}H_{34}$).

2. Cyclic structures seem to burn faster than straight-chain molecules, since TH Dimer ($C_{12}H_{20}$) reacted faster than n-dodecane ($C_{12}H_{26}$), and decalin ($C_{10}H_{18}$) reacted faster than n-decane ($C_{10}H_{22}$).

Both branched-chain and cyclic hydrocarbons contain tertiary hydrogen atoms, which are more reactive than the primary and secondary hydrogens on the straight-chain molecules.

BINARY MIXTURES

It has been shown (Table 3) that the pressurization rate obtained by the combustion of octane (C_8H_{18}) with nitric acid is measurably higher than that observed when high-molecular-weight hydrocarbons are substituted for the octane. Hexadecane ($C_{16}H_{34}$) was especially unreactive. It was to be expected, therefore, that blends of octane with hexadecane might yield pressurization rates between those of the two pure compounds.

Several octane/hexadecane compositions were tested with nitric acid/water (90/10% by weight) at an O/F volume ratio of 2:1. The results are presented in Figure 14 as plots of combustion pressure (at 2, 8 and 18 msec after the impact ignition pulse) versus the volume percent of octane in the fuel. Each experimental point represents the average of at least three firings.

At least three conclusions can be reached by inspection of Figure 14.

1. The pressurization rate of octane/nitric acid combustion may be moderated by the substitution of hexadecane for some of the octane.

2. The results of this substitution are more pronounced early in the combustion. This effect is shown clearly in Figure 15 where the ratio of the pressure obtained with pure octane to the pressure obtained with pure hexadecane is plotted against combustion time. It appears that, given enough time, the pressure ratio would be near 1:1.

3. The effect of an increment of hexadecane is greater at the higher octane concentrations.

One additional observation was that adding octane (10% by volume) produced a very beneficial effect on the ease and reproducibility of hexadecane ignition.

A second study was conducted with binary mixtures of n-octane with Decalin (decahydronaphthalene). Decalin is currently under investigation at NWC as a component in airbreathing missile fuels and has physical properties very much like those of JP-5. Moreover, it has superior chemical properties, including nonreactivity with nitric acid at room temperature.

Figure 16 shows plots of the combustion pressure, at 2, 8 and 18 msec after the ignition pulse, for several mixtures of n-octane and Decalin with nitric acid (90/10%). These plots are similar to the ones for octane/hexadecane mixtures and demonstrate that the combustion kinetics may be tailored by adjusting the relative amounts of Decalin and octane in the fuel mixture.

7

ACID CONCENTRATION

TH Dimer with nitric acid/water (90/10% by weight) at an O/F volume ratio of 2:1 was found to be a very smooth-burning, reproducible combination. Experiments were extended to include TH Dimer with nitric acid/water (83/17% by weight), and TH Dimer with red fuming nitric acid (RFNA) (20% NO_2 by weight). Both combinations were at an O/F volume ratio of 2:1.

Pressure-time curves for all three combinations are shown in Figure 17. The ranking of the three is not surprising, but the extreme reluctance of the propellant containing nitric acid (83/17%) to develop rapid combustion was not expected. Ignition always occurred, but pressurization was very slow. The RFNA curve represents the runs where ignition occurred immediately after the original impact. In most cases, the mixture was ignited by the rebounding weight with an energy of only about 10% of the original impact. Apparently, much of the original impact energy was absorbed by vaporizing and dissociating N_2O_4 ($N_2O_4 \rightleftarrows 2NO_2$), and mixing of the resulting NO_2 with the TH Dimer. Hydrocarbon/NO_2 mixtures are known to be extremely sensitive,[3] and ignition occurred readily upon the second impact.

SUMMARY AND CONCLUSIONS

A number of characteristics of high-pressure hydrocarbon/nitric acid combustion were clarified by the drop-weight-tester technique.

1. Burning rate is strongly dependent on the molecular weight of the hydrocarbon. For the normal alkanes, there are three pressurization regimes: (I) high-volatility fuels where the vapor-phase composition is so fuel-rich that ignition is extremely difficult, (II) rapid-burning, intermediate-volatility fuels, which produce a combustible-vapor phase prior to or shortly after impact, and (III) slow-burning, low-volatility fuels, which require a significant amount of the impact energy to volatilize the fuel and thereby create a combustible-vapor phase. Admittedly, this explanation of the volatility effect is speculative, but it does rationalize the data and is analogous to hydrocarbon/air combustibility.

There is an optimum molecular weight (around C_{10} to C_{12} for n-alkanes) at which the pressure data display the best reproducibility.

2. The pressurization rate depends on the O/F ratio. For the reactive fuels, there is an optimum O/F region (fuel rich) where combustion proceeds most reproducibly.

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[3] Davis, T. L. *Chemistry of Powder and Explosives*, Wiley, New York, 1943.

3. Oxidizer-rich combustion leads to a more serious metal erosion problem than is encountered under fuel-rich conditions. Whether oxidizer-rich propellants cause a significant problem in a gun remains to be determined. There is no evidence of excessive gun barrel erosion to date. Quite the contrary, the data suggest longer barrel life under all O/F ratio conditions compared to solid-propellant guns.

4. Branched-chain and cyclic saturated hydrocarbons burn faster and more reproducibly than their straight-chain counterparts.

5. The rate of hydrocarbon/nitric acid combustion can be tailored to some extent by adjusting the structures and relative amounts of the hydrocarbon fuel components.

6. The rate of hydrocarbon/nitric acid combustion is strongly dependent on the water concentration in the nitric acid. The addition of NO_2 to the acid increases the reaction rate, but may inhibit ignition under some conditions.
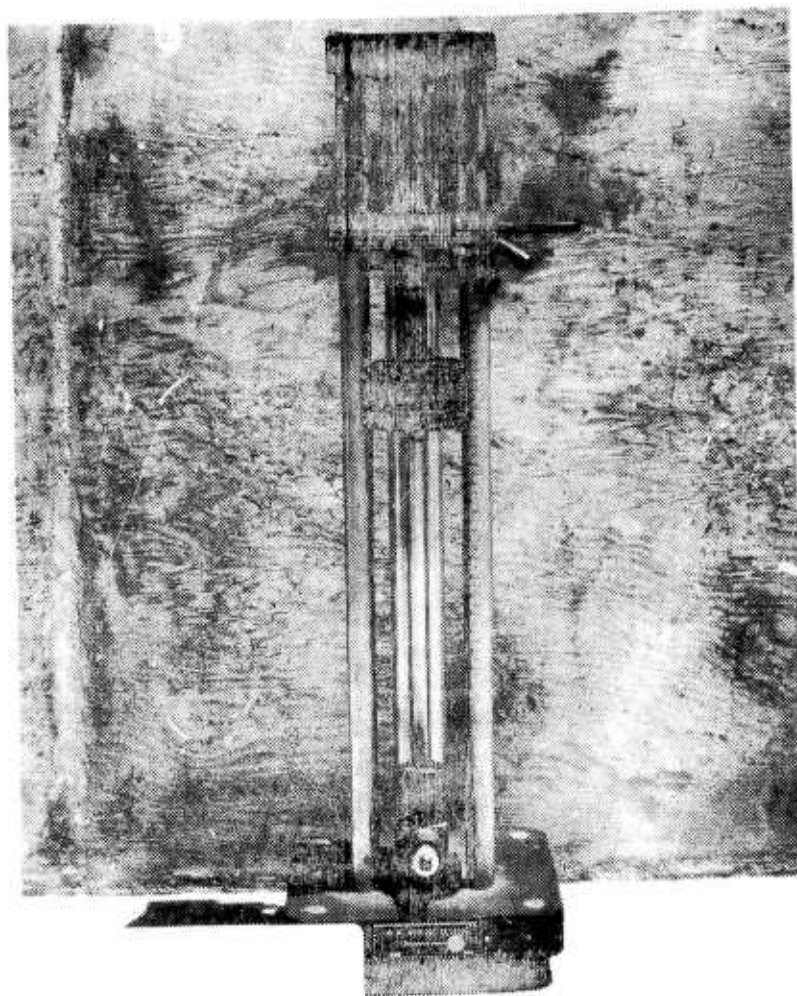
FIGURE 1. Drop-weight Tester.
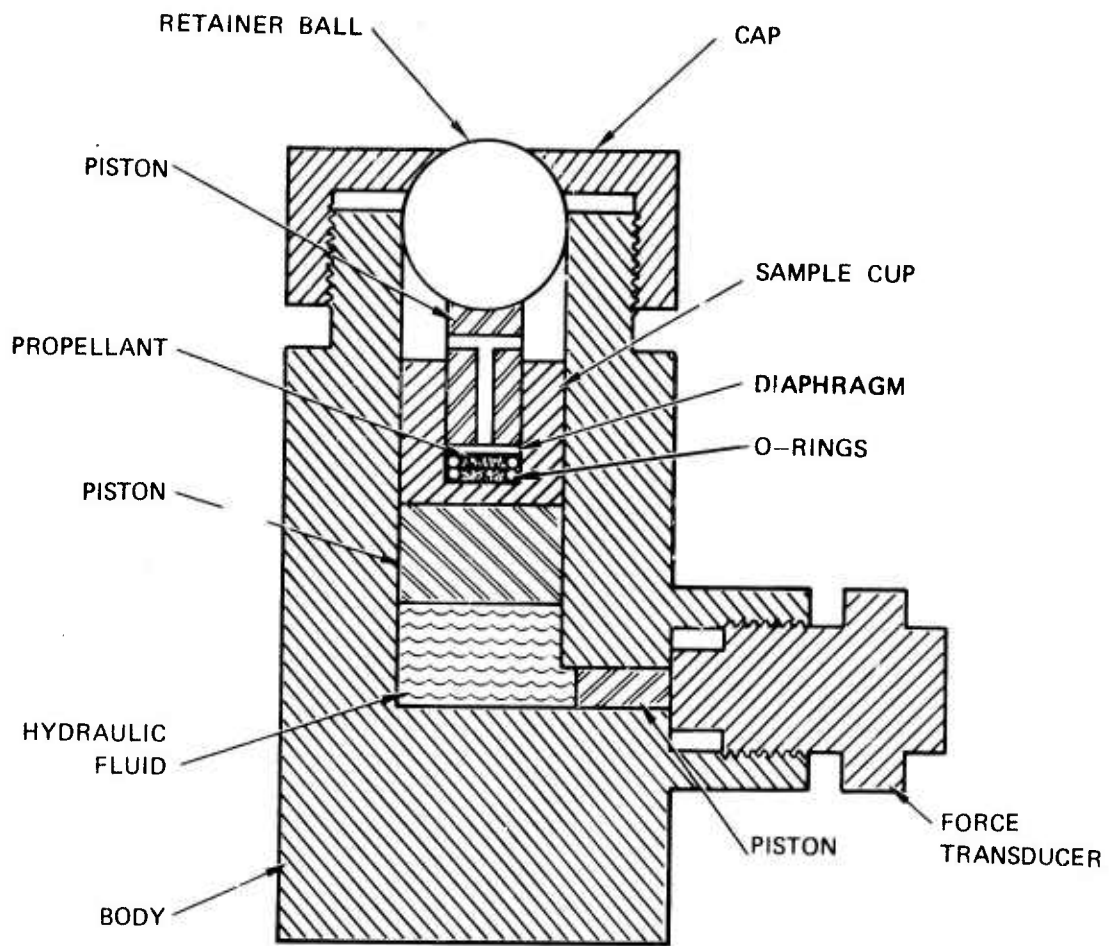
FIGURE 2. Technoproducts Olin-Mathieson Drop-Weight Tester Pressure Cell.
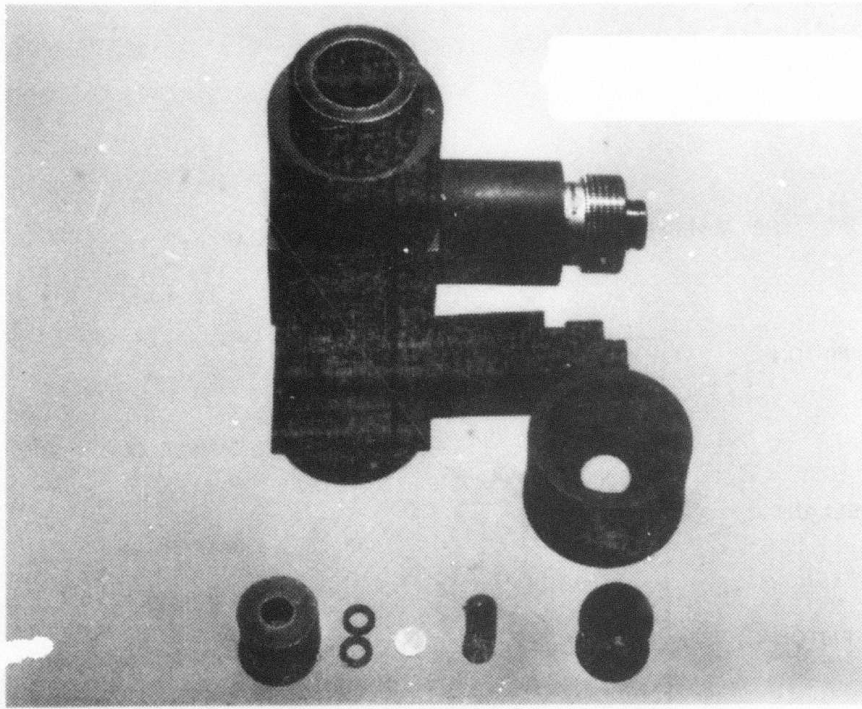
FIGURE 3. Disassembled Pressure Cell.
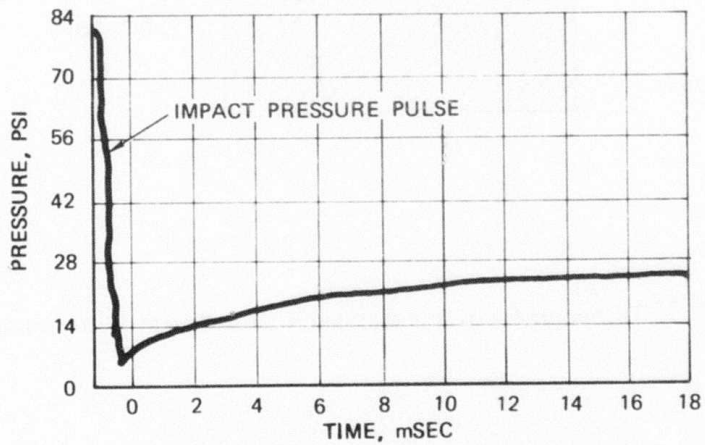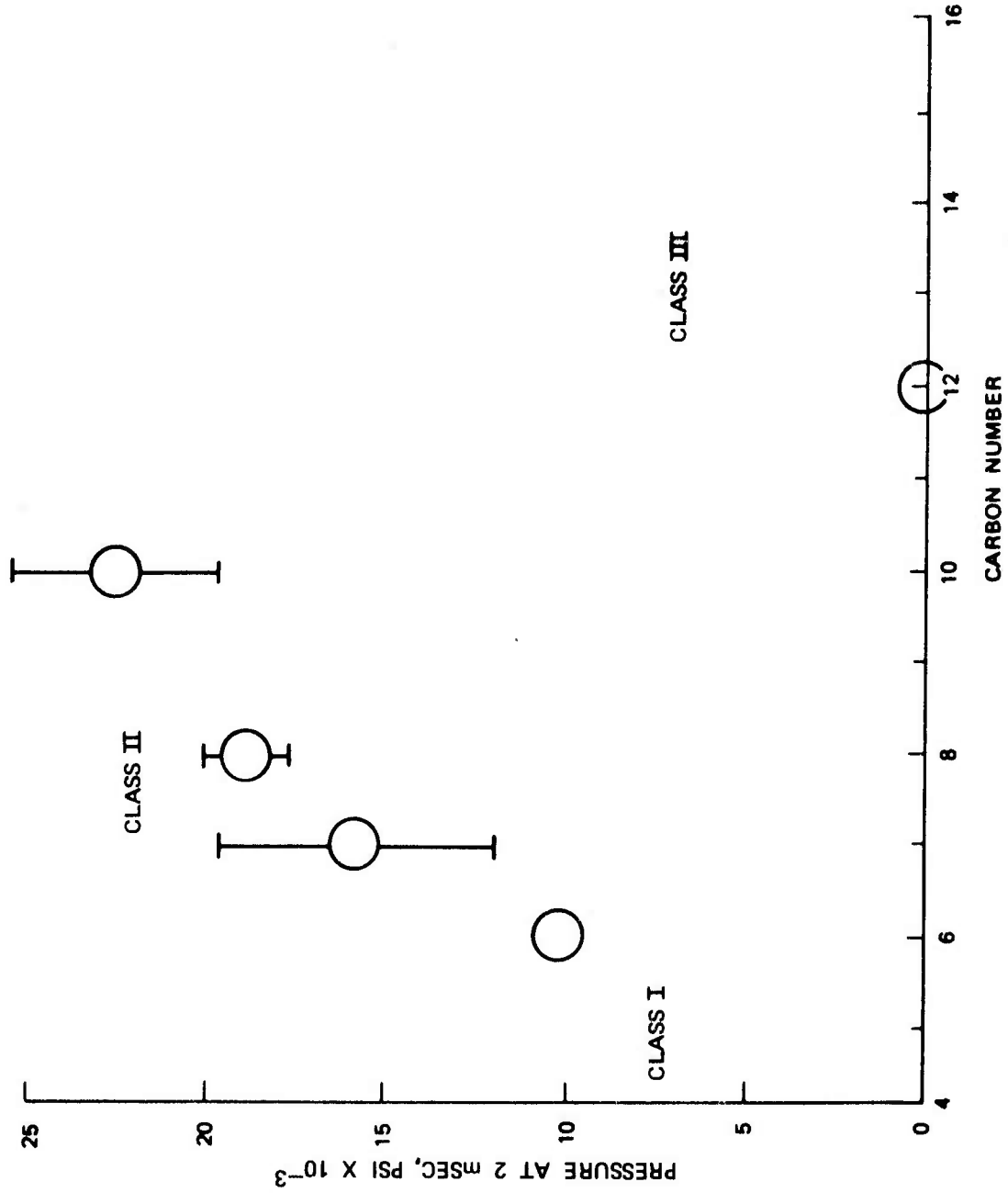

FIGURE 4. Typical Pressure Trace.

FIGURE 5. Effect of Carbon Number on Pressurization Rate for n-Alkane/Nitric Acid Combustion (O/F Volume Ratio = 1:1).
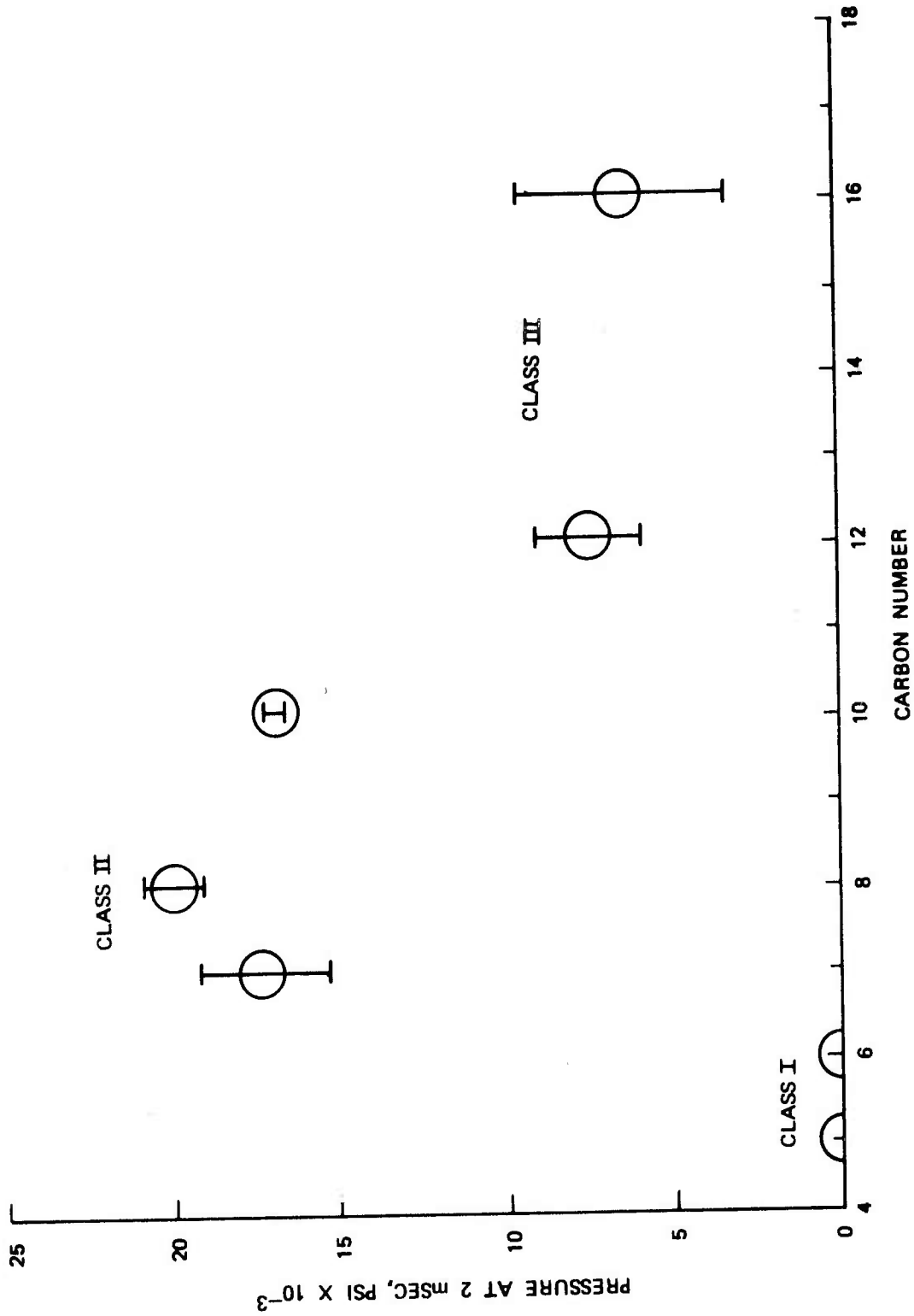
FIGURE 6. Effect of Carbon Number on Pressurization Rate for n-Alkane/Nitric Acid Combustion (O/F Volume Ratio = 2:1).
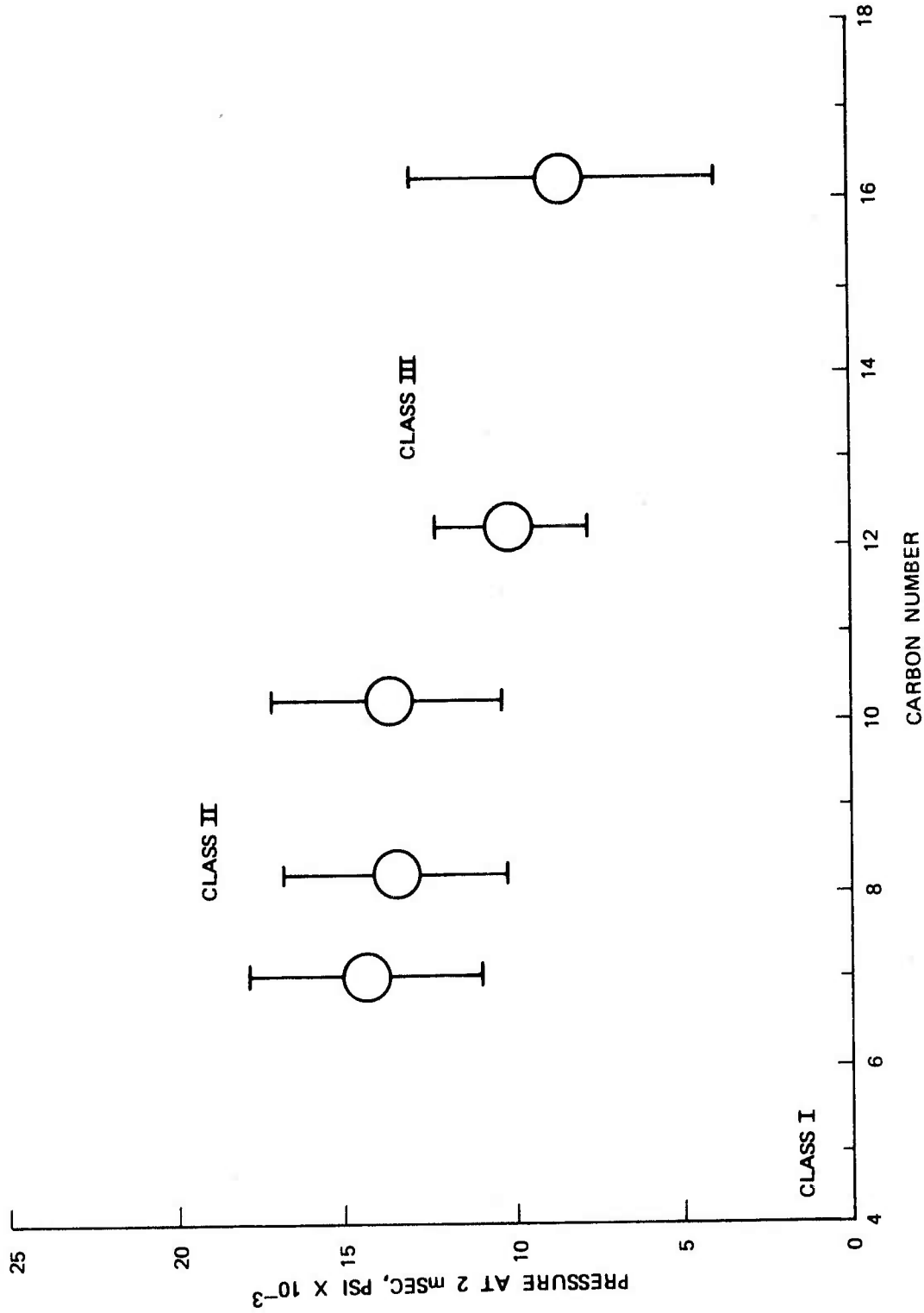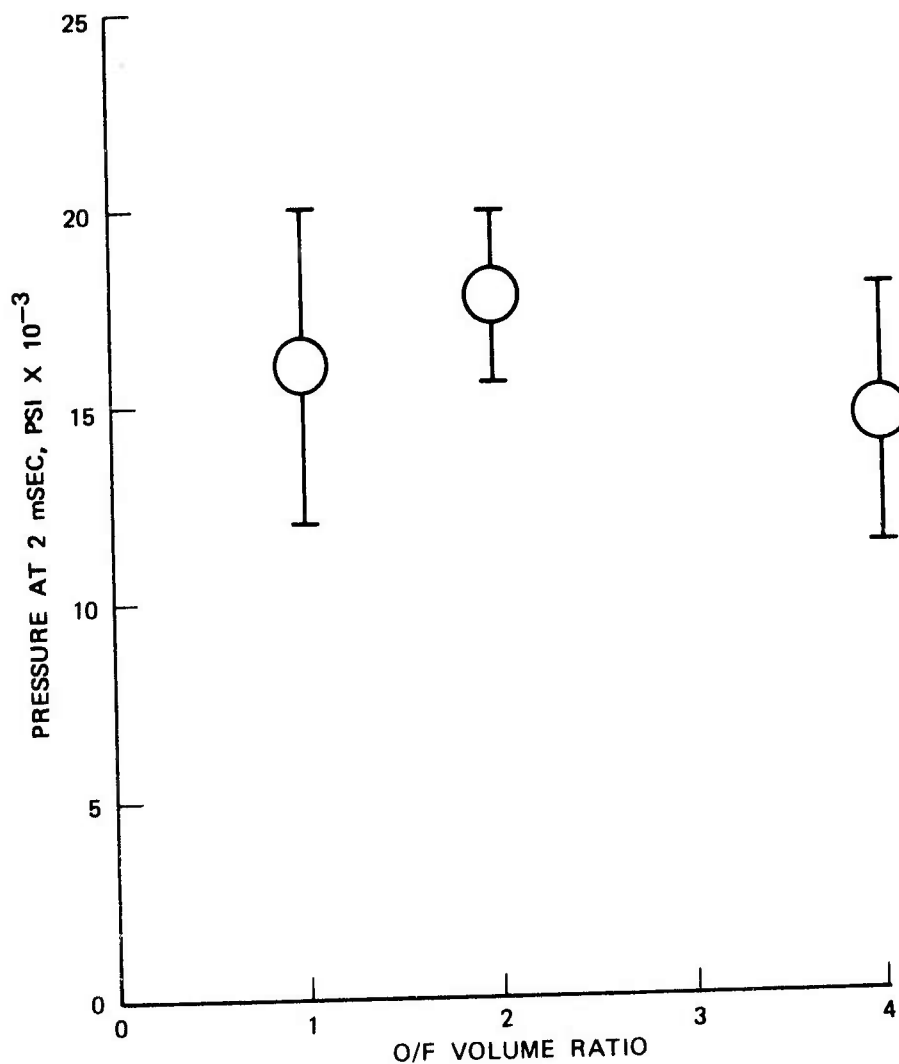
FIGURE 7. Effect of Carbon Number on Pressurization Rate for n-Alkane/Nitric Acid Combustion (O/F Volume Ratio = 4:1).

FIGURE 8. O/F Ratio Dependence for n-Heptane Combustion With
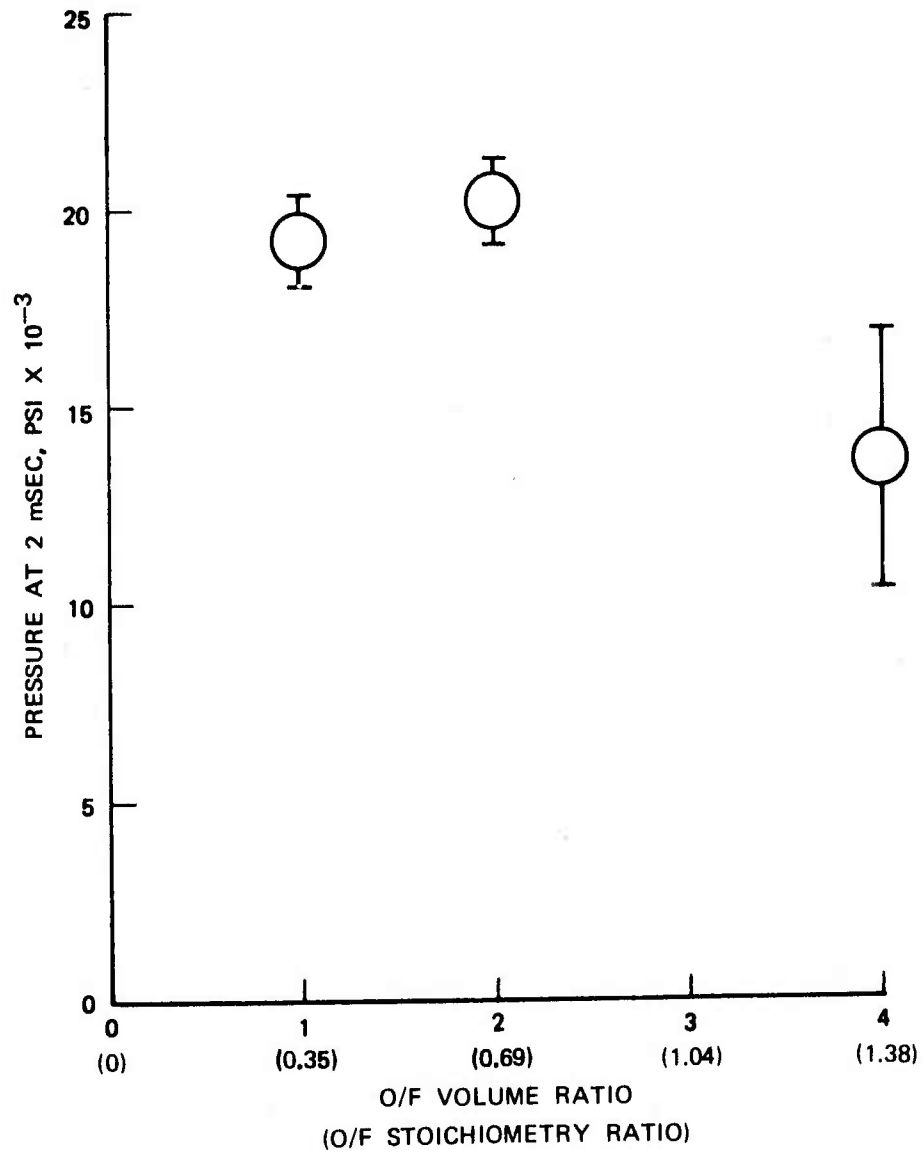Nitric Acid/Water (90/10% by weight).

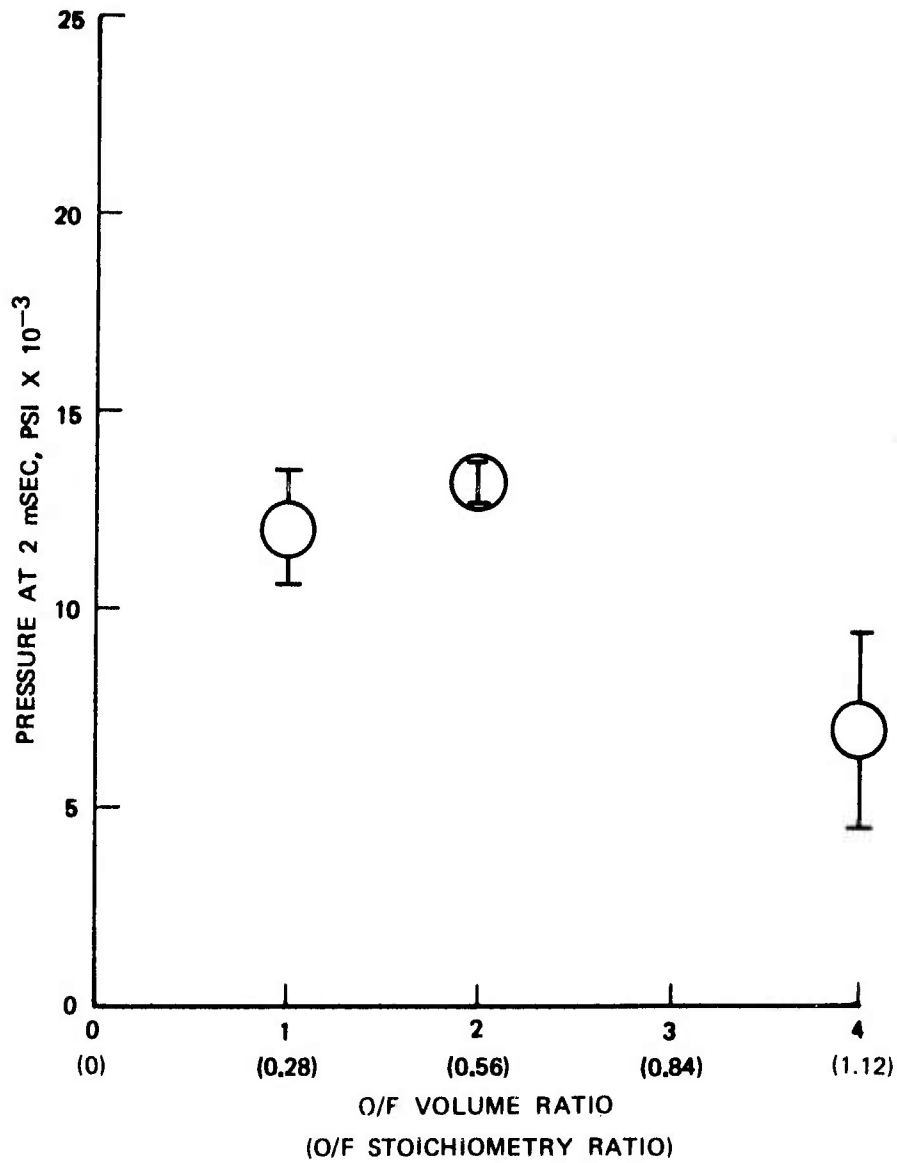FIGURE 9. O/F Ratio Dependence for n-Octane Combustion With Nitric Acid/Water (90/10% by weight).

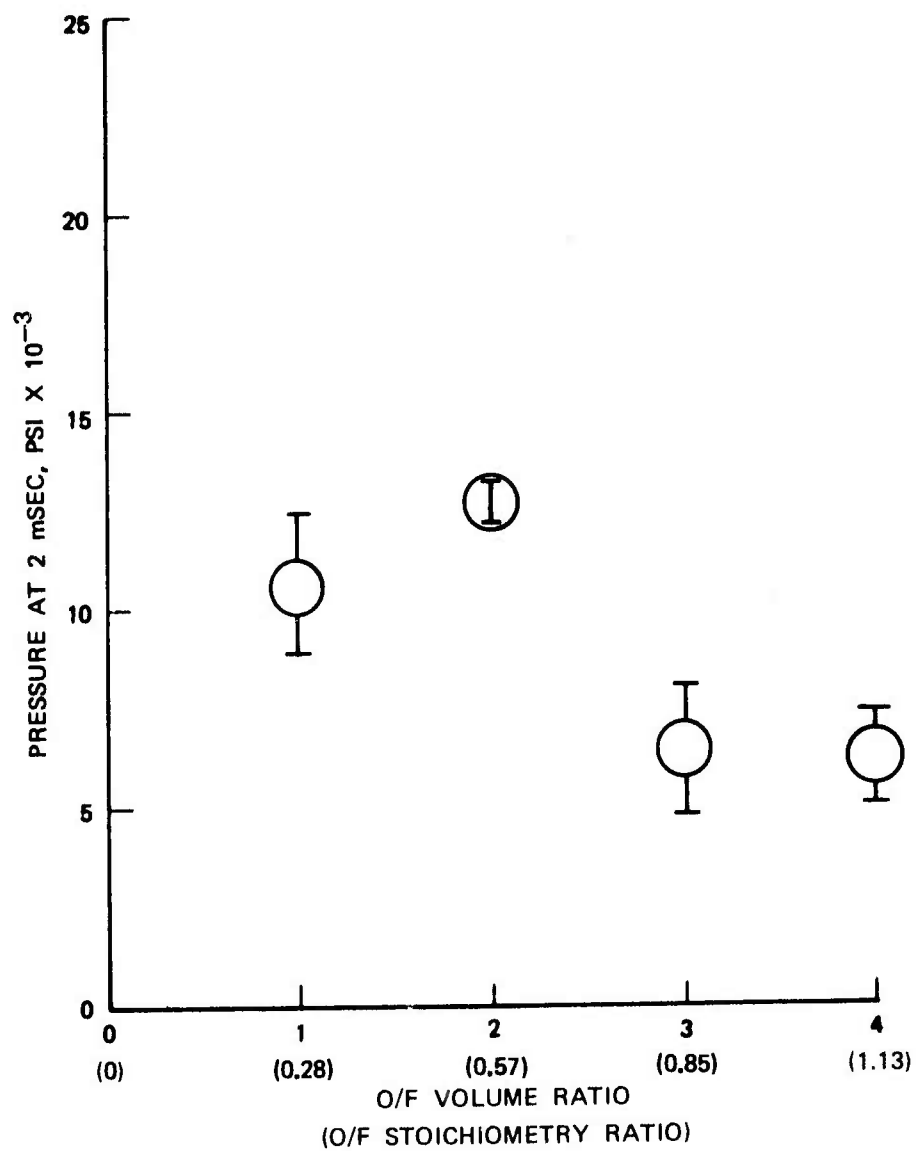FIGURE 10. O/F Ratio Dependence for TH—Dimer Combustion With Nitric Acid/Water (90/10% by weight).

FIGURE 11. O/F Ratio Dependence for Decalin Combustion With Nitric Acid/Water (90/10% by weight).
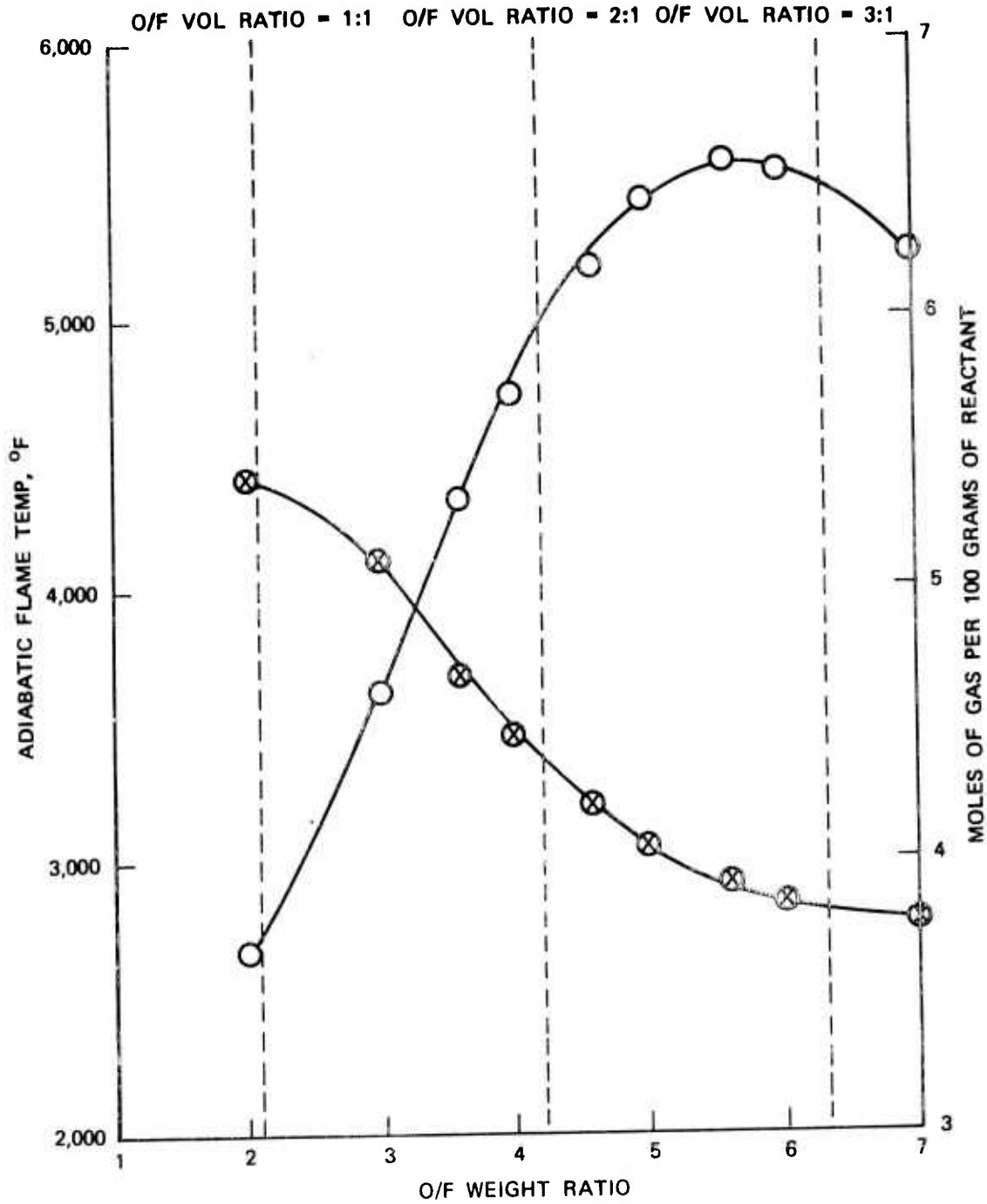
FIGURE 12. Thermodynamic Equilibrium Calculation of Adiabatic Flame Temperature and Moles of Gas for Combustion of n-Octane With Nitric Acid/Water (93/7% by weight) at 33,000 psi.
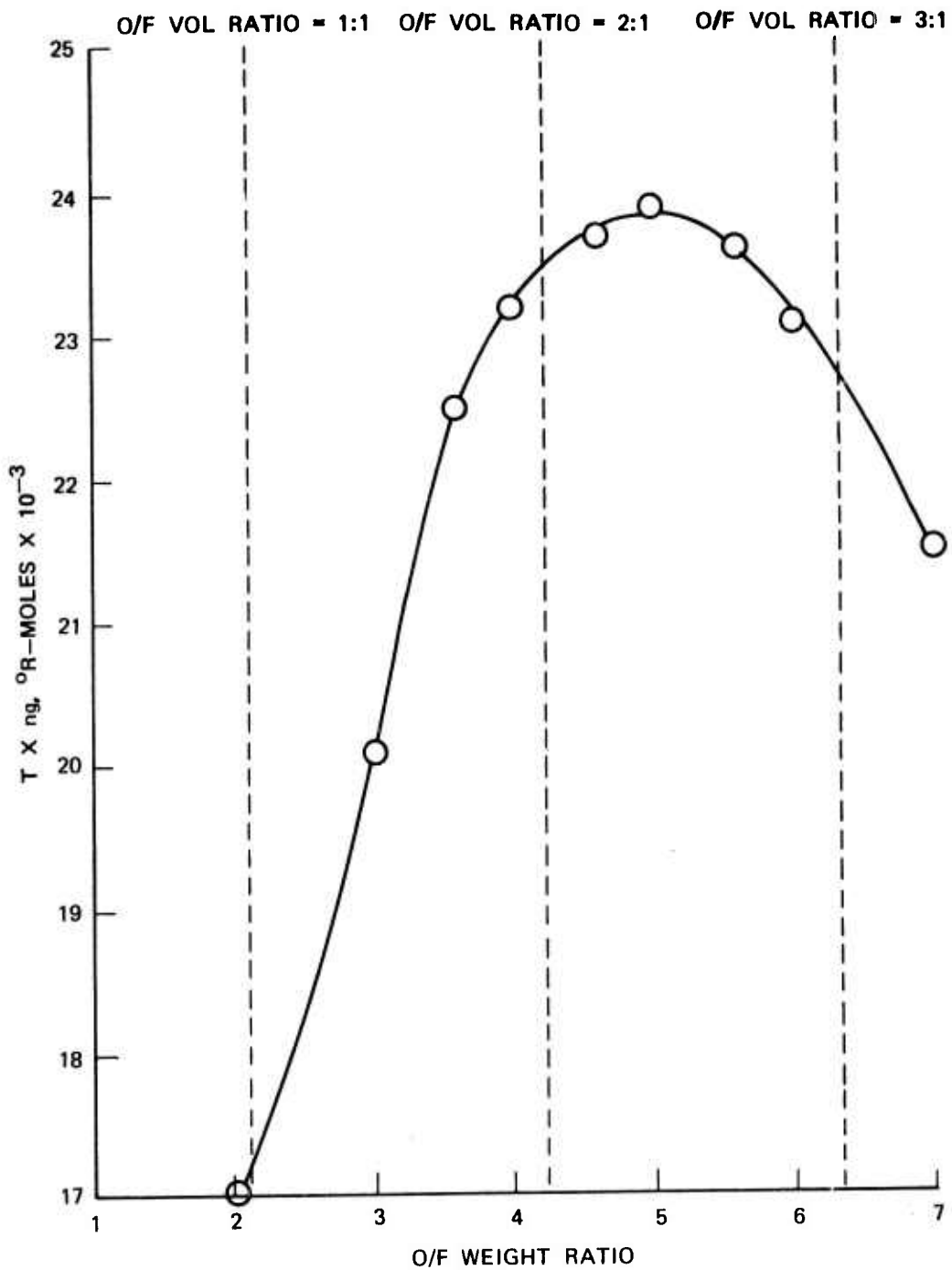
FIGURE 13. T x n_g Versus O/F Ratio for Combustion of n-Octane With Nitric Acid/Water (93/7% by weight) at 33,000 psi.
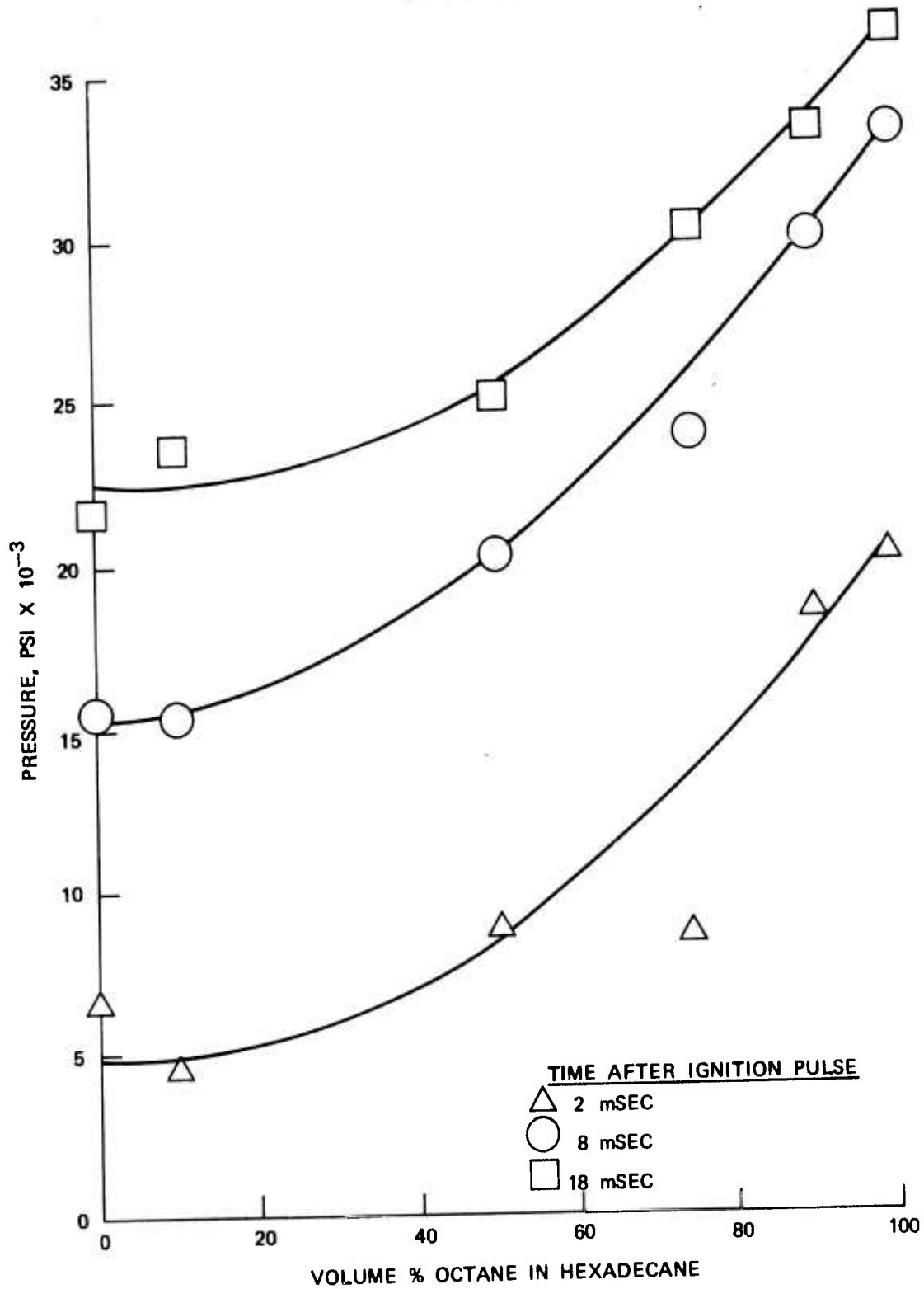
FIGURE 14. Dependence of Combustion Pressure on Composition of Octane/ Hexadecane Mixture. (The pressures plotted for 18 msec were the maximums obtained within the duration of the scope trace, 18 msec after the impact pulse. Some of the maxima occurred at less than 18 msec.)
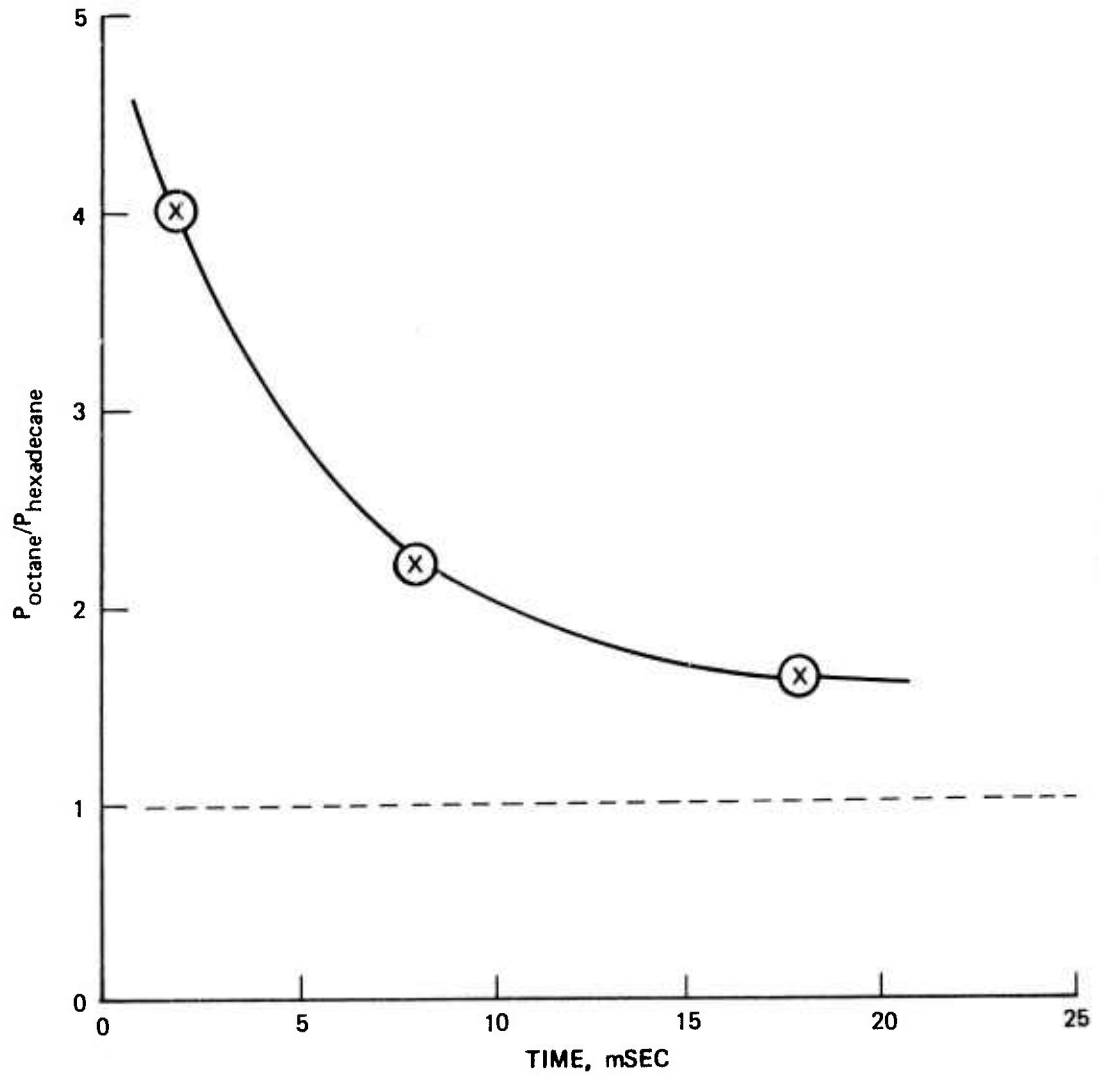
FIGURE 15. Ratio of Octane Combustion Pressure to Hexadecane Combustion Pressure as a Function of Combustion Time.
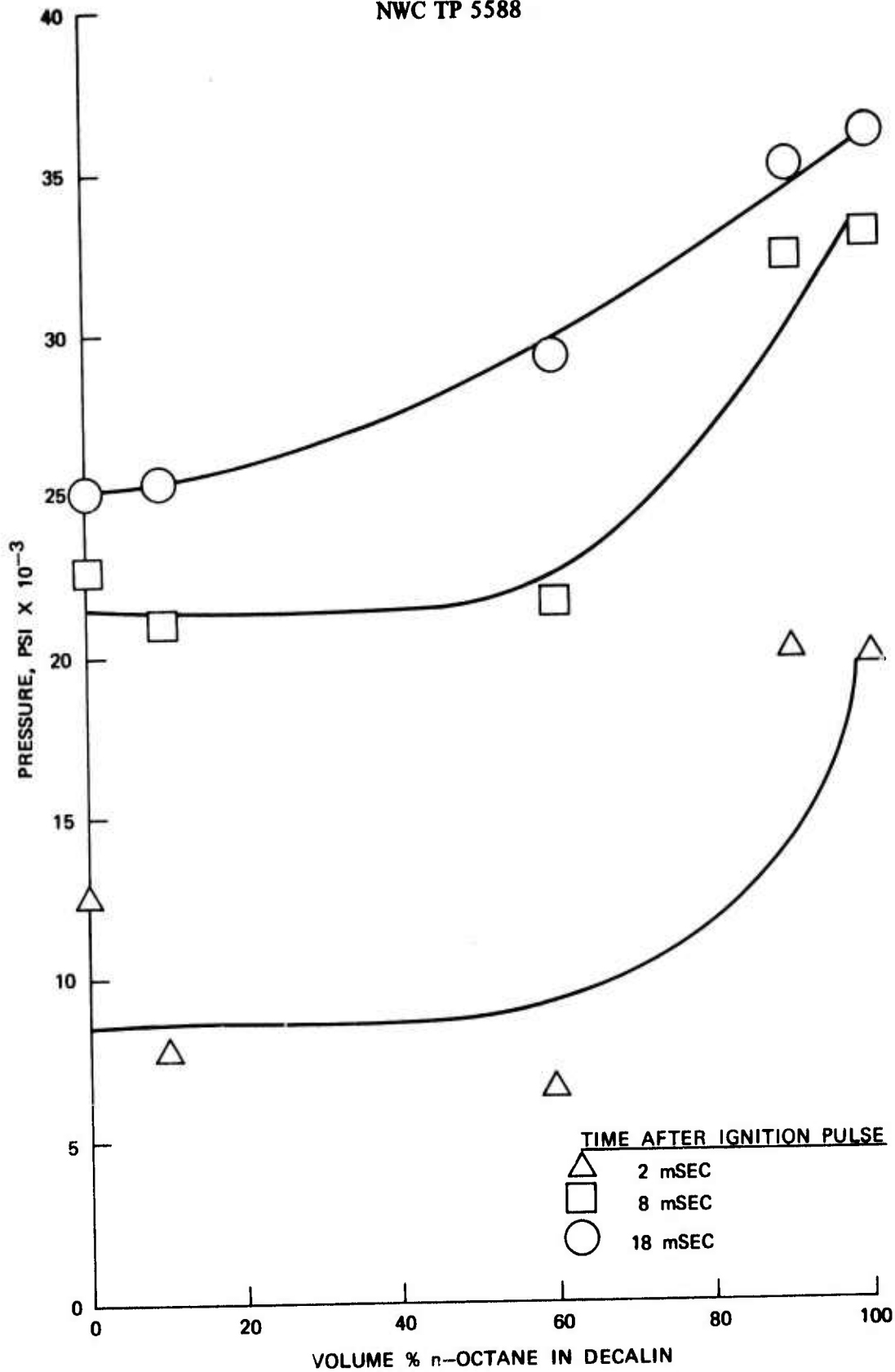
FIGURE 16. Dependence of Combustion Pressure on Composition of Octane/Decalin Mixture.
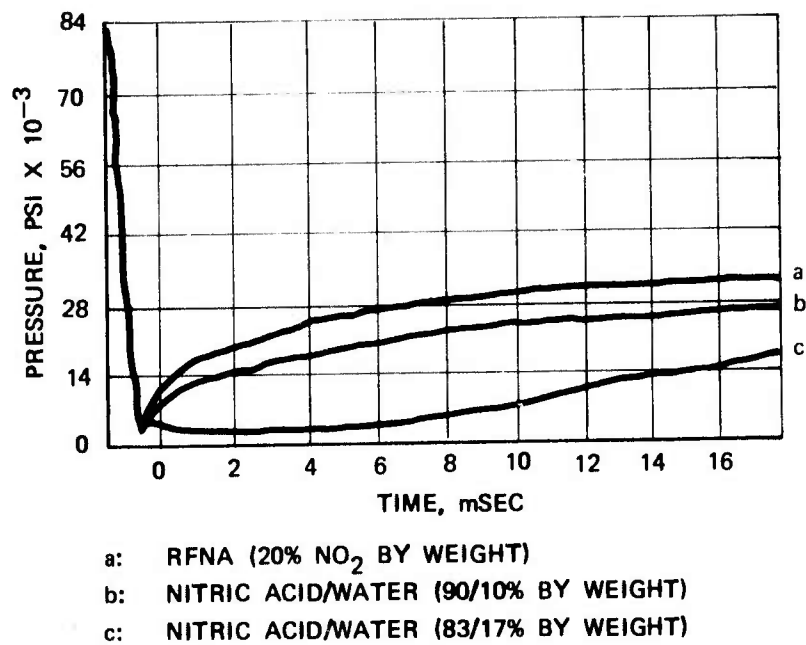
a: RFNA (20% NO_2 BY WEIGHT)
b: NITRIC ACID/WATER (90/10% BY WEIGHT)
c: NITRIC ACID/WATER (83/17% BY WEIGHT)

FIGURE 17. Pressure-Time Curves for Combustion of TH-Dimer With Various Concentrations of Nitric Acid.

TABLE 1. Fifty Percent Ignition Points of Hydrocarbons with Nitric Acid/Water (90/10% by weight) (O/F volume ratio = 2:1).

Hydrocarbon	50% ignition point
n-pentane	>150 kg-cm
n-octane	33 kg-cm
1,2,3-trimethylcyclohexane	33 kg-cm
2,6,11-trimethyldodecane	28 kg-cm
n-propyl nitrate	9 kg-cm*

* Technoproducts data for standard test at 70°F.

TABLE 2. Selected Properties of Some Hydrocarbon Fuels.

Compound	Formula	Mol. wt., g/mole	Density at 20°C, g/cc	Melting point, °C	Boiling point, °C
n-hexane	C_6H_{14}	86.18	0.65937	-95	68
n-heptane	C_7H_{16}	100.21	0.68376	-91	98.42
n-octane	C_8H_{18}	114.23	0.7025	-56.5	125-6
n-decane	$C_{10}H_{22}$	142.29	0.7300	-29.7	174.1
n-dodecane	$C_{12}H_{26}$	170.34	0.7487	- 9.6	216.3
n-hexadecane	$C_{16}H_{34}$	266.45	0.77331	18	287
cis-Decalin	$C_{10}H_{18}$	138.25	0.8967	-45.4	195
TH Dimer	$C_{12}H_{20}$	164.16	0.92	-40	- - -

TABLE 3. Reaction of Hydrocarbons with Nitric Acid.

Hydrocarbon	O/F Volume Ratio = 1:1		O/F Volume Ratio = 2:1		O/F Volume Ratio = 4:1	
	Pressure at 2 msec, psi	Maximum pressure, psi	Pressure at 2 msec, psi	Maximum pressure, psi	Pressure at 2 msec, psi	Maximum pressure, psi
n-pentane	· · ·	· · ·	· · ·	· · ·	· · ·	· · ·
n-hexane[a]	10,110	37,680	· · ·	· · ·	· · ·	· · ·
n-heptane	16,030±3,980	27,520±2,126	17,600±2,000	32,700±3,100	14,600±3,300	29,000±4,400
n-octane	19,150±1,160	30,730±2,840	20,200±900	36,400±1,400	13,600±3,300	26,800±3,500
n-decane[b]	23,000±3,080	33,750±1,150	17,100±200	29,200±1,700	13,820±3,500	29,900±3,200
n-dodecane	· · ·	· · ·	7,500±1,700	24,600±1,400	10,200±2,400	24,200±2,700
n-hexadecane	· · ·	· · ·	6,600±3,200	21,800±7,500	8,700±4,700	27,600±3,300
TH Dimer	12,100±1,400	23,100±1,300	13,200±500	24,300±400	6,900±2,500	16,800±1,800
cis-Decalin	10,700±1,800	25,300±1,500	12,800±470	25,200±1,500	6,180±1,100	22,200±3,900

[a] Out of many attempts only one ignition was obtained.

[b] The burning rate of decane was sometimes lower than reported here, corresponding more to the values shown for dodecane and hexadecane. On any given day the results were the same, but they varied from day to day, presumably because of the poor ambient temperature control in the laboratory. This phenomenon was not observed with the other fuels.

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