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INVESTIGATION OF DECOMPOSITION CHARACTERISTICS OF GASEOUS AND LIQUID NITROUS OXIDE

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Air Force Weapons Laboratory
Kirtland Air Force Base, New Mexico

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This report is a compilation of data obtained from Rocketdyne Division and Pratt & Whitney Aircraft Division. The Rocketdyne study examined liquid nitrous oxide stability with contaminant materials ranging from metal oxides to polymers at a temperature just below the critical point. Based on gas chromatographic analysis, negligible decomposition of the N_2O occurred in all cases. One material in these tests which showed degradation was polymer (AF-E-124X) developed for use with N_2O_4. In general, it is concluded that N_2O stability to catalytic and chemical decomposition is

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greatly increased in the liquid versus the gaseous state. The Pratt & Whitney effort concentrated on the decomposition characteristics of both gaseous and liquid nitrous oxide. The experimental program evaluated the ignition, flame speeds of induced reactions, and quenching characteristics of the decomposition reaction. The results demonstrate that gaseous N_2O can be ignited either by rapid compression or electric spark at elevated pressures. All attempts to initiate the decomposition by an exploding wire at 60°F proved negative, but it was shown that a minor reaction can be started by a No. 6 blasting cap inside the contained, high-pressure liquid. The results indicate that nitrous oxide may safely be handled under extreme conditions in the cooled liquid state but that great hazards exist in the gaseous condition at elevated pressure and/or temperature.

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This report was prepared for the Air Force Weapons Laboratory. Captain James K. Cawthra (LRL) was the Laboratory Project Officer-in-Charge.

This technical report has been reviewed and is approved for publication.

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PREFACE

The author extends special thanks to G. D. Lewis and P. L. Russell of Pratt and Whitney Aircraft Division and J. V. Lecce and R. W. Melvold of Rocketdyne Division for their contribution to this report.

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SECTION I

INTRODUCTION

The desirability of using nitrous oxide (N_2O) in chemical reactors has prompted an investigation into its safety and handling characteristics. At least one instance of an unexpected and violent decomposition reaction has pointed out the need for additional information defining the conditions under which N_2O can be decomposed. Therefore, the Air Force Weapons Laboratory (AFWL) sponsored an investigation to determine these conditions. That investigation is the subject of this report.

N_2O is widely used as a general anesthetic, mostly in dentistry, and is usually classed as a safe, nontoxic, nonflammable fluid. Its critical temperature is 97.7°F, and the critical pressure is 1050 psia. It is conventionally stored and shipped as an ambient temperature liquid under its own vapor pressure of about 750 psia. Its normal boiling point is -129°F. N_2O has a negative heat of formation of about 800 BTU per pound which it released upon decomposition to produce 1 mole of nitrogen and 1/2 mole of oxygen for each mole of nitrous oxide. If the nitrous oxide was initially at 80°F, the mixed nitrogen and oxygen products of decomposition will have a temperature of 3800°F if the reaction proceeded at constant volume or 3000°F if it proceeded at constant pressure. Because of this large energy release, approximately equal to that produced by the combustion of a stoichiometric kerosene-air mixture and about double that produced by exploding an equal weight of TNT, it is important to know how much energy it takes to ignite the decomposition and under what conditions it will propagate. The experimental program to obtain that information is described in the following sections.

SECTION II

PROGRAM PHASES

The test program was divided into three phases: (1) the ignitability of gaseous N_2O, (2) the ignitability of liquid N_2O, (3) the materials compatability of liquid N_2O.

The test apparatus is shown schematically in figure 1. It consists of a nitrogen purge system, two nitrous oxide supply systems to provide a range of filling-flow rates, and a set of reaction tubes, each containing two pressure transducers and a thermocouple at one end. An igniter was installed in each tube at the end opposite the instrumentation. Tubes of 1/2-inch, 1-inch, and 2-inch diameters were tested and during the last day of testing, a brief series of tests was conducted with a 6-inch diameter pipe 10 inches long to test a quenching correlation that had been developed with the smaller size pipes. Electrical ignition was provided by either (1) a variable energy, high-voltage supply firing a spark plug at values up to 7.3 joules or (2) by exploding a small coil of 0.020-inch diameter steel wire welded across spark plug electrodes by feeding it with current from a 300-ampere welder. In neither case was the energy at the ignition point measured. The quoted values for the spark plug are based on the voltage and capacitance of the variable energy supply. No value was calculated for the exploding wire, but in appearance it was a much more powerful ignition source than the 7.3-joule spark. A rough estimate of its value is 20 joules.

The temperature of the pipe could be increased above ambient by supplying electrical energy to a heating coil wrapped around it. In some cases, the pipe was chilled by packing it in dry ice. In all cases the N_2O supply was at ambient temperature, about 90°F, and the supply pressure was limited to the vapor pressure in the storage bottles. In heated or cooled tests, the ambient temperature N_2O was admitted to the test pipe and allowed to rest until it reached the pipe wall temperature. In the one high-pressure test conducted, the pipe was chilled to condense liquid N_2O in it and then heated to vaporize the liquid and expand it to the desired pressure.

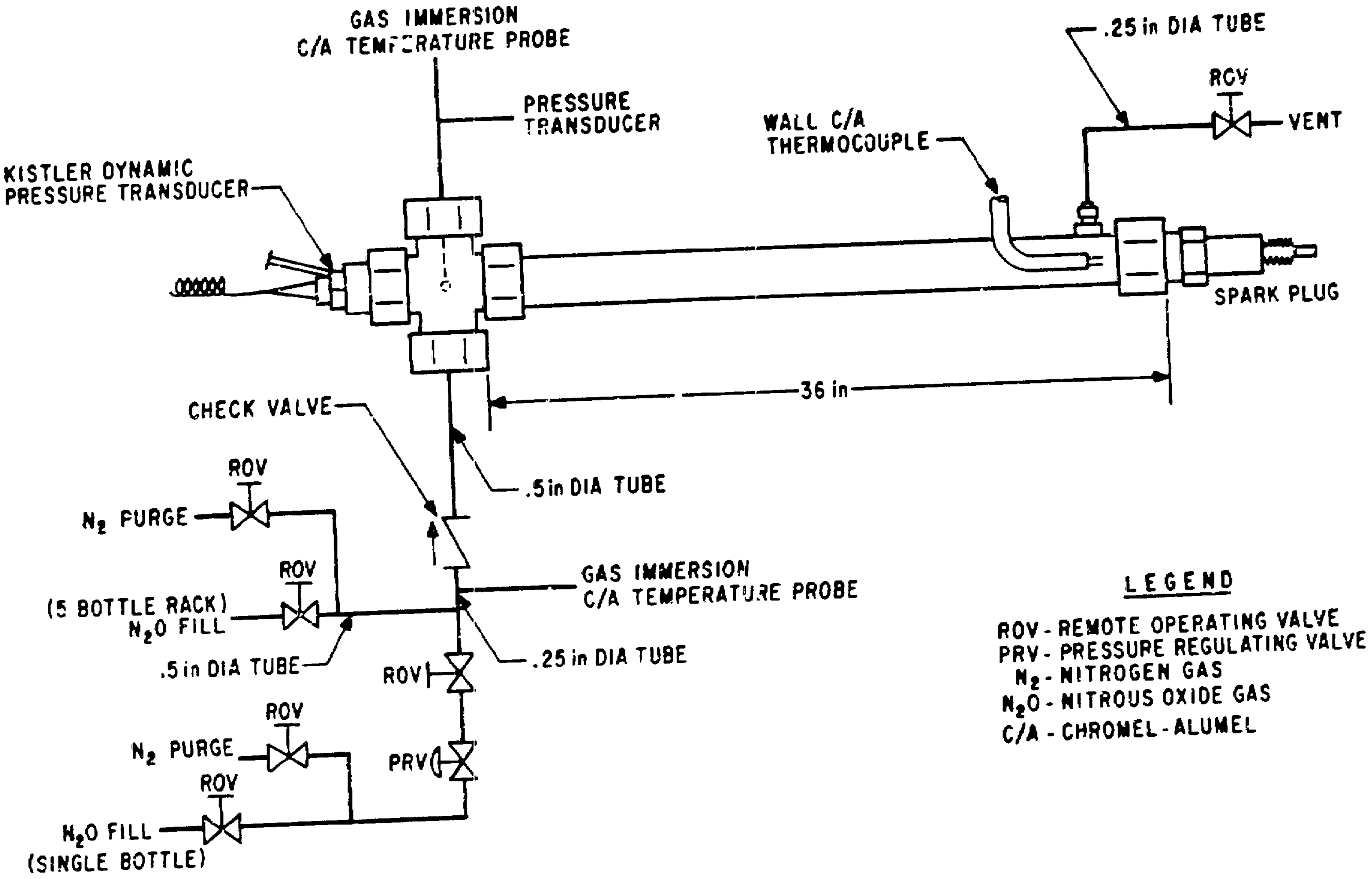

Figure 1. Test Apparatus

Pressure and temperature were recorded on an oscillograph. Although both a high-frequency Kistler pressure transducer and an oscillograph paper speed of 100 inches per second were provided at the start of the program, the decomposition reactions occurred so slowly that most measurements were made with a strain-gage-type transducer at an oscillograph speed of 4 inches per second. Temperature of the N_2O at the end of the pipe opposite the igniter was also recorded on the oscillograph.

SECTION III

TEST PROCEDURE, OBJECTIVES, AND RESULTS

1. TEST PROCEDURE

A test was conducted by first purging the test apparatus with nitrogen and then purging it with gaseous nitrous oxide. After the purge was completed, the pipe was filled with N_2O to the desired pressure and heated or cooled to the desired temperature. The oscillograph was then turned on and the igniter fired. Both the temperature and pressure before and after the test were recorded by hand so that decomposition efficiencies could be calculated. In many cases leaks in the system or an extremely low pressure rise prohibited reasonable determination of the combustion efficiency. After the data were recorded, the pressure was vented and the system purged with gaseous N_2O in preparation for the next test.

2. TEST OBJECTIVES

The test program had three primary objectives. These were (1) to measure the flame propagation rates, (2) to measure the energy required to start the reaction, and (3) to evaluate quenching effects.

a. Flame Propagation

Two different rates of flame propagation can be defined. The first is the observed speed of the reaction moving through the pipe (speed relative to the pipe), and the second is the speed of the reaction moving through the cold N_2O gas (speed relative to the cold gas). Because the volume of the decomposed gas is much greater than the volume of the N_2O that produced it (because of heat release, increased number of moles, and increased compressibility factor), the cold N_2O is pushed down the pipe ahead of the decomposition flamefront. For an ideal situation with a plane flamefront, 100 percent efficiency, and no heat loss, the observed propagation rate is about 10 times greater than the speed of the flame burning into the cold gas.

Flame propagation rate was measured by two methods. In the first, the distance between the igniter and the thermocouple was divided by the time required for the flame to travel that distance as indicated by the elapsed time between igniter firing and flame temperature indication at the thermocouple.

Figure 2 is a tracing of a typical oscillograph record obtained with a 2-inch diameter pipe. The time required for the flame to travel the 94 cm between igniter and thermocouple is taken as the time between the ignition signal (electrical induction from the spark current surge) and the abrupt rise in temperature. In this case the elapsed time was 1.2 seconds, and the observed flame propagation rate was 78 cm per second.

In the second method, both the rates of flame propagation through the pipe and through the cold mixture were calculated from the slope of the pressure versus time recording on the oscillograph. In some cases, usually where the reaction was vigorous and the propagation rate was high, it was impossible to pick a reliable slope near the beginning of the reaction. In other cases, however, ususally near flame propagation limits where the reaction was slower, the slope could be measured quite reliably. Figure 3 is a tracing of an oscillograph recording obtained with a 1-inch diameter pipe. In this test, the slope and the flame time of transit could be measured quite reliably.

b. Ignition Energy

Although measurement of ignition energy was a primary objective at the beginning of the program, it soon became apparent that very small amounts of energy would ignite the decomposition reaction and that the possibility of a violent reaction was far more closely coupled with the quenching characteristics of the N_2O than with its minimum ignition energy. In the area of auto-ignition by rapid compression, however, the energy required to ignite the mixture is of practical importance and this was investigated experimentally.

c. Quenching

The governing parameter controlling whether a decomposition flame would propagate through N_2O gas and even whether it could be ignited at all turned out to be quenching by heat loss to the metal pipe or, in compression ignitions while filling the pipe, to the colder inflowing N_2O gas. Quenching characteristics were evaluated analytically based on the observed quenching limits.

3. TEST RESULTS

The test results are grouped into the same three categories as the test objectives, i.e., flame propagation rate, ignition, and quenching.

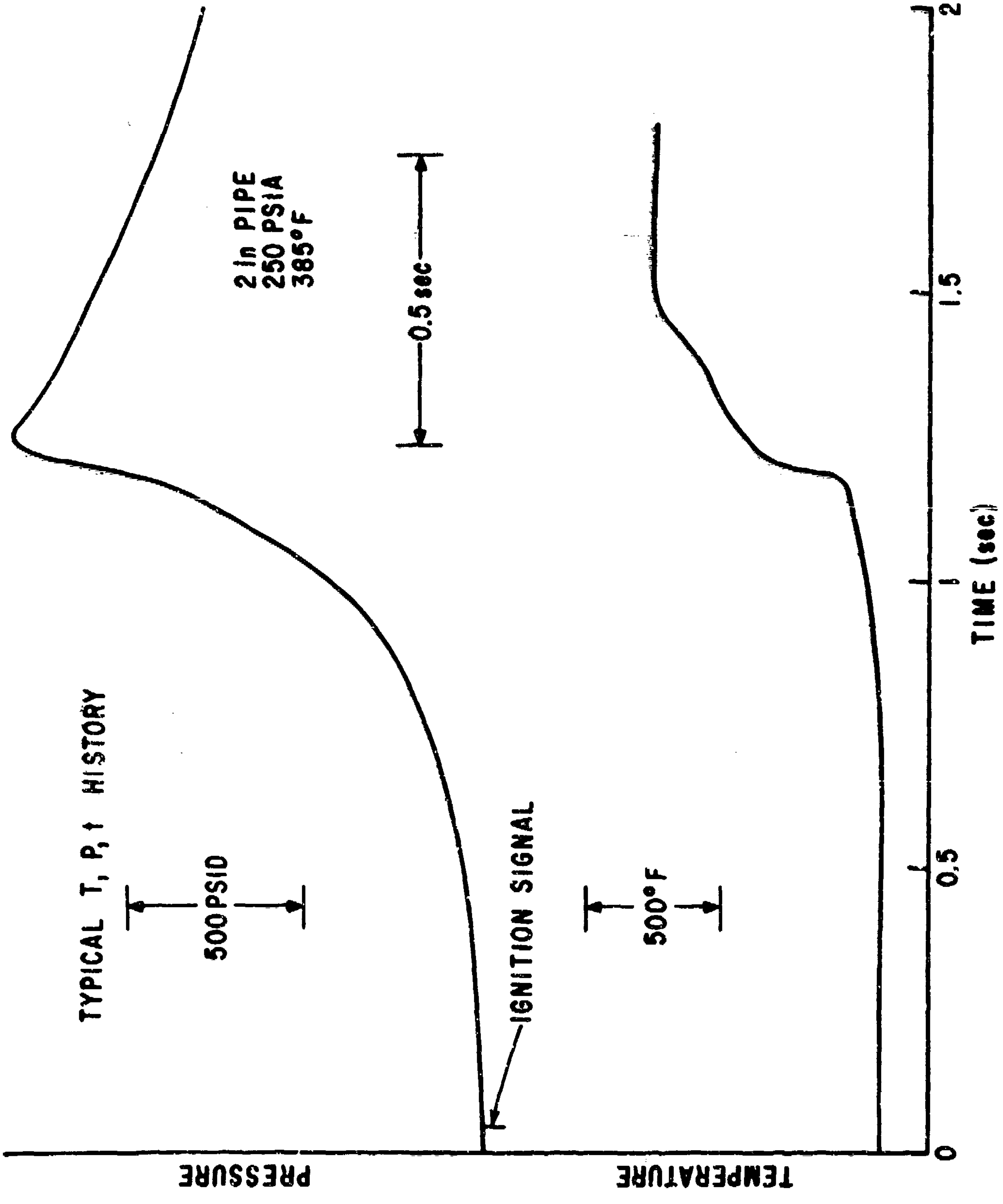

Figure 2. Oscillograph Tracing - 2-Inch Diameter Pipe

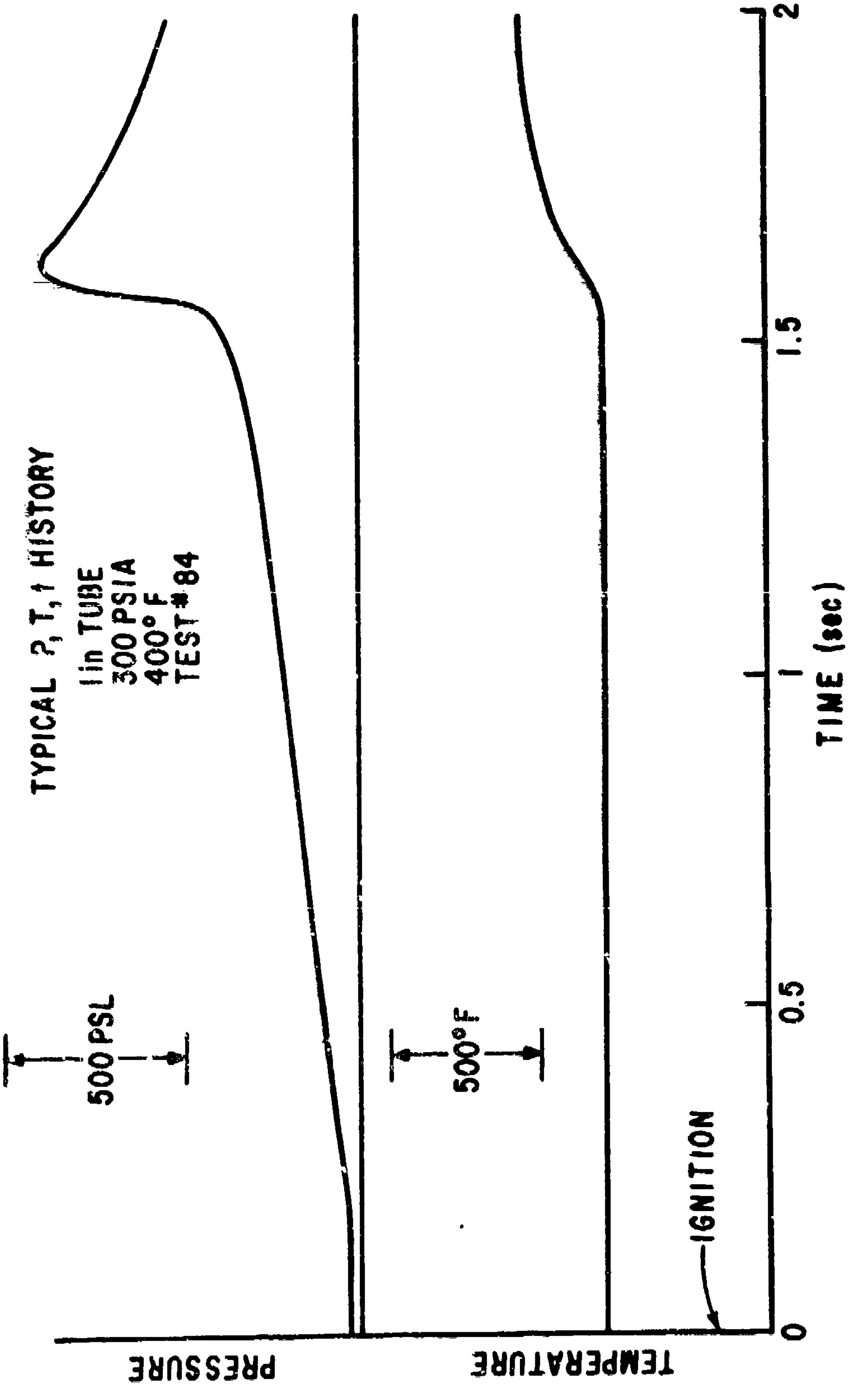

Figure 3. Oscillograph Tracing - 1-Inch Diameter Pipe

a. Flame Propagation

Flame propagation rate measurements were made in 2-inch diameter, 1-inch diameter, and 1/2-inch diameter pipes at temperatures ranging from ambient (90°F) up to 420°F. The data for the 2-inch diameter pipe are plotted in figure 4. The data fall naturally into two groups, those at ambient temperature and those at elevated temperatures. As the data show, increasing the temperature has a significant effect on flame propagation rate. Even the highest value measured, however, is about 30 times slower than typical values measured for propane-air mixtures at ambient pressure and temperature, even at a mixture ratio producing abou the same flame temperature as the N_2O decomposition. This great differenc is probably the result of the N_2O decomposition progressing purely as a thermal process, whereas the propane-air combustion is a combination of thermal and chain carrier processes. No tests were conducted in the 2-inch diameter pipes at higher pressures because of potential damage to the apparatus. The threshold value for flame propagation in the 2-inch diameter pipe at ambient temperature was 250 psia. At 240 psia the flame would never propagate, and at 250 psia it would propagate every time.

Decomposition efficiency was calculated for points where data were available, i.e., significant pressure rise and no leaks. The data are plotted in figure 5 as a function of temperature. The correlation is poor, probably as a result of trying to read small differences between large pressures. The data showed no correlation with any parameter other than temperature. The relatively low efficiencies most likely result from the strong quenching action of the pipe walls, especially at the lower temperatures.

The true flamespeed of the reaction through the cold N_2O gas was also calculated for tests where the slope of the pressure rise curve could be measured reasonably well. These results are presented in figure 6 as a function of pressure. The lowest values of 3 cm per second are much lower than the lowest values that have been observed in hydrocarbon-air combustion systems.

Similar results were obtained in the 1-inch diameter pipe as shown in figure 7. Again the temperature had a strong effect on flamespeed, and pressure had a lesser but still substantial effect. The minimum pressure at which decomposition could be sustained at ambient temperature was 410 psia. Similar minimum pressure limits of 200 psia and 130 psia were obtained at temperatures of 280°F and 410°F, respectively. In one test the pipe was

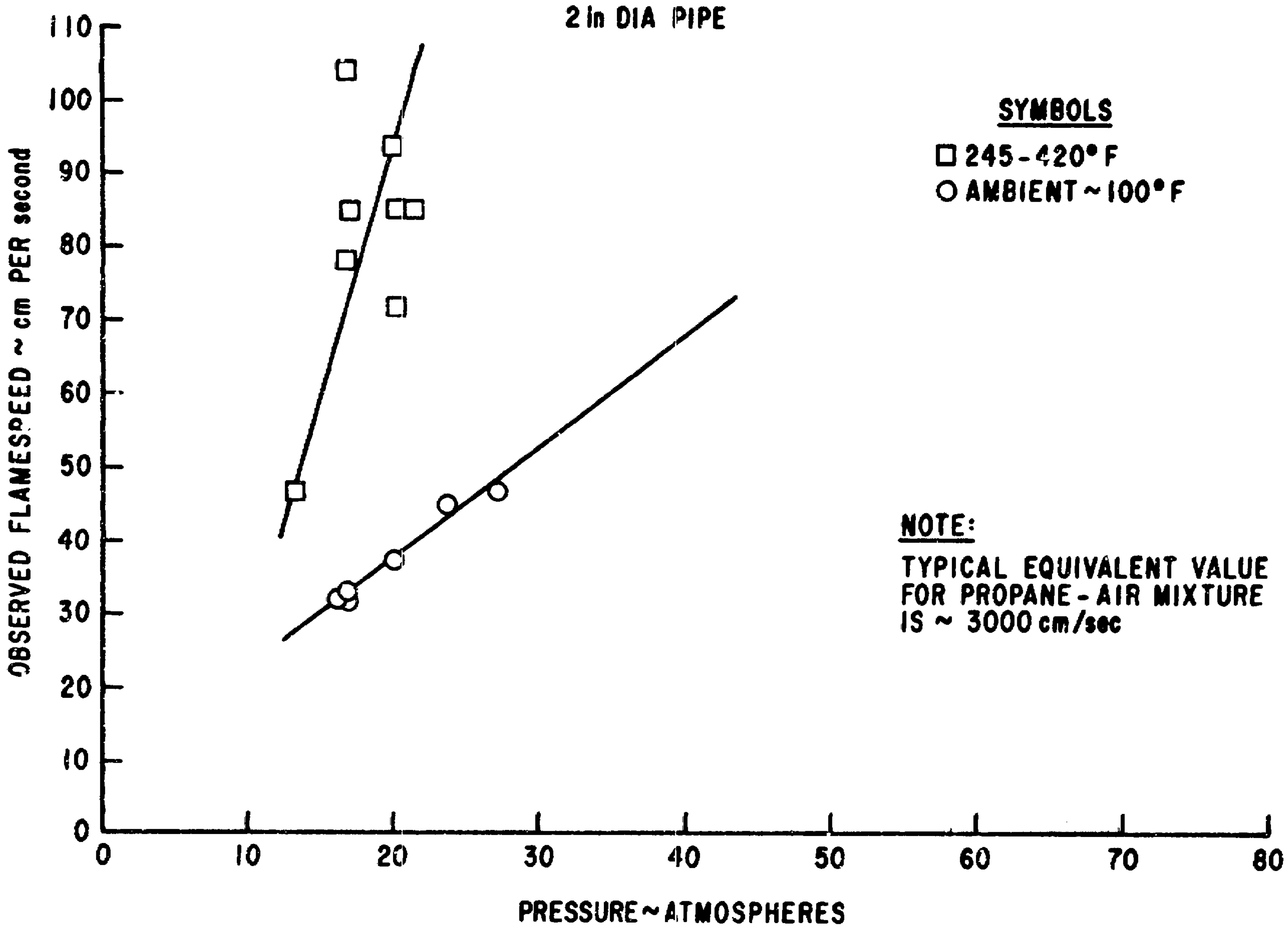

Figure 4. Flame Propagation Rates - 2-Inch Pipe

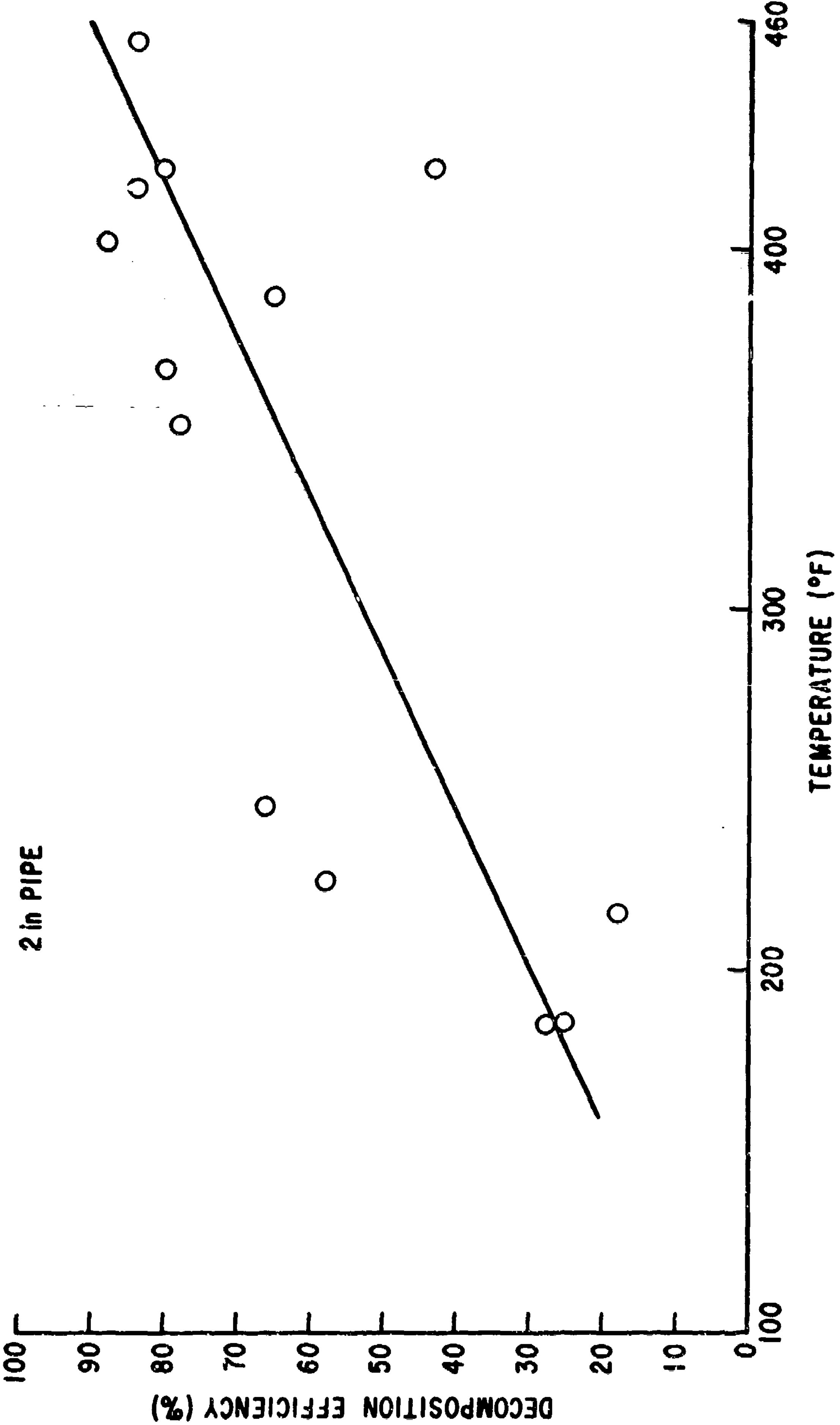

Figure 5. Decomposition Efficiency - 2-Inch Pipe

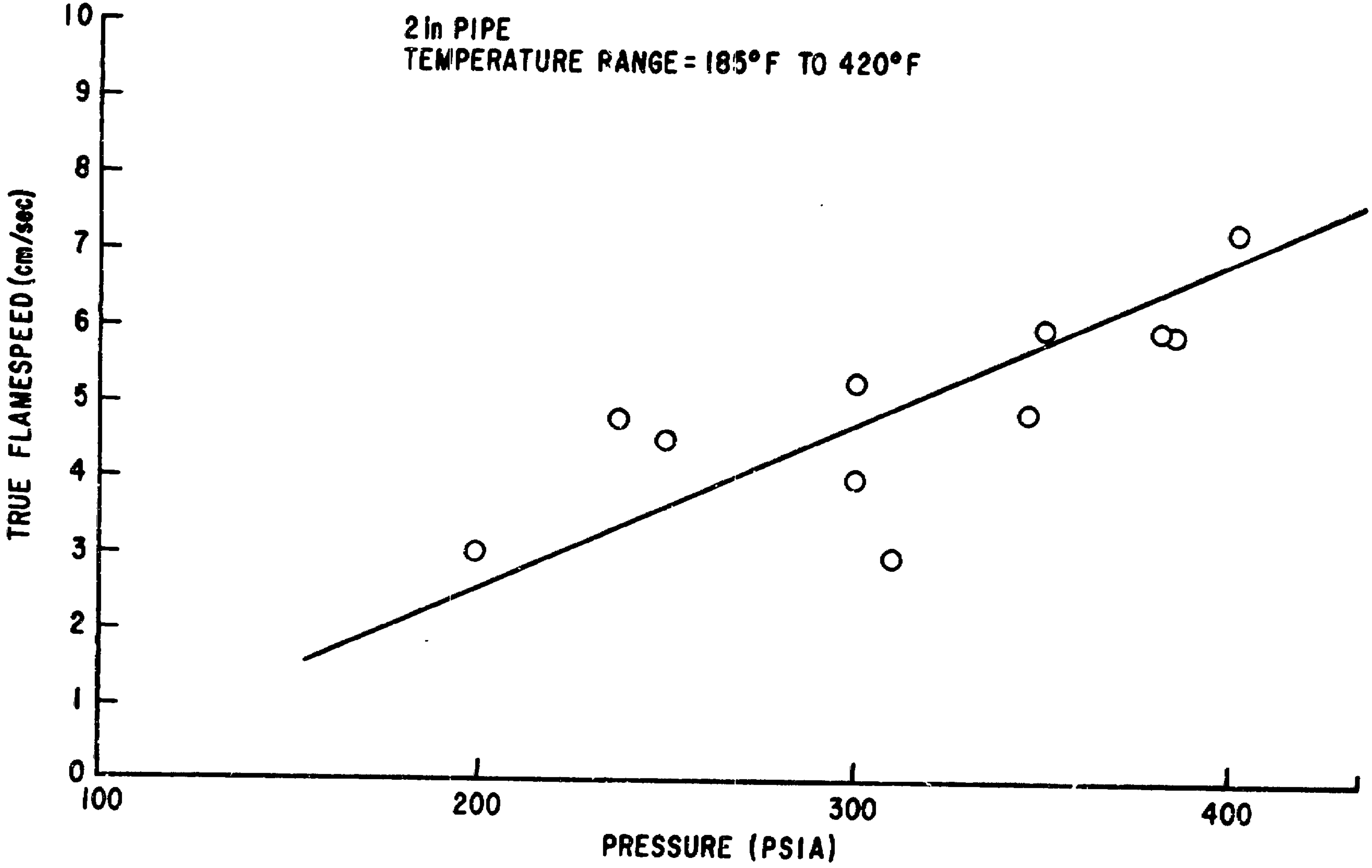

Figure 6. Flamespeed as Function of Temperature - 2-Inch Pipe

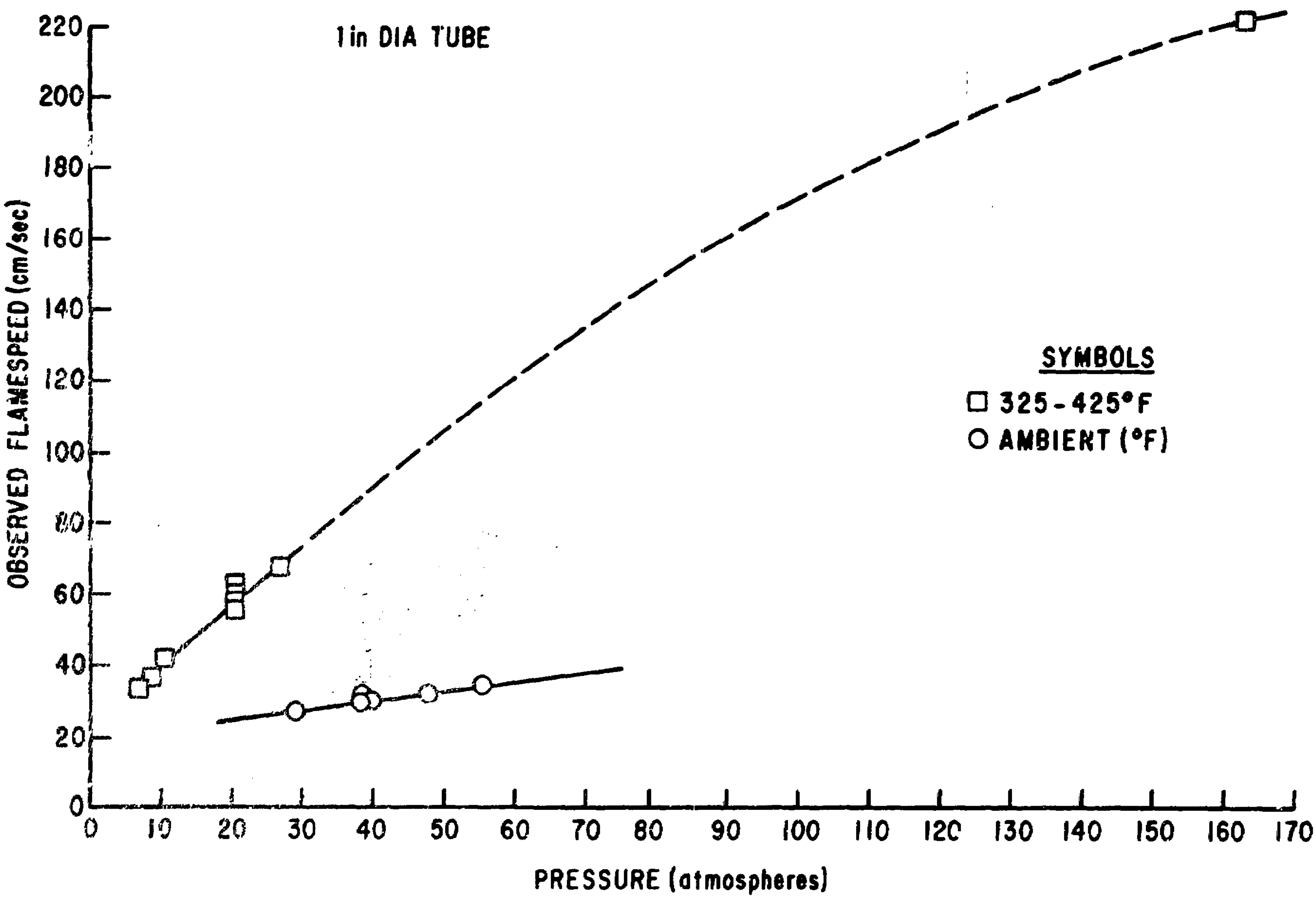

Figure 7. Flamespeed as Function of Temperature - 1-Inch Pipe

chilled to condense N_2O gas to a liquid, the inlet valve was closed, and the pipe was heated to produce preignition conditions of 2450 psia and 325°F. In this test, flame was relatively fast for N_2O, and the pipe burst near the midpoint from overpressure. The theoretical breaking strength of the pipe was 10,000 psid. The flamespeed as determined from the slope of the pressure rise curve was 222 cm per second which is a reasonable extrapolation of the data obtained at lower pressures. A tracing of the oscillograph recording for that test is presented in figure 8. At the end of the trace the galvanometer was deflected so fast that the oscillograph trace did not record on the light-sensitive paper. It is probable that the remaining N_2O in the pipe detonated at this point.

It was very difficult to ignite the N_2O in the 1/2-inch diameter pipe, and no ignitions were obtained at ambient temperature at pressures up to 1000 psia. Only in two tests was decomposition sustained, one at 400°F and 800 psi, the low-pressure limit at that temperature, and the other at 1000 psi and 400°F. The measured flame speeds are presented in figure 9 and are about the same ord order of magnitude as the lowest values recorded for the other pipe sizes.

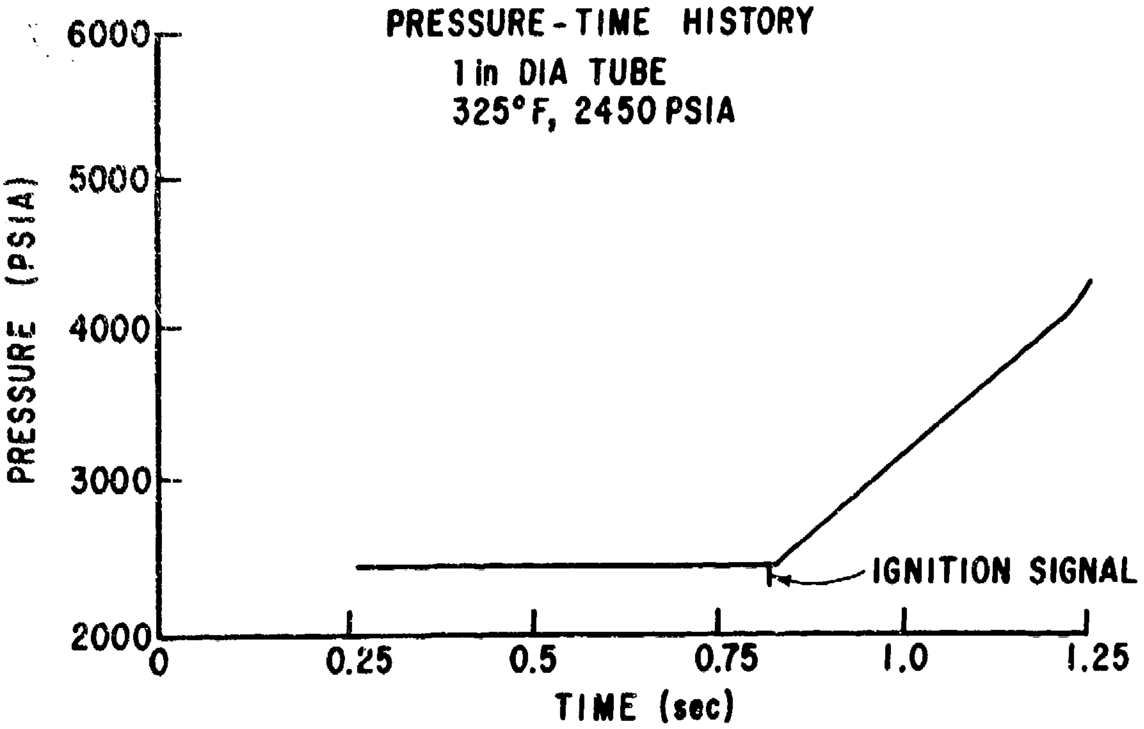

Figure 8. Pressure Time History - 1-Inch Pipe

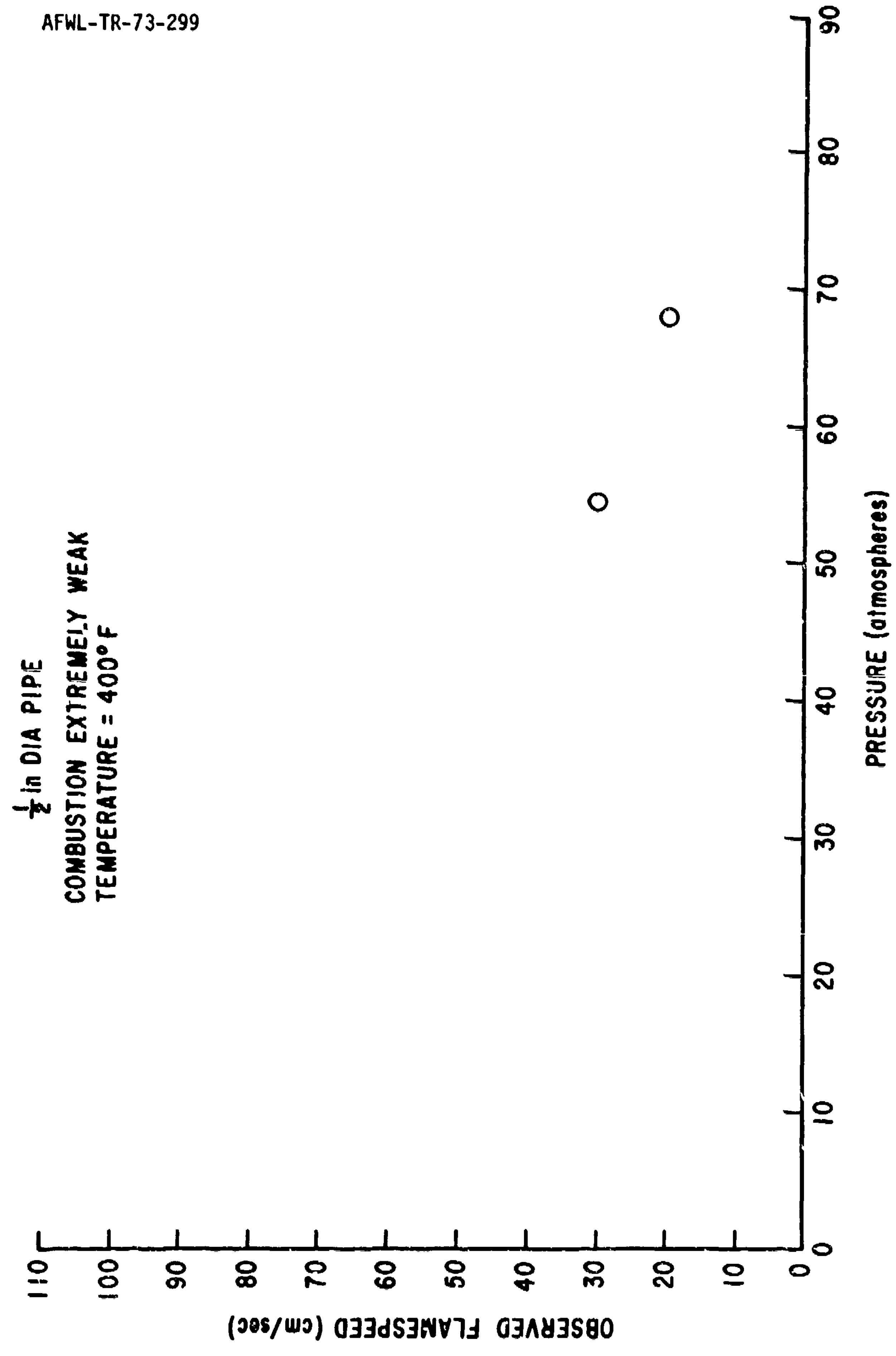

Figure 9. Observed Flamespeed - 1/2-Inch Pipe

b. Ignition Energy

The energy of the spark used to ignite the N_2O had no effect on the rate of flame propagation as shown in figure 10. No effort was made to pursue the determination of minimum electrical ignition energy further because the lowest value tested, 0.14 joule, is already so low that further refinement was not of practical interest. Instead, the effort was directed toward a better definition of autoignition by rapid compression.

Autoignition tests were conducted by preheating the pipe to the desired temperature and then suddenly opening the inlet valve to permit high-pressure (800 psig) N_2O at ambient pressure to flow into the test pipe until the pressures equalized and the flow stopped. Two different supply systems were used. One was fed through a 1/2-inch diameter tube from a bank of five N_2O bottles to produce a relatively high pressurization rate, and the other was fed through a 1/4-inch diameter tube and pressure regulator to provide slower pressurization rates. The results for both the 1-inch and 2-inch diameter tubes are presented in figure 11, where pressurization rate is plotted against pipe (not N_2O) temperature. No autoignitions were obtained in the 1-inch pipe. The data for the 2-inch diameter pipe show a trend of decreasing pressurization rate with increasing pressure. At 450°F pipe temperature, autoignition occurred at a pressurization rate of only 200 psi per second. This is extremely low compared to pressurization rates commonly used in pneumatic systems. Even at a pipe temperature of 50°F and ambient temperature, N_2O ignition occurred at a rate of only 1150 psi per second. Figure 12 shows a typical oscillograph tracing of an autoignition in the 2-inch diameter pipe. This figure shows an interesting characteristic that was observed in many of the autoignition tests. The pressure trace shows the relatively slow, linear increase in pressure with time as the N_2O flows into the pipe until autoignition occurs. At this point in time the pressure starts to rise more rapidly because of the combustion and takes an even greater slope as the fire reaches the opposite end of the tube where the thermocouple is located. After than, the pressure and temperature drop as the fire is apparently quenched by the cold N_2O gas still flowing into the pipe, and the pressure levels off at the bottle supply pressure. The pressure level at which the ignition occurs during the compression cycle is plotted as a function of pipe temperature in figure 13. It is interesting to note that if the gas reached pipe temperature before heating from isentropic

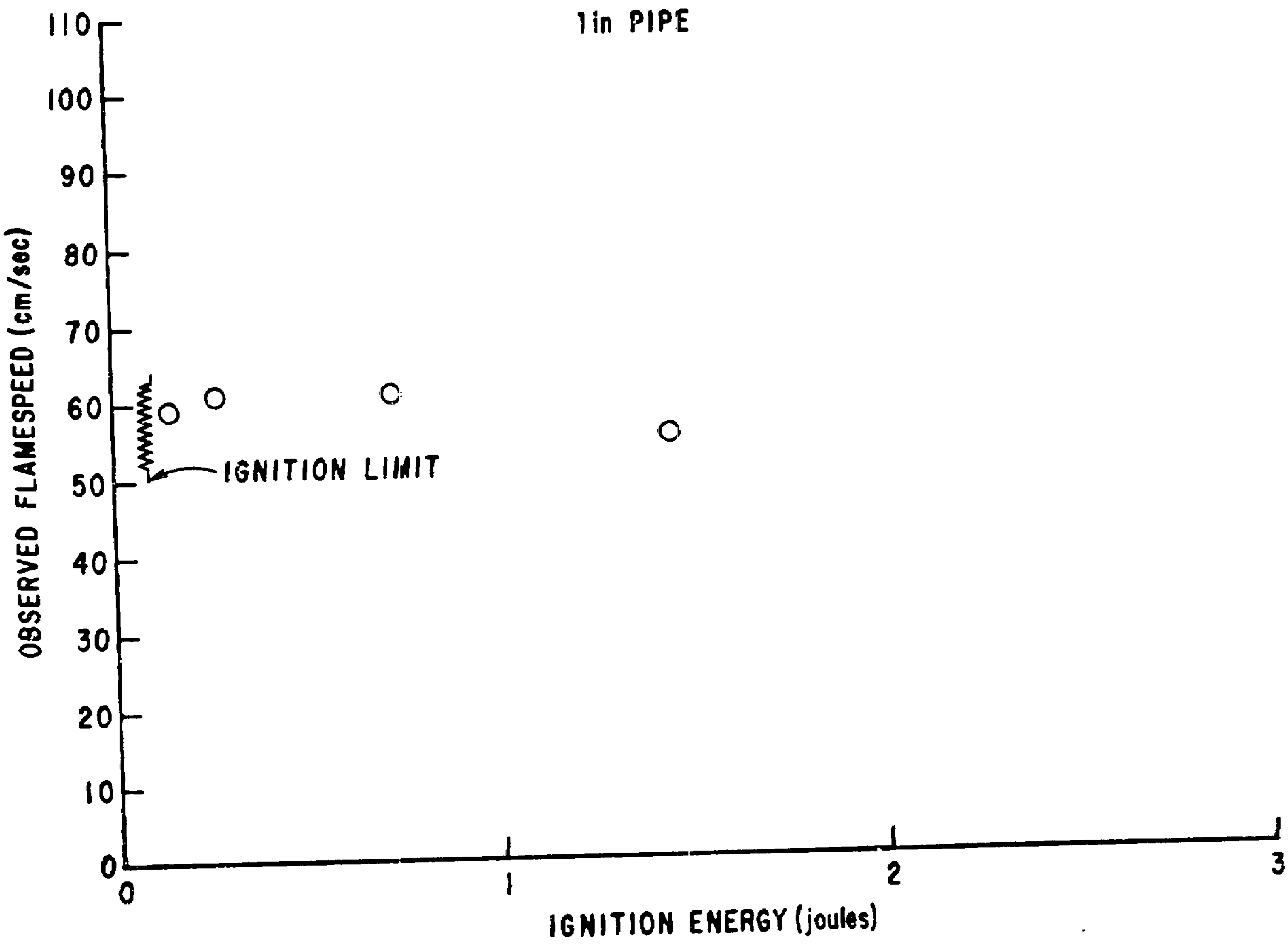

Figure 10. Observed Flamespeed - 1-Inch Pipe

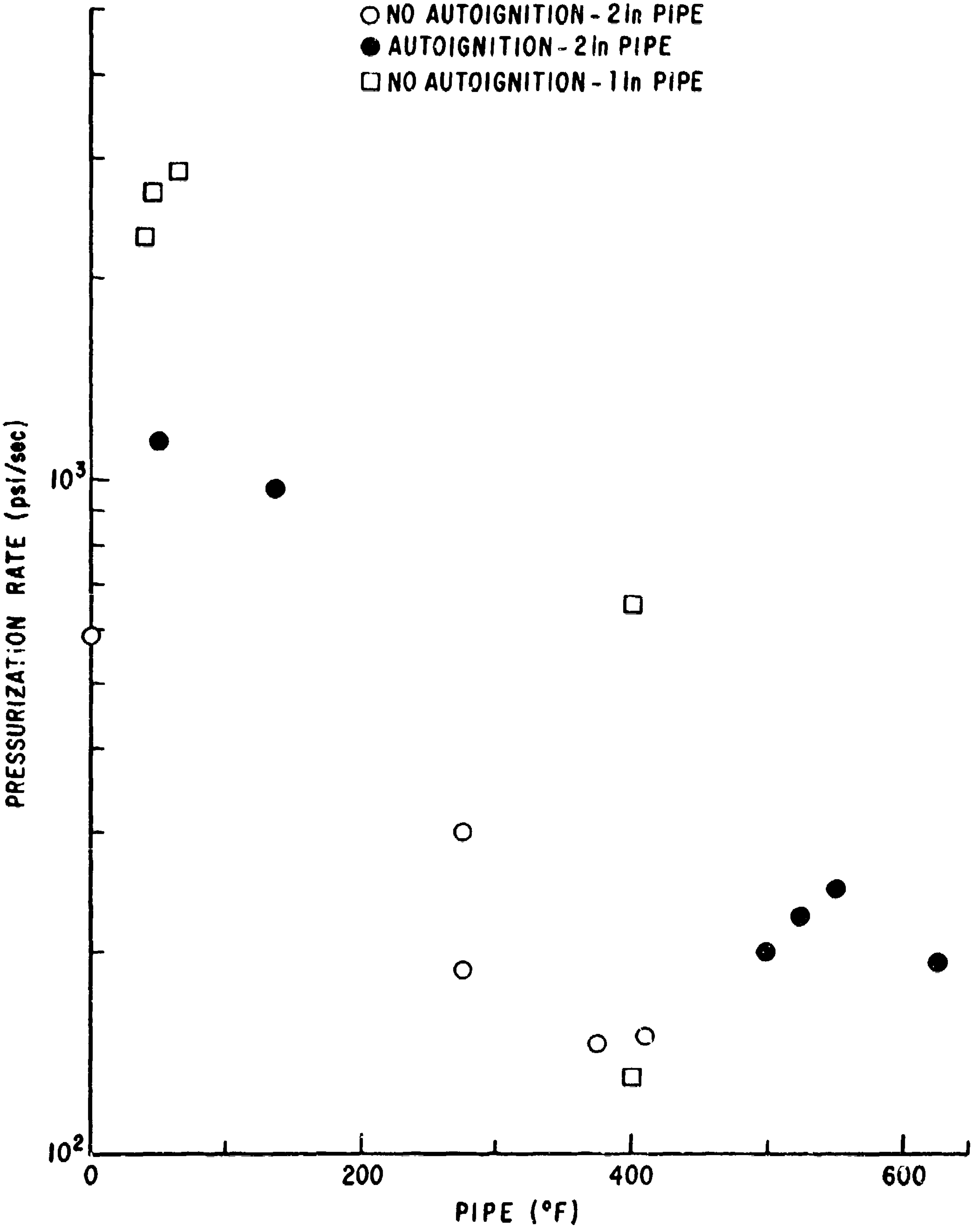

Figure 11. Autoignition - All Sizes

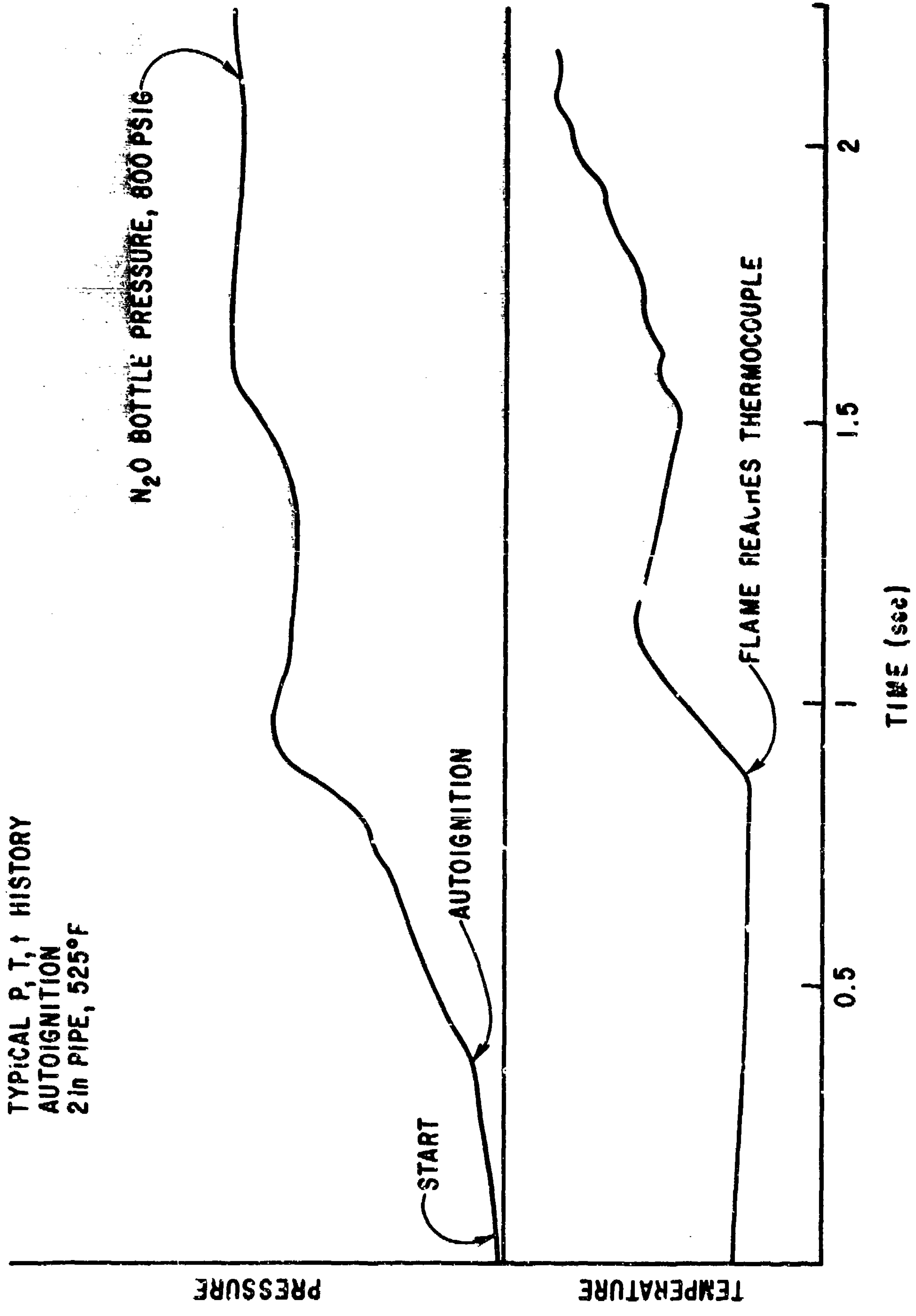

Figure 12. Oscillograph of Autoignition in 2-Inch Pipe

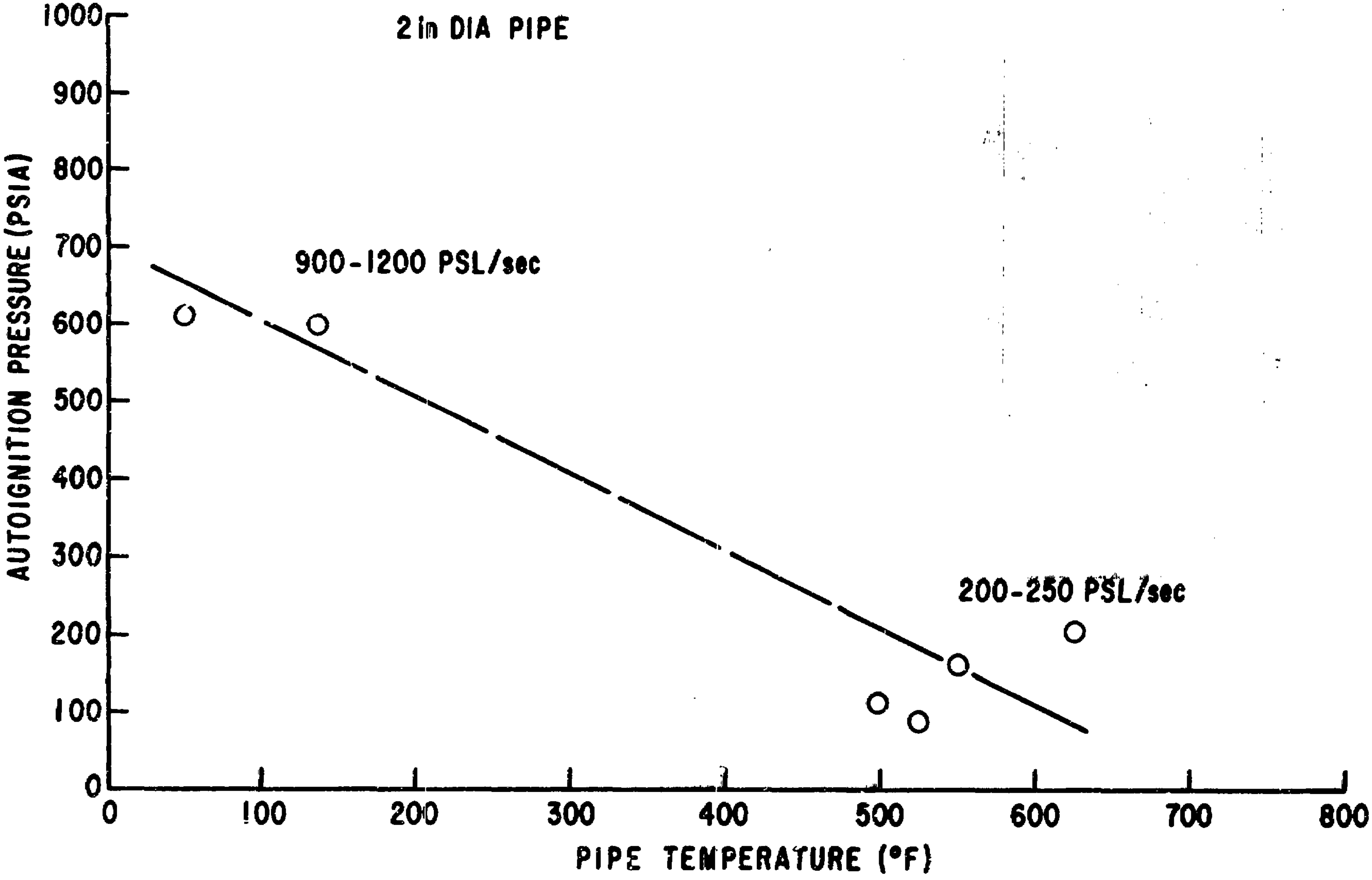

Figure 13. Autoignition Pressure versus Pipe Temperature

compression began, the maximum possible temperature at ignition is only 750°F for the datum point at 610 psia. This is remarkably low and emphasizes again that the decomposition is easy to ignite but also very easy to quench.

The ease of ignition is even more remarkable in light of the rate of heat loss from the compressed gas to the pipe. While the exact cooling rate is not calculable, data on the cooling of combustion products of a propane-air fire in a similar closed pipe showed temperature loss rates in the order of thousands of degrees per second. The results from another program (Edse-Ohio State) are presented in figure 14. Thus, the undoubtedly high cooling rate coupled with the low maximum theoretical temperature indicate that autoignition occurred somewhat below 750°F.

c. Quenching

As mentioned previously, quenching appeared to be the governing parameter in almost every test. A correlation was developed to express the quenching-controlled propagatiom limits in terms of pressure, temperature, and pipe size. The correlation has the form of equation (1)

$$1.1 \times 10^9 = (P)(T)^{2.5} \frac{(D)^{-2}}{(D - 0.38)} \qquad (1)$$

where

P is pressure in psia

T is temperature in °R

D is pipe internal diameter in inches

The correlation was based on the five limit data points previously mentioned; one from the 2-inch pipe, one from the 1/2-inch pipe, and three from the 1-inch pipe. Subsequent to the development of the correlation, additional experimental data obtained at Ohio State University (by Edse et al.) were located. These data fit the correlation well at temperatures above 250°F but disagree at lower temperatures (figure 15). Because the Ohio State data show that a fire will not propagate in a 1-1/2-inch diameter pipe at 100°F and 500 psi when P&WA data show it will propagate in a smaller 1-inch diameter pipe at the same temperature and only 400 psia, it must be concluded that the Ohio State data do not represent a real combustion limit in this range.

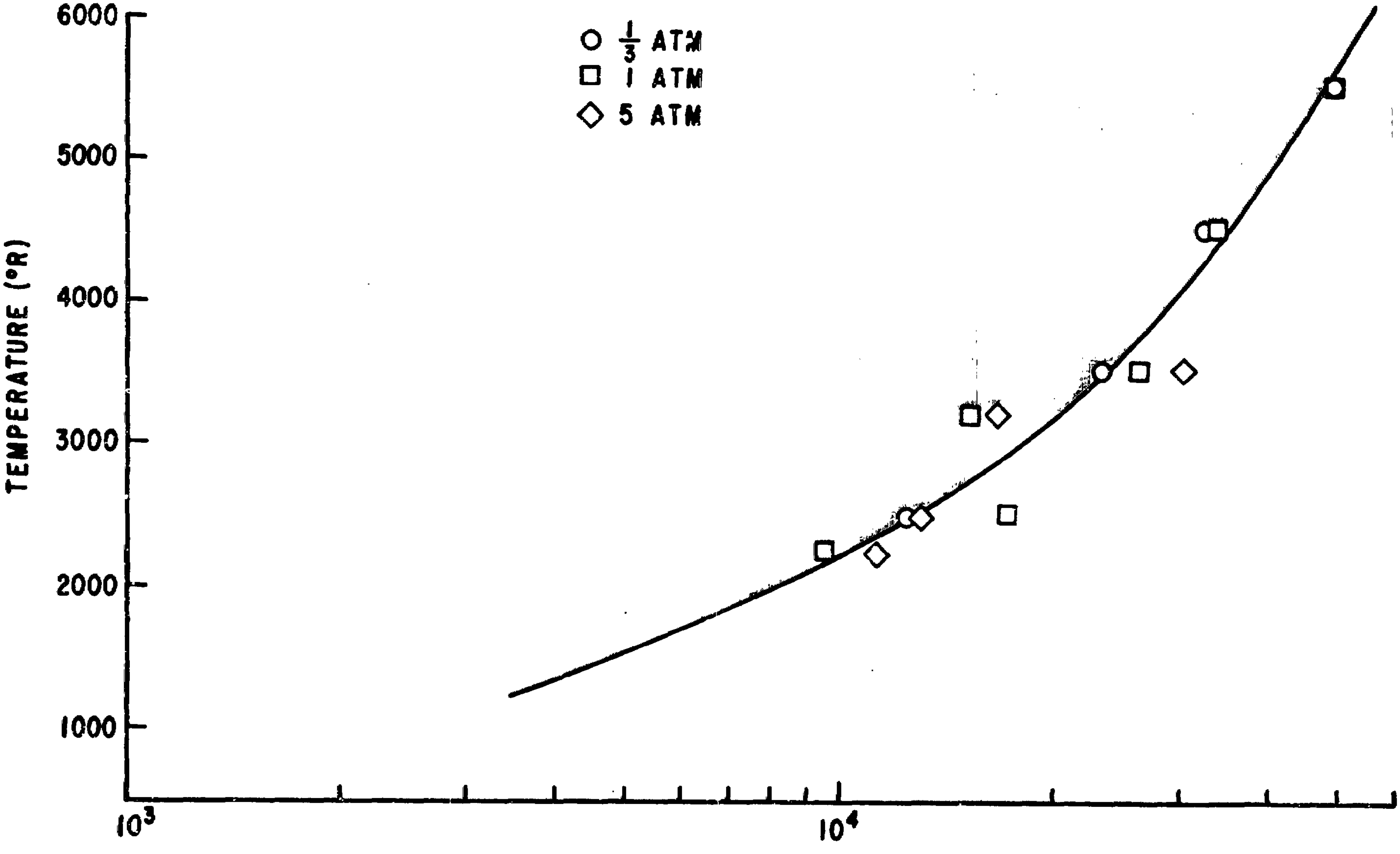

Figure 14. Ohio State Data

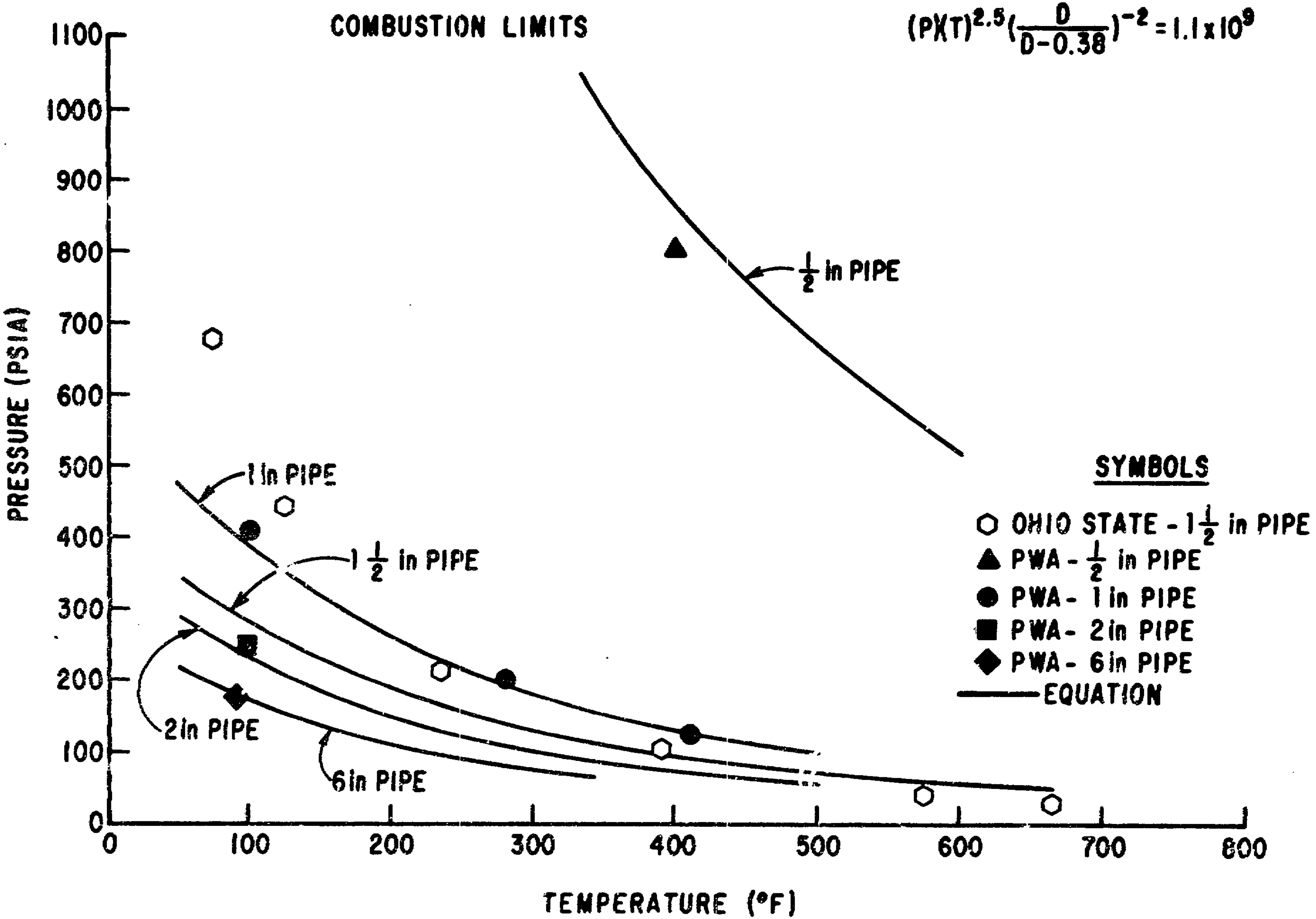

Figure 15. Combustion Limits

To further check the correlation, an additional test was conducted in a 6-inch diameter pipe at ambient temperature. The quenching was less severe in the larger pipe, and the limiting pressure not sharply defined. It did lie between 150 and 190 psia at 90°F, however, which is in reasonably good agreement with the correlation.

d. Anomalies

One disturbing observation during the gaseous test program was the rather frequent (about 10 percent of the tests) occurrence of unexplained events in two categories, spontaneous ignitions and spontaneous temperature rises. In the first category, sudden temperature and pressure spikes were sometimes observed while N_2O was being vented from the pipe. These anomalies generally occurred at low-pressure conditions where steady-state decomposition cannot be sustained. The other category consisted of unusual increases in pipe wall temperatures (by 20 to 50°F) during filling operations without any sudden pressure rise or other indication of a decomposition reaction. Both of these anomalies remain unexplained.

SECTION IV

N_2O-HELIUM MIXTURE IGNITABILITY

The ignitability of mixtures of helium and N_2O was evaluated in the apparatus shown schematically in figure 16. It consisted of a stainless steel chamber 10 inches long and 6 inches in diameter. Gaseous N_2O was supplied through a fitting at one end and helium was admitted through two tangentially mounted fittings located on the wall, near the center (axially) of the chamber. Threads to accommodate a modified spark plug were located in the center of the end opposite the N_2O admission port. To conduct a test, the chamber was thoroughly purged with N_2O and pressurized slowly, to avoid autoignition, to the full N_2O bottle pressure of about 700 psia. Helium was then admitted very rapidly through the tangential ports to thoroughly mix the two gases together. The igniter, which consisted of a coil of 0.020-inch steel wire welded to the electrodes of a spark plug and connected to a 300-ampere arc welder, was fired. Pressure and temperature in the chamber were sensed by strain-gage transducer and thermocouple and were recorded on an oscillograph. Eight tests were conducted and are listed in table 1. The test area is shown in figure 17.

As table 1 shows, the helium quenched the reaction until the partial pressure of the helium was reduced to 50 psia or 7.2 mole percent (0.70 weight percent). The chamber after test is shown in figure 18. The lowest value tested which did not ignite (test 7.01) had a helium concentration of 1.7 percent by weight. These results are consistent with the prior, pure N_2O tests which showed that the N_2O decomposition reaction is very easily quenched. Either pipe walls or a diluent gas act to absorb heat and quench the reaction. If it is assumed that the limiting value for helium to quench the reaction lies between the values of tests 7.01 and 8.01, or at about 1-1/2 percent helium by weight, the resulting constant pressure flame temperature must be reduced only from a theoretical 3070 to 2975°F to quench the reaction. Obviously, N_2O decomposition is a marginal reaction with just enough heat released to sustain itself over the convection, conduction, and radiation losses. It is highly probable that small amounts of other gases (for example, 7 weight percent nitrogen or 8 weight percent oxygen) would also quench the recaction at these conditions. At higher temepratures, of course, more diluent would be needed.

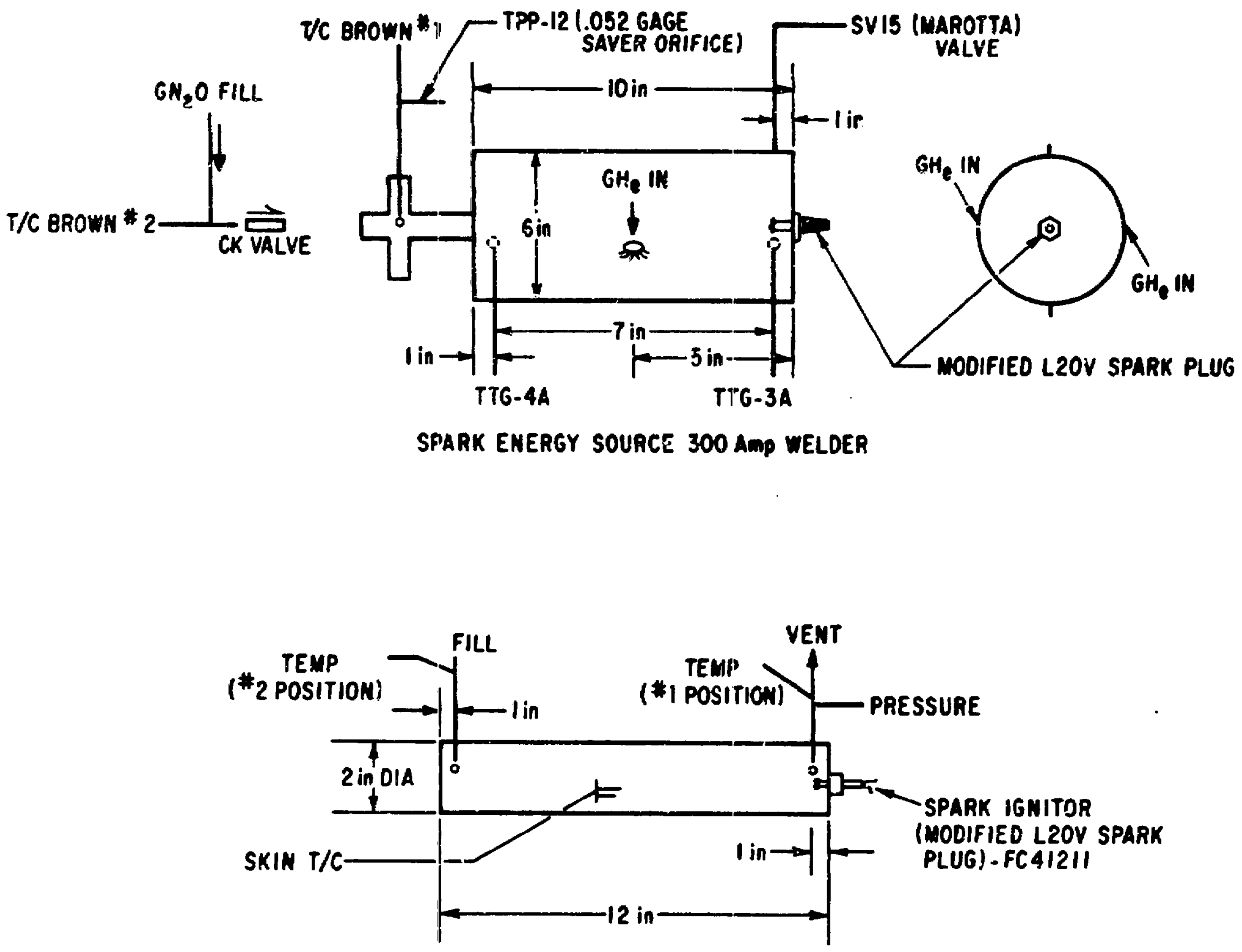

Figure 16. Ignition Apparatus

Table 1

TEST MATRIX GN_2O AND GHe MIX

Test No.	Partial pressure N_2O (psi)	Partial pressure He (psi)	P_{mix} (psia)	Temp (°F)	Comments
1.01	750	750	1500	80	No temperature or pressure rise
2.01	700	1400	2100	75	No temperature or pressure rise
3.01	700	800	2200	75	No temperature or pressure rise
4.01	690	710	1400	74	No temperature or pressure rise
5.01	690	410	1100	74	No temperature or pressure rise
6.01	690	410	1100	74	No temperature or pressure rise
7.01	670	130	800	73	No temperature or pressure rise
8.01	640	50	690	70	Mixture ignited; chamber ruptured; flame propagation rate: 40 cm/sec

Figure 17. Bunker

Figure 18. Test Chamber

1. LIQUID N_2O IGNITABILITY

Ignition tests of liquid N_2O were conducted in a pipe 2 inches in diameter and 12 inches long as shown in figure 19. Heavy caps were welded on both ends, and one end was threaded for a spark plug. One-eighth-inch nominal pipe fittings were used to fill and vent the test chamber. Internal and skin thermocouples measured the N_2O and metal wall temperatures. To conduct a test, the pipe was packed in dry ice, and N_2O was condensed until the pipe was full of liquid. The supply valve was then closed and water was sprayed on the pipe until both the liquid N_2O and the wall reached the desired temperature. Care was taken to ensure that both the wall and liquid N_2O were at the same temperature. Nitrogen pressure was then applied to the 1/4-inch diameter fill line about 20 feet from the test apparatus to compress the liquid N_2O and the N_2O gas remaining in the 20 feet of fill line to the desired test pressure. Two sets of ignition tests were conducted, electrical ignition tests and explosive cap ignition tests.

a. Electrical Ignition Tests

Four tests were conducted using the exploding wires and arc welder previously described. Slight pressure and temperature increases were recorded when the igniter was fired. In one case, the pressure increased momentarily from 1700 to 4000 psi, indicating a slight reaction near the igniter, but in the other three tests little or no pressure change was noted. There was no external sound or any other indication of a reaction. The tests clearly show that any N_2O which may be vaproized and ignited by the exploding wire is immediately quenched by the surrounding liquid. The test conditions are presented in table 2.

b. Explosive Cap Ignition Tests

The explosive cap ignition tests were essentially the same as the electrical ignition tests except that the exploding wire was replaced with a standard No. 6 blasting cap. The test results, however, are not quite as clear. In all, eight tests were conducted, four using water to provide a basis for comparison and four using liquid N_2O. The water test conditions and results are presented in table 3. The last test, 4.01, was conducted after the N_2O tests were completed to see if 50 grains of Primacord in addition to the six-grain cap would duplicate the damage that was produced by the N_2O.

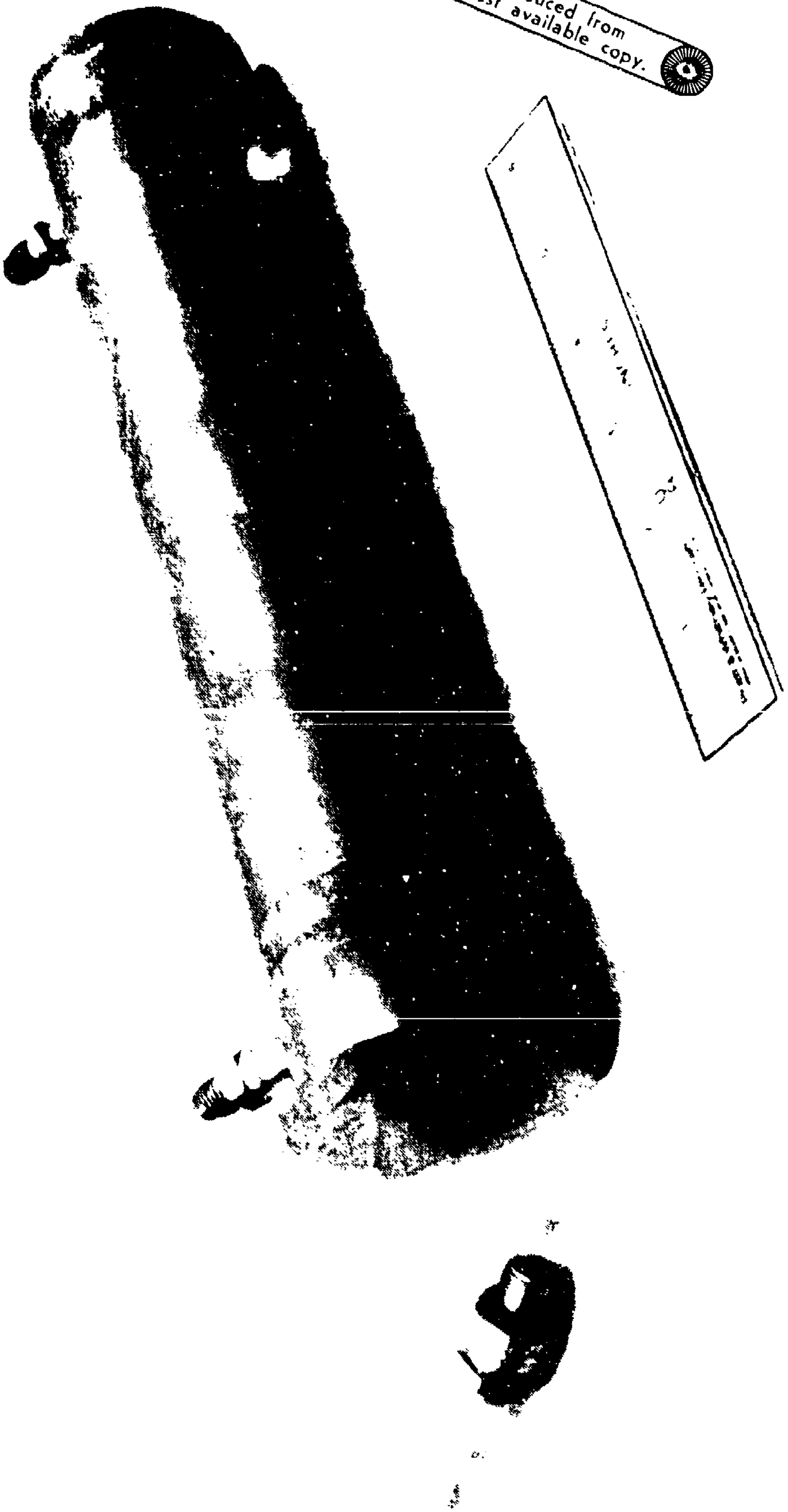

Figure 19. 2-Inch Pipe

Table 2

ELECTRICAL IGNITION TEST CONDITIONS

Test No.	Pressure (psia)	Temp (°F)	Temp wall (°F)	Comments
1.01	1700	60	60	No ignition; slight temperature rise; coil not burned completely
2.01	1700	60	60	Temperature to 200+ degrees; pressure to 4000; no change in wall temperature
3.01	1750	60	60	T/C No. 1 moved to No. 2 position; slight pressure rise note
4.01	1700	60	60	No pressure rise

Table 3

WATER BASELINE TESTS

Test No.	Pressure (psia)	Temp (°F)	Comments
1.01	1700	65	Very slight change in pipe diameter at explosive cap end
2.01	1700	65	Very slight change in pipe diameter at explosive cap end
3.01	1700	65	Very slight change in pipe diameter at cap end; conax fitting blown out
4.01	1700	65	Tube increased in diameter 4 inches down tube at cap end; did not rupture

After testing, the diameter had increased at the cap end, 4 inches down the tube, but the tube did not rupture. The other tubes were not changed in appearance by the testing except for a barely noticeable bulge near the end containing the cap.

Of the four tests using liquid N_2O, one bulged the tube slightly as in the first three water tests, and the other three ruptured the tubes. The three ruptured tubes were permanently stretched about 20 percent before rupture. The tube which was slightly bulged in the first test was also reused and ruptured in the last test. Figure 20 shows the remaining three tubes after test. Each of the tubes split open along the top but left both ends intact. In only the last case was the tube broken into two pieces. If the liquid N_2O had detonated, the energy released would have been equivalent to almost 2-1/2 pounds of TNT. This quantity of TNT would have broken the pipe into shrapnel and completely demolished the bunker where the tests were conducted. As shown in figure 17, the bunker was completely undisturbed at the completion of the test program.

The difference in results between the N_2O tests and the water tests clearly shows that some N_2O decomposition occurs. The relatively small amount of damage, however, also shows that the reaction is either slow or is quenched. It is postulated that the high compressibility of the liquid at this temperature results in high local temperatures as the shock wave passes through, resulting in partial decomposition. At lower temperatures where the compressibility should be greatly reduced, the amount of decomposition ought to be correspondingly lower.

One other explosive cap test was conducted in which the cap was taped to the outside of a 1-inch diameter pipe filled with liquid N_2O. This test produced a comprehensive deformation of the pipe in the vicinity of the cap but no evidence of N_2O decomposition. The five explosive cap tests are listed in table 4.

Figure 20. Ruptured Pipes

Table 4

EXPLOSIVE CAP TESTS

Test No.	Pressure (psia)	Temp liquid (°F)	Temp wall (°F)	Comments
1.01	1700	60	60	No detonation; however, tube diameter increased entire length
2.01	1700	60	60	Tube ruptured
3.01	1700	60	60	Tube ruptured
4.01	1700	60	60	Tube ruptured
5.01	760	-70	---	Cap taped to outside of tube; pressure spike to 2000 psia

SECTION V

MATERIALS COMPATIBILITY

High-pressure compatibility containers were used in this study (conducted under Rocketdyne G. O. 09518). Each container consisted of a 5000-psi Taber pressure transducer, an Autoclave Engineers safety head and valve, and a specimen ampoule. All but one of the container connections were the Autoclave Engineers medium pressure type (30,000 psi), a metal-to-metal connection. The ampoules were fabricated at the Rocketdyne machine shop with an end connection designed to mate with the Autoclave Engineers parts. Cylindrical ampoules were made of type 304 and 316 stainless steel and had the following dimensions: 5/8-inch ID, 1-inch OD, and 9-inch internal length. Total volume of each container was 52 cc (figure 21).

A stainless steel N_2O transfer system was designed and constructed for loading the compatibility containers with N_2O. Basically, the system consisted of an N_2O supply cylinder, a cylinder of known volume, pressure gages, valves, cold trap, and a vacuum pump.

Instrumentation was installed for monitoring the pressure and temperature of the compatibility containers. The outputs of the Taber pressure transducers were recorded on a Leeds & Northrup multipoint recorder. Bath temperature was recorded on a Hewlett-Packard strip chart.

1. TEST PROCEDURE

All parts of the compatibility containers which were to be exposed to N_2O liquid or vapor were thoroughly cleaned. The cleaning procedure involved washing the parts with trichloroethane and then with acetone. High purity methanol was used for the final rinse of the parts. The parts were then dried in a vacuum oven at 140°F for at least 30 minutes. This same procedure was used for cleaning the nickel, copper, and stainless steel screen specimens used as test impurities. Other test impurities used in this study but not requiring cleaning were ferric oxide, chrome oxide, and leak detector solution type FX 190, manufactured by 3-M Corporation.

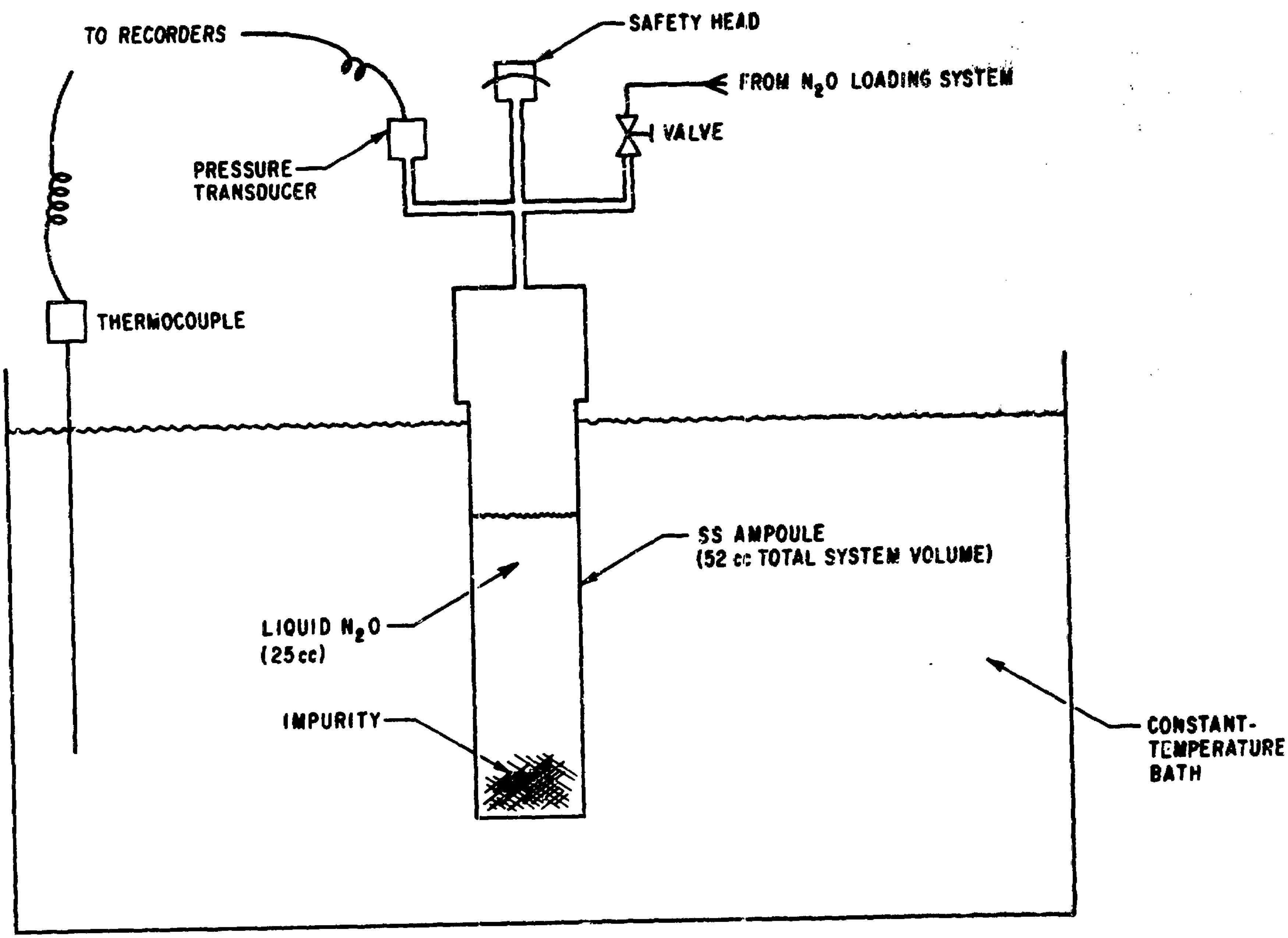

Figure 21. Materials Compatibility Chamber

Following the cleaning operation, the test impurities were inserted in the ampoules and the compatibility containers were assembled and leak checked. Eliminating leaks in the assembled units proved to be a problem, due principally to imperfections in the sealing surface of the purchased Autoclave Engineers adapters. These adapters, used to mate the ampoules to the remaining apparatus, had to be refinished at the Rocketdyne machine shop. Each of the assembled apparatuses was then pressurized with GN_2 to about 3600 psi and the pressures were monitored for several days. A Bacharach Industrial leak detector was also employed in the leak checking procedure.

The need for design and fabrication of high-pressure compatibility containers, and subsequent high-pressure leak checking, was based on requirements of the originally scheduled compatibility study. This study was to have involved measurement of decomposition of gaseous N_2O in contact with selected specimens at 240 atm (3528 psi) and 410°F. Immediately following the high-pressure leak checking but just prior to loading the test containers with N_2O, instruction was received to cancel the gaseous N_2O tests and redirect the test experimental effort to evaluate compatibility of liquid N_2O. The same test materials and contaminants were used in the new study, but they were immersed in liquid N_2O for a period of at least 4 days at a temperature somewhat below the critical temperature (309.6°K, 97.6°F); 85°F was selected. It was decided that the same test containers (already leak checked and with the specimens within) would be adequate for the new study.

The N_2O was loaded into the ampoules using a vapor phase transfer. A calibrated volume in the N_2O loading system was pressurized to a preselected value with the vapors above the N_2O liquid in a supply cylinder purchased from Matheson Gas Products. Since the starting material has an average purity of 98.5 percent (air being the chief impurity, according to the vendor), the N_2O was loaded into the test ampoules by condensation at -126°C from the calibrated volume. The noncondensable gases and some N_2O were pumped off, and then the ampoule valve was closed.

The compatibility containers, loaded with N_2O, were placed in an 85°F constant temperature bath, and the temperature and pressure were monitored for a period of 9 days. In addition to the six test impurities in contact with N_2O in 304 SS ampoules, one 304 SS ampoule and one 316 SS ampoule (both without any test impurity) were filled with N_2O and placed in the bath under the same test conditions.

2. TEST OBJECTIVES

The objective of this study was to determine the compatibility of liquid nitrous oxide in contact with several selected impurities specified in the contract work statement.

3. TEST RESULTS

After 30 days at 85°F, no pressure buildup was noted in any of the eight compatibility containers. The containers were removed from the bath, placed in dry ice, and then submitted for gas chromatographic (gc) analysis. Each ampoule was connected to the gc and then immersed in a methylcyclopentane slush bath (-142°C). At this temperature, N_2O has a vapor pressure of about 1 mm Hg. After opening the ampoule to the gc inlet system, the pressure was measured using a tilting McLeod gage. In most cases, the total pressure of the gases (comprised of N_2O, N_2, and O_2) was about 2 mm Hg for a total gas volume of 199 ml (gc inlet system and compatibility ullage volume).

Based on the results of the gc analyses, the total number of moles of noncondensables (N_2 and O_2) was combined, and using the decomposition reaction $N_2O \rightarrow N_2 + 1/2\ O_2$ or 1 mole of N_2O yields 1-1/2 moles of noncondensables, the percent N_2O decomposition was calculated for each ampoule (table 5).

Table 5

N_2O DECOMPOSITION DURING 30 DAYS AT 85°F

Impurity	Quantity (size)	Percent N_2O decomposition
Copper	1.22 gm (8 in^2)	0.7×10^{-3}
Nickel	9.39 gm (8 in^2)	1.6×10^{-3}
304 SS screen	1.68 gm (11 in^2)	0.9×10^{-3}
Ferric oxide	0.15 gm	3.1×10^{-3}
Chromic oxide	0.22 gm	1.6×10^{-3}
Leak check solution (FX 190; 3-M Corp)	0.2 gm	1.9×10^{-3}
304 SS (control)	---	1.9×10^{-3}
316 SS (control)	---	2.4×10^{-3}

From the table, it can be seen that the ampoule containing ferric oxide had the highest amount of N_2O decomposed, 3.1×10^{-3} percent. However, even this quantity can be considered to be negligible. Inspection of table 5 will show that even the test control ampoules had N_2O decomposition quantities of the same general magnitude as the ampoules containing test impurities. Table 6 lists several additional materials which showed no detectable decomposition. Three materials which were tested in liquid N_2O did suffer significant degradation. Those materials were buna-N, neoprene, and a polymer designated AF E-124X. The AF E-124X had been developed as a bladder material for use with N_2O_4. The materials showed no net effect on N_2O.

Table 6

ADDITIONAL N_2O COMPATIBILITY SUMMARY*

Sample	Total exposed hours
Inconel No. 1 HT	512
Inconel No. 2 HT	8
Inconel No. 3 HT	1839
Inconel No. 4 HT	1839
Inconel No. 5	1839
Inconel No. 6	1839
Stainless steel No. 1	8
Stainless steel No. 2	512
Stainless steel No. 3	1839
Stainless steel No. 4	1839
Stainless steel No. 5	1839
Stainless steel No. 6	1839
Aluminum No. 1	1149
Aluminum No. 2	1149
Aluminum No. 3	1149
Aluminum No. 4	1149
Aluminum No. 5	1149
Aluminum No. 6	1149
Teflon	1149
Delron	1149
Nylon	1149
Rolon A	1149
Vespel	1149
25 percent glass with Teflon	1149
Ray-Chem (wire)	1149
Brand-Rex (wire)	1149

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*Data taken at Beechcraft Corp, Boulder, CO.

No reaction was detected between N_2O and the above list of materials.

SECTION VI

CONCLUSIONS

1. Gaseous N_2O is easy to ignite, but the reaction proceeds slowly.

2. N_2O decomposition reaction is easily quenched.

3. Liquid N_2O is relatively insensitive to high-energy sparks or external shocks, but some decomposition can be induced by explosive caps.

4. Relatively small amounts of helium quench the decomposition reaction in ambient temperature N_2O.

5. Negligible decomposition of liquid N_2O occurs as a function of selected impurities such as ferric oxide, chromic oxide, a leak check solution, copper, nickel, and type 304 stainless steel screen (100 mesh).