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SUMMARY OF NITROUS OXIDE INVESTIGATIONS

July 1976

Final Report

Approved for public release; distribution unlimited.

AIR FORCE WEAPONS LABORATORY
Air Force Systems Command
Kirtland Air Force Base, NM 87117

AFWL-TR-75-231

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

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The Air Force Weapons Laboratory (AFWL) proposes to use nitrous oxide (N_2O) as an oxidizer in a combustion process for an airborne system. A number of problem areas were investigated by AFWL or by other organizations under contract to AFWL. This report summarizes the results of these N_2O investigations. Two of the programs investigated the decomposition characteristics of N_2O-CO mixtures. It was determined that the decomposition of gaseous N_2O is not difficult to initiate, but that it is also easily quenched. Tests with the N_2O-CO mixture revealed that this combination has significant explosive potential; however, it is difficult to

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initiate such an explosion. Another series of investigations addressed potential flow problems. The liquid N_2O in the system is pressurized by helium in the ullage of the tank and absorbs some of this He. With more than about 0.5 mole percent absorbed He, the effective throat area of the cavitating venturi used in the metering system is appreciably reduced. A second aspect of the flow studies concerned the possibility of N_2O solidification when it is dumped overboard under emergency airborne conditions. Tests confirmed the formation of N_2O snow when liquid N_2O was dumped into a vacuum chamber; however, no flow problems were encountered. The third group of tests evaluated materials compatibility with liquid N_2O. A selection of 34 materials were immersed in liquid N_2O, and three of the materials showed evidence of degradation. Another study involved impact tests of 19 materials immersed in liquid N_2O. No adverse effects were noted. Three materials were also tested for use as bladders which could potentially be used to pressurize the N_2O tank. One of these materials degraded considerably. The last group of research programs were designed to prove the stability of the combustion of CO using N_2O as the oxidizer and to verify the results of the scaled down flow tests. Acoustic stability was verified and exhaust gas uniformity was mapped by direct sampling for temperature and species.

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SECTION I

INTRODUCTION

Nitrous oxide (N_2O) is a well known compound that has been used primarily in three different fields. First, it is well known as a weak anesthetic for medical and dental use, and the physiological effects of nitrous oxide on the human body have therefore been extensively investigated. Second, chemists have been studying the decomposition of N_2O for more than 50 years (refs. 1 and 2). Data have been accumulated concerning reaction rates and conditions required for decomposition. Third, nitrous oxide is commonly used as a low temperature refrigerant. This use has stimulated a number of investigations into the thermodynamic and physical properties of N_2O. Reference 3 contains an extensive bibliography concerning N_2O thermodynamic and physical properties, and that bibliography has been included herein. It should be noted that the bibliographies included in reference 3 were compiled as part of the program described in this report.

The remainder of this report concerns a fourth use of N_2O, i.e., its use as an oxidizer in a combustion process. The particular way in which the Air Force Weapons Laboratory (AFWL) proposes to use the N_2O requires that it be loaded into a tank installed in an aircraft and that the N_2O then flow to a combustion chamber in metered quantities. This application brings up a number of new problems, which are listed below:

1. The fact that the N_2O is to be used as part of an airborne system elevates the safety criteria and focuses attention on the exothermic (and possibly explosive) decomposition of N_2O. In addition, possibilities of large N_2O spills exist, either during filling or due to system failure. The hazard presented by these spills must be evaluated.

2. A new group of flow problems arises from the system operating conditions. The N_2O tank will contain pressurized helium in the ullage volume, and this He will force the N_2O out of the tank, through the metering system, and into the combustor. The N_2O absorbs He, and if the absorbed He is subsequently desorbed during the flow or metering processes, the metering system may not function properly. In addition, this flow may affect the stability and/or the species distribution of the combustor. Thus, the effect of dissolved

helium must be evaluated. Another potential flow problem concerns dumping the N_2O from the airborne tank during emergency inflight conditions. The thermodynamic properties of the N_2O indicate that the liquid may solidify if expanded to the low ambient pressures that exist at high altitude. Solidification, in turn, can cause blocking of the lines and thus prevent dumping of the N_2O. This possibility must be investigated.

3. Materials used in the system which will be exposed to N_2O must be screened for compatibility. Little published N_2O compatibility data is available, therefore testing programs were required.

4. The AFWL system uses N_2O as an oxidizer in a combustion process. Questions exist concerning the characteristics and stability of the combustion when N_2O is used with available fuels. Thus, combustion stability must be investigated.

These problems were investigated either by AFWL or by other organizations under contract to AFWL. Several technical reports (refs. 4, 5, 6, 7, 8 and 9) have been published, and it is the purpose of this report to summarize the results of those investigations. Readers who desire more detail than is given here may obtain these reports from the Defense Documentation Center (DDC) or from the National Technical Information Service (NTIS) operated by the Department of Commerce. The experimental programs conducted were of a practical nature and were designed to yield data applicable to the system of concern to AFWL. It is hoped, however, that the information will prove useful to others who are involved with N_2O flow systems.

SECTION II

DECOMPOSITION AND EXPLOSION HAZARDS OF N_2O

Two programs concerned with reactions of N_2O were sponsored by AFWL. The first investigated the decomposition characteristics of gaseous and liquid N_2O, and was done by the Pratt and Whitney Aircraft Division of United Technologies Corporation. This work is described in reference 4.

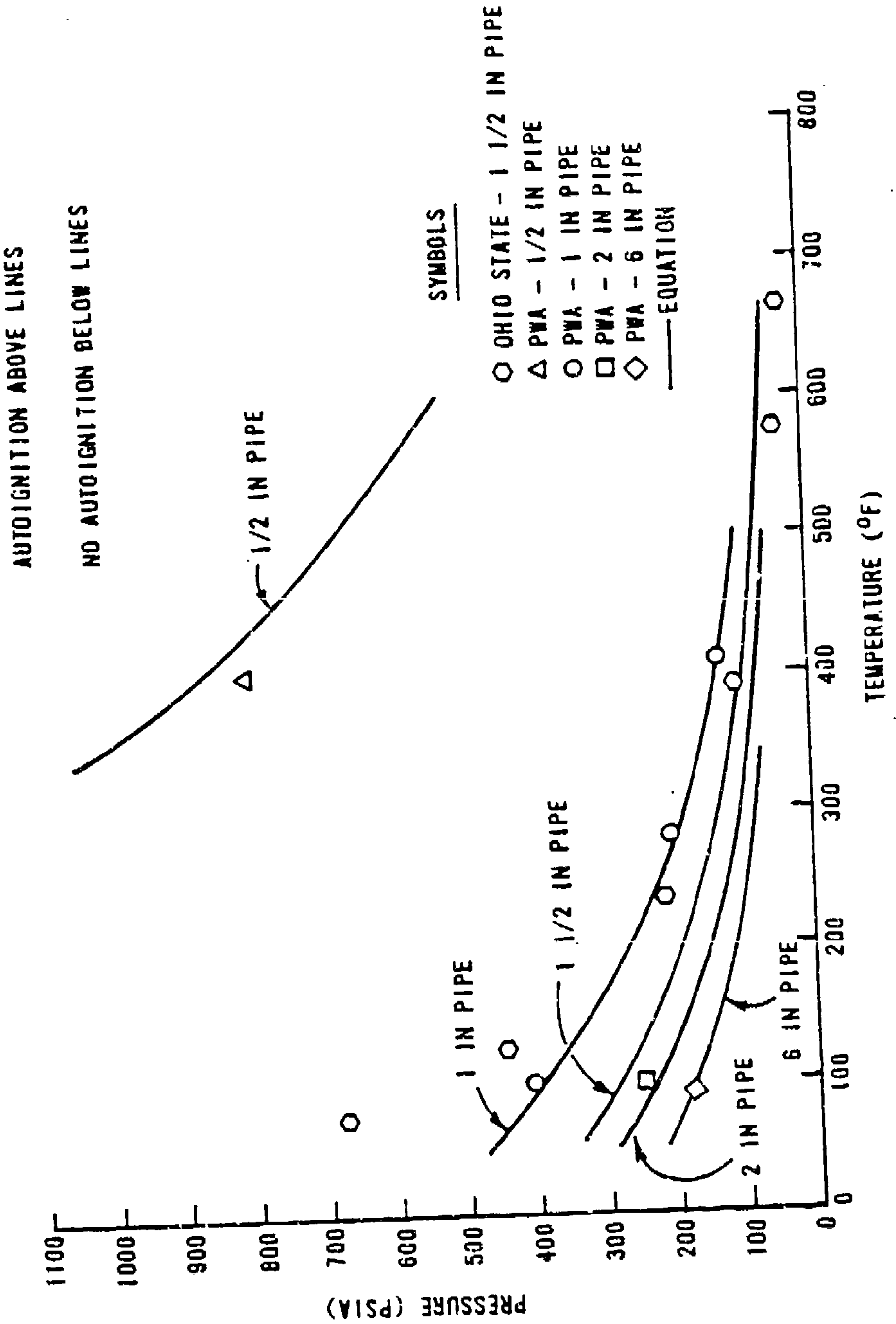
The second program was concerned with detonation of N_2O-CO mixtures in the liquid and gaseous phases. Both of these compounds are used in the airborne system, and it is necessary to know the consequences of mixing them inadvertently, as in the case of a spill. Testing of the explosive potential of these mixtures was done by the Air Force Rocket Propulsion Laboratory and is described in reference 5.

1. DECOMPOSITION OF GASEOUS NITROUS OXIDE

An experimental program designed to investigate some of the parameters pertinent to N_2O decomposition is described in reference 4. The parameters of interest in that program were flame propagation speed, ignition energy required to initiate the decomposition reaction, and the quenching effects of both the container holding the N_2O and of helium added to the N_2O.

The experimental procedure for determining decomposition parameters consisted essentially of filling a pipe with gaseous N_2O to a predetermined pressure, firing a spark plug at one end of the tube, and measuring the rate at which the dissociation reaction propagated down the tube. Details of the test setup are given in reference 4. It was found that the decomposition reaction was easily initiated in gaseous N_2O by an electrical spark and that the propagation rate of the reaction did not depend on the energy of the electric spark.

Following the spark ignition tests, experiments were conducted to determine the autoignition characteristics of the N_2O decomposition reaction. The procedure for the autoignition test was to heat the pipe to a predetermined temperature and then quickly admit N_2O from a tank at 800 psia and room temperature. The pipe pressure at which the decomposition reaction began was recorded, for the predetermined pipe temperature. Results of the autoignition tests are presented in figure 1. The points labeled "PWA" come from the work done by



Fogure 1. Autoignition Limits for N_2O

Pratt and Whitney Aircraft (ref. 10), and the points labeled "Ohio State" are from reference 11.

The data in figure 1 show that pipe diameter, along with pressure and temperature, is a significant variable. The effect of pipe diameter indicates the important role of quenching in inhibiting the dissociation reaction. As pipe diameter decreases, the surface to volume ratio of the pipe increases. Thus, if decomposition in a small pipe is compared with that in a larger pipe, the surface to volume ratios indicate that a greater fraction of the heat released by the dissociation will be conducted away by the small pipe than by the large pipe. This heat loss tends to quench the reaction and, as a result, higher temperatures and pressures are required to sustain a decomposition reaction in a small tube (fig. 1).

The sensitivity of the N_2O dissociation reaction to quenching was further demonstrated by test designed to dissociate the N_2O in the N_2O-He mixtures. In these tests, the gases were placed in a 6 inch diameter reaction chamber, and spark ignition was attempted. Results are shown in table 1. It should be noted that the partial pressure of the N_2O in each of these mixtures falls within the autoignition range define in figure 1, at room temperature. Thus, the N_2O in each of the mixtures in table 1 would have decomposed if the helium had not been present. The reaction was quenched in seven of the eight tests by the presence of the helium.

In summary, the decomposition of gaseous N_2O is not difficult to initiate, but it is also easily quenched.

A small amount of liquid ignitability testing was also done in this program. Two-inch diameter pipes were filled with liquid N_2O, and ignition of the N_2O decomposition reaction was attempted with both exploding wires and blasting caps. The exploding wire tests indicated that a small amount of dissociation occurred but the reaction was quickly quenched by the liquid N_2O. Detonation of the blasting caps, however, caused sufficient N_2O decomposition to rupture the pipe.

2. BLAST HAZARDS OF N_2O-CO MIXTURES

The details of the experimental program for determining the blast hazards of N_2O-CO mixtures are given in reference 5. The initial part of this work involved detonation of liquid mixtures which were held in cylindrical containers 3 feet in diameter. The total liquid depth was approximately 1 foot, and

Table 1

TEST CONDITIONS FOR GASEOUS N_2O - He MIXTURES

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

explosion of the stoichiometric liquid mixture was attempted by means of either two blasting caps or a blasting cap and a squib. The results of six tests are shown in table 2, and it is seen that while fires occurred in all cases, explosions only occurred in two cases, and both of these used two blasting caps for ignition. The results also show no significant differences due to loading both compounds simultaneously into the tank, or first loading one and then the other. Reference 10 contains data detailing the intensity of the explosions at various distances from the device.

The second set of tests simulated a situation in which the N_2O and CO were unconfirmed. Streams of each liquid were sprayed into a nylon tent that measured 12 by 12 by 8 ft., and ignition was accomplished by one blasting cap and one squib. No explosions were experienced during the four tests, although fire occurred in each of the tests.

Table 2

RESULTS FROM IGNITION OF LIQUID MIXTURES OF N_2O - CO

Test Condition	Test No. 4	5	6	23	24	25
Both Propellants	yes	yes	yes	yes	yes	LCO only
Fill Method	seq.	sim.	sim.	sim.	seq.	----
Ignition Source	2 caps	2 caps	2 caps	1 cap 1 squib	1 cap 1 squib	1 cap 1 squib
Results						
Fire	yes	no	yes	yes	yes	yes
Explosion	yes	no	yes	no	no	no
Explosive Yield	38%	0	16%	0	0	0

* Seq. = sequential; sim. = simultaneous

SECTION III

FLOW STUDIES

Section I of this report briefly described two types of flow problems to be encountered by the airborne N_2O system. The first involved absorption of helium by the N_2O, followed by desorption during the flow or metering processes. The desorption could interfere with proper metering. Two programs dealing with this problem were sponsored by AFWL; one was done by the National Bureau of Standards (NBS) (ref. 6), while the other was done by the Rocketdyne Division of Rockwell International (ref. 7). The solidification problem described in section I was the subject of an in-house study at AFWL (ref. 8).

1. PHASE EQUILIBRIUM OF THE He-N_2O SYSTEM

The logical starting point for a study involving the absorption of a gas by a liquid is to determine the solubility characteristics at equilibrium, therefore the NBS study investigated the equilibrium solubility of helium in liquid N_2O. This was done over a temperature range of 351° to 513°R, and a pressure range of 26 to 2,000 psia. Both solubilities and Henry's Law constants were determined.

The results showed that the equilibrium solubility of He in N_2O varied from 0 to 4.18 mole percent for the temperature and pressure ranges investigated. Thus, a considerable volume of helium may be released during the flow and metering processes if the N_2O is saturated with helium initially and the helium then leaves the solution due to decreasing system pressure. The Henry's Law constants were found to be highly temperature dependant, but exhibited only a weak pressure dependance.

2. DESORPTION DUE TO PRESSURE DROP IN THE FLOW LINES

Knowledge of the equilibrium solubilities enables one to calculate the pressure drop of flowing N_2O that is helium saturated, as a function of pipe pressure if equilibrium is assumed, the problem is a classical two phase flow problem, complicated by the continuous release of helium as the N_2O progresses down the pipe. The results of a pressure drop analysis for this situation is shown in figure 2, taken from reference 6. The flow rate on which the analysis is based was selected by appropriate scaling of the airborne system. The

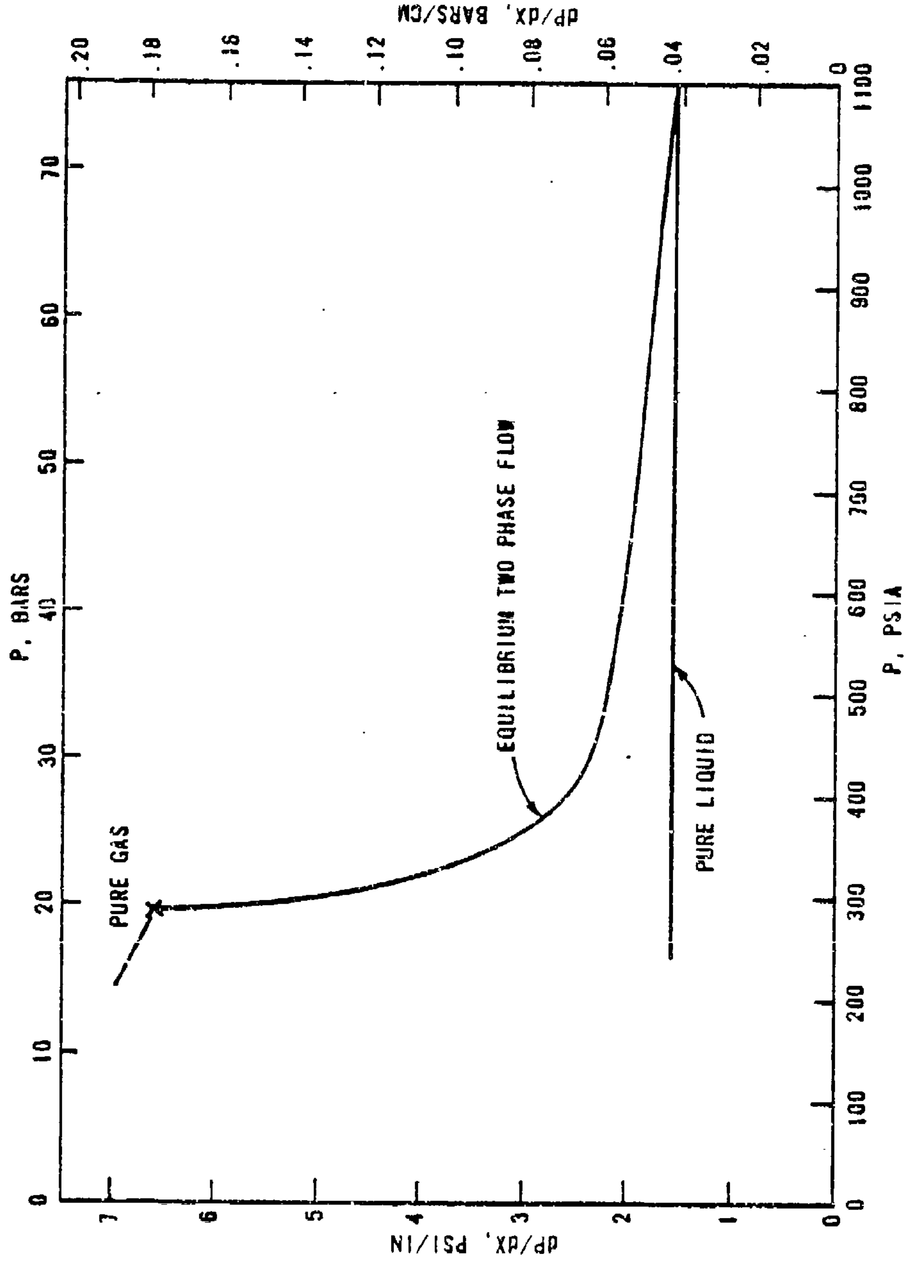


Figure 2. Axial Pressure Gradient as a Function of Local Pressure

analysis shows that the pressure gradient does not rise steeply until the fluid pressure falls below about 400 psia.

Flow experiments were run using both helium saturated N_2O and unsaturated N_2O. The minimum pressure reached in any of these tests was 650 psia, and no difference was discerned between the pressure losses of saturated and unsaturated N_2O. This is not surprising considering the results in figure 2 and the pressure range of the tests. Pressures below 1,000 psia are not anticipated in the airborne system.

3. DESORPTION EFFECTS DUE TO PRESSURE DROP IN A NOZZLE

One of the basic components of the metering system is a cavitating venturi. This device causes a rather sudden drop in the static N_2O pressure, and there was concern that this sudden pressure drop would cause helium desorption from the liquid. If the desorption occurred, the gaseous helium would tend to choke the flow of liquid N_2O through the venturi throat and thereby interfere with proper functioning of the metering system. Calculations in reference 6 indicated that significant choking would occur if equilibrium conditions prevailed. Thus, the last portion of the NBS study was concerned with N_2O flow in a venturi.

The facility used for testing flow in a venturi is shown schematically in figure 3. The pressure drop labeled ΔP_4 is the pressure drop across a flowmeter, and the flow rate is proportional to $\sqrt{\Delta P_4}$.

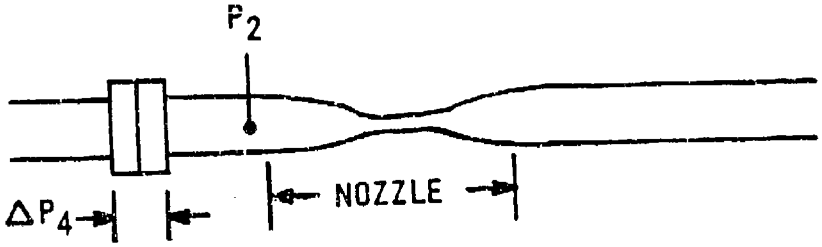


Figure 3. Schematic Diagram of the Test Facility

In order to determine the effect of helium on flow choking, flow tests were run using both saturated and unsaturated N_2O. It was found that the flow rate of the helium saturated N_2O was 9% to 12% lower than that of the unsaturated liquid. Typical test results are shown in figure 4. These results show

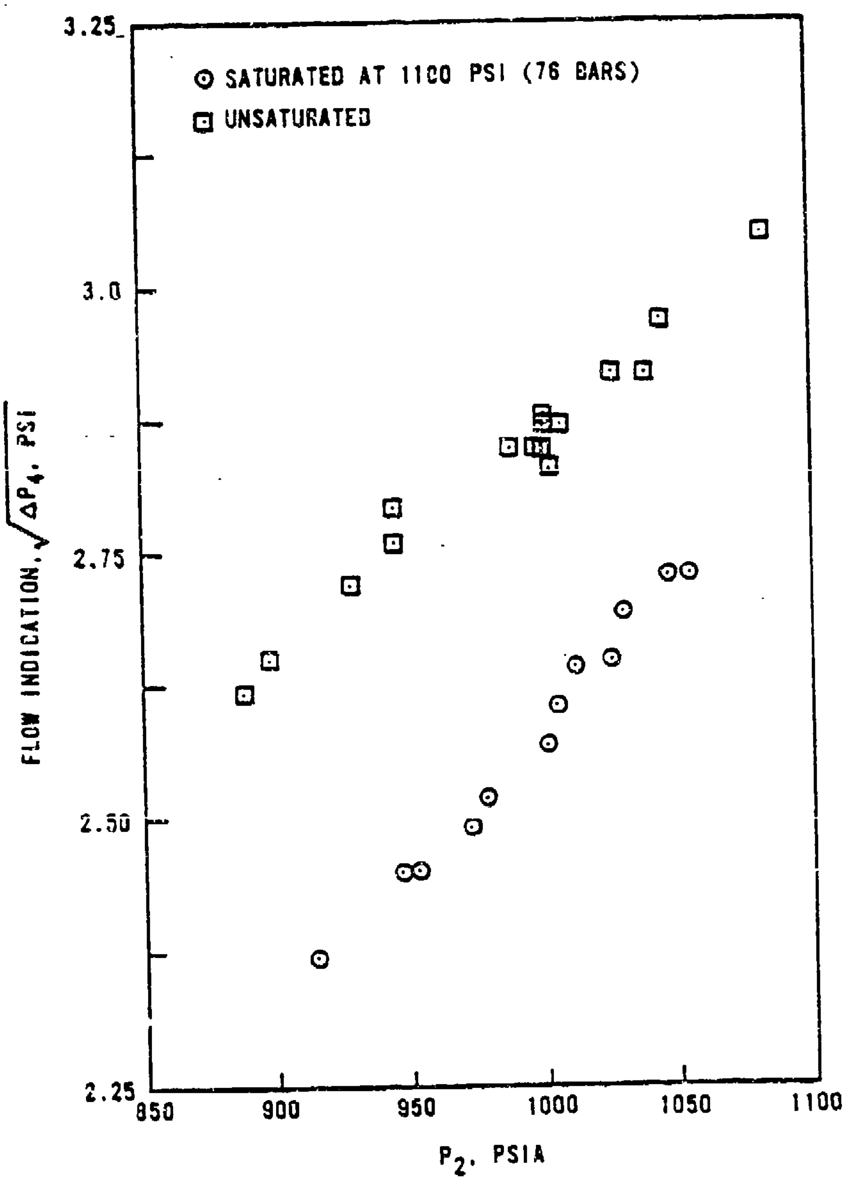


Figure 4. Flow Rate Indication as a Function of Liquid Nitrous Oxide

that the flow of saturated N_2O through the venturi is neither equilibrium flow nor frozen flow, but is rather something in between the two. Thus, there is some effect due to helium desorbing from the liquid N_2O, but the effect is much less than that predicted for equilibrium flow conditions.

4. FULL SCALE FLOW TESTING

The NBS tests described above were conducted in a scale model facility. The fact that desorbed helium had an effect on venturi flow rates indicated that some full scale testing was necessary to ensure proper functioning of the airborne system. AFWL funded Rocketdyne to conduct full scale flow tests, and the results of that program appear in reference 7. The Rocketdyne full scale flow testing is summarized in the following paragraphs:

"Oxidizer flow rate to the combustor is controlled by a cavitating venturi located between the main valve and the primary injector. This is a 2-inch, variable area unit* in which the throat area is determined by the position of a movable pintle. The venturi was calibrated with gaseous nitrogen (using NBS-traceable sonic venturi meters as standards) prior to the start of the present program, to obtain a curve of effective throat area as a function of pintle position.

"The cavitating venturi was used in GDL combustor Tests No. 22 through 35. Nitrous oxide flow rates in all these tests were measured with turbine flowmeters. On the basis of these measured flow rates, the effective throat area of the cavitating venturi was calculated in each case. Results are plotted in figure 5 together with the GN_2 calibration curve. All flows of neat N_2O fall on a line which is slightly displaced from the calibration curve. When the N_2O is (partially) saturated with helium, however, the effective throat area of the cavitating venturi is a function of the proportion of helium dissolved. Thus, in Test No. 26 with about 0.5 mole percent He in the N_2O, there is no discernible change in effective throat area. In Test No. 30, with about 1.0 mole percent He, the effective throat area is decreased by about 2.4 percent, while in Test No. 35, with about 1.4 mole percent He in the N_2O, the effective throat area is decreased by about 6.7 percent. Manifestly, if the helium content of the N_2O is greater than about 0.5 mole percent, the cavitating venturi calibration applicable to neat N_2O is not valid. The error introduced is a function of the amount of helium dissolved in the nitrous oxide."

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*Model 610479C, Fox Valve Development Company, Hanover, New Jersey 07936

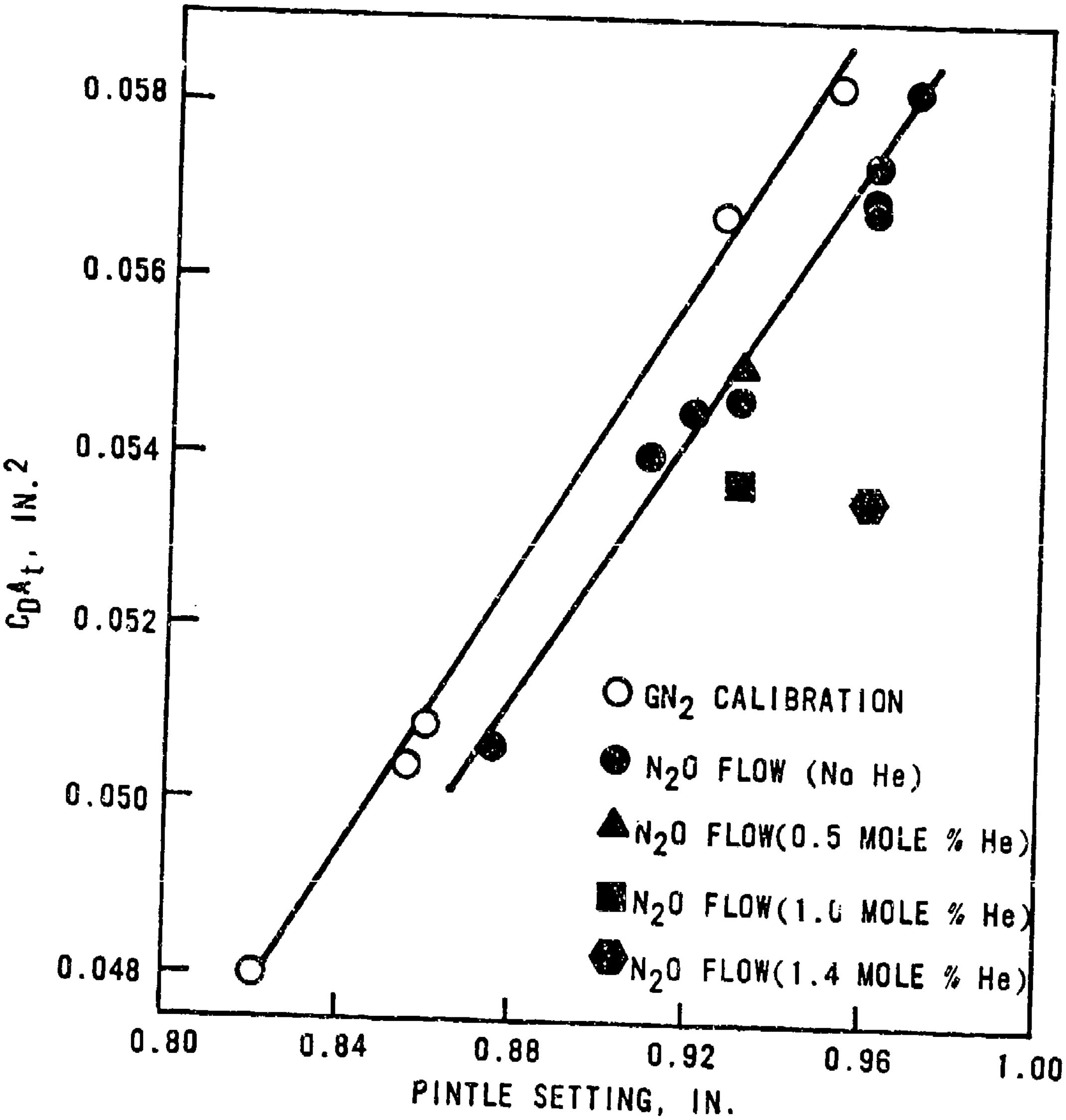

Figure 5. Effective Throat Area of 2-inch Cavitating Venturi as Function of Pintle Setting

5. SOLIDIFICATION OF N_2O DURING FLOW

Under emergency airborne conditions, it may be desirable to dump the N_2O overboard. The thermodynamic properties of N_2O indicate that the dumping operation might cause problems due to the possibility of N_2O solidifying in the dump line. The nature of the problem can be best explained using the pressure-enthalpy diagram in figure 6.

The airborne N_2O tank is maintained at 0°F. The tank contains liquid N_2O, with gaseous N_2O in the ullage volume, and both the liquid and gas are saturated. The liquid and gas states are shown as states 1 and 2 in figure 6. If the tank is pressurized to 2000 psia by adding gaseous helium to the ullage volume, the state of the liquid moves to state 4. The partial pressure of the N_2O vapor should not be greatly affected by the presence of helium and will therefore be at about 279 psia (which is the N_2O vapor pressure at 0°F).

In order to dump the liquid N_2O, it is necessary to open a valve in the line going from the tank to the outside of the airplane. The liquid flow will be almost adiabatic; therefore, the flowing N_2O will follow the vertical path shown from state 4 in figure 6. The ambient (airborne) pressure will be less than the triple point pressure of 12.7 psia, therefore figure 6 predicts that the N_2O will be a solid + vapor mixture upon exiting from the line. If there is no helium in the ullage volume, the adiabatic expansion will again end in the solid + vapor region.

The AFWL test program described in reference 8 simulated operating conditions by dumping N_2O from a tank into a vacuum chamber. It was found that solidification did not occur in the lines but did occur just after the N_2O entered the vacuum chamber. Thus, while large quantities of N_2O snow formed in the vacuum chamber, no flow problems were encountered. Figure 6 shows that venting N_2O gas to ambient should not cause any solidification problems in the lines, and this was confirmed by the test program. Excessive gas venting did, however, cause evaporation of the liquid N_2O in the tank. The cooling associated with the evaporation caused freezing of the liquid in the tank. Thus, the liquid dump mode should be used for emergency emptying of the N_2O tank, and not the gaseous vent mode.

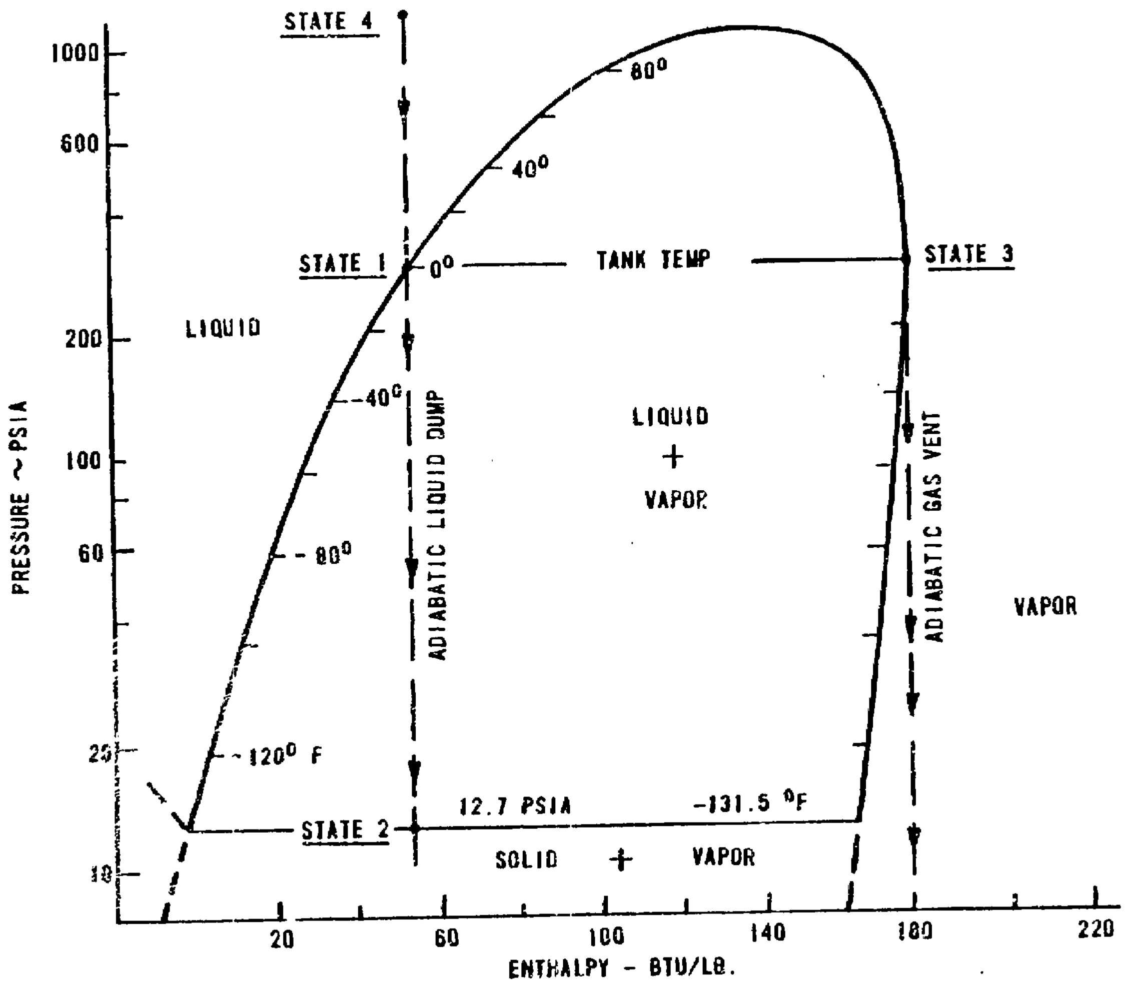

Figure 6. Pressure - Enthalpy Diagram of N_2O

SECTION IV

MATERIALS COMPATIBILITY

The concept of materials compatability has two meanings when applied to N_2O systems. First, the materials must function properly in the liquid N_2O environment, and second, the surfaces of the materials must not catalyze the N_2O dissociation reaction. The catalysis problem has long been recognized but does not appear to be severe (ref. 12). AFWL has sponsored experimental work covering both of these aspects of materials compatability.

Tests were performed on a number of materials to determine their effect on N_2O decomposition. Reference 4 describes testing of 34 materials. Each of these materials was placed in a container of liquid N_2O at 85°F for 30 days, after which the concentration of N_2O decomposition products was measured. The extent of the decomposition was found to be negligible in all cases. The materials tested included metals, metal oxides, and polymers. The materials were inspected for evidence of degradation caused by the 30 day immersion in liquid N_2O, and it was found that three polymers were affected. These were buna-N, neoprene, and an elastomer designated as AF E-124X.

Impact tests of 19 materials immersed in liquid N_2O were conducted at the White Sands Test Facility by NASA (see ref. 13). These tests consisted of placing 1/2 inch diameter specimens in a cup like device, immersing the cup and specimen in liquid N_2O, and dropping a 20 pound (1/2 inch diameter) plummet on the specimen. The energy of the plummet at impact was 367.5 ft-lb/sq in. These tests were conducted in a unique pressurized facility. The results showed that there were no effects to the materials and no problems with N_2O dissociation. The materials tested included metals, alloys, polymers, and grease.

The problems caused by helium solubility in liquid N_2O were discussed in section III. One possible method of avoiding these problems would be to confine the helium in inflatable bladders, thereby eliminating direct contact between the N_2O and the helium. Inflation of the bladders with helium would force N_2O from the tank, and this method would replace direct pressurization with helium. AFWL initiated an in-house program to evaluate potential bladder materials experimentally. The results of this work are reported in reference 9.

Three materials were tested in the bladder program. These were FEP Teflon, AF-E-332 (a silicone rubber), and Arrowhead Rubber AP 2962. The tests consisted of immersing the specimens in liquid N_2O at room temperature for several days and then comparing the mechanical properties of specimens. It was found that the tensile properties and resistance to cyclic strain of both FEP Teflon and AF-E-332 were practically unaffected by exposure to liquid N_2O, but the properties of the Arrowhead Rubber degraded considerably. These tests eliminated the Arrowhead Rubber as a possible bladder material.

SECTION V

OPERATION AND STABILITY OF COMBUSTION

The stability of the combustion of CO, using N_2O as the oxidizer, was investigated by Rocketdyne under contract to AFWL. The results are in reference 14. In this work, a gas generator (combustor) was fabricated based on a two-stage concept, consisting of a primary combustor in which gaseous N_2O was reacted with a mixture of CO and H_2, and a secondary mixer in which N_2 was added to the primary combustion gases. The combustor used an impinging doublet injection of gaseous reactants to produce a high combustion efficiency and temperature and composition uniformity across the flow exit cross section.

Rocketdyne has also completed a program designed to test several of the combustor components. Bomb tests were conducted to demonstrate selected combustor technology. Bomb tests were conducted to demonstrate the stability of the combustor using gaseous CO and gaseous N_2O injection. The igniter operation was characterized by the gaseous N_2O/CO combination. In further work at Rocketdyne, liquid N_2O has been used as an oxidizer and reacted with CO. Over 30 stable tests have been completed and confirmed by exhaust gas sampling of concentrations and temperatures.

The combustion research is still being carried out under contracts with Rocketdyne. In addition, independent programs are being conducted as ongoing IR&D programs by Rocketdyne and by Pratt & Whitney Aircraft.

SECTION VI

CONCLUSIONS

The design considerations of a system for the storage and use of N_2O raised a number of questions which had no available answers. To obtain the answers, experimental programs were either performed or funded by AFWL. These programs have been briefly summarized in this report, with more detailed descriptions of the work included in the referenced documents. This information should be useful for the design of future N_2O systems.

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