SECTION I
INTRODUCTION

At about 1645 hours on Friday, 6 July 1973, a heated, pressurized tank of nitrous oxide (N_2O) exploded and burned on B-8 test stand, where preparations were underway for testing an N_2O-CO laser combustor. Although there were no personal injuries, significant property damage occurred.

A preliminary report has already been prepared and distributed. This final report corrects several small errors in the preliminary report, adds some subsequently obtained data and appends reports prepared by Mr. John Chamberlain on N_2O reaction characteristics, by the P&WA™ Materials Laboratory regarding its analysis of several involved items, and by the Test Department, presenting a detailed description of the damage and test history.

SECTION I
INTRODUCTION

At about 1645 hours on Friday, 6 July 1973, a heated, pressurized tank of nitrous oxide (N_2O) exploded and burned on B-8 test stand, where preparations were underway for testing an N_2O-CO laser combustor. Although there were no personal injuries, significant property damage occurred.

A preliminary report has already been prepared and distributed. This final report corrects several small errors in the preliminary report, adds some subsequently obtained data and appends reports prepared by Mr. John Chamberlain on N_2O reaction characteristics, by the P&WA™ Materials Laboratory regarding its analysis of several involved items, and by the Test Department, presenting a detailed description of the damage and test history.

SECTION II
NITROUS OXIDE PROPERTIES

Nitrous oxide is a colorless, nontoxic gas with a slightly sweetish taste and odor. It is normally shipped as a liquified compressed gas under its own vapor pressure of 745 psig at 70°F. Specific gravity of the liquid is 1.27 and the critical temperature is 97.7°F. It is reported to be stable and comparatively unreactive at ordinary temperatures but begins to decompose at an appreciable rate above about 1050°F. Although one source classes N_2O as nonflammable, another source states that it can form explosive mixtures with air. A number of experimenters report experimental data on the relatively slow reaction between carbon monoxide and nitrous oxide in stoichiometric proportions at temperatures between 960 and 1020°F. At higher temperatures, autoignition and explosion resulted. Another source reports autoignition of the decomposition reaction in N_2O alone at 617°F and 21.4 atm although the delay time is not noted. Impact tests, providing a pressure ratio of 3.66 on N_2O at temperatures up to 400°F, showed some pressure rise from decomposition but no autoignition or detonation. Isentropic compression over a pressure ratio of 3.66 from an initial 400°F would produce a momentary compression temperature of 769°F.

From this mixture of data it appears that N_2O can decompose exothermally and that it has an autoignition temperature near or above 600°F. The large differences in reported results (autoignition at 617°F and decomposition starting at 1050°F) implies that unknown factors such as surface catalysis, purity, pressure, vessel size and heat loss rate may strongly affect the results.

Thermodynamic calculations based on the average of several slightly different heats of formation available in the literature give a temperature rise resulting from complete constant volume decomposition of 3545°F. This rise will provide a decomposition temperature of 3940°F for the initial temperature of 395°F, which existed when the explosion occurred. In a constant volume container, allowing for both increased number of moles of gas as well as the increased temperature, 100% decomposition would produce a pressure ratio of 7.7. With an initial pressure of 1300 psig, this ratio would result in a final pressure near 10,000 psig.

It must be concluded from the available data that N_2O alone (1) can be ignited under certain conditions and (2) has sufficient energy to create substantial damage.

SECTION III
TEST APPARATUS

A schematic drawing of the equipment associated with the explosion is presented in figure 1. The pressure vessel holding the N_2O gas was made of 304 stainless steel, had walls 1-1/2 in. thick and had four access openings. Two 3-in. flanged connections were welded, one each, to the top and bottom of the tank. In addition, the tank had been drilled and tapped at two points for a 1/8-in. pipe thread. An immersion type chromel-alumel thermocouple was mounted in the upper threaded hole and a pressure sensing line was connected to the lower one. Gaseous N_2O was admitted to the tank through a 3/4-in. stainless steel line connected to the top flanged fitting and N_2O was withdrawn from the tank through the bottom flanged fitting. Both the supply line to the top and the run line from the bottom were connected to pressure relief valves set to open at 1375 psig. The tank is reported to have been hydrostatically tested to its working pressure (1800 psig at 70°F) in October 1972.

Surrounding the pressure vessel is a large (about 2,000 gal) bath composed of a 50-50 mixture of ethylene glycol and propylene glycol. This mixture has a flash point of 500°F and a fire point of 600°F. Three electric heaters were mounted, projecting radially inward at 120-deg circumferential positions, well below the tank and run line to heat the bath. The top of the bath was covered with aluminum and a nitrogen blanket filled the space between the glycol mixture and the aluminum cover. In addition to heating the N_2O storage vessel, the glycol mixture was pumped through a trough surrounding the run line to heat it prior to testing. Thermocouples measured run line temperature, bath temperature and N_2O tank temperature. N_2O tank temperatures and glycol bath temperatures always agreed within 5°F but the run line temperature was usually about 80°F lower because of heat losses.

Moving downstream from the tank, the N_2O passed successively through a hand valve, a turbine type flowmeter, a remotely operated pneumatic valve, a screen type (100 mesh) filter and the final control valve. All components of the system were stainless steel except for the thermocouple in the run line which was barewire chromel alumel. The system was completely lox-clean and thoroughly dried and purged before being charged with N_2O.

SECTION IV
OPERATING HISTORY

Two significant changes made this test different from prior tests. The first was the elevation of the N_2O temperature. Prior tests had used N_2O at 220°F and pressures up to 1500 psig. Conditions for this test were set at 400°F and 1300 psig. The other difference was the removal of remote operated valve (ROV) 311 and the substitution of a new valve with a stronger actuator. On prior tests with the old ROV 311, the valve actuator would not exert enough force to overcome the force produced by the tank pressure holding the valve closed. As a result, it was necessary to install a small bypass line and hand valve around the old ROV 311 to equalize pressure on both sides of the valve before it was opened. For this test, a new ROV 311 with a stronger actuator was installed and the bypass line was removed.

Filling the tank with N_2O and heating the bath started simultaneously on 2 July 1973. N_2O was pumped into the tank from small gas cylinders using a Haskel dry pump. Charging the tank was completed at a pressure of 1080 psig and 260°F. Heating operations were interrupted for the Independence Day holiday and resumed on Thursday, 5 July. The time-temperature history of the N_2O tank is presented in figure 2 for the 16+ hours prior to the explosion. The data points indicate a gradual leveling off of the temperature rise rate as would be expected from the increasing heat losses at the higher temperatures. There is no evidence of an increased heating rate that should be evident if any decomposition were occurring. In addition, the measured final pressure of 1300 psig agrees within experimental error with the calculated final pressure of 1290 psig based on the pressure and temperature when filling was completed. However, the tank was vented once to reduce the pressure by 10 psig and there is some evidence that a pressure relief valve might have had a very small leak. In addition to monitoring the pressure and temperature, samples of N_2O were withdrawn from the tank and analyzed for decomposition products, N_2 and O_2. Samples were taken on 3 July at 1330 hours, 220°F; 3 July at 2100 hours, 260°F; 5 July at 0800 hours, 225°F; and 6 July at 1045 hours, 375°F. None of the samples showed any evidence of N_2O decomposition within the accuracy limits of the analysis.

SECTION V
POST EXPLOSION OBSERVATIONS

Although the area showed evidence of both explosion and fire, explosive damage dominated the scene. Except for the cutting torch type of action caused by hot, decomposing N_2O flowing through holes in the tank, fire damage was minor. Figure 3 shows the general area looking from the B-8 stand gate. Helium (left) and nitrogen tube trailers partially block the view of the N_2O tank. In the foreground (right) is an angle iron that formerly supported the aluminum cover and catwalk over the glycol bath. A piece of the aluminum cover is also visible between the fire truck and the blockhouse. Figure 4 shows the catwalk, which weighs about 300 lb and which landed on the other side of the blockhouse. Figure 5 shows the top of the N_2O tank. The hole on top was formerly a 3-in. flanged connection and the hole visible partly down the side of the tank was formerly a 1/8-in. pipe-threaded connection. The thermocouple and pipe fitting, which were blown out of the hole by the force of the explosion were recovered intact. A similar size hole is near the underside of the tank (out of view in the photograph) where the pressure sensing line was formerly attached. The line itself, with the 1/8-in. pipe fitting still attached is visible as the smallest of the stainless steel lines near the top of the bath. The line which was formerly used to supply gaseous nitrous oxide (GN_2O) to the tank is also visible in the photograph. The end of the line formerly connected to the tank clearly shows evidence of rupture from excessive internal pressure. Closeup photographs of the end of the vent line and the pressure and thermocouple pipe fittings are shown in figure 6.

The pipe threads show evidence of being strained as they were forced from the tank by excessive internal pressure, presumably shearing the threads out of the softer tank material in the process, but they do not show any evidence of heat. Even the Teflon tape that was wrapped around the threads as a sealant was not damaged, proving that the fitting temperature never exceeded about 700°F. The main run line is still attached to the bottom of the tank and shows no visible indication of damage other than a small bulge. The flowmeter vanes, however, do show discoloration from excessive heating and the screen located between ROV 311 and CV 1758 has been burned. Neither the flowmeter nor the screen show any evidence of pressure deformation. Both pressure relief valves, which were set to open at 1375 psig, have been damaged. The Teflon seat is completely burned out of one and the metal seat is burned on the other. The N_2O tank itself was shifted within the bath by the propulsive force of the N_2O issuing through the holes.

In addition, the manufacturer's tag, which was welded to the tank at the corners, has had the weld broken. Comparison of the present edge of the tag with the location of the broken weld on the tank indicates that the tank yielded 0.31 in. in 6 in. (5% yield). After the tank is removed from the bath, it will be measured to determine the total amount of deformation. A photograph of the tag is presented in figure 7. The calculated pressure to produce this permanent deformation is about 6000 psig.

SECTION VI
DISCUSSION

The very rapid onset of the explosion without any prior indication of N_2O decomposition plus the occurrence of the explosion immediately after valve actuation very strongly implies that the violent decomposition reaction was ignited by the valve actuation event rather than being the result of a slower decomposition that grew to ignition conditions. The data available in the literature indicate that a rapid decomposition reaction can be ignited at temperatures somewhere above 1000°F. It is postulated that opening ROV 311 allowed hot N_2O to suddenly pressurize the hot (315°F) ambient pressure nitrogen gas trapped in the run line between ROV 311 and CV 1758. Raising the pressure of this nitrogen isentropically to the tank pressure of 1300 psig would increase its temperature to 2050°F, well above the N_2O ignition temperature. The ignited N_2O could then burn back through the run line to the main tank and ignite the bulk of the mixture. The temperature rise resulting from the N_2O decomposition is equivalent to that produced by burning JP-4 and air at a fuel-air ratio of 0.054. If the gas had been a JP-4 air mixture, a similar result would have been expected.

The delay time between valve actuation and explosion can be composed of two parts, (1) the ignition delay time between heating and the appearance of fire, and (2) the time required for the flame front to move from the ignition point back to the tank. The burning rate, S, can be expressed by equation (1)

$$S = \frac{S_{obs}}{1 + (a/A)(3T_2/2T_1 - 1)} \qquad \text{Eq (1)}$$

where S_{obs} is the distance from ignition to tank (47 ft) divided by the time between ignition and explosion, ft/sec

a/A is the ratio of the flame front area to the pipe cross section area - this characteristically lies between 1.1 and 1.4

T_2 is the temperature after decomposition $\sim$°R

T_1 is the temperature before decomposition $\sim$ °R

Using a value for a/A of 1.25 and bracketing the uncertain delay time with values of 1 and 5 sec give lower limit value of S equal to 1.2 fps and an upper limit value of S equal to 6.0 fps. Both these values are reasonable when compared to flame

propagation rates for hydrocarbon-air combustion flames producing a similar heat release.

It should also be noted that a several seconds delay between valve actuation and explosion is characteristic of compression-induced explosions in pressure gage tubing experienced by the writer over 20 years ago when working with N-propyl nitrate (N-P-N). In that instance, the flow of liquid N-P-N compressed air in the pressure gage line, heated it as a result of the isentropic compression, and then was ignited by the hot air. Delays of several seconds were common between the time the flow valve was opened and the explosion occurred.

The burned condition of the screen located between ROV 311 and CV 1758 shows that very high temperature gas existed at that location although there is no proof of whether the burning occurred before or during the tank explosion. The burned screen is, however, consistent with the theory that ignition originated near CV 1758.

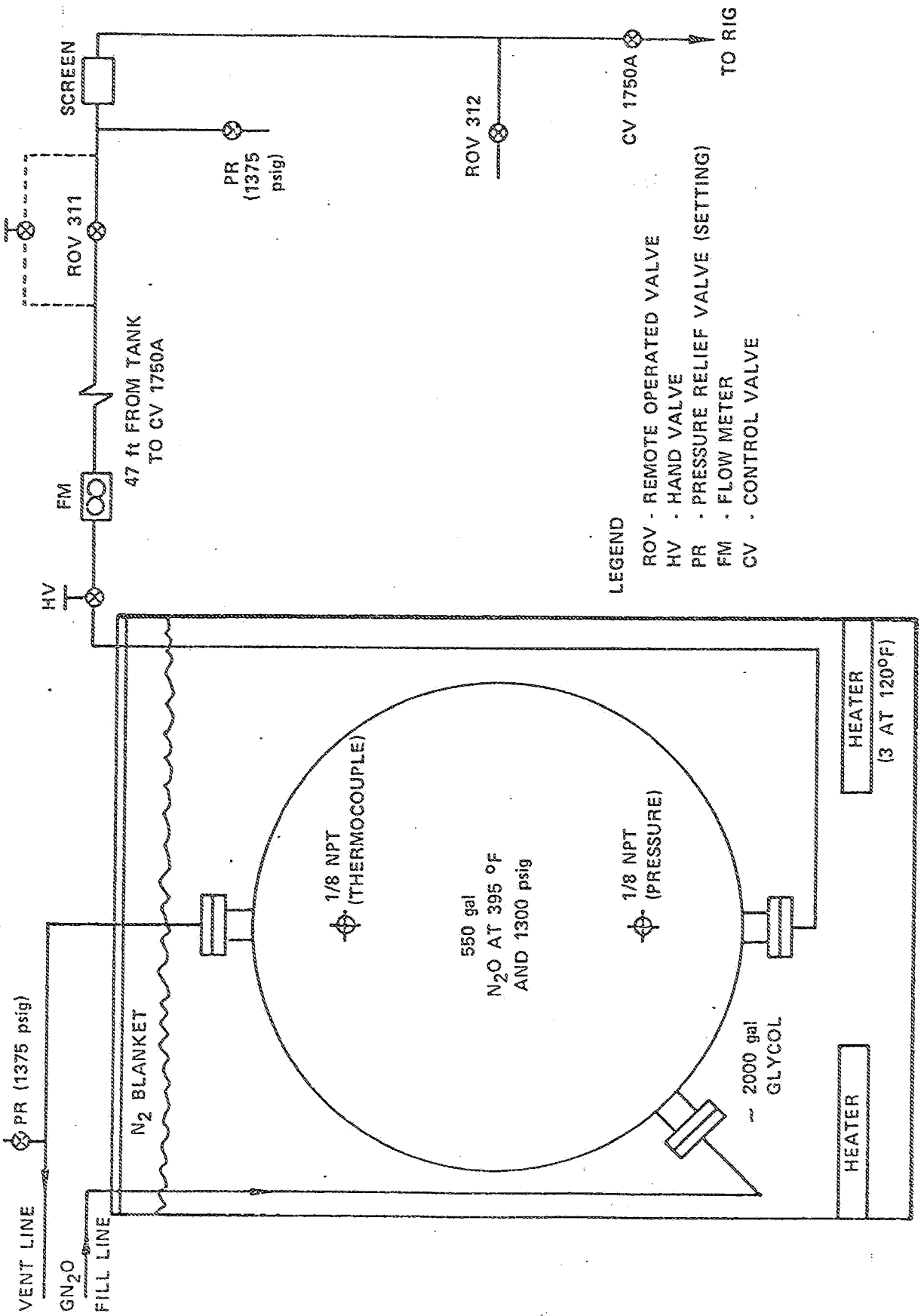
The burned condition of the pressure relief valve connected to the tank top indicates that the explosion was not instantaneous. It appears to have been similar to a conflagration rather than a detonation. Because the vent line was ruptured at the tank by overpressure, yet the valve seat was burned, the tank pressure must have exceeded 1375 psig (the valve opening pressure) long enough to flow hot N_2O through the line to the valve (~0.07 sec) and burn the seat before the pressure reached the value that burst the line. A sample of the line has been hydrostatically tested to 9500 psig without failure, indicating that significant heating of this line must have occurred to have it burst with the pressures theoretically possible.

As an interesting comparison, the energy released during decomposition of 450 pounds of N_2O is slightly greater than that released by the explosion of 1000 pounds of TNT.

SECTION VII
CONCLUSIONS

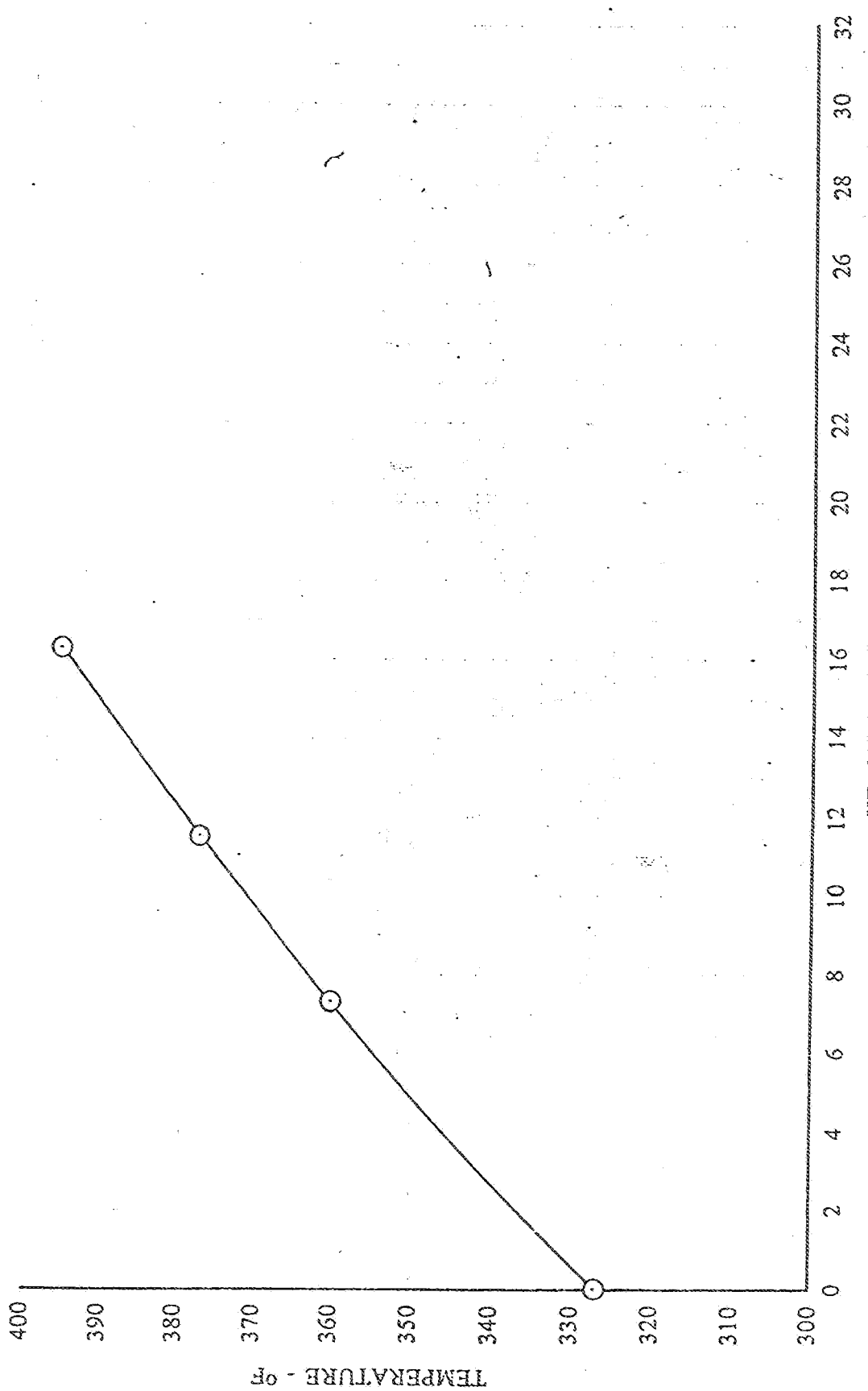
It is concluded that:

1. The explosion was most likely caused by ignition of the N_2O decomposition reaction near CV 1758 by the rapid compression of nitrogen gas trapped between CV 1758 and ROV 311.
2. The explosion probably resulted from a change in operating procedure, i.e., the failure to slowly equalize pressures across ROV 311 through a small bypass line, and is not the direct result of operating at the higher temperature.
3. Although the higher temperature probably did not directly cause the explosion, lower ignition energies are required at higher temperatures. Therefore, increasing the temperature directly increases the hazard.



FD 72157

Figure 1. Equipment Associated With Explosion



DF 96917

Figure 2. Temperature vs Time History of N_2O Tank For 16+ Hours Prior to Explosion

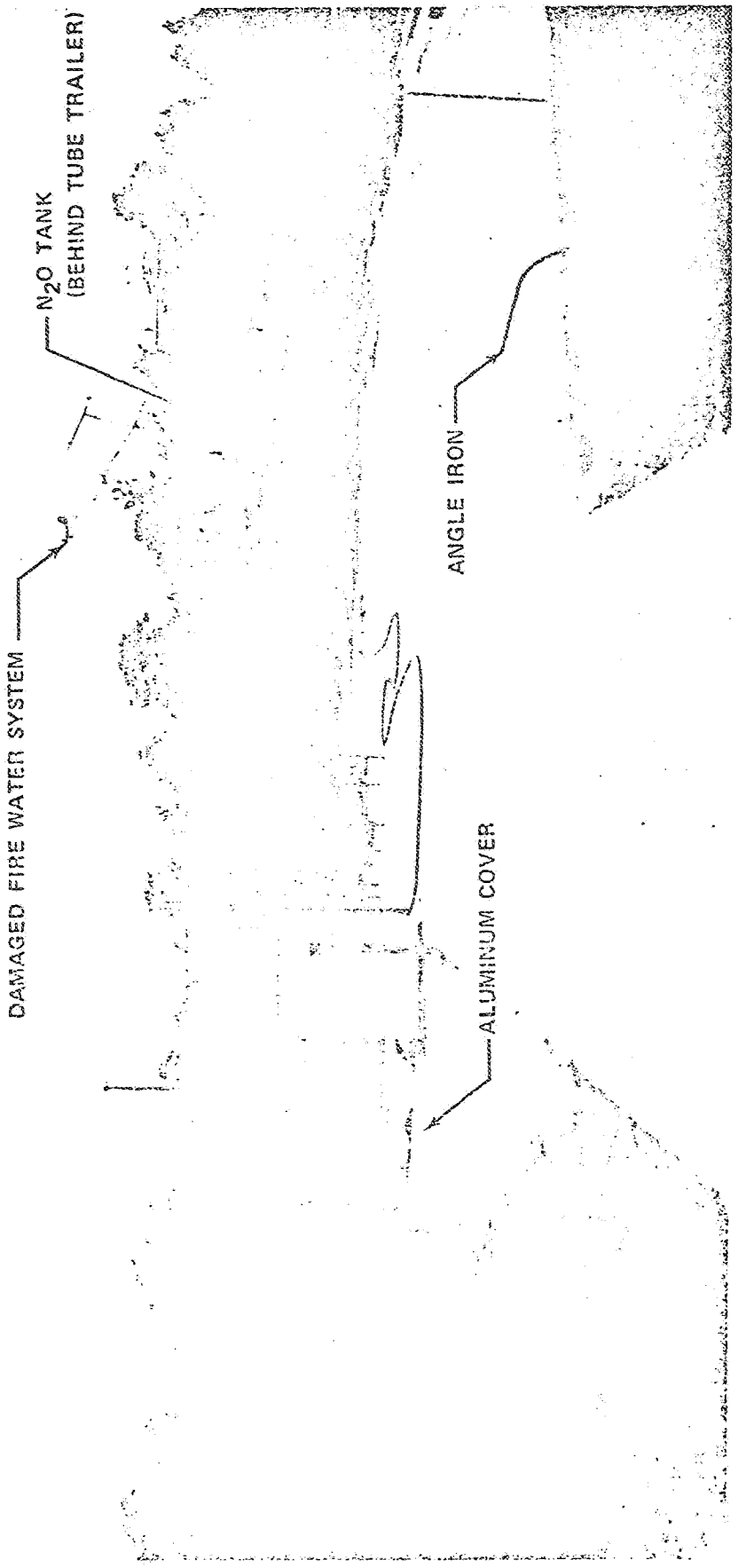
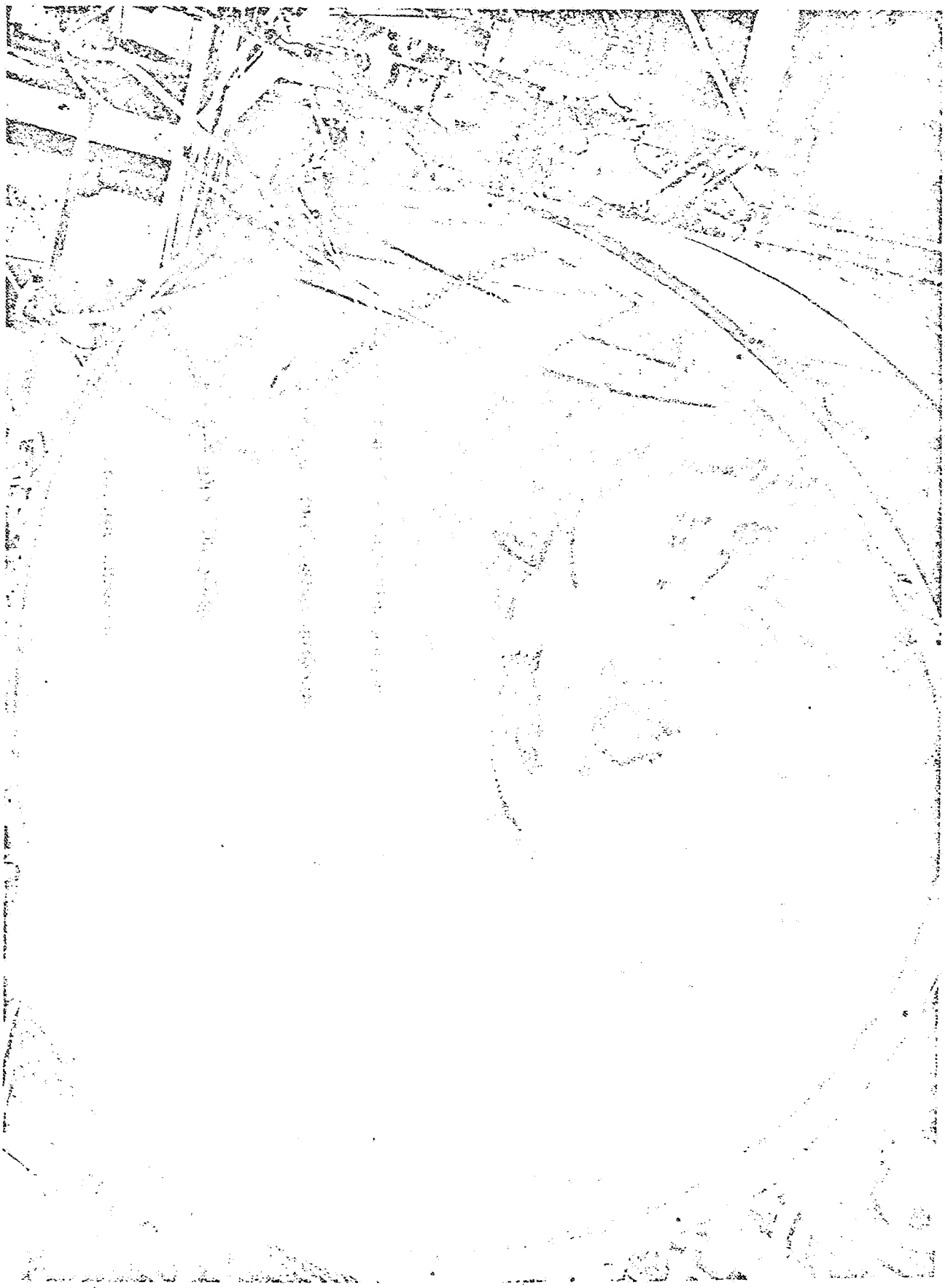


Figure 3. General B-8 Stand Area After Explosion

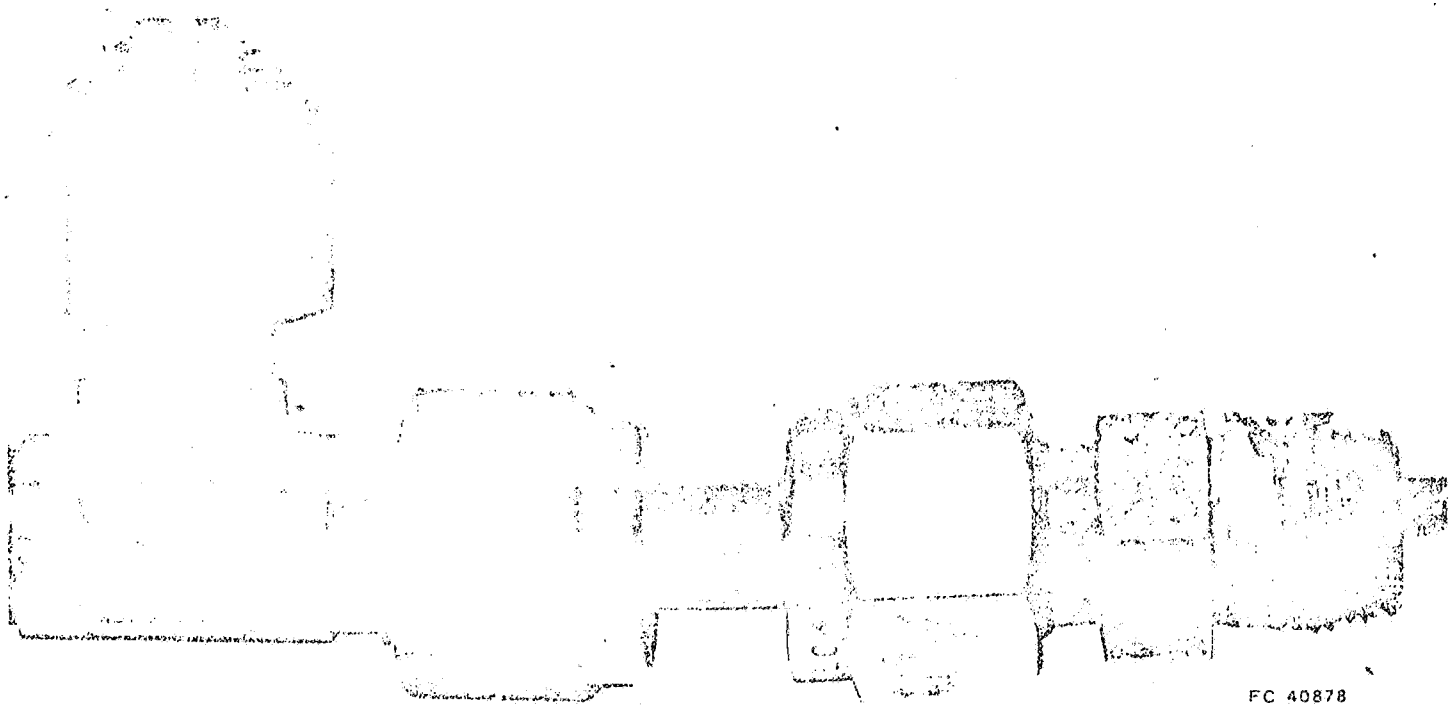
FC 40867

Figure 4. 300-lb Catwalk, Which Landed On Other Side Of Blockhouse



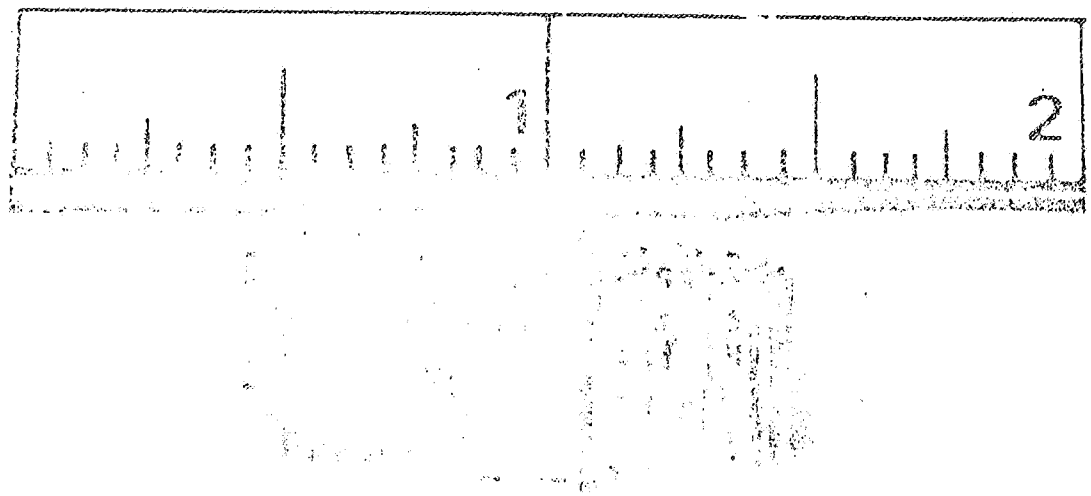
FD 72159

Figure 5. Top of N_2O Tank



FC 40878

THERMOCOUPLE FITTING



PRESSURE GAGE FITTING FC 40879

END OF VENT LINE FC 40877

Figure 6. End of Vent Line and Pressure and Thermocouple Pipe Fittings

FC 40887

Figure 7. Manufacturer's Tag With Broken Weld

INTER-OFFICE CORRESPONDENCE

Pratt & Whitney Aircraft
FLORIDA RESEARCH AND DEVELOPMENT CENTER

UA

To G. D. Lewis Date July 18, 1973

From J. Chamberlain Ext

Subject Preliminary Review of N_2O Reaction Characteristics

cc: W. Alley
E. Bailey
R. Carroll
H. Kobush
A. Masters
J. Matheson
W. Missimer
R. Schmidtke
M. Schilling
C. Staley
G. Zewski

This memo discusses the decomposition characteristics of N_2O as it relates to explosion hazards. It is concluded that:

1. Decomposition of N_2O can produce explosive forces comparable to those that can be produced by burning common fuel-air mixtures. Theoretical pressure ratios from decomposing N_2O gas can exceed 10 to 1 in closed volumes.

2. N_2O is orders of magnitude less reactive than common fuel-air mixtures. This makes it hard to achieve any significant reaction at ambient pressure, but at high pressures, such as 1,000 psia, it can be ignited easily and decomposition "flames" will burn through it, much as common fuel-air mixtures can be ignited and will display propagating flames at atmospheric pressure.

3. We need more information on the effects of pressure, temperature, and perhaps materials on the ignition energy required to ignite N_2O and the quenching distances which will allow or stop the reaction.

Theoretical Decomposition

As noted below, the theoretical heat release due to the decomposition of N_2O ($2\ N_2O \rightarrow 2\ N_2 + O_2$) is 800 BTU/lb, which is about 2/3 as large as the theoretical heat of combustion of a stoichiometric mixture of any common hydrocarbon fuel, such as JP or gasoline with air. Consequently, the theoretical Δt is very large, though less than that of gasoline-air. The heat release per unit volume (or per mol) is about the same, however, because N_2O is denser than air. As a liquid, or near the critical point, the heat release per unit volume is higher. The similar (or higher) heat release per mol leads to similar (or higher) expansion ratios of the gases due to burning, as shown below. Thus, the potential pressure rise or explosion force from N_2O decomposition is as great (or greater) than that from burning hydrocarbons and air.

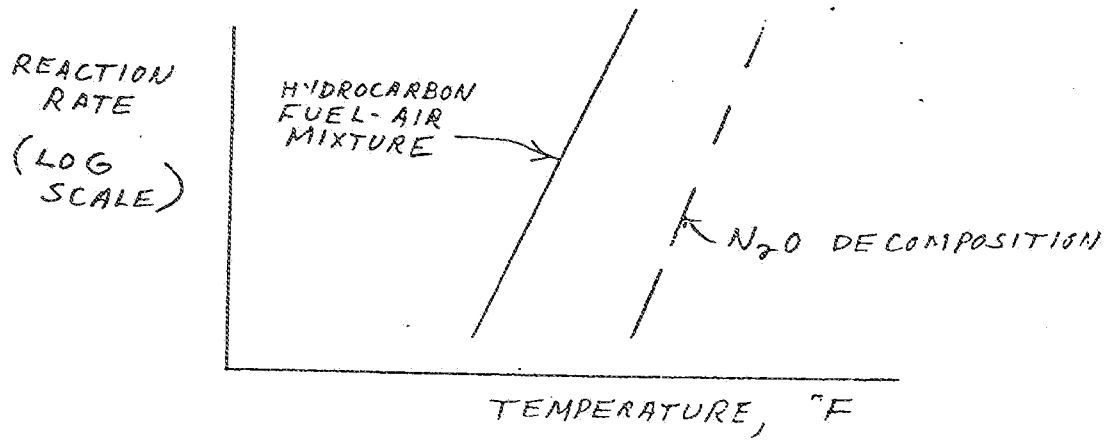
	N_2O	Stoich. Gasoline-Air Mixture
Heat of Reaction	800 BTU/# (92 BTU/STD Cu.Ft.)	1,260 BTU/# (96 BTU/STD Cu. Ft)
ΔT (Initial T = 140°F)	2,800 °F	3,500 °F
Mols of Products Per Mol of Initial Gas	1.5	1.07
Volume of Products Per Unit Volume of Initial Gas	8.5	7.3

$$= \left(\frac{T_2 \times MOLS_2}{T_1 \quad MOLS_1} \right)$$

The theoretical pressure ratio due to the N_2O decomposition (or fuel-air burning) in a constant volume container is somewhat greater than the above volume ratios, typically by 30 or 40%. The exact value is a function of the initial pressure and temperature, but can easily exceed 10. Actual values are less than the theoretical values by an amount which depends on the combustion efficiency and the heat losses to the walls during the reaction. Note that even if there are large heat losses and the reaction is incomplete, pressures in a tank of N_2O which decomposes could easily rise by a factor of 5 times or perhaps more, thus creating dangerous explosive forces. If the tank then leaks or ruptures, very hot, highly oxidizing gases are sprayed about, providing both the oxygen and the heat for ignition to cause rapid combustion of any nearby combustible materials, or even of the tank materials. Thus, both severe explosion and fire hazards are presented if rapid decomposition occurs, so rapid decomposition must be avoided.

Actual Chemical Reaction

N_2O decomposition, like hydrocarbon-air reaction, proceeds at a rate which increases exponentially as temperature increases, but the N_2O reaction rates are very much lower at any given temperature:



As with hydrocarbon-air mixtures the rate of reaction is so low at low temperatures that the heat generated is lost to the surroundings, and the temperature of the N_2O fails to rise much. However, if the temperature is raised high enough, the reaction rate becomes sufficiently rapid so that the heat generated exceeds losses to the surroundings, and the reaction "takes off"; once some part of the mixture gets this hot, that part decomposes (or burns) rapidly until it approaches completion, and the very hot products heat and ignite the adjacent gas. A flame front is thus formed which propagates through the mixture. N_2O decomposition flames are visible. Ordinarily, these would progress slowly through the N_2O (feet/sec) but if the rate of reaction is high enough and the length of pipe is high enough, detonations can take place, as observed in Ref. 1. Either way, high temperature and pressure increases can be caused in enclosed volumes.

Another measure of combustibility which is directly related to the above reaction rates is "ignition lag time", the time which exists after a mixture is heated to a given temperature before it appears to burst into fire. Theoretically this could be computed from known reaction rates. Fig 1 presents some data. Though data on N_2O is very sketchy and hydrocarbon-air ignition lag times vary with the fuel used and the measuring technique, the N_2O ignition lags are clearly orders of magnitude larger than those of hydrocarbon-air mixutres. Perhaps 1000 to 1 is the approximate ratio. Put another way, N_2O required 600° to 800°F higher temperature for a given ignition lag time.

Very little ignition lag data exists on the lag times beyond 10 seconds or so, and practically none beyond 100 seconds. If the reaction rates are so slow that the lag times should be longer, most the heat generated in the gas is lost to the vessel, and the gas temperature never rises high enough to cause ignition.

Chemical reaction rates and ignition lags are a function of pressure. R. Carroll states that it has been determined that the N_2O reaction, like gasoline-air combustion, is a 'second-order' reaction. This means that the rate of reaction increases with the square of the pressure level. Since the mass in a given volume increases with the first power of pressure level, the % of a volume which reacts per second should increase in proportion to the pressure level. Thus, the rate of temperature rise should be proportional to the pressure level and the ignition lag should be proportional to 1/pressure level.

At pressures of the order of 1,500 psia, the ignition lag of N_2O obtained by extrapolation of the above data would be about 10,000 seconds at 400°F. Since lag times below 10 to 100 seconds would be required for spontaneous ignition, there would seem to be no risk of spontaneous ignition at these conditions. It would appear that at 1,500 psia, spontaneous ignition would become likely at about 800 psia. These values are for stagnant gases in tanks. In flowing systems, the heat transfer to walls is greatly increased and spontaneous ignition would be suppressed to far higher temperatures.

Flame Propagation

Assuming that the bulk of the N_2O is not heated to high temperatures, rapid decomposition can only occur through 1) ignition and 2) propagation of the

reaction through the N_2O. Consequently, an understanding of these phenomena is of great importance. For propagation to occur, the already-reacted gas must conduct enough heat into the next element of unreacted gas to ignite it before too much heat is lost to the walls. Thus, the speed of the reaction, the temperature reached by the reaction, and the diameter of the duct determine whether the fire will be propagated or quenched.

With N_2O, the reaction rates at low temperatures are of the order to 1/1000 of that of hydrocarbon-air mixtures, and the maximum temperatures reached are more limited, so the average and peak reaction rates must be lower than these normally associated with atmospheric pressure hydrocarbon-air mixtures by a factor of the order of 1000 times. A Stoichiometric methane-air mixture will just barely propagate a flame through a 0.1" dia tube at 1 atmosphere pressure and 60°F. The marginal duct diameter for propagation presumably varies inversely as the pressure varies. If it is assumed that the above 1000-to-1 ratio of reaction rates exists and that reaction rate times duct diameter is a constant, then one would conclude that 1,500 psia N_2O would require about a 1" duct diameter for propagation of decomposition. This "guess" value may be substantially in error, but it is obvious that N_2O decomposition is vastly more easily quenched than hydrocarbon-air flames, and that one could stop propagation of an N_2O decomposition by installing a tube bundle of sufficiently small diameter tubes in an N_2O line. The tubes may not have to be extremely small. At atmospheric pressure, the tube diameter may have to be extremely large, such as 100", for a reaction to be initiated and propagated.

Ignition gets rapidly easier as pressure is increased, so even though it may be nearly impossible to ignite N_2O at atmospheric pressure, it should be easy to ignite in sufficiently large pipes at high pressure. Probably the most likely source of ignition in such a system would be rapid compression of some gas in a line when a valve is opened, admitting high pressure gas to a closed line previously at low pressure. This is a very powerful ignition source, and is presumed to be the cause of ignition in B-8 stand. At high pressures, where relatively little ignition energy is required, ignition might also be caused by electrical spark (spark plug), static electricity discharges resulting from high velocity flow, mechanical sparks due to valve parts rubbing or pump parts rubbing, heat from compression, heat from hot surfaces (such as rubbing pump parts), or conceivably from catalytic activity. Again, ignition becomes more and more likely as pressure is increased, and more and more care is required.

If the pipe or volume where an ignition source might exist is small enough, ignition will not occur and there is no hazard. Similarly, one can prevent the the spread of decomposition and the related pressure rise by inserting a tube bundle of sufficiently small tubes in the line, as discussed before. Both ignition energy required and critical pipe diameter are increased if temperature is decreased, but we do not know how powerful this effect is. With hydrocarbon fuel-air mixtures, a change of 200° in gas temperature only changes these 10 or 20%.

Tests

To refine our evaluation of possible hazards in N_2O systems of various designs, it is strongly recommended that tests be made to determine the approximate

energy required to ignite N_2O at various pressures and temperatures and the tube diameter above which propagation is possible at these temperatures and pressures. These tests could be done quite simply using lengths of pipe fitted with a spark plug and a pressure gage. Initial tests could be made with the pipes at ambient temperature and later tests with the pipe heated to 400°F or so. Some variations in pipe material or wall coating should be made to check for catalytic effects.

It may be desirable to run a few tests with a bundle of tubes inserted into a larger tube to prove that this "flame arrestor" will really stop a flame propagation which approaches it.

John Chamberlain

John Chamberlain

REFERENCES

1. E. Stokes Fishburne and Rudolph Edse, "Chemical Kinetics of Nitrous Oxide Decomposition at Elevated Pressures and Temperatures," USAF Aeronautical Research Laboratories report ARL 62-430, prepared by Ohio State University, Sept, 1962.

2. Richard E. Thomas, "Experimental Study of the Use of Nitrous Oxide in a hypersonic Wind Tunnel", USAF Aeronautical Research Laboratories report arl 62-420, prepared by Ohio State University, September, 1962.

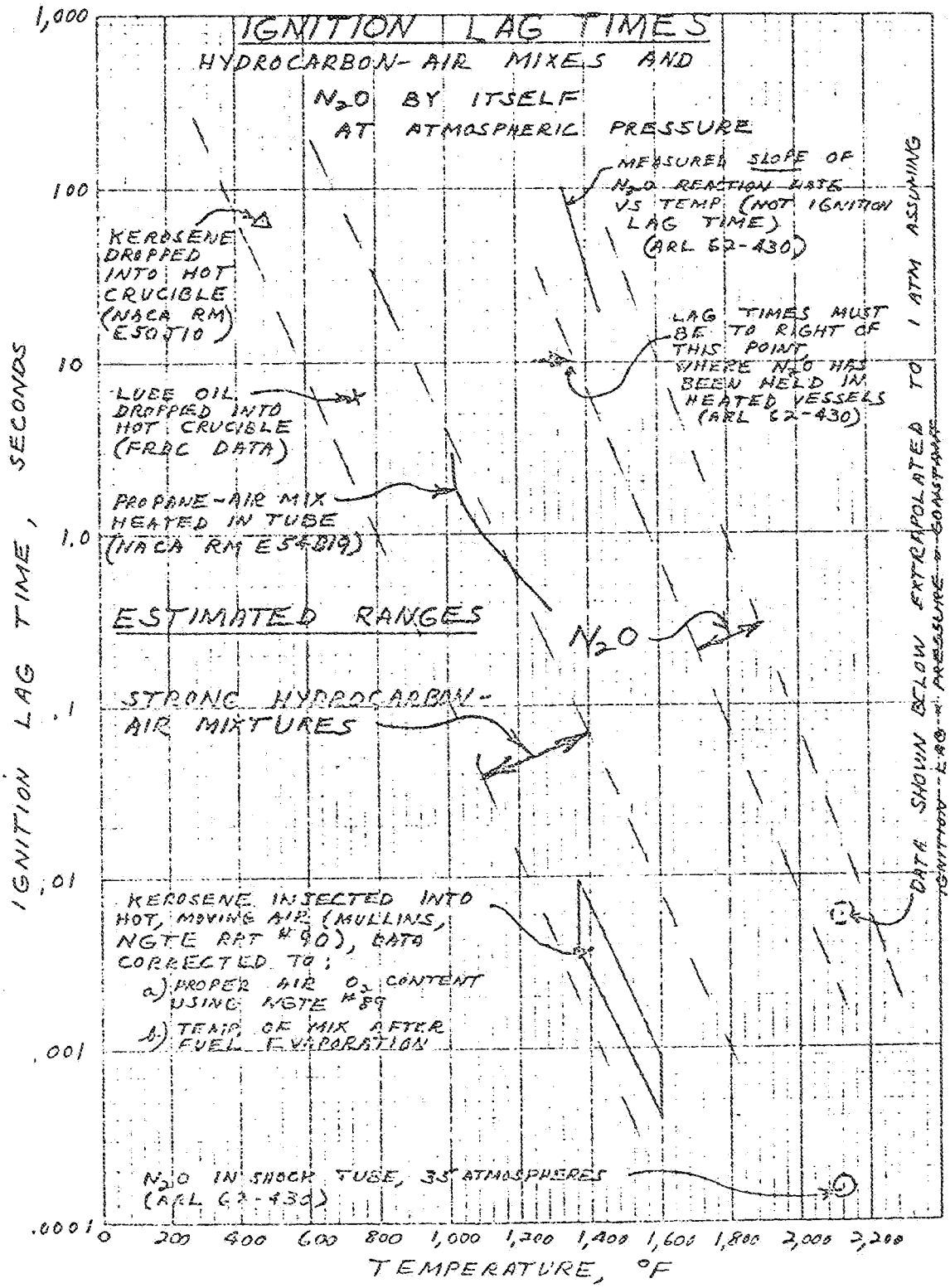
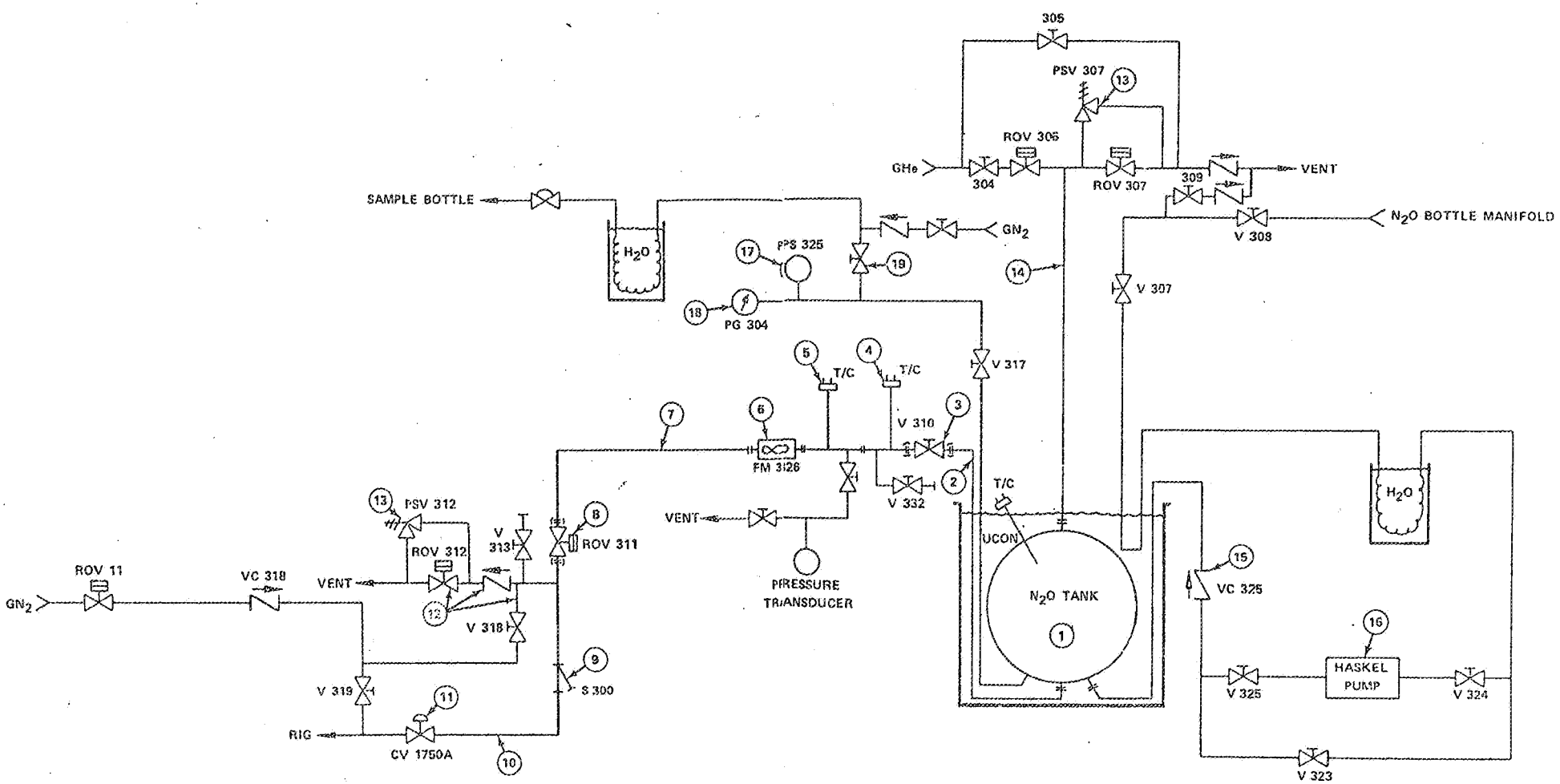

IGNITION LAG TIMES
HYDROCARBON-AIR MIXES AND
N_2O BY ITSELF
AT ATMOSPHERIC PRESSURE
MEASURED SLOPE OF N_2O REACTION RATE VS TEMP (NOT IGNITION LAG TIME) (ARL 62-430)
KEROSENE DROPPED INTO HOT CRUCIBLE (NACA RM E50J10)
LAG TIMES MUST BE TO RIGHT OF THIS POINT WHERE N_2O HAS BEEN HELD IN HEATED VESSELS (ARL 62-430)
LUBE OIL DROPPED INTO HOT CRUCIBLE (FRDC DATA)
PROPANE-AIR MIX HEATED IN TUBE (NACA RM E54B19)
ESTIMATED RANGES
N_2O
STRONG HYDROCARBON-AIR MIXTURES
KEROSENE INJECTED INTO HOT, MOVING AIR (MULLINS, NGTE RPT #90), DATA CORRECTED TO:
a) PROPER AIR O_2 CONTENT USING NGTE #89
b) TEMP OF MIX AFTER FUEL EVAPORATION
N_2O IN SHOCK TUBE, 35 ATMOSPHERES (ARL 62-430)
DATA SHOWN BELOW EXTRAPOLATED TO 1 ATM ASSUMING IGNITION LAG ∝ PRESSURE ^ CONSTANT
IGNITION LAG TIME, SECONDS
1,000
100
10
1.0
.1
.01
.001
.0001
0
200
400
600
800
1,000
1,200
1,400
1,600
1,800
2,000
2,200
TEMPERATURE, °F

FIGURE 1.

ATTACHMENT NO. 4 - TMR 8216



FD 72161
730208

Pratt & Whitney Aircraft
FLORIDA RESEARCH AND DEVELOPMENT CENTER

Lab. No. 18102

Part No.

Account No. 1128-47-852-62

Engine Type Laser

MATERIALS DEVELOPMENT LABORATORY
WORK REQUEST AND REPORT

Requested by W. Trowsdal Department Test Operations

Send Reports to R. Rankin, T. Pruitt, G. D. Lewis, J. Chamberlain, E. Pinsley, N. Bennett, S. Devine, D. Smith, F. Regali, F. Hahn, G. Brown

Part Name Oxidizer Tank Mfg Source Pottstown Metal Products Works

Ident & Serial No. Mfg. S/N 5676 / S/N 3057 Matl Source

Material & Process Specs 304 Stainless Heat Code, Lot, or Heat No.

Engine No. B-8 Stand Part Time

Operation Data Tank was pressurized with Nitrous Oxide to 1300 psig and 400F when tank failed.

Work Requested Investigate the cause of tank failure.

REPORT:

See attached sheets and tables.

Reported by P.Klier, R. Morgan, R. Meehan Approved by [signature] Date Released 7/20/73
(dk)
Figures:

PWA 1003c REV 5-63

CONCLUSION:

The exact cause or causes of the system failure was not determined from the study made. Since more work with nitrous oxide (N_2O) is predicted, further testing with this propellant system should be performed by MDL.

This program includes a scaled down system simulation of the stand's test condition using an ultra clean system and the evaluation of the effect of possible contaminants in this system.

Also, a thorough study of hardware efficiency should be made to determine the strength of the system fabrication and combustion characteristics of N_2O.

INTRODUCTION:

The nitrous oxide system at B-8 stand was almost at required test conditions when a catastrophic failure occurred. The tank was 40OF and 1300 psig pressure with the two-inch run line at approximately 37OF. The run line was pressurized downstream as far as valve ROV-311. The line from ROV-311 to CV-1750A had been maintained at ambient pressure following the initial pressure check of the system.

Four samples of N_2O from the run tank had been taken from the pressure readout line and analyzed by gas chromatography. See data in Table I. The results of these analyses produced no evidence of decomposition or disassociation. The slight scatter in oxygen and nitrogen content is well within the limits of experimental error and sampling technique.

In an effort to equilibrate temperatures in the run line prior to test, hot gas from the tank was admitted to the non-pressurized segment of line. Approximately 5 seconds after the ROV-311 valve signal light indicated an Open position, system failure occurred. The 3" line at the top of the tank and both 1/8" threaded fittings to pressure and temperature readouts failed resulting in large holes in the tank wall.

Some damage occurred in the 2" run line; the Teflon seat of valve ROV-311 disintegrated and the stainless screen filter exhibited a few localized areas of ignition. The ball bearing assembly of the flow meter was rendered inoperable due to the complete burnout or disintegration of the bearing cage. See Table II for composition of the bearing cage material.

REPORT:

The cause or causes of failure are not known although there are several suspect areas in the N_2O system. Examination of the hardware of B-8 stand and subsequent analyses of some objects found in the system are described in Table II.

There were three separate failures of the oxidizer tank, but the sequence of failures or possibility of simultaneous failures was not determined.

The areas encompassing a 3" long neck flanged fitting and two 1/8" pipe threaded fittings ruptured causing a catastrophic failure of the tank. Rapid release of the heated pressurized gas produced high temperatures which was followed by the ignition of the UCON 50-HB-280X oil used as the system heating medium.

Analyses of the N_2O gas prior to tank failure produced no evidence of decomposition so it may be assumed that the system was stable as regards the contained oxidizer.

Pressurization of the run line may have caused an abnormally high temperature resulting from rapid compression, to propagate a thermal or shock wave of sufficient force to cause structural failure of the system.

The presence of UCON oil on the inner threads of a pipe plug adjacent to a screen filter should be considered as a possible start of reaction. UCON oil at ambient temperatures and pressures is considered compatible with N_2O, but information is limited on the compatibility under high temperature and pressure conditions.

It is known, from the appearance of the filter screen, that ignition did occur at this location in the system. The presence of small fragments of screen wire a considerable distance upstream of the filter indicate a tremendous back surge of pressure toward the tank. This pressure surge is further substantiated by the presence of particles, identified as a type of fluorocarbon, in the valve body of HV-310.

There was no pressure or temperature data available to substantiate the extent of pressure and temperature rise immediately prior to failure. A considerable pressure surge must have occurred to cause the structural failure as the system had been pressure tested prior to this failure.

Metallurgical examination of a segment of the tank shell showed no evidence of intergranular attack which, if present, could possibly account for failure at the top of the tank.

Figure 1 depicts an enlarged view of the screen wire fragments similar to the inner screen of the run line filter.

Table I

Analyses of Nitrous Oxide from B-8 Run Tank

Sample	Time & Date	Tank Temperature, F	Tank Pressure, psig	Sample Composition	
				Oxygen + Argon, ppm	Nitrogen, ppm
1.	1330 hrs, 7/3/73	--	900	93	1505
2.	2100 hrs, 7/3/73	260	900	56	1635
3.	0800 hrs, 7/5/73	225	850	35	1455
4.	1045 hrs, 7/6/73	375	1300	54	1578

Table II

Analyses of Deposits and Materials from B-8 Stand System

Relative Location of Sample Specimen	Specimen Analysis and Observation
1. Inner screen from run line filter.	Fe/Cr, Ni (Elements in descending order of concentration). Similar to 302 or 304 S.S.
2. Outer housing of run line filter.	Fe/Cr, Ni (Elements in descending order of concentration). Similar to 302 or 304 S.S.
3. Metal chips caught by run line filter.	Fe/Cr, Ni, Cu (Elements in descending order of concentration).
4. Black magnetic particles removed from valve bodies of RLV-311 and CV-1750A. Some of the particles appeared to be segments of fine screen mesh.	Fe/Cr, Ni, Mo (Elements in descending order of concentration).
5. White plastic appearing particles and black magnetic particles from valve body of old HV-310 valve. Black particles similar to magnetic particles from RLV-311 and CV-1750A.	Infra-red analysis of white particles displayed absorption bands common to fluorocarbons. Could possibly be Teflon. Spectro analysis of black particles Fe, Ni/Cr (Elements in descending order of concentration).
6. Very small white particles and black slightly magnetic particles from weld cap of old HV-310 valve.	Insufficient quantity of white particles for analysis. Spectro analysis of black particles Fe, Ni/Cr, Mo (Elements in descending order of concentration).
7. Two 1/8" - 27 NPT S.S. adapters from N_2O tank shell. One for pressure readout and one for temperature.	Fittings made from 300 series stainless, threads were not destroyed and still retained the Teflon tape used for sealant.
8. Segment of tank shell adjacent to area of 3" 1500# RTJ long welding neck flange where one tank failure occurred.	Metallurgical examination revealed no evidence of intergranular attack.

Relative Location of Sample Specimen	Specimen Analysis and Observation
9. Model 2½ 5118-B flowmeter mounted in 2" run line. Ball bearing assembly in flow meter suffered complete loss of cage material and discoloration of impeller blades.	A ball bearing assembly of same type as used in flow meters was examined to determine cage material composition. Spectro analysis - Al, Mg Cr/B, Na, K, Cu, Si, Fe, Zr, Sn, Pb (Elements in descending order of concentration).
10. 2" stainless pipe plug located adjacent to in-line screen filter which suffered burn-through in several areas.	The plug threads were contaminated with UCON oil.

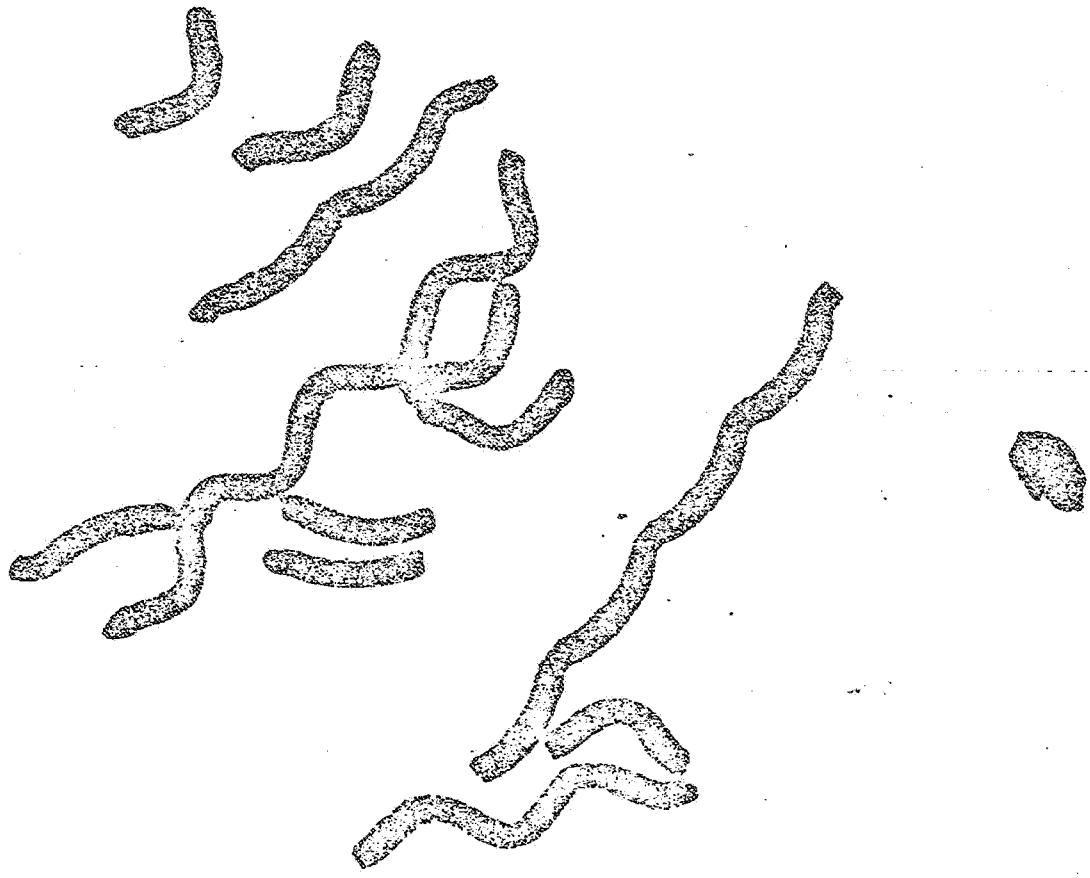


Figure 1. 300 Series Stainless Steel Screen Wire Residue From Valves RLV 311 and CV 1750A FAE 131127

TEST MALFUNCTION REPORT

Number 8216 Interim

Date 7-20-73

Test Stand B-8

Rig/Engine Number 33817-12

File Number 8.8

Test Number 93.01

Type of Program:

R & D, Advanced Combustor

Component or System which Malfunctioned:

Hot nitrous oxide system.

Description of Malfunction:

Two to five seconds after the N_2O run line block valve (ROV 311) was opened to pressurize the line up to the flow control valve (CV 1750), there was a loud explosion followed by fire that covered the test stand and control room. The stand operator closed ROV 311, turned on the fire water and called the Fire Department. One-half hour later all of the fire on the stand and all of the brush fires around the stand were extinguished. A fire hose was required to put out the fire inside the glycol tank as the fire water manifold directly over the tank was damaged by the explosion.

Inspection of the test stand revealed the explosion originated from inside the N_2O run tank and/or its surrounding heated glycol tank. The glycol tank top was blown off and scattered over the test stand. Most of the contents of the glycol tank (2000 gallons of UCON 50-HB-280X) were blown out thru the top of the tank. The burning glycol then rained down over the stand.

The N_2O run tank had 3 round flame-cut holes in it. These were at the two 1/8" NPT taps on the side and where the 3" - 1500 lb. long neck flange was welded on the top (the flanged assembly has not been found). The 1/8" pipe tap 45° from the top was burned out to a 6" diameter hole, the 1/8" pipe tap 30° from the bottom was burned out to a 2 1/2" diameter hole and the 3" flanged nozzle was burned out to a 4" diameter hole. The tank shifted inside the glycol tank with enough force to bend the support legs on the N_2O tank and elongate the glycol tank. This movement was away from the holes in the side of the tank. The N_2O tank, a spherically shaped tank 62" I.D x 1 1/2" wall thickness, was expanded 5% in circumference which indicated a severe overpressurization.

The N_2O run line and surrounding glycol trough was intact with glycol still in the trough.

Conditions of the test stand at the time of the explosion were as follows:

1) The N_2O run tank was pressurized to 1300 psig with gaseous N_2O at 395°F. The tank was being heated to 400°F in preparation for the first test of this rig mount. This was the first time the tank had been heated above 240°F.

2) The pre-run stand countdown was complete up thru pre-run purge, BDR cals and valve and abort checks.

3) N_2O run line glycol trough temperature was 310°F.

4) N_2O run line temperature at the unjacketed flowmeter section was 250°F.

A summary of equipment damage is as follows:

1) The paint is discolored and burned on B-5, 7 and 8 stands, the control room and on tube trailers 471 and 472.

2) 55' of black iron fire water pipe over the glycol tank - broken and bent.

3) 2 stand light fixtures - broken.

4) 50' of 3/4" electrical conduit - bent.

5) 200' of electrical wire - burned.

6) 4' of 6" flex duct on input building pressurization blower - burned.

7) 3 transducers - overheated and shorted out.

8) 90 instrumentation cables - burned.

9) Weather van propeller and cable - burned.

10) Glycol tank top - the portions of it that have been found are bent.

11) N_2O run tank and run line - 5% permanent elongation of the metal.

12) N_2O flowmeter bearings.

13) Plastic valve seals in ROV 311, PSV 307 and PSV 312 - missing, cracked and eroded (respectively).

14) N_2O run line screen - burned.

15) 20' of 3/4" SS tubing, 20' of 1/2" SS tubing, and 20' of 1/4" SS tubing and fittings on N_2O tank vent and pressure gage lines ruptured and bent.

16) Three 12 KW Chromalox immersion heaters (for glycol bath) - bent.

17) 2000 gallons of UCON 50-HB-280X - burned.

18) 500 lbs. of N_2O - burned

The advanced combustor rig which was mounted in the stand test position was not damaged.

Immediate Action Taken:

The N_2O and glycol systems were inspected externally and nothing was found that could have caused the explosion. The entire N_2O system was inspected internally and evidence was found of fire inside the run line as far down the line as the screen between ROV 311 and CV 1750. The source of the overpressurization or fire could not be positively determined. A detailed report of the external and internal system inspections and a chronological sequence of events leading up to the malfunction is attached (attachments 1, 2 & 3 respectively).

The Materials Lab was requested to run an analysis of particles found in the system, the flowmeter bearing cage material, the screen material, and a one foot square specimen of the N_2O tank material to determine if there was a catalyst for N_2O decomposition present in the system. MDL aslo inspected the tank, runline, and the fittings that blew out of the 1/8" pipe taps in the tank. A MDL report is to be issued on what was found.

Project Engineering has conducted an independent investigation to determine the cause of the explosion. The preliminary report, written by Mr. G. D. Lewis, dated 7-10-73, concludes that the N_2O decomposed rapidly and ignited due to isentropic compression of the gasious nitrogen in the pipe downstream of ROV 311 when it was opened. A final report will be written by Mr. Lewis after all other reports are complete.

After completing the inspections and reviewing the sequence of events leading up to the malfunction, it was determined that there were additional possible causes that should be considered. They are as follows:

1. Even though there was no evidence of dissociation from samples taken from the N_2O tank during heatup (last sample at 1045 hrs. 7/6/73), the fact remains that from 0730 hours on 7/6/73 until the malfunction occurred at 1637 hours temperature continued to increase, at a decreasing rate, but pressure remained constant at 1300 psi. This was probably due to the relief valve leaking. The tank was vented at 0730 hours from 1310 psi to 1300 psi. The relief valve is located close to the vent valve and could have been affected by flow in the line while venting the tank. The valve was found to be leaking at 1400 hours on 7/6/73. This clouds the issue, somewhat, as to whether or not there was any dissociation of the N_2O in the tank prior to the malfunction.

2. The screen downstream of ROV 311 may have been contaminated. There is no way to prove or disprove this other than that the system and all components were cleaned and maintained Lox clean. This screen had not been removed for cleaning since October of 1972 when the hot N_2O system was installed. It is a parts catcher type screen to protect the rig and is not inspected unless upstream valve seat or flowmeter problems occur. Any contaminants stopped by the screen could have been ignited by the temperature rise due to compression of the ambient pressure gas (GN_2 and possibly some N_2O) resulting from the opening of ROV 311. There also was no sign of heat downstream of the screen.

3. The high velocity, high temperature flow over the screen wire may have heated it to the ignition temperature of N_2O.

4. The bare wire c/c thermocouple upstream of the flowmeter may have acted as a catalyst. This thermocouple is in a section of unjacketed pipe and was at 250°F prior to opening ROV 311. Opening ROV 311 would flow 400°F N_2O past it.

5. The flowmeter bearing cage material may have burned or acted as a catalyst. It is not known at this time what the cage material was. The cages are missing from both bearings. This material could have been burned out after ignition occurred.

6. The rubbing of ROV 311 plug against the body may have raised the temperature locally above the ignition temperature of the N_2O, however, there was no sign of heat in this area. Airco has reported compressor explosions when pumping bone dry N_2O to 1200 psig and have eliminated the problem by adding a soapy water solution to the N_2O.

7. UCON may have leaked into the line downstream of ROV 311. This section of line is immersed in the glycol trough and is left vented when not testing. The fittings on the line were pressure checked at ambient temperature, but may have leaked when heated to 310°F. The temperature rise from compression of GN_2 when ROV 311 opened could have heated the UCON to its spontaneous ignition temperature. This temperature is above 700°F in air according to the literature. The temperature at which spontaneous ignition would occur in high pressure N_2O is not known.

Long Range Action to be Taken:

1. Project Engineering is in the process of defining programs required to determine completely the behavior of nitrous oxide, what effect various materials have on nitrous oxide, and what effect nitrous oxide has on various materials over a range of pressures and temperatures. A better understanding of nitrous oxide is required prior to rebuild of B-8 stand or any other nitrous oxide system.

2. The B-8 N_2O system will be redesigned with a much greater regard for system and personnel safety.

T. M. Pruitt
S. L. Devine

cc: Chief, Experimental Test Laboratories
Chief, Facilities Engineering & Design
Area Supervisor
Senior Operations Engineer
Operations Engineer
W. C. Missimer
S. Bonifazi
G. D. Lewis
E. P. Granberry
Foreman

ATTACHMENT #1

N_2O AND GLYCOL SYSTEM EXTERNAL INSPECTION

External inspection showed the following:

1. The entire N_2O run line and glycol trough were intact from HV 310 to the rig. There was no evidence of N_2O leakage from the line and the trough still had glycol in it.

2. The N_2O run tank had 3 round holes in it having a flame out appearance. These were at the two 1/8" NPT pipe taps on the side and on the top where the 3" - 1500 lb. flange was welded on. The 1/8" NPT tap 45° from the top, used for the tank C/A immersion type thermocouple, was burned to a hole 6 inches in diameter. The 1/8" tap 30° from the bottom which had been the pressure gage tap was burned out to a hole 2 1/2" in diameter. The hole in the top was not cut past the O.D. of the pipe welded in it. The tank surface around this hole is badly eroded indicating flame had deflected off the bottom surface of the 3" - 1500 lb. flange prior to its blowing off. A small area of the remaining pipe is rolled back and shows a clean break indicating the flange separated from the tank at this location. The pressurization gas distribution tee welded inside the tank under this flange and both halfs of the 3" flange assembly have not been found.

   There are short hairline cracks over the entire top half of the sphere. The circumference of the tank has expanded from 206 inches, measured on an identical tank on B-5 stand, to 212 inches (just above the weld). The tack welds on the left side of the vendors name tag are broken and have a 1/4" gap in them. Shiny unoxidized tank surface previously under the tag now extends 5/16 inches past the tag on the left side. The two tack welds on the right side of the tag did not break. This was 5/16 inches elongation over the 6 1/2 inch tag width.

3. The N_2O run tank shifted to the south inside the glycol tank with enough force to bend the N_2O tank legs and elongate the glycol tank. This movement was away from the 2 holes burned on the side of the tank. It is not known if the tank movement was due to a glycol explosion or the reactive force of the burning N_2O flowing through the holes in the tank.

4. The 3/4" 0.049" wall 304 SS pressurization/vent tube connected to the top of the tank was ruptured and badly eroded on the ruptured end.

5. The 1/4" 0.035" wall SS tubing connected to the 1/8" NPT pipe tap 30° from the bottom of the tank was blown completely out of the glycol tank and coiled up. The 1/4" AN to 1/8" pipe thread adapter that screwed into the tank was still on the end of the tubing and showed no sign of damage. The teflon tape on this fitting was not discolored.

6. The Haskel pump discharge tube (1/2" - 0.035" wall 304 SS) to the 2" - 900 lb. flange 30° from the bottom of the tank was still connected to the tank and was not damaged.

7. The 2" run line was still connected to the 3" - 1500 lb. flange at the bottom of the tank. There was no sign of hot gas leakage at this flange.

8. The N_2O tank thermocouple was found on the stand with the 1/8" pipe fitting still attached to it. The threads on this fitting are rolled back indicating it blew out of the tank. The fitting is not burned. The thermocouple is bent but not burned.

9. The aluminum glycol tank cover has a slight amount of black discoloration and is bent and gouged in an area which was directly over the tank top flange. This indicates there was flame hitting the aluminum at this location for a short period of time prior to the flange hitting it when the explosion occured.

10. The south side of the inside of the glycol tank has black flame impingement marks at the same elevation as the N_2O tank top. This indicates flame came out of the tank top and deflected off the 3" - 1500 lb. flange prior to the flange blowing out.

ATTACHMENT #2

N_2O SYSTEM INTERNAL INSPECTION

Since nothing could be concluded from an external inspection of the system, other than it had seen severe overpressurization, an internal system inspection was conducted. The items inspected are numbered on the attached system schematic, (Attachment #4).

Results of this inspection are as follows:

1. N_2O tank - A one foot square section of the tank top was cut out and sent to MDL for an intergranular corrosion test. This test showed no intergranular corrosion. The inside surfaces of the tank are not corroded or eroded. There is some discoloration from the UCON solution that leaked through the hole burned in the bottom after the fire. Ref. Photos.

2. The run line from the tank to HV 310 was cut at each elbow, the old HV 310 was cut out of the pipe and the cap was cut off old HV 310 bonnet. Several chunks of white teflon (largest about ¼ X 1/8") and several pieces of magnetic material that looked like screen wire were found in the old HV 310 body and bonnet. (Sent to MDL for analysis). The globe type valve body (old HV 310) is badly discolored on the rig side of the valve and slightly discolored on the tank side. The valve seat is not eroded. The discoloration is bluish/black indicating high temperature gas went through it towards the tank. The pipe is not discolored except in the horizontal section under the N_2O tank where the glycol/firewater solution ran in it after the fire. (Ref. Photo FC 40890.)

3. Removed bonnet from HV 310. No particles or discoloration were found except the bottom of the gate was brown indicating high heat. Reference Photo FC 40951.

4. C/A thermocouple downstream of HV 310 was discolored bluish/black and bent slightly back towards the tank. The T/C is a 1/8" immersion type with an immersion depth of 1 3/4". The thermocouple sheath is either stainless or inconel. (Ref. Photo 40952).

5. Bare wire C/C thermocouple upstream of the flowmeter is discolored bluish/black. The T/C is 1/8" diameter with an immersion depth of 1/2". All of the T/C material is intact. (Ref. Photo FC 40953).

6. Potter flowmeter - (2½" PWF-3) was disassembled in the Instrument Lab. A small piece of white teflon was found in the meter prior to shipment to the lab. The flowmeter blades and flow straighteners were not damaged. The bearing cage material was missing from both ball bearings, but all of the balls were still in the races. The tank side of the flow straighteners and blades were discolored bluish/black indicating hot gas flow thru the meter towards the rig. The rotor hub was discolored yellowish/green. The meter was sent to MDL for analysis of discoloration and bearing cage material. Ref. Photos FC 40959, 40960, 40961, and 40962.

7. The run line from the flowmeter to ROV 311 was discolored bluish/black at the 3" to 2" reducer. No particles or other discoloration was seen.

8. Removed and disassembled ROV 311. Found numerous magnetic metal particles in the body. Some looked like screen wire and some looked like slag. The teflon stem seat ring was missing and the lock wire (304 SS) on the 4 bolts on the end of the plug was slightly brown. There is a galled mark on one side of the plug and a matching line on the valve body(parallel to stem movement) indicating rubbing when operating. The particles were sent to MDL for analysis. Ref. Photo FC 40874.

9. Removed run line screen element. Found the wire mesh and perforated back-up metal burned through in two places on the downstream side and the wire mesh burned in one place on the upstream side. The screen wire is 50 x 50 mesh, plain square weave 302 or 304 stainless steel. The back-up metal is 1/32" thick, 304 or 304 series stainless with four 3/16" diameter holes per inch. The wire screen and the back-up metal are both discolored (black). There is no indication of screen damage due to excessive flow. The screen housing contained many magnetic pieces of screen wire and slag from the burned screen, which were sent to MDL for analysis. (MDL reported that stainless steel can become magnetic when it is burned). There was no damage to the screen housing or signs of heat in the housing. The plug threads were contaminated with UCON, but this could be expected since the entire housing was submerged in UCON. The copper/teflon seal between the plug and screen housing showed no signs of overheat, burning or damage. There was also no damage or sign of heat on the screen near the top where the screen is in contact with the plug.

   The screen had not been removed for cleaning since October of 1972 when the hot N_2O system was installed. It is a parts catcher type screen to protect the rig and is not inspected unless upstream valve seat or flowmeter problems occur. Ref. Photos FC 40954, 40955, 40956, 40957.

10. Removed the glycol trough from the section of pipe between the screen and CV 1750 and cut the pipe upstream of CV 1750 and at the screen. There was no discoloration or particles in the pipe.

11. Removed the bonnet from CV 1750 and found several small pieces of teflon in the body. There was no discoloration in the body or on the copper plug. The pieces were sent to MDL for analysis.

12. Removed ROV 312, check valve V 313, and the 1/2" tubing from the run line to these valves. Found no discoloration in the valves or tubing. ROV 312 was not damaged.

13. Removed run line and tank relief valves and sent them to R/S to check the relieving pressure. Both valves had been set at 1375 psig the week of June 25, 1973. The 1/4" run line relief lifted at 875 psig. Disassembled it and found the Kel-F seat eroded, but no discoloration. The tank relief lifted at 1550 psig. Disassembled it and found a strip of the teflon seat chipped off and black discoloration of the brass body and plug. The tank relief was leaking thru prior to the explosion as indicated by condensation dripping off the valve and the 3/4" tubing to the valve being warm to the touch. This was observed 2 1/2 hours prior to the explosion.

14. Cut the tank pressure/vent tubing in several places. It is eroded internally back from the tank 7" and is discolored black inside (approximately 48" from the tank). This indicates burning N_2O flow through the tube towards the relief valve prior to the tube blowing off the tank. (Ref. Photos FC 40877, FC 40875).

15. Disassembled Haskel pump discharge check valve and found no damage or discoloration.

16. Disassembled Haskel pump. (It was valved off from the tank at the time of the explosion and was not in operation). Found no evidence of damage or anything that might have come from, or through, the pump to cause the N_2O to ignite or decompose.

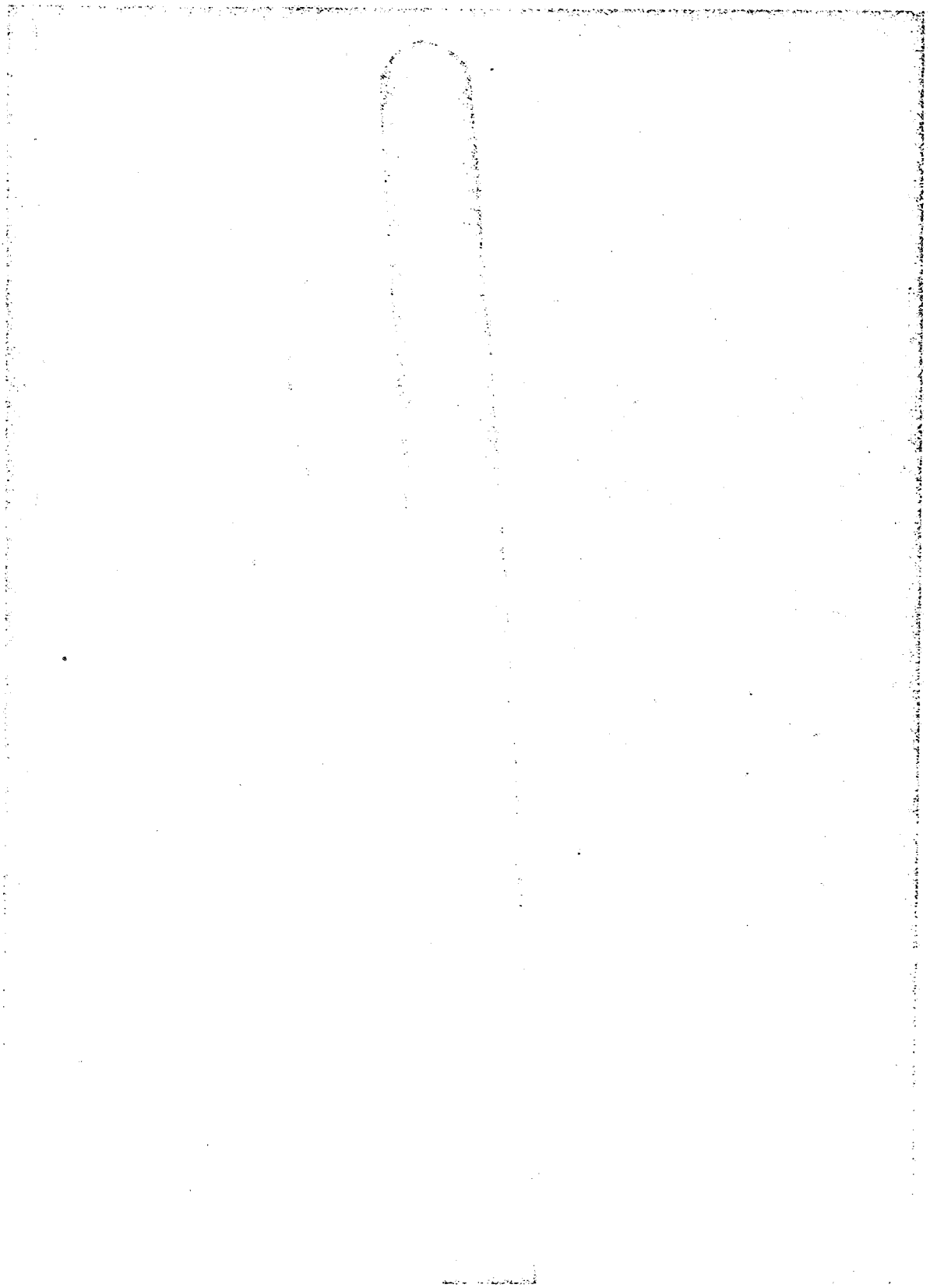
17. Checked pressure switch PS 325 in Lab and found nothing wrong with it.

18. Tracked N_2O tank pressure gage and found it read 25 PSIG high at 1200 and 1400 PSIG. The gage is mounted in the control room window with over 100' of tubing between it and the tank.

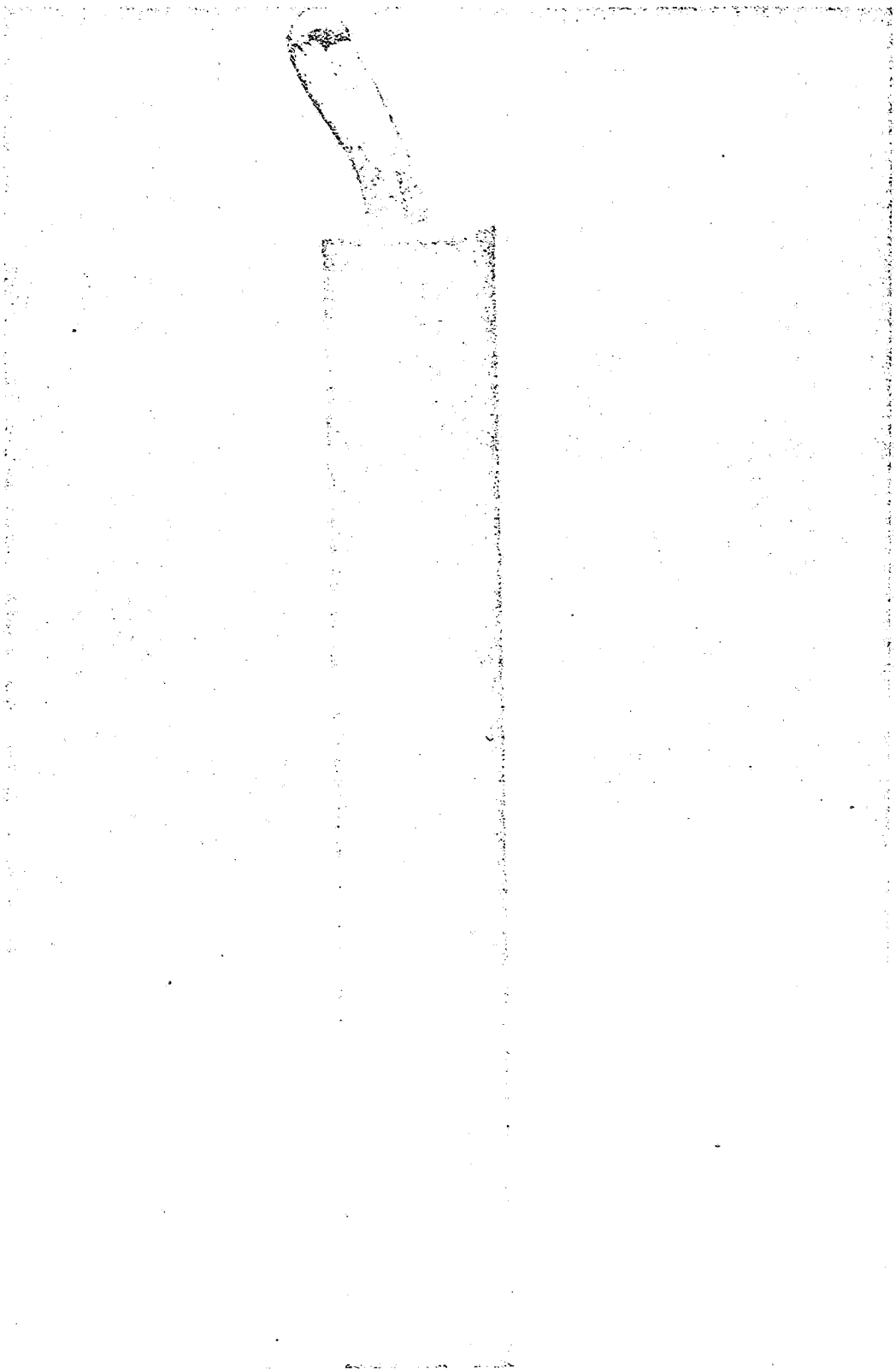
19. The N_2O sample system was valved off when the explosion occured and has not been disassembled.

FC 40890. Old HV310

FC 40951. Bonnet From HV310



FC 40952. Thermocouple Sheath



FC 40953. Bare Wire C/C Thermocouple

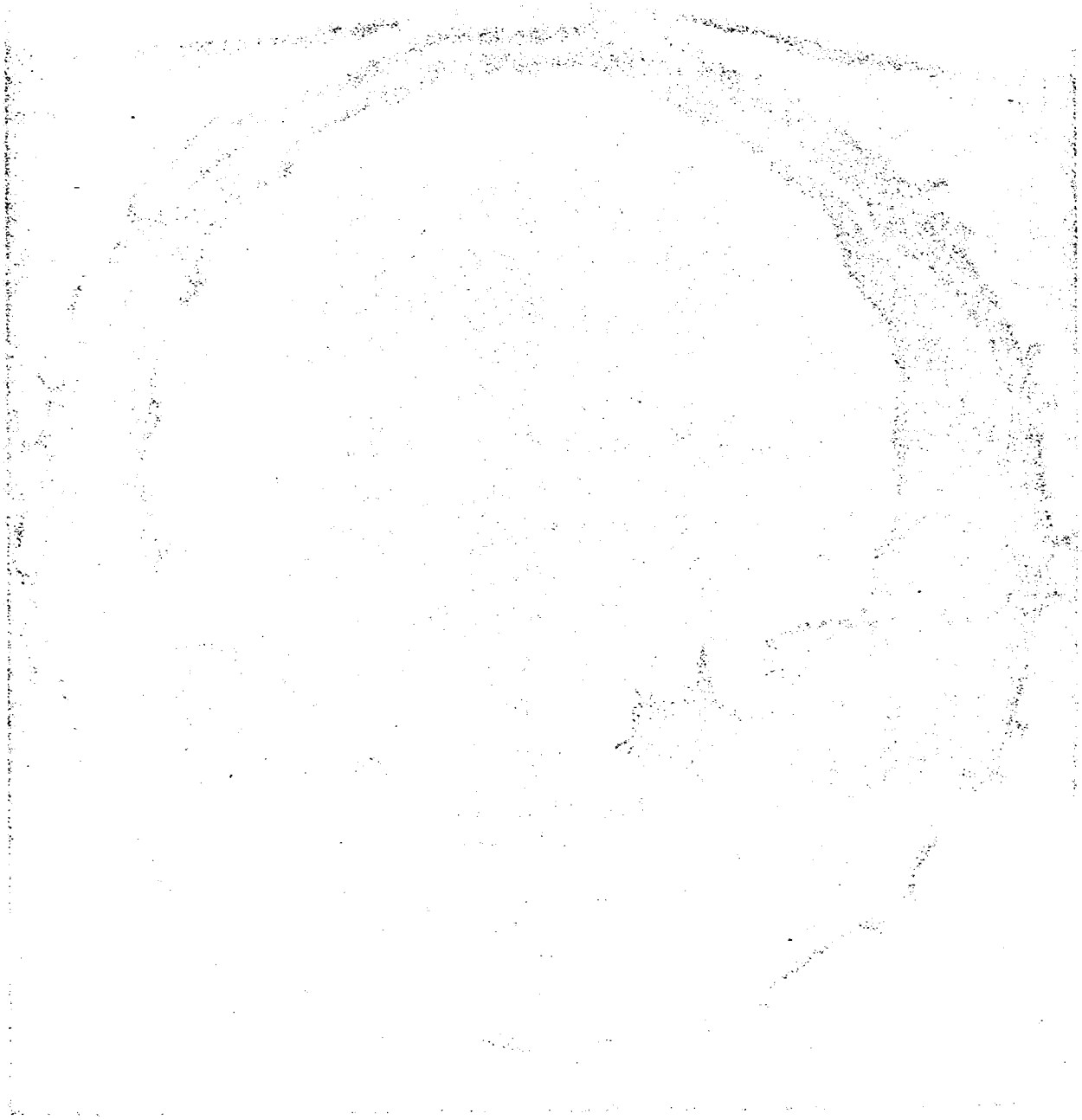
FC 40959. Potter Flowmeter Assembled

FC 40960. Potter Flowmeter Disassembled

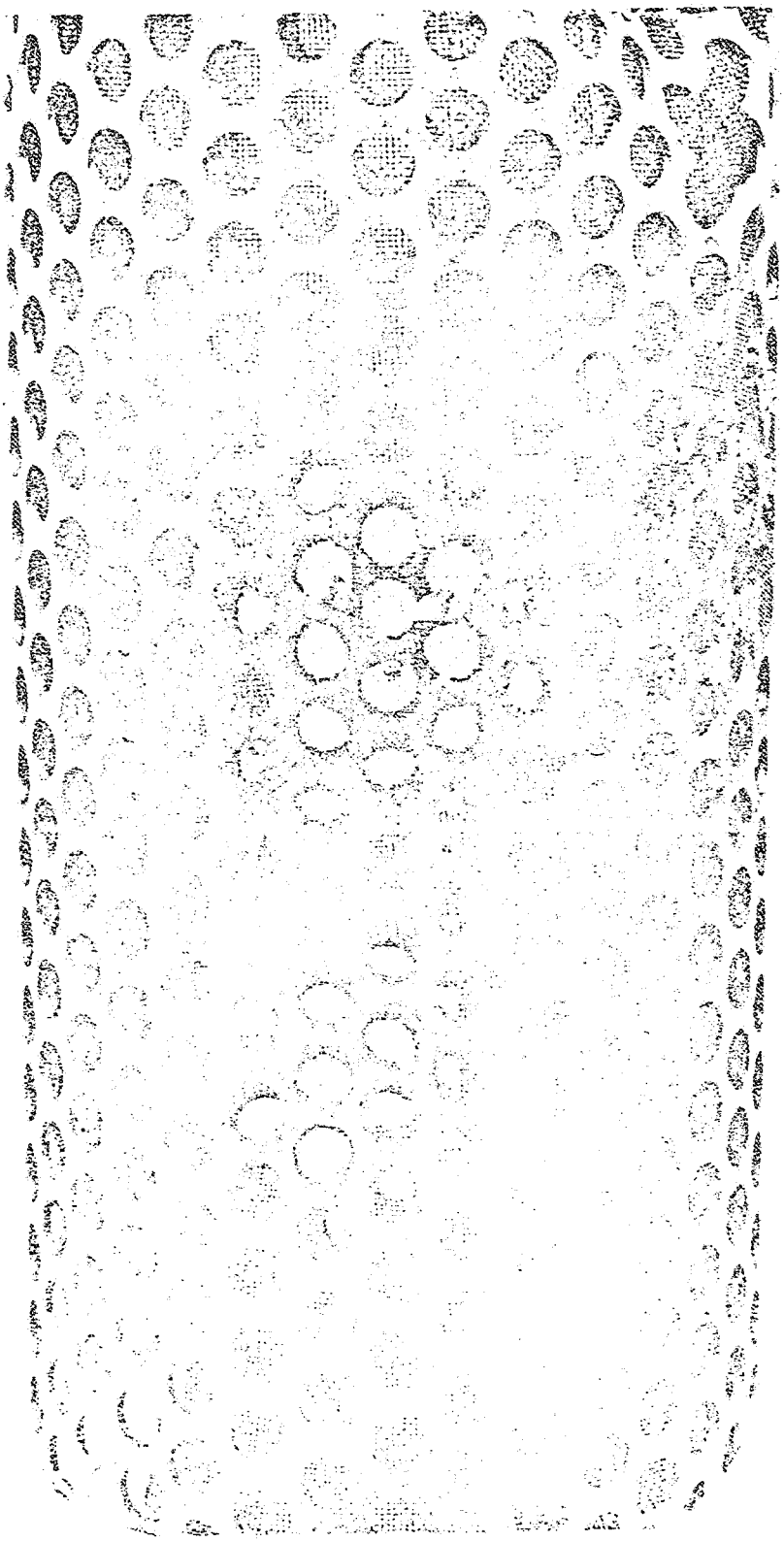
FC 40961. Potter Flowmeter Blades (View 1)

FC 40962. Potter Flowmeter Blades (View 2)

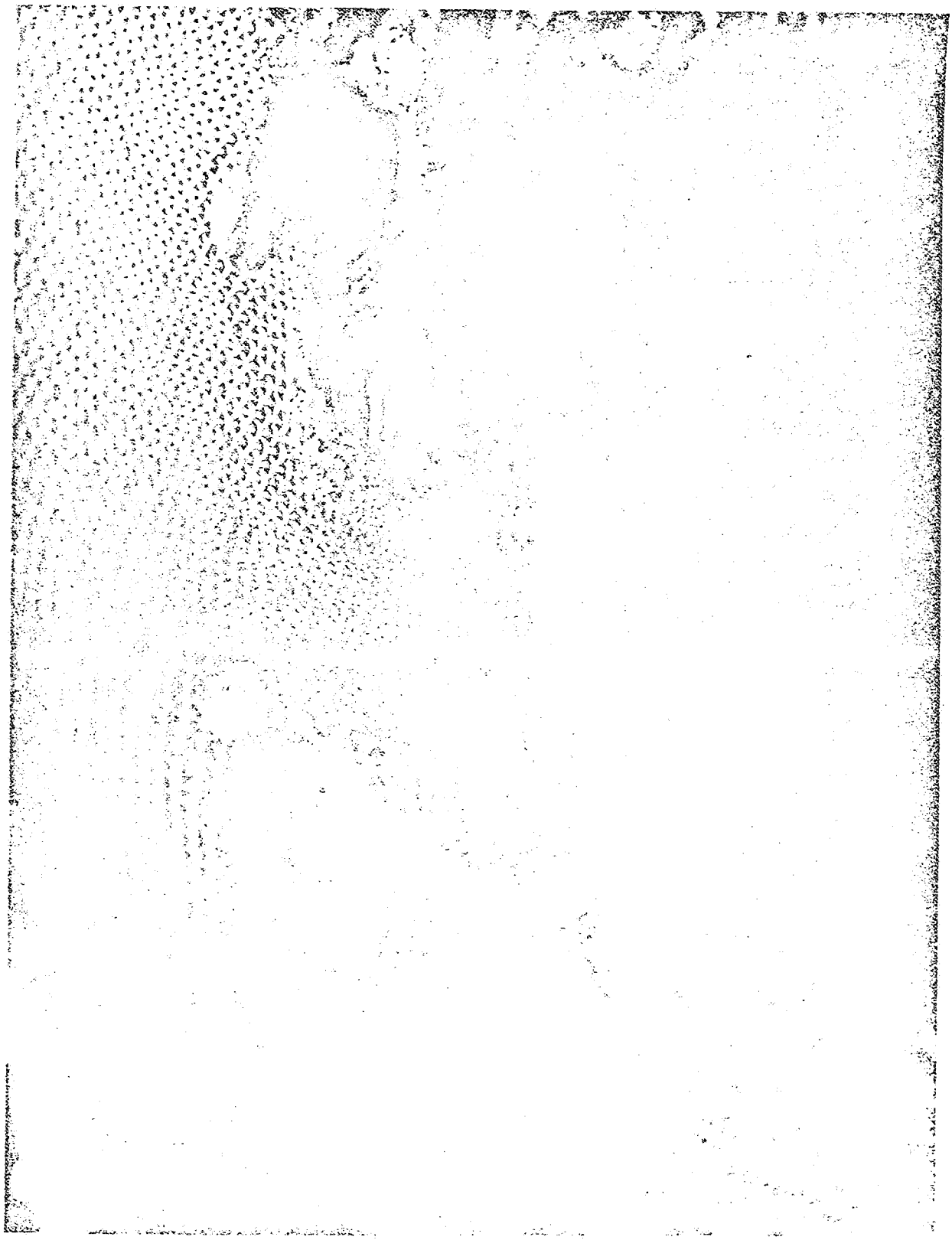
FC 40874, ROV 311



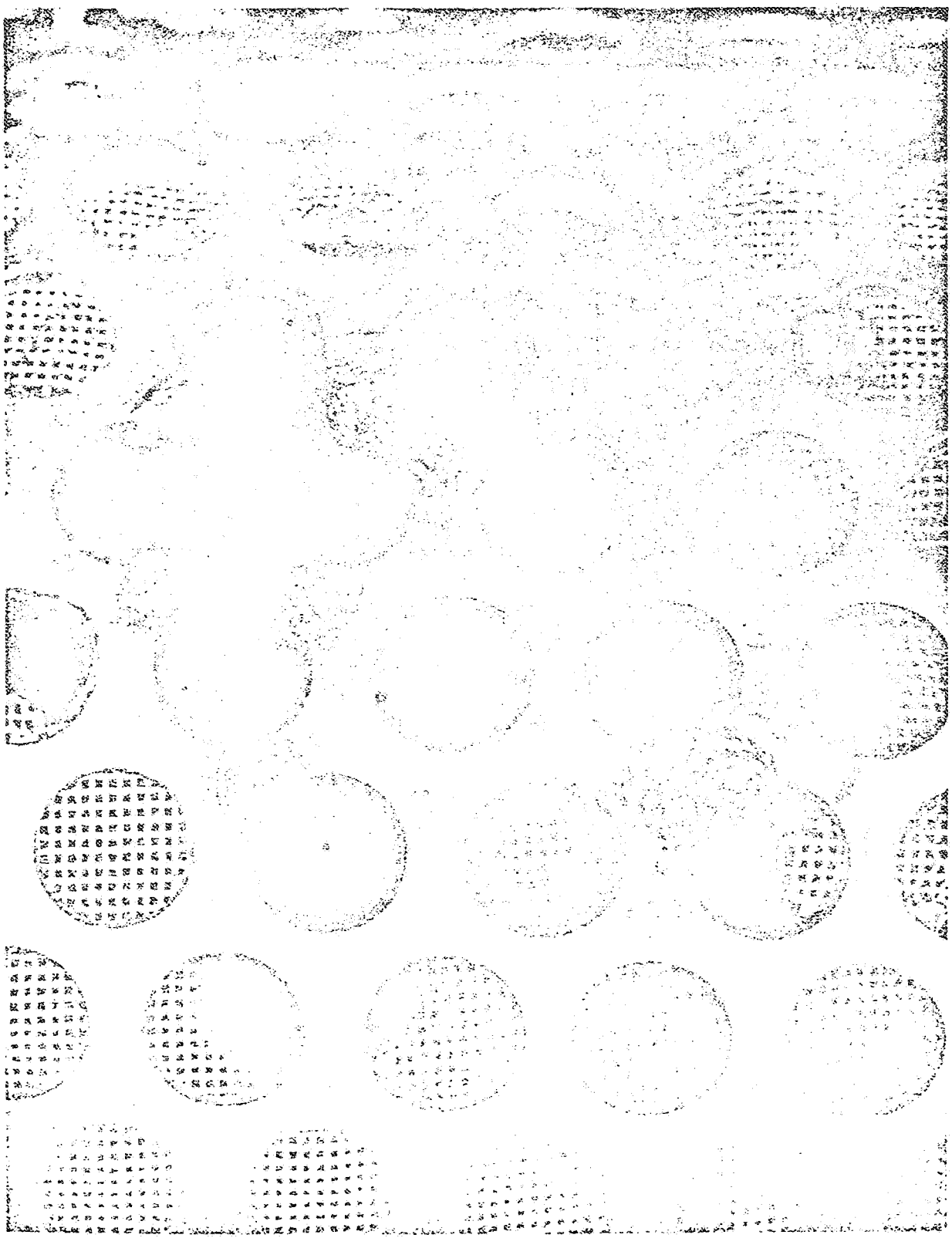
FC 40954. ROV 311 Screen Filter (View 1)



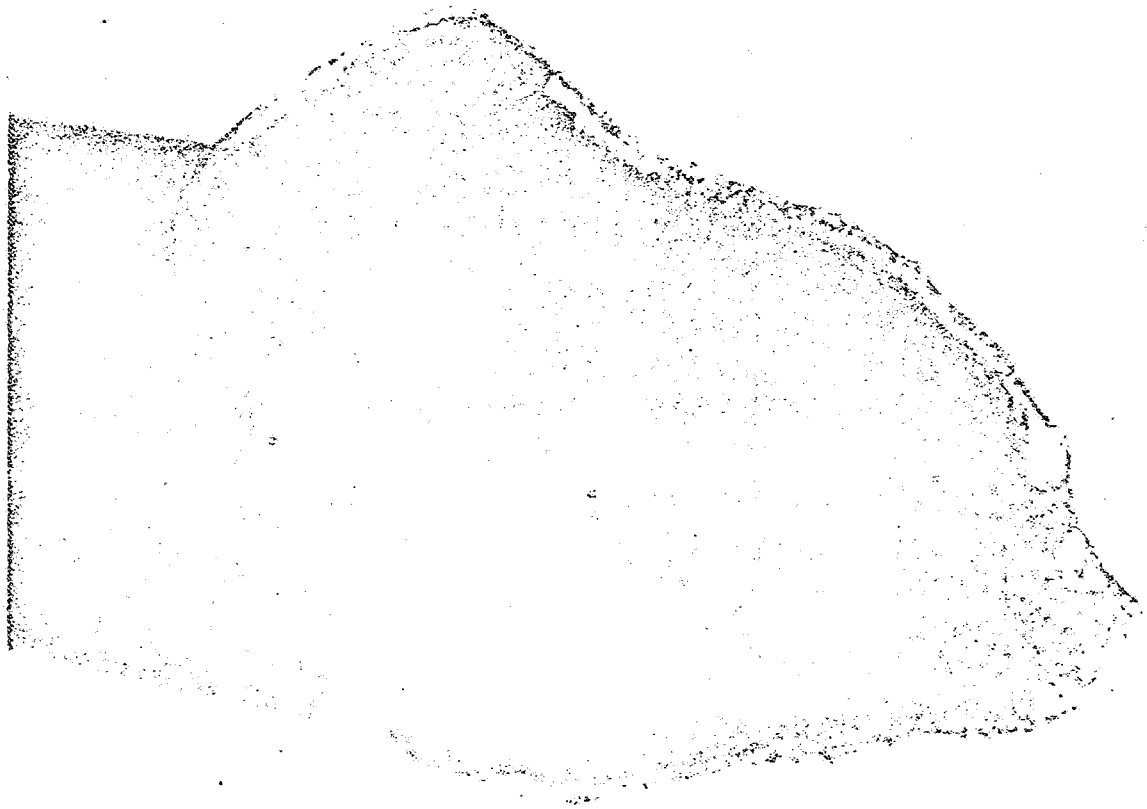
FC 40955. ROV 311 Screen Filter (View 2)



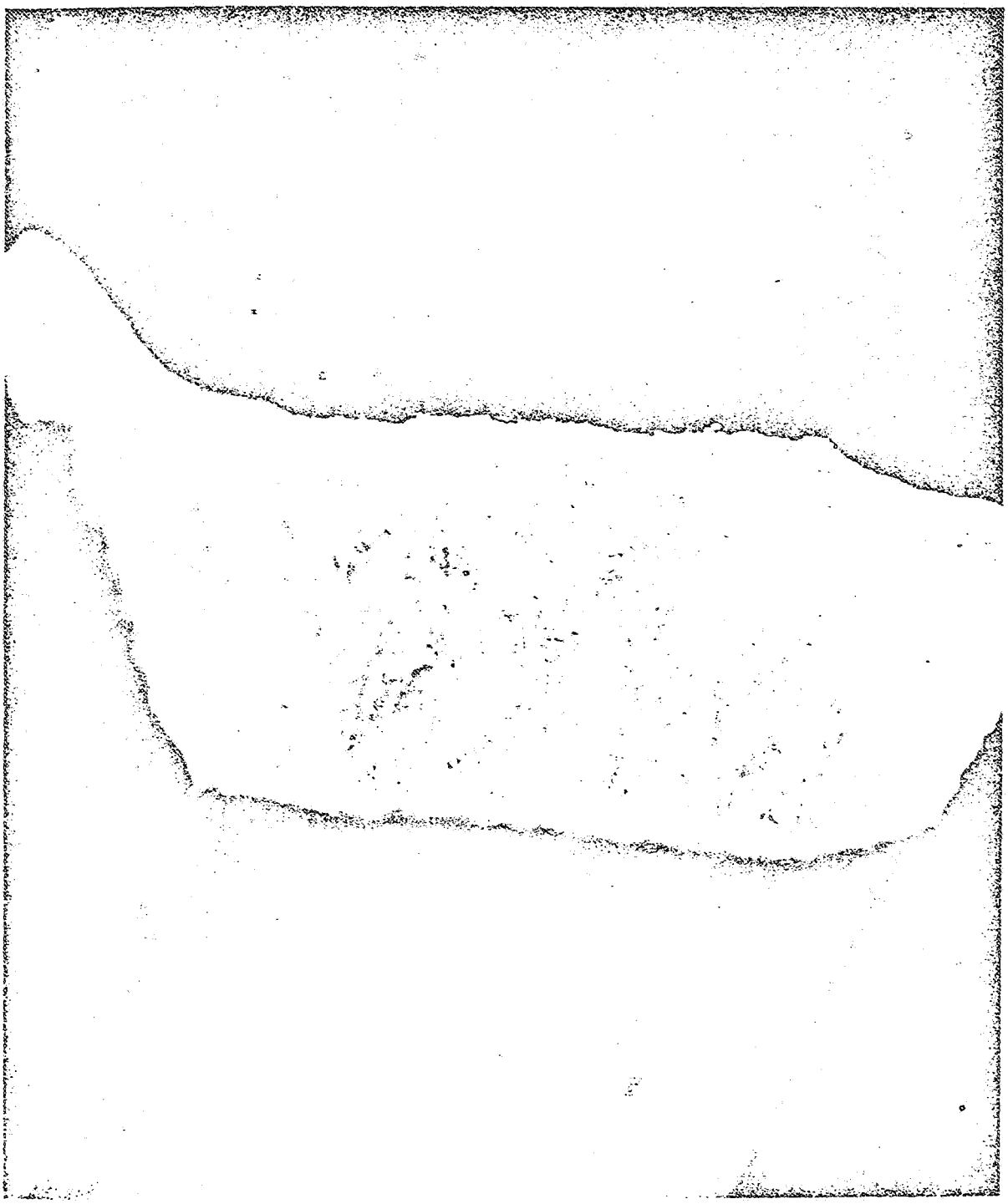
FC 40956. ROV 311 Screen Filter (View 3)



FC 40957. ROV 311 Screen Filter (View 4)



FC 40877. Tank Pressure/Vent Tubing (View 1)



FC 40875. Tank Pressure/Vent Tubing (View 2)

ATTACHMENT 3

Chronological Sequence of Events Leading Up To N_2O System Burnout on B-8

Stand 7-6-73

6/7/73 Request received from Project Engineering to determine what operating temperature and pressure the existing N_2O system would operate at with flow rate comparable to previous testing. Temperature was the prime requirement. (390° - 400°F). Flow and pressure were negotiable.

6/7/73 Issued IOM to Facilities Engineering to provide study of B-8 N_2O system to determine maximum operating temperature and corresponding pressure of existing system.

6/7/73 Limiting factor, as far as temperature was concerned, was determined to be the heat transfer fluid (Dow Chemical Sentinel Antifreeze) - Literature indicated a 330°F boiling point.

6/8/73 Determined that if another means of heating the tank could be used, we could meet requirement of 400°F in the tank. Tank pressure would be limited to 1300 psi because of increased tank temperature.

6/12/73 Received notification from Project that money is not available for continued combustor testing.

6/12/73 Received notification for Project, late 1st shift, that money is now available for additional combustor testing and that test estimate is required for high temperature N_2O system.

6/14/73 Results from heating a sample of the existing heat transfer medium were found to be 340°F boiling point.

6/13/73 thru 6/19/73 Three means of raising the tank temperature to 400°F were investigated. They were as follows:

1) Changing the heat transfer medium to another medium that was capable of operating at 400°F, compatible with nitrous oxide, and could be delivered in time to meet schedule.

   a) Several phone calls were made trying to locate a medium without any success. On 6/18/73, Union Carbide recommended their UCON-50-HB-280X. (A polyalkylene glycol fluid composed of a 50-50 mixture of ethylene glycol and propylene glycol with an oxidation inhibitor added.)

   b) Several phone calls were made between 6/13/73 and 6/25/73 to determine if the UCON fluid was compatible with N_2O. From these phone calls we determined that N_2O and the UCON can form a flammable mixture and would burn if we had a source of ignition; otherwise, we had no problems. A GN_2 blanket over the UCON was suggested.

2) Heating the tank with GN_2 flowing thru electric powered Chromalox heaters and blowing on and thru the tank.

   a) Facilities investigated to see if it could be done using heaters that were available on E-10 and B-8. It was determined it could be done.

3) Heating the tank with electrical strip heaters.

   a) Phone calls revealed that special heaters would be required and power would be a problem.

All three methods had advantages and disadvantages.

	Advantages	Disadvantages
1) New heat transfer medium UCON 50-HB-280X	1) Stable heat source takes a long time to get appreciable Delta T. 2) Minimum time required to go from existing system to new system. 3) Quick delivery of product (one week ARO) 4) No changes would be required in operational technique.	1) Compatibility with N_2O might be a problem. 2) Cost - 35¢/#
2) Heating tank with GN_2	No compatibility problem.	1) Needed design which would be time consuming. 2) Needed mechanical and electrical installation which would be time consuming. 3) Different operational technique. (Would require new procedures). 4) Might require 3 shifts coverage in fuel farm to maintain and monitor GN2 flow. 5) Could deplete GN_2 system and delay or shut down other test stands. 6) In the long run may be more costly than buying a new heat transfer fluid.

	Advantages	Disadvantages
3) Heating the tank with electric strip heaters	1) No compatibility problem.	1) Needed mechanical and electrical design. 2) There would be an installation problem which would require special heaters. 3) May not be able to complete in time to meet test schedule. 4) There was a possibility of getting hot spots in the N_2O tank and piping causing localized dissociation.

6/13/73 thru 6/25/73 Also during this period, several phone calls were made to determine if there was a problem operating N_2O at 1300 psig and 400°F. All available literature indicated there was no problem with dissociation until the temperature reached approximately 1000°F. From this survey it was concluded that we were well under the range at which dissociation starts. Available literature did say that certain catalysts tend to lower the temperature at which dissociation starts. (662°F being the lowest value mentioned but not indicating if pressure had an effect).

6/19/73 Completed Rocket Test Estimate No. 337. Program was estimated with two Options as follows:

a) Option I - N_2O tank temperature of 400°F

Replace existing medium with new heat transfer medium - UCOB 50-HB-280X.

b) Option II

Run the tests with the existing system which we could do at 325°F.

6/20/73 Received go-ahead from Project Engineering for 400°F system. Project indicated it was of utmost importance to get 400°F. Had to have 4 tests run by July 6. Initiated paperwork to buy UCON and additional nitrous oxide required for the test program.

6/20/73 Purchase Order placed for UCON.

6/20/73 Purchase Order placed for additional N_2O.

6/20/73 Issued EWR 114481 to foreman to drain glycol system.

Located a 2" Pacific gate valve to install in place of leaking HV 310. Issued EWR 114484 to foreman to have the new HV 310 sent to R/S to disassemble it to verify all parts are Lox compatible, clean it for N_2O service and hydro it at 3345 psig.

6/21/73 Sent new HV 310 to R/S. Foreman wrote EWR to Shop (W.O. 6-299).

Started draining glycol.

6/22/73 Completed draining glycol system. Discovered that the section of run line that HV 310 is in could not be removed from the glycol tank for cleaning due to interference with glycol tank - would have to remove N_2O tank in order to get the pipe out. Decided to pull the top works off the old valve, weld a cap on it and install the new valve in the horizontal section of run line down stream of the old valve.

Made fire water check. Discovered part of the N_2O run line is not covered with spray.

6/25/73 Received requirement to add sample system to N_2O tank so that gas samples could be taken during tank heatup and after test.

Issued EWR 115001 to install GN_2 purge system for ullage space of glycol tank. Stand crew cut out section of run line upstream of the orifice for new HV 310 installation. Prepared end of pipe for welding Grayloc hub on. Stand crew cut off threaded section of old HV 310. Prepared end of bonnet for weld.

6/25/73 Welded Grayloc hub on pipe at new HV 310 location and 2" Sch. 40 pipe cap on old HV 310 bonnet. Requested radiograph inspection of welds as we could not hydro the system.

Issued EWR to stand crew to install a heat exchanger coil in a drum of water upstream of the N_2O Haskel pump to prevent exceeding 275°F pump discharge temp. limit.

Issued EWR 101481 to rotate firewater nozzles to cover entire glycol/N_2O system.

Issued EWR 101482 to reset N_2O tank and run line relief valves at 1430 psig (design pressure at 400°F) operating pressure was limited to 1300 psi.

Discovered old HV 310 was rated to only 1245 psig at 400°F.

Removed trough cover at ROV 311 and discovered the valve and run line flanges were 600 lb. ASA 304L which are only rated at 725 psig at 400°F. Checked ROV 311 pressure rating and found that it was a CPC valve that was probably not rated for 400°F.

6/26/73 Issued EWR 120751 to replace ROV 311 304L flanges with 316 or 347. Located new flanges and sent to shop for cleaning.

Discussed ROV 311 seat working press with Facilities. They did not have any C.P.C. valve info and had the same Jamesbury info we had that said seat was rated at 0 psid at 400°F.

Decision was made to lower relief valve setting to 10% over 1245 psig max working pressure of old HV 310. Issued EWR 115019 to reset relief valves PSV 307 and PSV 312 to 1375 psi.

Received UCON heat transfer fluid.

Stand crew started glycol blanket purge system and continued Haskel pump heat exchanger installation.

Removed and disassembled ROV 311 - seat looked like nylon which is good for 180°F at 1300 psig.

Issued EWR 120750 for N_2O tank sample system installation.

6/26/73 Rig arrived.

New HV 310 pipe weld was good, old HV 310 cap weld was bad.

O/E looked for replacement ROV 311 in used stores, LPRF, B Area. Could not find one that would fit in the glycol trough that was also rated for 400°F service. Did find a Pacific valve on B-32 stand with a Kel-F seat that was good for 350°F.

6/27/73 Cut the cap off HV 310 bonnet and rewelded it.

Discussed use of the 2" Pacific valve at 400°F with Facilities Engineering. Decided to change Kel-F seat to Teflon and use it in place of ROV 311.

Removed valve from B32 and sent it to R/S for rework.

Reinstalled N_2O tank and run line relief valves after they were reset in R/S.

Started rig mount.

Made radiograph of HV 310 bonnet cap weld. Radiograph was good this time.

Issued EWR 120764 to press. check N_2O system at 1300 psig. (Ambient temp GN_2)

Stand crew cut the N_2O run line up and downstream of the old ROV 310 and prepared ends of pipe for welding Grayloc hubs on for new ROV 311.

Made up the Grayloc flanges at the new HV 310. Started welding the glycol trough together at this location.

6/28/73 Pressure checked N_2O run tank at 1300 psig and vented down to 10 psig.

Completed N_2O sample system up to regulator.

Electricians megger checked glycol heaters - they checked OK.

Started filling glycol tank.

Removed N_2O tank sample system and sent it to R/S for cleaning.

Rocket Support welded Grayloc flanges on N_2O run line up and downstream of ROV 311.

Stand crew worked new aluminum top for the glycol tank.

Issued EWR 120770 to blow down the N_2O run line after welding.

Stand crew and electricians worked ROV 311 inst. air system mods to convert from 125 to 750 psi air.

6/29/73 Completed glycol tank ullage purge system.

Completed glycol tank top installation.

Turned on glycol tank ullage GN_2 purge.

Continued tanking glycol.

Made radiographs of ROV 311 pipe flange welds.

ROV 311 pipe weld radiographs were good.

Completed tanking glycol.

Turned on glycol heaters at 1710.

Started glycol circulating pump.

Pump motor circuit breaker tripped on overload - secured heaters at 1815 while electrician changed overload heaters.

Started heaters and pump again about 2000. Glycol heated up to 110°F at 2300 when heaters and pump were secured. Had 10 psig of GN_2 in N_2O tank at this time.

Blew out N_2O run line with GN_2 from flowmeter down thru the open flange upstream of ROV 311 using a 1/4" tube hooked to the flow meter upstream pressure tap.

Blew out the pipe downstream of ROV 311 backwards using ROV 11 purge exiting at ROV 311 downstream flange.

Test Support installed ROV 311.

Press. checked ROV 311 for leak thru at 1300 psi - no leak.

Press. checked N_2O run line from new HV 310 to CV 1750 at 1300 psi - repaired all leaks.

Could not hook up ROV 311 bypass line as the 1/4" tap upstream of ROV 311 was cut out with the old ROV 311 upstream flange. Could have plumbed it to the flowmeter press. tap, but since it was questionable whether we needed it at all the decision was made to leave it disconnected and see if ROV 311 would open OK without it (without overscaling the flowmeter).

Reinstalled Haskel pump heat exchanger tubing after return for R/S where it was LOX cleaned.

Electrician wired up ROV 311 limit lights and the new 750 psi crescent valve for ROV 311.

Did not heat glycol over the weekend.

7/2/73 Started heating and circulating glycol at 0730. Tank was at 105°F.

Reinstalled N_2O tank sample system after return from R/S where it was LOX cleaned.

Vented N_2O tank to 0 psi, purged tank with N_2O, and tanked to 740 psi at 140°F. This required ten 64 lb. "K" bottles of N_2O.

R/S welder worked glycol trough installation at ROV 311.

N_2O temp. at 140°F - 1400 hrs.

Glycol temp. at 150°F - 1400 hrs.

Project requirement for 1st test is 275°F.

N_2O temp at 160°F at 1630.

Project decided to make the first test with 400°F N_2O. Requested that we hold N_2O press and temp as is until rig mount is further along.

Stopped heatup and N_2O tanking at 860 psi and 200°F.

Found glycol heater thermostat didn't work.

Completed glycol trough installation at ROV 311 - flowed glycol thru trough for leak check.

Installed ROV 311 support bracket.

At 2345 hrs. N_2O temperature was 200°F.

Turned all heaters off at midnight.

7/3/73 N_2O tank pressure dropped 110 psig over 3rd shift, found some small leaks in sample system. Also found HV 310 had leaked thru and pressurized the run line. Found that ROV 311 was open when the limit lights indicated closed - the limit lights were installed backwards. ROV 311 was open all day Monday and the HV 310 leak thru had pressurized the system up to CV 1750. Closed ROV 311 and vented N_2O off downstream of ROV 311. Left run line pressurized between HV 310 and ROV 311.

Took N_2O tank sample.

Received EWR 120780 from Project to heat N_2O to 270-280°F by 2400 hrs. 7/3/73, and hold at this temperature over July 4th holiday. Project wanted to heat up as high as possible over the 4th using the one thermostatically controlled heater with the stand unattended. Test Operations would not make the initial system heatup without a mechanic at the stand to observe for problems. Project decided they would not attempt to get overtime approved so it was decided to heat as high as possible Tuesday and hold this temp. over the 4th.

Completed ROV 311 750 psi inst. air system.

Installed new glycol heater thermostat.

Took another N_2O tank sample at 2115 hrs.

Secured all glycol heaters at 2345 hrs., couldn't get the thermostat to work.

N_2O run tank temperature at this time was 265°F.

7/5/73 At 0700 hrs. N_2O temperature was 220°F, glycol was 225°F. (Since the glycol was cooling down along with the N_2O tank this indicates the N_2O temp reads 5° lower than glycol when they are both at the same temperature).

Turned all 3 glycol heaters back on to continue heatup.

Took another N_2O tank sample at 0800 hrs.

Pumped N_2O to 1080 psig at 260°F - this would bring press. to approximately 1300 psi at 400°F.

Received EWR from Project to put strip heaters on rig inlet pipe from CV 1750 to rig to heat this section to 400°F.

Started circulating glycol thru the run line trough.

Overtime approved for 2 stand crew to standby 3rd shift for N_2O system heatup.

N_2O temperature 275°F at 1515 hrs.

Opened HV 310.

Removed thermostat from B-5 and installed in B-8 glycol tank well. This one worked OK.

N_2O temp. 327°F at 2345 Hrs.

7/6/73 Continued N_2O heatup thru 3rd shift. 345°F and 1260 psig at 0315 hrs.

At 0630 hrs., glycol temp was 365°F and N_2O tank press was 1300 psig.

At 0700 hrs. N_2O temp was 360°F.

At 0730 hrs. tank press was 1310 psig - <u>vented to 1300 psig with ROV 307.</u>

Took N_2O tank sample at 1045 hrs.

N_2O tank temp 377°F at 1115 hrs.

N_2O tank temp 390°F at 1510 hrs.

Installed two 150 watt 110 volt strip heaters on the N_2O inlet line downstream of CV 1750 and wired them to the STR rig strip heater variac, clamped a C/A T/C under one heater and heated the line to 400°F.

Started stand countdown for run at 0730 hrs.

Received program change later to install one explosive pulser, take combustion gas samples during test and extend run time to 3 seconds on first hot run.

Revised operators countdown to include the changes.

Stand crew worked combustion gas sample system hookup and checkout.

At 1400 noticed N_2O tank relief valve leaking thru slightly - it was dripping condensation and the 3/4" tubing to it was warm to the touch.

Completed controls valve check at 1500 hours and BDR cals at 1450 hours.

Cal ambient data printout later showed that N_2O tank temp was 399°F and N_2O flowmeter was 248°F at this time.

Completed O/E valve and abort checks at 1520 hours.

Completed pre-run purge.

Glycol temp at 400°F at 1630 hours and N_2O temp at 396°F at this time.

N_2O run line glycol temp was 310°F at this time.

Decided to warm up the run line by flowing N_2O thru it out ROV 312. Cleared stand of personnel to allow pressurizing N_2O line up to control valve.

When ROV 311 was opened at 1637 hrs. the explosion and fire occurred.