CONSIDERATIONS IN USING JOULE-THOMSON COOLERS

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ABSTRACT

Much has been learned from experience about considerations that are important in the design and use of JT cooling systems. This paper presents a discussion of some of the parameters that are important in system design and operation such as the effect of contaminants in the gas, heat transfer ΔT between the liquid cryogen and the dewar wall, dewar thermal effects, temperature stability with demand flow and fixed flow control, and the use of selected gas mixtures. Practical aspects of using JT coolers focus on the gas supply system and operative procedures that are needed to avoid having contaminants freeze out and also provide long-term reliability.

INTRODUCTION

Joule-Thomson coolers (a.k.a. JT cryostats) provide a fast-acting source of refrigeration by expanding a compressed gas isenthalpically to achieve temperatures in the range of the gas's saturation pressure. They ideally lend themselves to miniaturization for lightweight, portable applications. They require little maintenance, and have a very long shelf life. Cryostats can be designed with a fixed high flow rate for fast cooldown followed by a short run, or with demand flow control for moderately fast cooldown and extended run time.[1] They are essentially vibration and noise free. Most JT cryostats can operate in any orientation, and in very severe thermal and G-load environments.

The JT cryostat typically comprises an inlet gas connection, a particle filter, a counterflow heat exchanger, and a nozzle. It may also include an exhaust gas connection. Strictly speaking, the term "cryostat" means these items are assembled and mounted in a dewar. The dewar typically comprises a vacuum-insulated container, having an inner containment for the counterflow heat exchanger and exhaust gas, and a "cold end" mounting for the device to be cooled.

JT cryostats can be applied as open- or closed-cycle refrigerators. Either one requires a supply of pure, high-pressure

gas. The open-cycle gas is supplied either from a small high-pressure bottle for portability, or continuously from a compressor. Conversely, the closed-cycle gas is recycled from an initial stored quantity. The exhaust gas is directly collected, compressed to high pressure and resupplied to the JT cryostat.[2] A multi-stage compressor is required for the closed-cycle system. This adds bulk, requires relatively high input power and cooling, reduces reliability, and inhibits fast cooldown. Consequently, most JT cryostats are applied as open-cycle coolers.

Having a JT cryostat cool a device such as an IR detector in a satisfactory manner requires an understanding of some of the problems that can be encountered and the design and operating procedures that have to be considered to avoid such problems . This paper discusses the following factors that have been found to be the most significant:

- Gas purity
- Gas supply system
- Handling
- Temperature stability
- Heat transfer of liquid cryogen
- Dewar effects.

In addition, a brief description is presented on the general characteristics of using a gas mixture.

Gas Purity

The gas type, supply pressure, ambient temperature, volume, and JT cryostat design determine what operating conditions will result. Most constituents in the gas that have freezing points warmer than the saturation temperature, or particulates which could physically restrict gas flow, are contaminants that can prevent successful operation.[3] Particulates can permanently restrict flow if they become lodged in the heat exchanger inlet passage or nozzle. Condensible contaminants that crystallize in the upper temperature region of the heat exchanger inlet passage are usually carried downstream where they collect and restrict the nozzle. For a given concentration of condensible contaminant, the "dew" point temperature increases with increasing pressure, thus freezing sooner at higher operating pressures and lower ambient temperatures.

The susceptibility of a JT cryostat to clogging is dependent on the nozzle size and flow rate. We define the nozzle size by the parameter C_0 which is the flow rate of nitrogen (N_2) in sL/min that is measured when the supply pressure is 7 MPa exhausting to 0.1 MPa with both the JT cryostat and gas at 20-24 C. It has been found that an orifice smaller than 0.07 mm or a flow rating, C_0, less than 0.5 sL/min will require a higher gas purity than is practical to achieve. Clogging of a fixed nozzle occurs either abruptly during cooldown, or cyclically during operation in which the flow declines like a damped

oscillation. Several methods have been tried to impede clogging of fixed flow JT cryostats by trapping the solid or frozen contaminants in the cold end of the heat exchanger with large flow area labyrinths[4] or thread filters.[5]

Demand flow JT cryostats are more tolerant to contaminants because their temperature-sensitive flow control nozzle will increase flow in response to the warming caused by clogging, thereby purging contaminants. We have found that the minimum acceptable purity for N_2 entering a nominal 200 mW demand flow JT cryostat must be at least 99.998% (as total assay), and the maximum acceptable levels for the common contaminants in N_2 are found to be: 2 parts per million by volume (ppmv) water vapor (H_2O), which is equivalent to a dew point of -71 C at 0.1 MPa; 2 ppmv carbon dioxide (CO_2) or carbon monoxide (CO); 3 ppmv total hydrocarbons (THC); 3 ppmv chlorofluorocarbons (CFC); and, 6 μm maximum diameter particles. Typically, air, oxygen (O_2), argon (Ar), helium (He), and neon (Ne) do not affect performance at low concentrations. It is found that by doubling the cryostat's nominal cold flow rate, and thereby halving the available run time from a fixed gas volume, operation is acceptable with 4 ppmv H_2O. As the level of H_2O is further increased, the frequency of temperature excursions also increases.

There are no general industrial, military, or federal purity specifications for JT gases at present. A few specific military and commercial JT cooler specifications coincide with the minimum acceptable levels described. Mil-P-27401, Grade C N_2 is sometimes specified, but its purity does not meet the acceptable levels described. Industrial gas suppliers present purity in a variety of ways.[6] Most of them use grade names and total purity assay. "Commercial purity", > 99.995%, contains up to 10 ppm H_2O. "Ultra-pure carrier" grade, > 99.998%, or "ultra-high purity", > 99.999%, typically contain < 2 ppm H_2O. However, grade names and purities vary among suppliers and gases, and the total purity may be based on a weight analysis which may appear purer than a volume analysis (ppmv = ppmw x MW gas / MW impurity). Also, the total purity assay may not include all the impurities pertinent to the application. Some industrial gas suppliers will provide an actual impurity analysis. Unfortunately, the analysis may not detect the low levels required, and it may not be for the gas as it exists in the actual vessel being supplied. Furthermore, impurities will outgas from the vessel's internal surfaces as the supply pressure decreases during use, changing the purity ratios. From a practical stand point it has been found that all of these gases can be used satisfactorily if a clean adsorber is used in the gas supply line. Higher purity gases reduce the frequency of having to regenerate the adsorber.

Gas Supply System

It is essential that all contaminants in the gas supply system be either eliminated or reduced to acceptable levels. The gas supply

system typically consists of the gas source, a pressure regulator, adsorbers with filters, and an interconnecting line to a small gas bottle, a start valve, and another gas line to the cryostat (See Figure 1). The gas source is typically a pressurized tank purchased from a gas supplier, but it may be compressed locally from a low pressure gas source, ambient air, or a liquid cryogen, and purified. For gas compression, non-oil-lubricated compressors are recommended to minimize hydrocarbon contamination.

The gas supply system design should consider the following:

(1) Safety: Capable of operating safely with the intended gas and pressure. Consideration must be given to the maximum working pressure at the high temperature ambient, the number of filling/venting cycles, applicable transportation codes, burst fragmentation, and fire safety (Ref.: ASME Sec. VIII, Div. 1; ANSI B31.5; DOT Hazardous Materials Regulations; Mil-STD-454; Mil-STD-1522).

(2) Leak Tightness: For short-term applications, a soap solution leak check is acceptable with the system pressurized with the process gas at the working pressure @ 21 C. For long-term applications, a He mass spectrometer should be used with the system pressurized with He to the maximum working pressure. Thermal cycling beforehand at high- and low-temperature ambients is recommended.

(3) Permeation: Minimize permeation by using corrosion resistant metals and metal sealed joints.

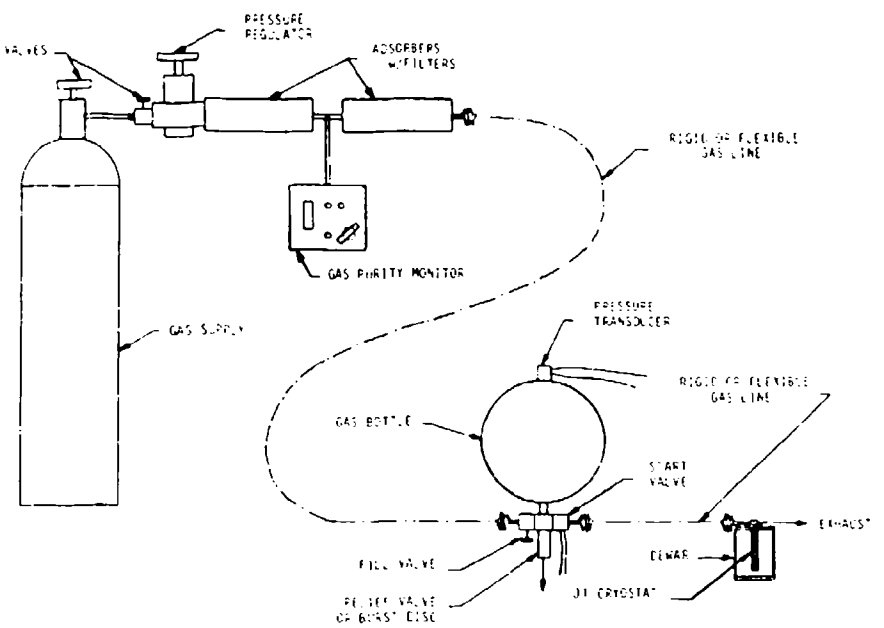

Fig.1. Typical JT Open-Cycle Refrigeration System

(4) Cleanliness: Cleaned for oxygen use is preferred.

(5) Simplicity: Minimize the number of joints, connections, dead flow volume, and internal surface area.

(6) Outgassing: Use low vapor pressure materials, such as metals, and purge the system prior to use. A typical outgassing rate of 1×10^{-5} torr L sec^{-1} cm^{-2} at 1 L/min flow represents a 1 ppm level of impurity.[7]

The choice material for interconnecting plumbing in most applications is austenitic stainless steel (AISI 300 series) for strength, corrosion resistance, and non-magnetism. Easy machining type 303 has poor welding characteristics to other stainless steels. Types 321, 316L, and 304L are better choices for welded joints because they will have minimal grain boundary chromium carbide precipitation during welding, and therefor better resistance to corrosion and cracking. Passivation and electropolishing are preferrable post-weld treatments because they enhance corrosion resistance, and remove micron-sized particles and crevices.

Copper and copper alloys outgas more than stainless steel and have been found to promote contamination abruptly after long-term use. Copper (Cu) oxidizes readily and continually in air. After the corrosion penetrates to the internal surface, H_2O permeates directly into the process gas. The plumbing must then be replaced. For these same reasons, a JT cryostat should be stored in a dry, inert environment to reduce corrosion of any copper in the heat exchanger.

Polymeric and elastomeric materials (i.e. plastics, Teflon, Nylon, silicone, nitriles, rubber, etc.) should also be avoided in JT gas systems. They are hygroscopic, and have high permeation and outgassing rates. They are fundamentally a source of hydrocarbon and chlorofluorocarbon contaminants. It is important to understand the properties of such materials which may exist in valves or pressure regulators.

Seamless tubing is preferred for interconnecting piping, although welded and drawn tubing is acceptable. All tubing, valves, gauges, and regulators should be leaktight and clean. Cleaning should consist of purging, scrubbing, vapor degreasing, or ultrasonic cleaning with solvents such as ethanol (denatured) or methanol, followed by Freon (TF or TMC) or 1,1,1 trichloroethane, followed by a pure N_2 purge at < 7 MPa. Vacuum baking afterward is beneficial.

All joints and connections in the plumbing should be inert-gas welded (e.g. TIG), fluxless brazed (e.g. vacuum), or dry metal sealed (e.g. Swagelok fittings, metal o-rings, MS or AN fittings, indium gaskets, etc.). Tapered pipe threads should be avoided because they can develop leaks without a sealant, and most pipe sealants contain Teflon. However, if pipe threads are used, an epoxy adhesive is a preferred

sealant. Elastomeric o-ring seals should be avoided. However, if they are used, the o-ring material should be carefully selected (e.g. Viton is acceptable for some applications), vacuum baked, and a very thin film of very low vapor pressure grease (e.g. Apiezon N) applied to the o-ring. Should a joint require soldering or brazing with an acid flux, such as with stainless steels, it is very important to use the least active flux possible. Neutralize the acidic residue immediately after making the joint (e.g. hot distilled water has been found to work better than many formulated neutralizers), then promptly clean as previously described.

An adsorber is required in the gas supply system. Adsorbers typically contain a molecular sieve which will adsorb primarily H_2O, and to a much lesser extent CO_2 and trace oils. The adsorber will improve or maintain the gas purity for a period of time, after which it begins to break through with contaminants. The amount of adsorbed H_2O is a function of the inlet gas purity, adsorbent mass, and initial adsorbent dryness. A typical adsorber, 32 mm diameter x 160 mm long and capable of operating @ 60 MPa, has a rating of 45.3 kSL of processed gas with 10 ppmv H_2O inlet purity. Adsorbers which have not been exposed to oils or other hydrocarbons can be regenerated by heating and purging, or vacuum baking, and re-used many times. If the adsorber breaks through with hydrocarbons due to excess exposure, then it has to be replaced. For long-term operations, it is recommended that two adsorbers be placed in-line downstream of the gas source, and routinely rotated with a third adsorber (i.e. the upstream adsorber is withdrawn and regenerated, the downstream adsorber is moved upstream, and the third, regenerated adsorber is placed in the vacant downstream position). It is recommended that a gas purity monitor be located between the two adsorbers to detect early breakthrough of the upstream adsorber.

A filter is required in the gas supply system downstream of every adsorber, unless a filter is part of the adsorber. Filters typically remove smaller particles from a gas than from a liquid. Therefore, it is important to understand the filter being used, and the pressure drop it imposes at the anticipated flow rate. APD Cryogenics supplies a porous stainless steel filter integral with their adsorbers and on the gas inlet of every JT cryostat. These filters typically have a 1 um nominal and 3 um absolute gas filtration rating.

The most direct method of identifying unacceptable levels of contaminants in the gas supply system is by using a gas purity monitor. It is important to routinely sample the gas purity nearest the JT cryostat. The APD Cryogenics gas purity monitor, p/n 2501060, will freeze up within 5 min if > 2 ppmv H_2O or > 1 ppmv CO_2 is present in 35 MPa N_2 (See Figure 2). Although it has not been calibrated for other contaminants, it is more sensitive to them than are most cryostats, providing a quick go no-go gage of overall gas purity. A hygrometer, gas chromatograph, or other gas sampling devices can be employed to gain a more complete analysis.

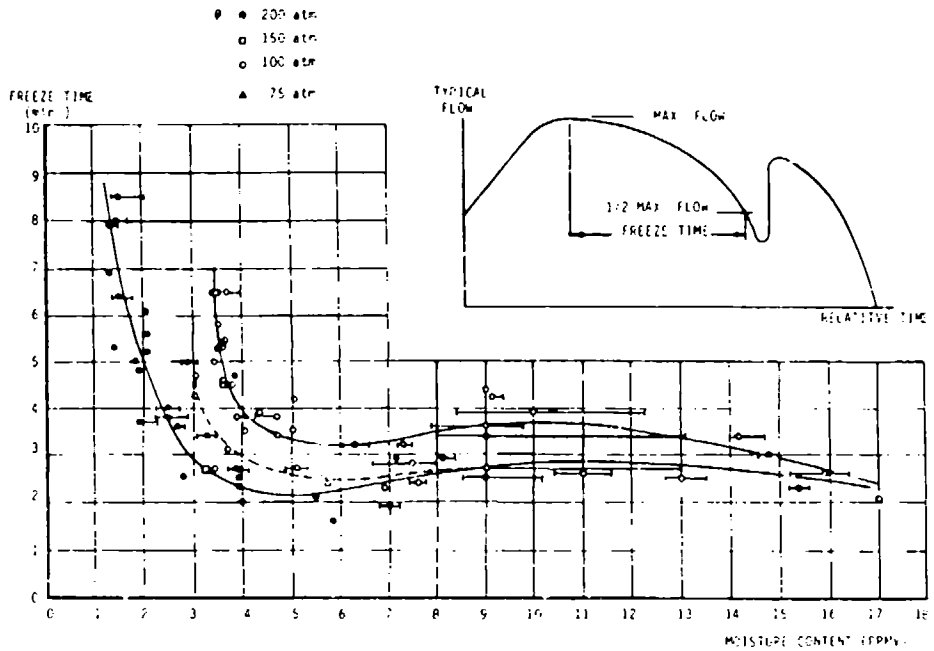

Fig.2. Gas purity monitor.
Freeze time vs. moisture content in nitrogen.

Handling

It is important to minimize the exposure of JT Cryostats to H_2O and other contaminants. Various storage methods can be employed to control their exposure. Unprotected storage in a typical laboratory is inadequate. The first improvement would be to seal the JT cryostat in a water vapor-proof bag. A dust-free package of dessicant could be sealed inside the bag to adsorb H_2O. A second improvement would be to seal the JT cryostat in a room temperature dry box, either purged with pure N_2 or dessicated. A third improvement would be to store it in a heated dry box, thereby degassing the surfaces. A fourth improvement would be to store it in a vacuum oven, thereby withdrawing the outgassed vapors. An oil-less, two-stage 30 L/min diaphragm pump @ < 25 torr on an oven at 70 C has been found to reduce the H_2O level to a few ppmv. It is important to keep the vacuum pump as close as possible to the oven, and the overall system clean and leaktight. Oil-lubricated vacuum pumps should be limited to 0.20 torr, and have a foreline trap to inhibit oil from backstreaming into the oven.

Before operating a J cryostat, purge the gas supply system and cryostat for several minutes with the pure process gas @ < 3 MPa. This flushes air and outgassed contaminants from the piping system. It is

also important to properly warm up the JT cryostat and dewar after each operating cycle. Cryopumping can occur on the exhaust side of the heat exchanger which can condense H_2O into the cold end area as it warms. This H_2O can freeze on the nozzle on subsequent cooldowns and cause erratic performance. Such adverse effects can be controlled by exhausting to a controlled dry environment, by using exhaust check valves to prevent backstreaming, or by continuing the supply gas at the end of the operating period at reduced pressure, < 3 MPa, until the cold end is > 273 K. Low level contaminants in the gas supply may accumulate, and backstreaming may occur during long operating periods (i.e. days/weeks). Therefore, it is usually necessary to warm up the system periodically.

When a JT cryostat has been exposed to excess H_2O, vacuum baking @ < 25 torr and < 80°C overnight is recommended. Alternately, it can be purged with pure N_2 at < 3 MPa. Contaminants that accumulate in a porous filter are difficult to completely remove, and usually require replacing the filter. Purging or vacuum baking may temporarily clean a path through a filter loaded with condensible contaminants, but the residual boundary contaminants will continue to mix with the incoming purer gas. Flushing with a liquid solvent is not recommended because the liquid will flush trapped particles downstream, and the solvent will be difficult to remove from the filter afterward.

Temperature Stability

The temperature stability of a JT cryostat-cooled dewar and load is dependent upon stable heat loads and a stable flow. Changes in heat load result in a change in the ΔT between the device being cooled and the liquid cryogen. Changes in flow rate have a small effect on heat transfer ΔT as discussed in the next section, but do change the pressure drop in the exhaust side of the heat exchanger. A change in the pressure of the liquid, whether it is caused by a change of pressure drop, altitude, or a valve in the vent line, will cause a change of temperature. For LN_2 at 77.4 K the change is 0.088 K/kPa. The nozzle flow is a sonic compressible gas condition, proportional to supply pressure and nozzle temperature as $P/T^{0.5}$. Therefore, as the supply pressure varies, as a pressure regulator fluctuates, or as a demand flow nozzle varies, so will the stability of the cold end temperature. These effects may cause a slow temperature change as the gas supply pressure decays in a bottle or a rapid change if the demand flow control adjusts suddenly.

A fixed flow JT cryostat or a dual-orifice type will generate an excess amount of liquid cryogen. The steady flow rate and excess liquid provide very stable temperatures, usually < 0.2 K/s variations. A demand flow JT cryostat will generate liquid cryogen proportional to the heat load, minimizing gas consumption, but the flow rate will vary, causing temperature fluctuations. Improved design and manufacturing of demand flow JT cryostats has resulted in routine temperature stability of < 5 K above the saturation temperature while maximizing gas

utilization. Better stability, within 1 K of the saturation temperature, can be obtained by increasing the average flow rate after cooldown, thus trading gas consumption for stability. This also reduces the susceptibility to clogging.

Heat Transfer of Liquid Cryogen

Gas consumption is minimized by designing the dewar cold end to have the lowest thermal mass and steady-state heat losses possible while providing the required structural support. For cylindrical plug-in JT cryostats, this is accomplished in general by minimizing the diameter of the heat exchanger sleeve. However, if the cold end cap is bonded directly to the end of the minimum diameter sleeve then the surface area may not be great enough to reduce the heat transfer ΔT to an acceptable level across the liquid film produced by the cryostat.

Figure 3 shows the design of a test sleeve that has been used to measure the heat transfer ΔT and test results for sleeves having inside diameters of 3.30 mm and 5.18 mm. Accurate measurements are obtained by brazing a flat copper plate to the end of a 0.076 mm wall stainless steel tube. The temperature of the liquid in the sleeve is determined by measuring the pressure in the heat exchanger mandrel, P1, which has no gas flow. The temperature of the copper end piece is determined by measuring the pressure of liquid in the vapor lab, P2, which is machined into the copper. Tests were run with N_2 at supply pressures in the range of 34 MPa to 10 MPa using fixed flow JT cryostats and a demand flow type all venting to 0.10 MPa. Measurements were also made with LN_2 in the sleeves. The sleeves had static losses of about 200 mW to which additional heat was applied via heater wire wound on the copper end piece. Test results show that the ΔT is greater for the smaller diameter sleeve, as expected, and is less for the high velocity liquid produced by the cryostat than for static LN_2. In either case the ΔT can be significant.

The data for the demand flow JT cryostat includes the ΔT due to pressure drop in the heat exchanger exhaust which tends to be small relative to the heat transfer ΔT. Conversely, for fixed flow JT cryostats the elevation of the saturation temperature due to pressure drop in the heat exchanger exhaust can be larger than the heat transfer ΔT, especially at high supply pressures. Actual values are a function of the heat exchanger design and nozzle size. An elevation of 1.6 K was measured for the 3.30 mm dia. cryostat at 34 MPa supply pressure.

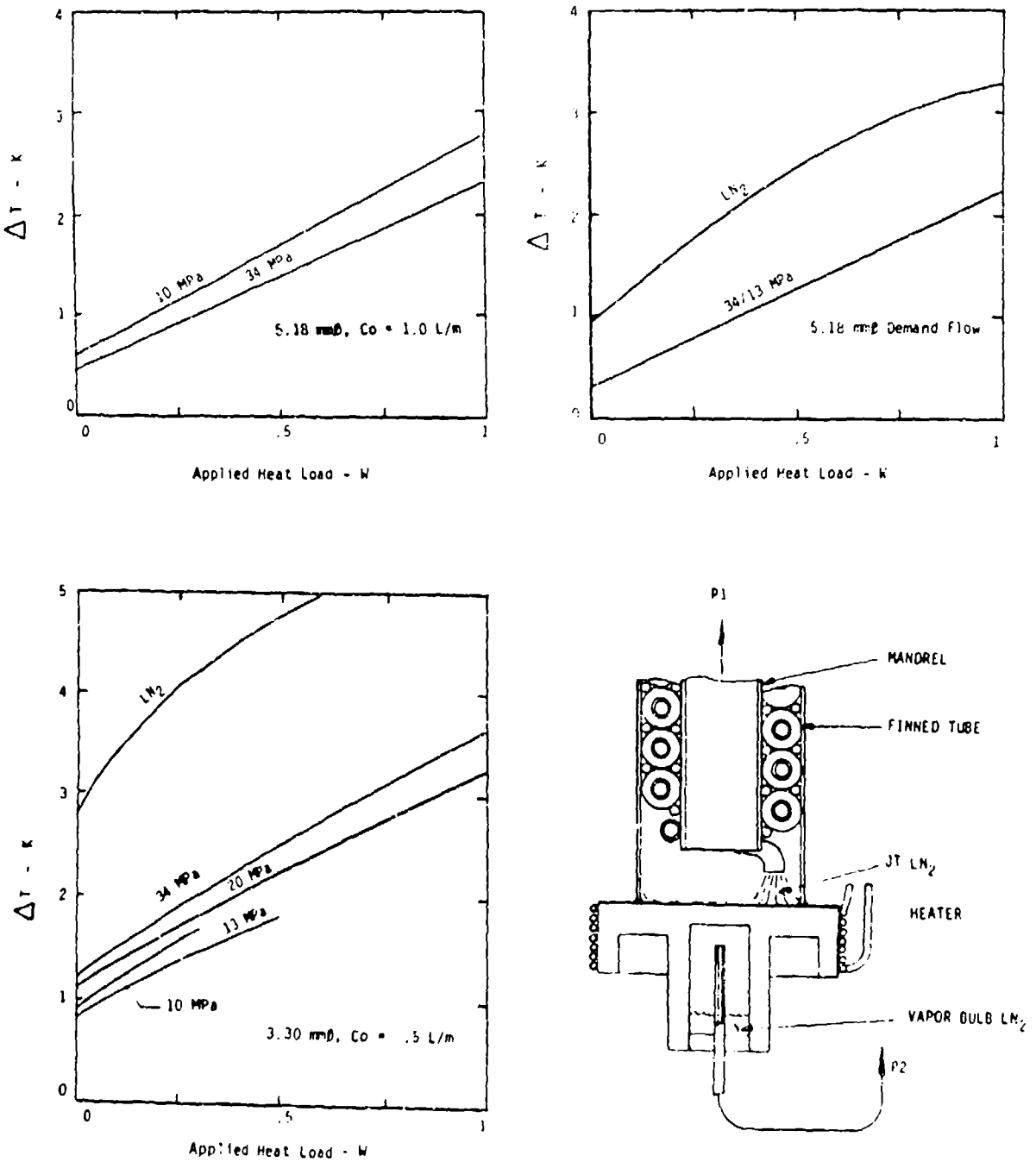

Fig.3. Measured values of the temperature difference between the LN_2 in the cryostat sleeve and LN_2 in a vapor bulb in copper end cap vs. applied heat load. Static dewar loss is 0.2 W.

Dewar Effects

The dewar is essentially a vacuum-insulated container. Proper dewar design for JT cryostats should include the following:

(1) For cylindrical plug in, finned-tube JT cryostats: A bore with +/- 0.0127 mm diameter tolerance, straightness within 0.001 mm/mm of length, and a depth such that the nozzle tip installs within 0.5 - 1.5 mm of the inside face of the cold end.

(2) Minimized conduction heat losses entering the cold end, with vacuum insulation < 1×10^{-3} torr over the ambient temperature range for the intended life, and radiant heat shielding.

(3) Minimized cold-end volume, maximized heat transfer surface area, and minimized load thermal mass. For complex load geometries, the component materials should have high thermal diffusivity, and the arrangement should attempt to minimize thermal gradients especially during cooldown. It has been found that demand flow JT cryostats will regulate flow (i.e. refrigeration) according to the local heat load which may be much less than the total load, thus extending the cooldown. This is due to poor thermal communication between the total load and the cooler.

The thermal response of a plug-in JT cryostat in a given dewar can be characterized by measuring the following:

(1) the temperature sensor's sensitivity with respect to temperature, its repeatability, and stability with liquid cryogen inside the dewar bore;

(2) the cooldown time from the ambient temperature by injecting liquid cryogen into the dewar bore; and

(3) the steady state heat loss over the lower cold end half of the bore using a liquid cryogen. (Plot the volume of boiloff gas vs. time. Calculate the ave. boil-off rate during the "lower half" period, ignoring the non-linear rate near the end. Multiply by the latent heat of vaporization and the NTP density for the cryogen used. For LN_2: heat loss [mW] = boil-off rate [sL/min] x 3862.)

Gas Mixtures

A large number of gas mixtures have been tested in JT cryostats of different efficiencies. In general, it is found that adding a gas such as CH_4 to N_2 provides a refrigeration effect approximately in proportion to the sum of their separate refrigeration effects, but the operating temperature can be closer to the saturation temperature of the

colder constituent. If the heat load is increased, or the efficiency of the heat exchanger is decreased, then the saturation temperature increases toward the higher temperature constituent. Adding a gas such as hydrogen (H_2) or Ne to N_2 can reduce the boiling temperature below 77.4 K while venting to atmospheric pressure.

Figure 4 shows the results of a test with a mixture of 0.1 Ne/0.45 Ar/0.45 N_2 from a 60 mL bottle charged to 42 MPa using a fixed flow JT cryostat venting to 0.10 MPa and a repeat test with pure N_2. The following observations apply to this test and are generally true for all of the tests with mixtures:

(1) Initial cooldown is faster, but the time to 80 K is the same for the mixture compared with pure N_2.

(2) There is a time delay for the mixture to reach minimum temperature due to the time it takes to reach equilibrium concentrations in the liquid.

(3) The temperature increases with time after reaching minimum temperature because the heat load is increasing relative to the rate at which refrigeration is produced. The increasing temperature is superimposed on a small decrease in temperature due to the decreasing gas pressure from a fixed volume and decreasing pressure drop in the heat exchanger exhaust as the flow rate decreases.*

(4) The temperature of the mixture rises at the end of the test toward the NBP of the higher temperature constituent, in this case Ar, and holds there while the residual liquid, rich in Ar, evaporates.

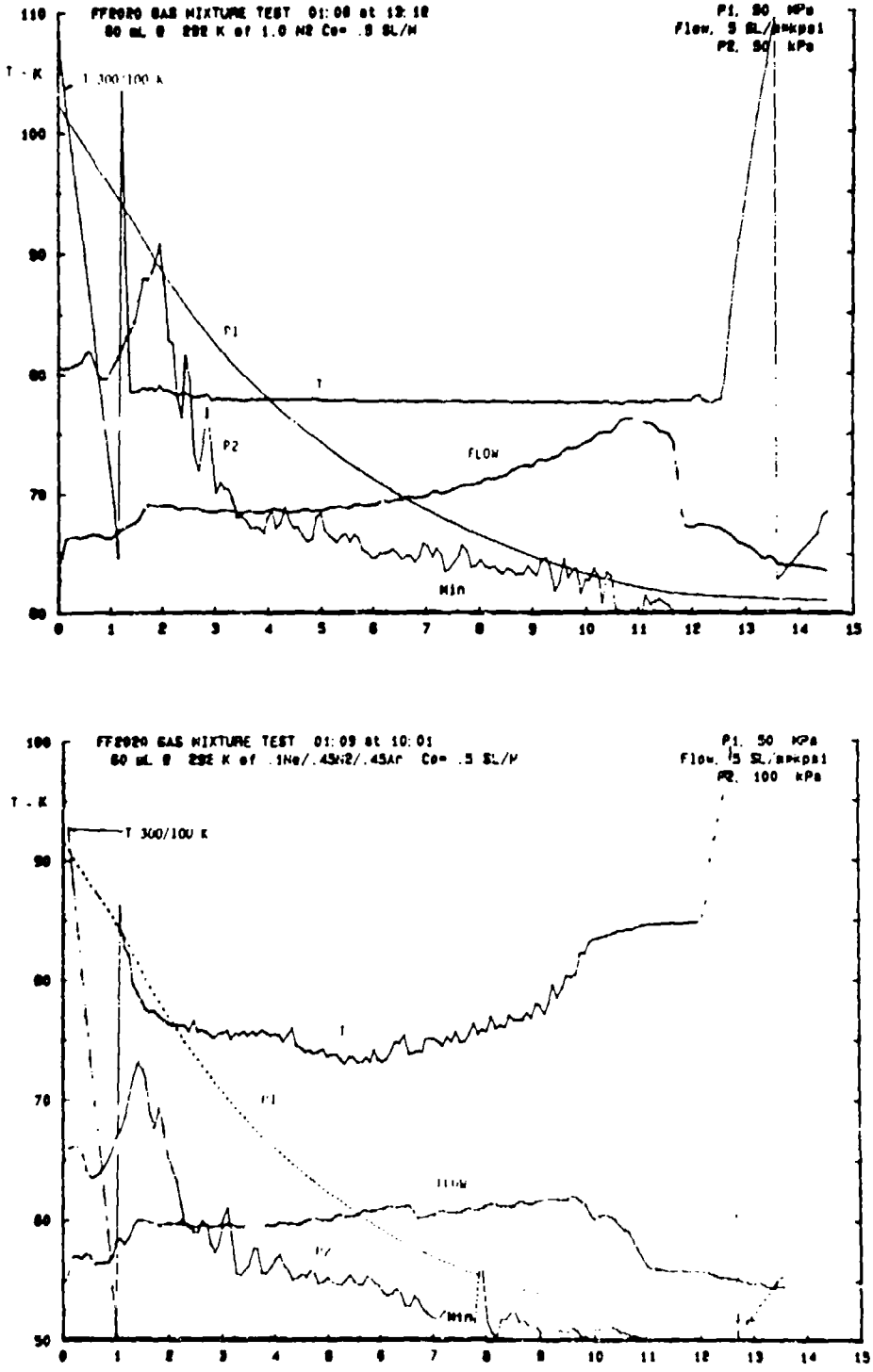

Fig.4. Temperature, T, bottle pressure, P1, pressure over the liquid, P2, and normalized flow vs. time for pure N_2 (upper) and a mixture of .1 Ne/.45 N_2/ .45 Ar^2(lower) venting to atmospheric pressure.

SUMMARY

JT cooling systems are relatively simple and versatile but have frequently presented problems for the user because of their sensitivity to contaminants. It is hoped that the experience which is presented in this paper will help users design and operate JT systems with a high degree of success.

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