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SRS HISTORY AND EXPERIENCE WITH PALLADIUM DIFFUSERS (U)

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SRS HISTORY AND EXPERIENCE WITH PALLADIUM DIFFUSERS (U)

SUMMARY

Palladium and palladium-silver diffusers have provided effective service in the Tritium Facilities at the Savannah River Site for almost forty years. The early multistage palladium diffuser was slow and required extensive maintenance. The diffuser was water cooled, relied on O-ring seals, and was connected in stages. Repairs required considerable disassembly which added to environmental releases. The lifetime of the diffusers varied from four months to over five and a half years. The last of the multistage palladium diffusers was taken out of service in 1985.

The Resource Services Incorporated (RSI) diffusers came into service in the mid 1980s. These units use a 75% palladium 25% silver alloy for the diffusion coil and have a single stage that can be multiply connected as required. The RSI diffusers have replaceable heaters and thermocouples. Thirteen RSI diffusers are currently in service. These diffusers have been reliable, each averaging 19.1 months between failures. However, this failure rate is believed to be high due to mercury attack. The four RSI diffusers installed in the Replacement Tritium Facility, which now have about Twelve months of tritium service, are expected to have a considerably longer service life due to the absence of mercury in the process. Reliability data from these stages can be made available.

The Savannah River Site has evaluated other diffuser designs and manufacturers. Two diffusers produced by Johnson Matthey Inc. did not fail after four years of service but had a lower than expected throughput rate. An Adolph Coors Co. diffuser, similar in design to the RSI diffuser, was installed in 1993. The single stage unit had some initial problems due to insufficient clearance between the heater and the heater well. This caused the heater to warp and wedge itself into the well and prevented easy replacement. Future service data from this unit can be made available.

Review of the history of diffusers in the Tritium Facilities at the Savannah River Site reveals several recurring failure modes. The palladium or palladium-silver alloy coils failed by steam attack at grain boundaries and by attack from liquid mercury used in diffusion pumps in the processes. The palladium to stainless steel braze joint has been a recurring mode of failure of diffusers, especially in the past fifteen to twenty years. Thermocouples and heater elements fail and are replaced routinely. Corrosion or stress corrosion cracking by cooling water was a failure mode of older diffuser types. The older types also had polymer O-rings that failed and were regularly replaced. The ancillary pumps and valves used with diffusers also frequently failed by localized aqueous or mercury corrosion or stress corrosion cracking. Thirteen design recommendations are suggested based on these failure modes.

Alternatives to palladium or palladium-silver diffusers have been developed at the Savannah River Site over the past ten years, utilizing metal hydrides to separate hydrogen isotopes from inert gases. Two of the early alternatives relied on palladium

supported on Kieselguhr (calcined diatomaceous earth) as the absorbing material. While these processes have been effective, their ability to achieve low levels of hydrogen in the byproduct is somewhat less than that of a series of diffusers due to the relatively high equilibrium pressure of hydrogen over palladium even at low temperatures. One of these processes, the Primary Separator, is in use in the Replacement Tritium Facility just ahead of the diffusers thereby significantly reducing its load and improving its performance. Other metal hydride separator systems with lower equilibrium hydrogen pressures are being evaluated, including zirconium-iron alloys and titanium sponge.

INTRODUCTION

The Savannah River Site (SRS) has processed tritium in support of national defense programs since 1955. Palladium diffusers have been used extensively for separating hydrogen isotopes from inert gases (such as argon, helium, and nitrogen). In almost forty years of service, the design of the diffuser has been steadily improving. Several diffuser designs from different manufacturers have been evaluated at SRS. The operating experience gained from these designs together with failure analyses performed on failed units have led to several recommendations for improved diffuser designs and operating methods. This experience gained at SRS and the following recommendations form the basis of this report.

Even though palladium diffuser technology has proven to be reliable, SRS has examined several alternative technologies over the past several years. This report will also review some of these promising alternatives.

DIFFUSER DESIGNS AND OPERATING EXPERIENCES

Multistage Palladium Diffuser (MPD)

The early diffusers used at SRS were single-stage, water-cooled units designed "in-house" by E.I. Du Pont de Nemours & Co. (prior SRS contractor). After 1958 these units were redesigned as multistage diffusers. Each stage of the Multistage Palladium Diffuser (Figure 1) consisted of one 0.59 inch (1.5 cm) outside diameter pure palladium diffuser coil 32 feet (9.8 meters) long with a surface area of 4618 cm^2 and a wall thickness of 0.025 cm. Stainless steel jumpers connected each stage, which was supported on a ceramic core in its own flanged section. The palladium coil was isolated electrically from its shell. The tube was heated electrically by its own resistance to 375 °C to 425 °C by connecting the tube center to a power source and grounding each end of the tube. Each stage was made of stainless steel, had a water cooling jacket, and connected using O-ring seals. Water cooling of the shell reduced diffusion losses through the shell and prevented heat damage to the O-rings [1].

The Multistage Palladium Diffuser system was slow and hard to maintain. Repairs required disassembling a large portion of the stack to access a single stage. This exposed large, contaminated surfaces to the atmosphere, adding to environmental

releases. The diffusers being water cooled led to localized corrosion or stress corrosion cracking similar to that observed in pumps [2]. In addition, these diffusers had polymer O-ring seals which failed by becoming brittle during exposure to high temperature for excessive time [3]. Water condensed from the process has been found in the Du Pont type diffuser [4], which could have led to internal corrosion if halogen ions were present. The last of the MPD stages was taken out of service in 1985.

Resource Services Incorporated (RSI) RSD-100-SRP

The Du Pont diffusers were replaced by more compact modular diffusers fabricated by Resource Systems, Inc. (RSI) in the middle 1980's. The diffusion coil in this design is a 75% palladium and 25% silver alloy commonly used for hydrogen purifiers because it is stronger than pure palladium. The alloy is also more permeable to hydrogen isotopes for a given tube wall thickness and has about twice the tensile strength [5].

The Model RSD-100A-SRP diffuser is commercially available from RSI as a standard design hydrogen purifier. Figure 2 shows a cross-sectional view of the unit. The diffuser consists of five parallel permeation coils that are fabricated from palladium - silver alloy tubing. Each tube is 11-feet (335 cm) long and has a 0.125-inch (.3175 cm) OD with a 0.004-inch (.011 cm) wall thickness. The coils are parallel-mounted in a single chamber to reduce the pressure drop associated with the relatively high tube-side gas flows.

The diffuser is heated by five replaceable cartridge heaters mounted in wells which are coaxial with the five permeation coils. Each heater is provided with an axial iron/constantan thermocouple to allow the heater temperature to be monitored for failure. A failed cartridge can be replaced without shutting down the entire system. Also, operation can continue on the four remaining heaters until the fifth one is replaced. A sixth thermocouple is inserted in a well near the center of the pentagonal cluster of permeation coils. This thermocouple is used for temperature control of the unit. The manufacturer's recommended operating temperature of the unit is 425°C. The diffuser is surrounded by vermiculite insulation in a metal skin.
Feed gas enters the feed inlet tube, passes into the feed manifold, and is distributed into the five parallel permeation coils. Hydrogen isotopes permeate through the palladium-silver coils into the pure gas chamber, leaving byproduct material (helium) and impurities in the coils. The pure gas is evacuated through the product outlet tube and is collected in tanks prior to processing. Byproduct and impurities are discharged from the coils into the byproduct chamber. This gas is subsequently fed to the next RSI diffuser in series or to collection tanks.

RSI diffusers have all welded containment. They are not susceptible to cooling water and gasket leaks like the Multistage Palladium Diffuser. Also, the stages of the MPD are bolted to form a single unit. The other stages must be unbolted for repair or replacement of a defective stage. The RSI design allows each stage to be repaired independently. Because the RSI diffuser is commercially available, replacement parts

are less expensive than the specially designed MPD. The cost of replacing a MPD stage is estimated at $35,000 while replacing an RSI stage is estimated at $20,000. The RSI diffusers have been in service in the Tritium Facility since 1983. A total of thirteen stages are currently installed, nine in the old 234-H facility and four in the new 233-H Replacement Tritium Facility. Between November 1985 and June 1994, 56 RSI diffuser stages were procured by SRS: four were installed in the 233-H Replacement Tritium Facility, three are kept as spares, and the remaining 49 were used in the 234-H facility. These units have been reliable, each diffuser averaging 19.1 months between failures. These failures are mostly due to the presence of mercury in the Tritium Process (mercury diffusion pumps). The mercury attack of the palladium-silver alloy corrodes the tubing, shortening its life. There were frequent failures of the stainless steel to palladium-silver transition joint in the earlier RSI models [6]. Savannah River Site personnel worked with RSI to improve the braze design to prevent this type of failure [7]. The four stages installed in the Replacement Tritium Facility should experience an extended service life because of the absence of mercury in that process.

Johnson Matthey Incorporated

Two diffusers manufactured by Johnson Matthey, Inc. (JMI) were installed in the Tritium Facility diffuser system and tested as possible replacements for RSI diffusers. A second source was sought because of concern over cost, quality (braze joint failures), and delivery of RSI diffusers.

The JMI diffuser incorporated several design improvements over the RSI diffuser (Figure 3). The feed inlet tubing was coiled around the diffuser shell, under the insulation, to preheat the feed gas prior to entering the palladium coil. The JMI diffuser used only a single heater cartridge to heat the coils. The burnout of a heater cartridge was rare. A single heater allows for better control of the palladium-silver coil temperature.
Tests at SRS revealed that the JMI diffuser was not equivalent to the RSI diffuser in separation performance. When operated at the same feed rate, the product rate of the JMI diffusers was about half that of RSI diffusers. Much of the product remained in the byproduct stream. When operated at the minimum byproduct rate, the JMI diffusers had about one-fourth the product rate of RSI diffusers and still did not produce acceptable byproduct. The JMI diffusers were designed for permeation from the shell to the coil rather than from the coil to the shell as in the RSI diffuser designs. The JMI design allows hydrogen isotopes in the feed to bypass the palladium coil and show up in the byproduct [8].

The two JMI diffusers were used in Tritium Facility for four years. During that time, no failures of the units were recorded.

Adolph Coors Company

The Adolph Coors Company was contracted to manufacture a palladium diffuser similar to the RSI unit. This was done to develop an alternate commercial manufacturer. The Coors design incorporated the preheater and single heater design of the JMI diffuser but retained the coil-to-shell product gas pathway of the RSI design (Figure 4).

In the Coors unit the tight tolerance fit between the heater cartridge and its well has become a problem. A tight fit is required to maximize the heat transfer efficiency. However, when the stage is heated, the well and/or the heater cartridge can warp. The heater can become wedged in the cartridge, rendering the unit useless. Adjusting the tolerance will allow the replacement of heater cartridges, extending the usage of the palladium diffuser, while still providing for adequate heater performance.

The Coors diffusers were installed into the Tritium Facility diffuser system in 1993. Performance testing of the diffusers is continuing.

Pretreatment Systems

All gas to be fed to SRS diffuser systems is analyzed by mass spectrometer. This prevents a flammable mixture of hydrogen and oxygen from being fed to the diffuser. In the old 234-H process, the feed gas flows in turn through a hot uranium bed (U-bed), a zeolite bed , and finally through a sacrificial palladium bed. The U-bed reduces moisture in the feed stream, and this bed must be discarded after the uranium is completely oxidized. This bed is used when the feed gas contains an excessive amount of moisture. The zeolite bed also removes moisture at roughly ambient temperature, and if the temperature of this bed rises significantly, this is a sign that the previous uranium bed has become oxidized and must be replaced. Finally, the sacrificial palladium bed contains palladium supported by an alumina substrate, to trap any impurities that could react with the palladium silver diffuser tube alloy. The new 233-H Replacement Tritium Facility uses only the sacrificial palladium bed to treat the diffuser feed, because the 233-H process has much less moisture. The 233-H process confined in a nitrogen glovebox system that prevents air from entering the process and subsequently forming moisture.

DIFFUSER FAILURE MODES

Palladium and Palladium-Silver Diffusion Elements

Two failure mechanisms have occurred with the palladium and palladium-silver alloy coils in Savannah River Site Tritium Facilities: steam attack of the grain boundaries, and pitting or slow crack growth caused by contact with mercury [6]. Steam attack is caused by hydrogen isotopes reacting with excessive oxygen in palladium to form molecular water at grain boundaries. The water molecules accumulate, and form steam cavities

that open the boundaries and lead to failure. Steam attack is a recognized failure mechanism of palladium diffusers [9], and also occurs in copper. Water vapor in the process stream is not required for this reaction to occur, sufficient oxygen in the inlet stream or oxide inclusions in the palladium will cause the reaction. Liquid metal embrittlement [10] and liquid metal corrosion [11] are well known failure modes of metals and alloys.

Examples of Diffuser Failures

The second stage of a diffuser failed in 1963 when a large hole formed in the palladium coil. The failure analysis revealed grain growth and grain boundary damage near the hole [12]. These observations are consistent with steam attack.

A new palladium diffuser coil failed during testing in 1975, and inspection of the failure revealed intergranular cracking and palladium oxide near the fracture surface [13]. The leak was located by pressurizing to 7 psig (106 kPa). Prior visual attempts to find the leak in this coil failed. Additional tests with other diffuser coils showed the coils could be made to fail by formation of steam cavities at grain boundaries, if the coil was first oxidized at 850°C in air and then exposed to 5 atmospheres (506 kPa) deuterium at 425°C for one hour [13]. To reduce the chance of palladium coil failure, it was recommended that the original palladium should contain as little oxygen as possible, and that the amount of oxygen the diffuser is exposed to be as low as possible.

All five stages of a Resource Systems, Inc. (RSI) diffuser failed within ten months of installation in 1984. Failure analysis of Stages 2 through 5 revealed two features of the palladium-silver alloy [5]. Stages 2 and 3 had large holes surrounded by blistered metal, located near the stainless steel to palladium-silver braze joint. Stages 4 and 5 had cracked brittle areas that leaked at locations not associated with the braze joint. The report [5] suggested that the failure mechanism of Stages 2 and 3 was associated with an unknown impurity introduced while forming the braze joint because of the observed proximity of the failure to the joint. However, the observations in the report are more consistent with the creation of steam causing intergranular cavitation in all four stages examined.

Other diffuser coil failures are associated with mercury [6]. For example, investigation of one failure revealed compounds of mercury on the palladium and on the ceramic support used for the old multistage DuPont designed diffuser [14]. In another case, investigation of a coil that leaked in 1966 [15] revealed discoloration of the coil indicating a reaction had occurred with mercury [16]. A large amount of mercury flowed out of this stage when it was removed [15].

Examples of failure of palladium coils at Savannah River Site by hydride formation during inadvertent cooling of the membrane have not been found. This is unusual, because this failure mode is expected, assuming occasional inadvertent cooling of the palladium. One letter describing Savannah River Site experience with diffusers states

that most palladium coil failures resulted from mercury, and others were pinhole leaks possibly from hydriding [17]. However, the evidence found since then and discussed above reveals that steam attack and not hydriding is the most likely cause of the failures not caused by mercury attack. Heaters, heater controllers, and thermocouples have all failed on occasion, but apparently insufficient hydrogen isotopes were present in the palladium coils to cause hydriding at the lowest temperatures reached. Multiple thermocouples are normally placed in the diffuser, so that the control function of a failed thermocouple can be replaced by a backup. Eventually all must be replaced during routine maintenance [18].

The lifetime of palladium tubes in the older DuPont design varied from four months [19] to over five and a half years [15]. The lifetime was determined by the environment of the particular coil, especially to the extent of oxygen and mercury exposure.

Ancillary Equipment Failures

Many material failures in the diffuser system have involved ancillary components to the actual diffuser assembly. Most commonly, booster pumps before and after the various diffuser stages have failed in several ways. Many pump failures have been caused by liquid metal embrittlement or pitting, resulting in a slowly growing crack or pit that finally forms a leak path. Since many of these older pumps are water cooled, instances of slow crack growth by stress corrosion cracking have also been observed. This cracking can cause both cooling water and air leaks into the process. Some types of pumps contain bellows, and fatigue failure of bellows was commonly observed [18]. Mercury and compounds of mercury have plugged Sprengel pumps (the roughing pump for the mercury diffusion pumps) [20]. Valve filters have been plugged by these compounds [19]. Diffuser efficiency can be adversely affected if the pumping speed of pumps before or after the diffuser is lower than expected. Lower mercury diffusion pump speed was caused by dross in the mercury [21] and by excessive mercury evaporation during pumping.

DESIGN RECOMMENDATIONS FOR TRITIUM DIFFUSER SYSTEMS

1) Employ easily replaceable heaters and thermocouples, to reduce cost and worker exposure when heater and thermocouple replacement is required.

2) Heat with multiple heater elements and redundant control thermocouples to minimize the likelihood of a single heater or thermocouple failure leading to cooling the diffuser to a temperature where hydride phases can form, which can crack the tube.

3) Do not water cool diffusers, if possible. If water cooling must be used, the chemistry of the cooling water must be controlled to mitigate the potential for stress corrosion cracking.

4) Design the joint between the stainless steel and the palladium-silver alloy to minimize thermal stress. Since braze joints have been problematical, consider an alternative palladium-silver to stainless steel joint, such as a solid state resistance weld or an explosive bond.

5) Monitor and control the feed gas chemistry to prevent failure and to reduce aging of the diffuser coil. The technical basis for permissible oxygen (and water) concentrations exposed to the diffuser needs to be firmly established. Use various types of getter beds to decrease the amount of oxygen and waters that the coil is exposed to.

6) Do not use mercury diffusion pumps.

7) Do not use polymers in any part of the process unless absolutely necessary. If polymers are used (gaskets, valve parts, etc.), regular, preventative maintenance and replacement will be necessary.

8) Install an automatic pump down subsystem in the diffuser system, to remove hydrogen isotopes from the diffuser should the temperature go below a set value. This pump down system should be powered by an uninterruptable power supply, should the facility power fail.

9) Avoid locations in the design where water can condense and collect in process lines. This is especially true if active cooling (water, for example) is used, which can locally condense waters in the process stream and possibly lead to corrosion.

10) Include adequate on-line pump diagnostics, to facilitate determining the location of a bad pump. Diffuser performance can be limited by pump performance on the feed, product, and byproduct lines.

11) Use a modular design, with several diffuser units attached together to enable replacement of any diffuser with minimal effort and time. This also allows diffuser operations to continue by "valving out" a broken unit.

12) Include an air bleed to the inlet side of the diffuser to allow regeneration by reaction of hydrocarbons on the palladium-silver surface with air at elevated temperature.

13) Ensure that the purity of palladium-silver diffuser coil alloy is very high, especially regarding oxygen and oxide inclusion content.

ALTERNATIVE TECHNOLOGIES APPLIED AT SRS

As discussed in the above sections, palladium-silver diffusers can be high maintenance items which may require frequent replacement. Due to their relatively slow separation rate and maintenance requirements, hydrogen isotope purification can often became the

bottleneck in a process. It is, therefore, desirable to replace or supplement the diffusers with new and improved purification processes.

Three development projects were planned in the early 1980's at SRS to develop metal hydride purification processes to replace palladium diffusers. The first was a hydrogen pump/purifier to produce pure hydrogen isotopes for the cryogenic distillation columns. This separator was developed, installed, started up and has been used since 1987. The second project was a primary separator for use upstream of the diffusers to take over the main separation duty from the diffusers. The primary separator was installed in the Replacement Tritium Facility and successfully started up early this year. The third project was an advanced hydrogen isotopes purifier to supplement the primary separator and completely replace the diffusers. This advanced purifier is still under development.

Cryogenic Still Feed Pump/Purifier

Before the pump/purifier was installed, hydrogen isotopes were fed to the cryogenic still by expanding the gas from large tanks pressurized to slightly above atmospheric pressure. Feed to the still must be free of condensable gases, such as moisture and air, and a minimum amount of helium. To avoid in-leakage of air, the tanks were kept at positive pressures. Storing tritium above atmospheric pressure creates the undesirable potential for tritium release to the environment. To maintain a low helium concentration, the gas must be processed quickly, otherwise the accumulation of helium makes it necessary to recycle the gas for helium separation by the palladium-silver diffusers. The still feed pump/purifier was developed to avoid the need for both storing tritium above atmospheric pressure and reprocessing the accumulated helium.

The cryogenic still feed pump/purifier was developed and implemented in 1987. It is a bed of palladium supported on kieselguhr (Pd/k) that pumps and purifies hydrogen isotopes fed to the cryogenic distillation column. Kieselguhr is a highly porous diatomite and is used for its high surface area (8 m^3/g) and hydrophobic nature. The Pd/k contains 50% palladium by weight. The particle size of the Pd/k is between 250 and 600 micron. The unit consists of three vessels connected in parallel (Figure 5). Each vessel is heated by electrical heaters in heater wells, and cooled by Freon™ cooling coils wrapped on the outside of the vessel wall. A secondary vessel is used as a vacuum jacket for thermal insulation and containment [22].

A typical run includes three steps: absorption, inert pump-off, and desorption. In the absorption step, the vessels are chilled by the cooling coils. Gas in storage tanks is drawn to the unit by the palladium absorption of the hydrogen isotopes. Inerts, which are not absorbed by the palladium, are left in the void space of the Pd/k bed. The flow of gas to the bed will continue until the tanks are empty, the bed is saturated, or the accumulated inert gas pressure is equal to the tank pressure. In the inert pump-off step, the inert gas accumulated in the Pd/k vessels is pumped out to inert storage tanks. The absorption step and the inert pump-off steps may have to be repeated several times if

the feed gas contains a high level of inerts. In the desorption step, the cooling is stopped and the heaters are turned on to desorb the hydrogen from the Pd/K. The high purity hydrogen gas is fed directly into the cryogenic still.

Primary Separator (Flow-through Bed)

The flow-through bed is a semi-continuous version of the feed pump/purifier discussed above. It is part of the gas purification process in the Replacement Tritium Facility (RTF) which successfully started in 1994. The flow-through bed was designed to take over the main inert separation duty thereby reducing the load on the diffusers in the RTF. The diffusers now only have to remove low levels of hydrogen from the gas passing through the flow-through bed.

The flow-through beds are U-shape columns filled with palladium supported on kieselguhr (Figure 6), the same as that used in the still feed pump/purifier. The U-shape column achieves the desired length-to-radius ratio without increasing the height, so that the unit can easily fit in a glovebox. Each column is heated and cooled by hot and cold nitrogen circulating in a jacket. Thermocouples in wells are placed along the length of the columns for the detection of the absorption front. In the RTF three U-shape columns are used to form one unit. The first two columns are connected in parallel, while the third is in series.

A typical operation cycle consists of an absorption step and a desorption step. In the absorption step, the column is first cooled to less than -20 °C. The feed gas in a tank is then expanded into the column. The hydrogen is absorbed by the palladium while the inert gas continues to flow through the column. A pump at the exit end of the unit draws the inert gas and sends it to the diffuser for further hydrogen cleanup. This absorption step continues until the feed gas is exhausted or until the bed is full (saturated). Hydrogen is first absorbed at the inlet end of the column, forming a saturated section. This saturated section extends progressively toward the outlet end of the column. Most of the hydrogen absorption occurs at the front of the saturated section (the absorption front). The heat of absorption at the absorption front results in a temperature increase which produces a temperature peak of up to 30 °C. The detection of this temperature peak allows for precise indication of the absorption front and is used to determine and control the saturation of the column. When a temperature peak is detected near the exit end of the column, the absorption front is about to break through and the column is desorbed.

In the desorption phase, the column is heated to desorb the hydrogen from the palladium. The gas pressure generated by desorption is up to 1 atmosphere (101 kPa) and is used to transfer the hydrogen to a storage bed or a tank. After desorption, the column is regenerated and is ready to return on-line. Normally, two units are used and are connected in parallel. One unit is on-line while the other one is being regenerated or on standby [24].

The flow-through bed in the RTF was started up successfully in January of 1994 and has been operated routinely. Hydrogen isotope and helium purities have typically been better than expected, and have exceeded design goals.

Advanced Hydrogen Isotope Purifier

The cryogenic still feed pump/purifier and the flow-through bed discussed above are both based on palladium supported on kieselguhr. Both processes are very efficient at removing most of the hydrogen (better than 99.5%) from the feed and producing high purity hydrogen. However, they can only reduce the hydrogen level in an inert stream to about 0.1%. A palladium-silver diffuser is required to further reduce the hydrogen level to less than 0.05%. Further reduction of tritium down to part per million levels is often desired. It is impractical for diffusers to achieve part per million levels of hydrogen in inert streams. It is, therefore, highly desirable to develop an alternative process which can both replace the function of a diffuser and reduce the hydrogen in an inert stream down to part per million levels. Testing of low pressure metal hydrides and commercially available getters for this purpose at SRS are being performed.

$NdCo_3$

Neodymium-cobalt ($NdCo_3$) was selected from a series of low pressure metal hydrides for bench scale test to separate hydrogen from helium. The tests consisted of processing batches of hydrogen and helium gas mixtures through a column filled with $NdCo_3$ powder. The results show that a 50% hydrogen in helium feed stream is purified to a helium stream of purity greater than 99.98% (0.02% hydrogen). The column was operated at room temperature and at a flow rate of 30 liter/min/cm^2. The column was regenerated by heating to 150 °C under vacuum to desorb the hydrogen. These results indicate that a $NdCo_3$ column can produce an inert stream with purity matching that of a diffuser, although it cannot achieve part per million levels of purity [25].

SAES Getters

The ability of SAES St 198™ (ZrFe) to remove low levels of hydrogen from a nitrogen stream was tested. Nitrogen containing about 100 part per million deuterium was passed through a packed bed of St 198 granules. The column was maintained at 250 °C in one case and at 350 °C in the other. In both cases, the deuterium level was reduced to less than the detection limit of the mass spectrometry of 0.5 part per million. The results showed that St 198 can strip hydrogen from nitrogen or inert streams down to the part per million level and should be further investigated [26].

Titanium Sponge

Titanium is known to absorb hydrogen at high temperatures and the resultant hydride has a very low equilibrium pressure (10^{-14} Pa). It has the potential for stripping tritium from inert gas streams. Tests showed that titanium sponge absorbs hydrogen readily at 200 °C after it has been activated at 500 °C under vacuum. It also absorbs hydrogen

readily at room temperature if the activation temperature is increased to 700 °C. Titanium has a very high capacity for hydrogen, up to 4% by weight. The capacity is reversible if the hydride is regenerated at 700 °C under a vacuum of 1 torr (133 Pa) or better. Half of its capacity (2%) is reversible if the regeneration temperature is decreased to 500 °C. These data indicate that when kept at about 200 °C, titanium should be able to strip hydrogen from an inert gas stream. An experimental unit has been set up and preliminary results are expected later this year.

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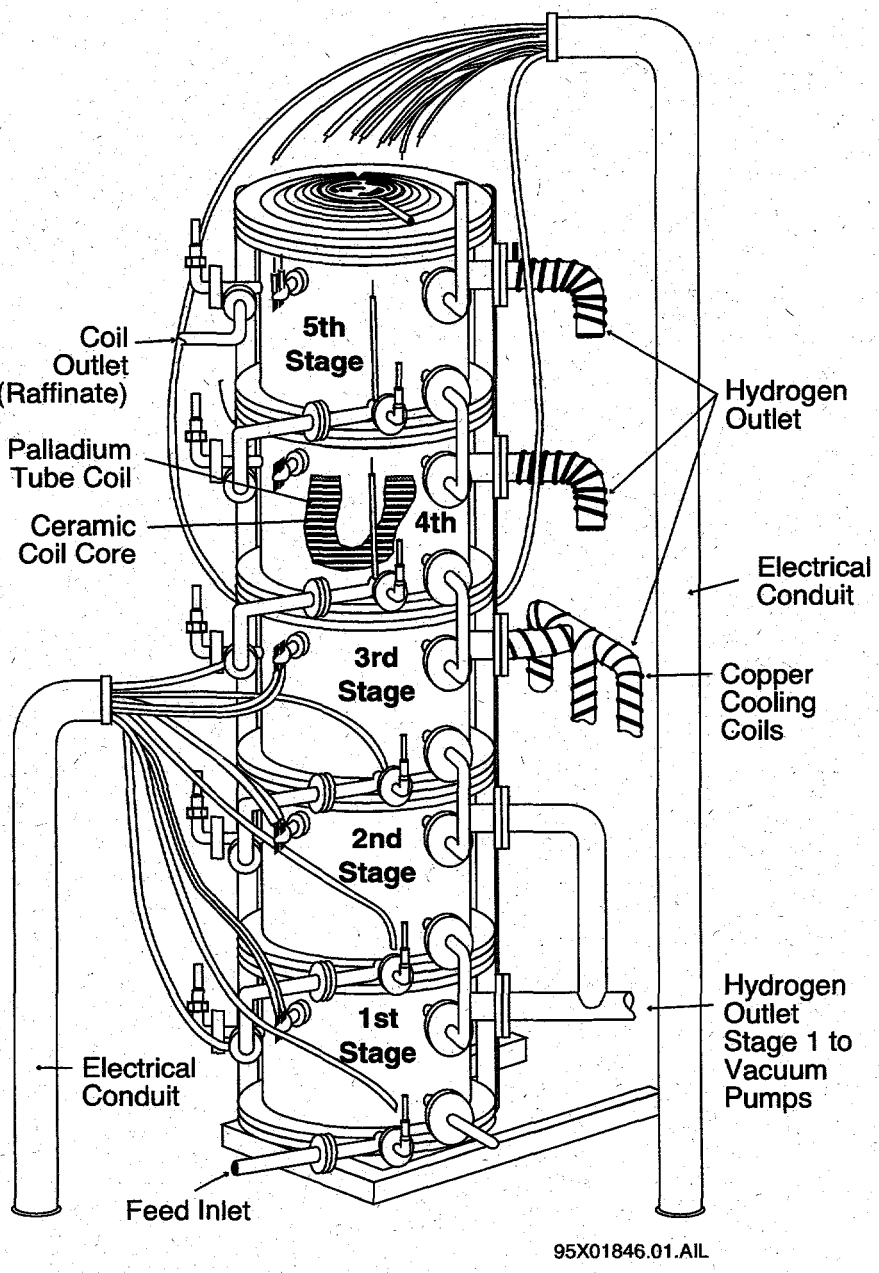

Figure 1. Multistage Palladium Diffuser

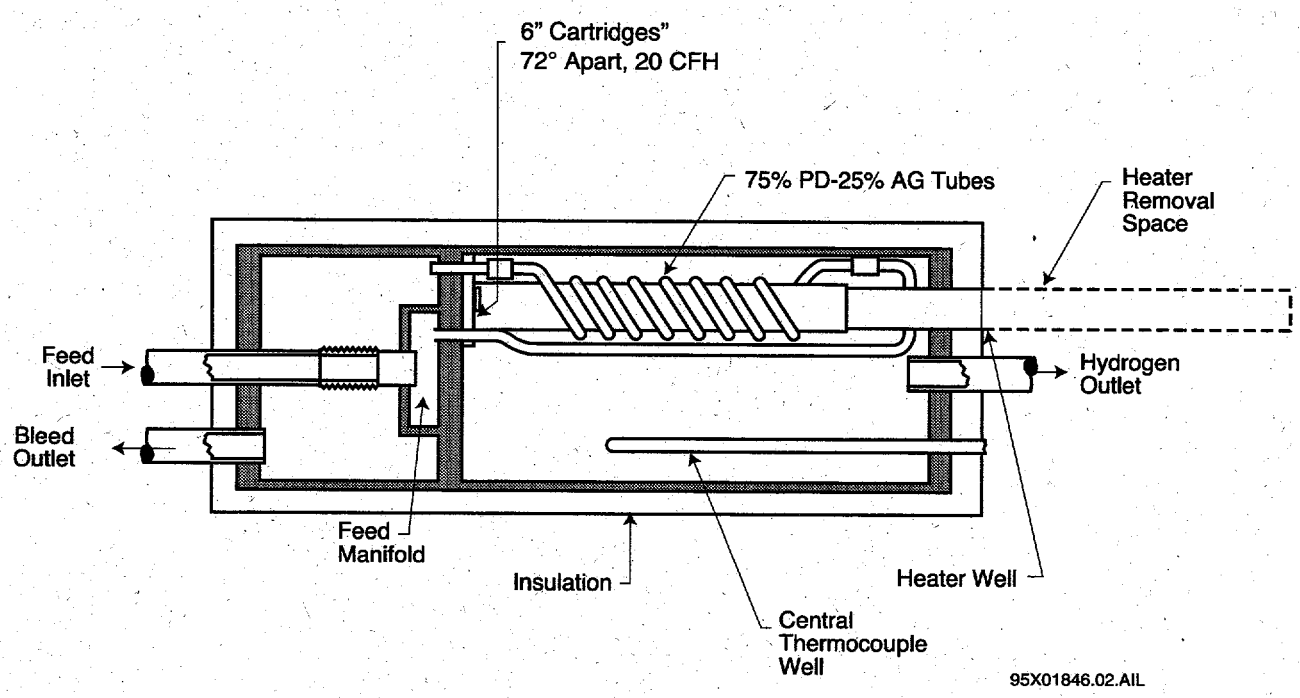

Figure 2. Single Stage RSI Diffuser

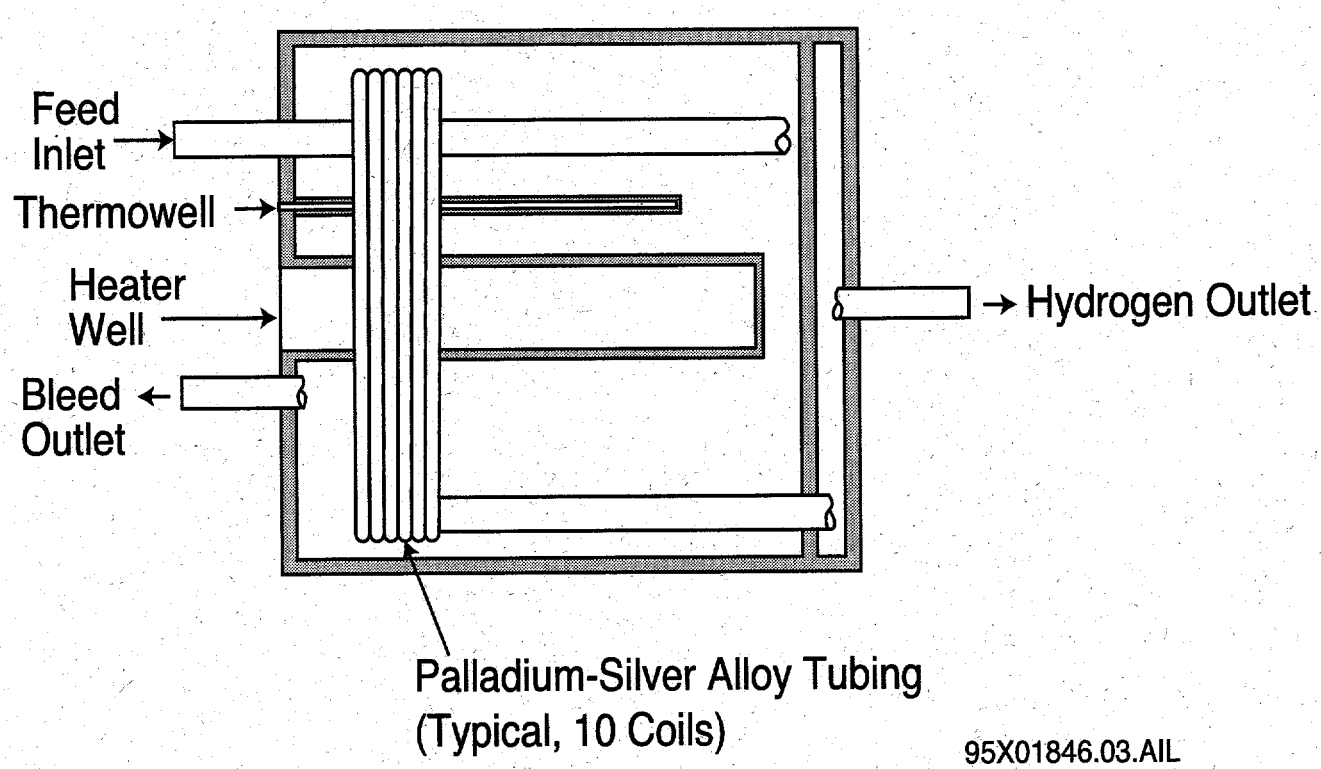

Figure 3. Johnson Matthey, Incorporated (JMI) Diffuser

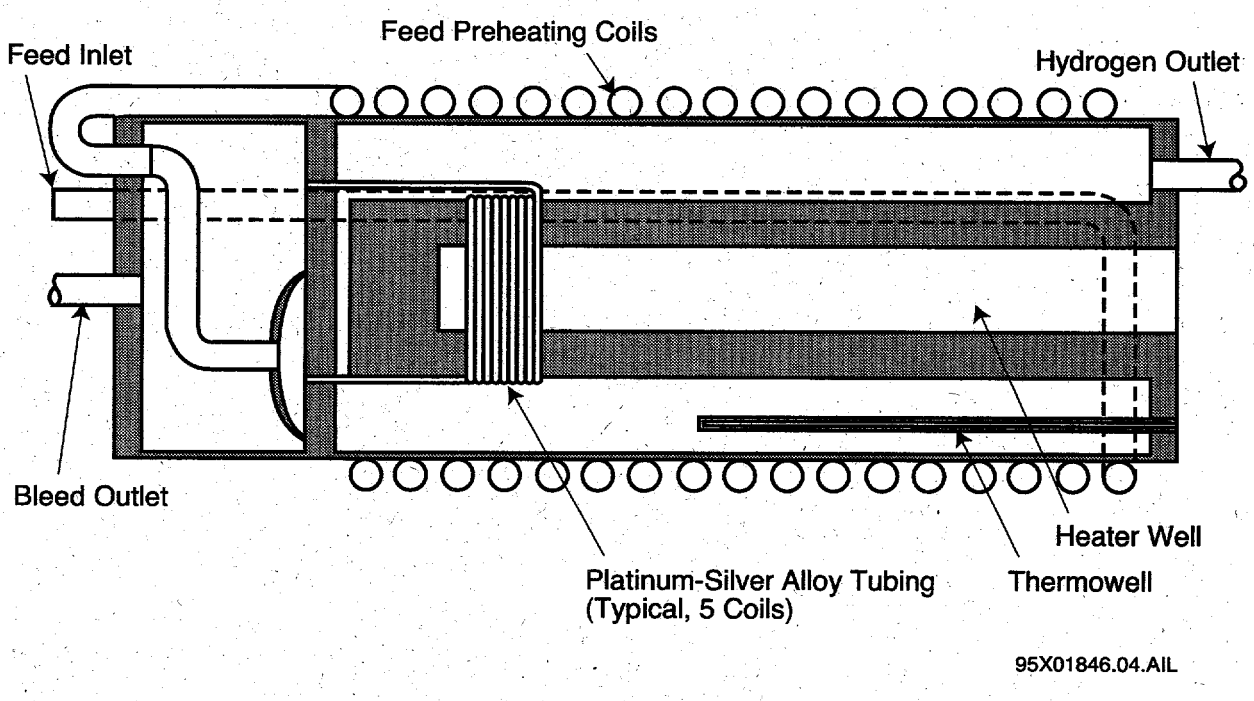

Figure 4. Adolph Coors Company Diffuser

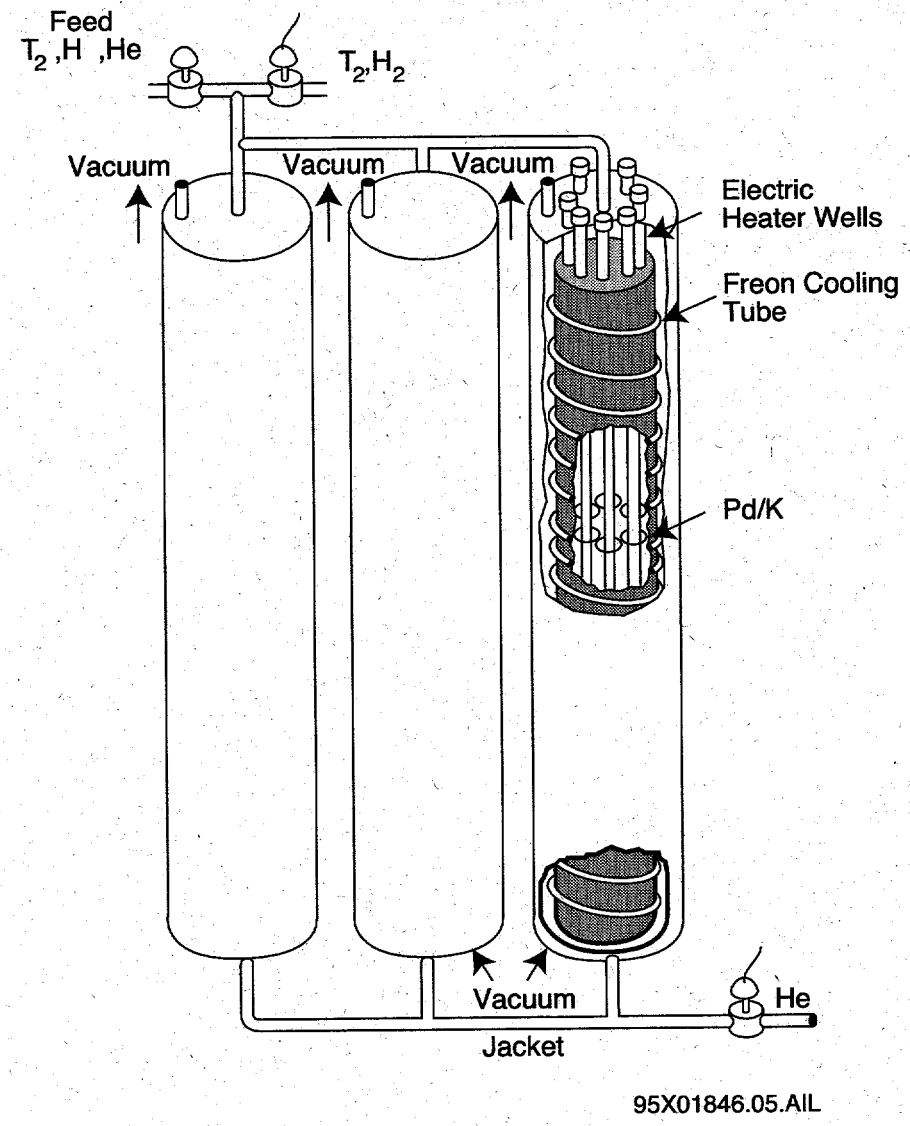

Figure 5. Still Feed Pump Separator - Design

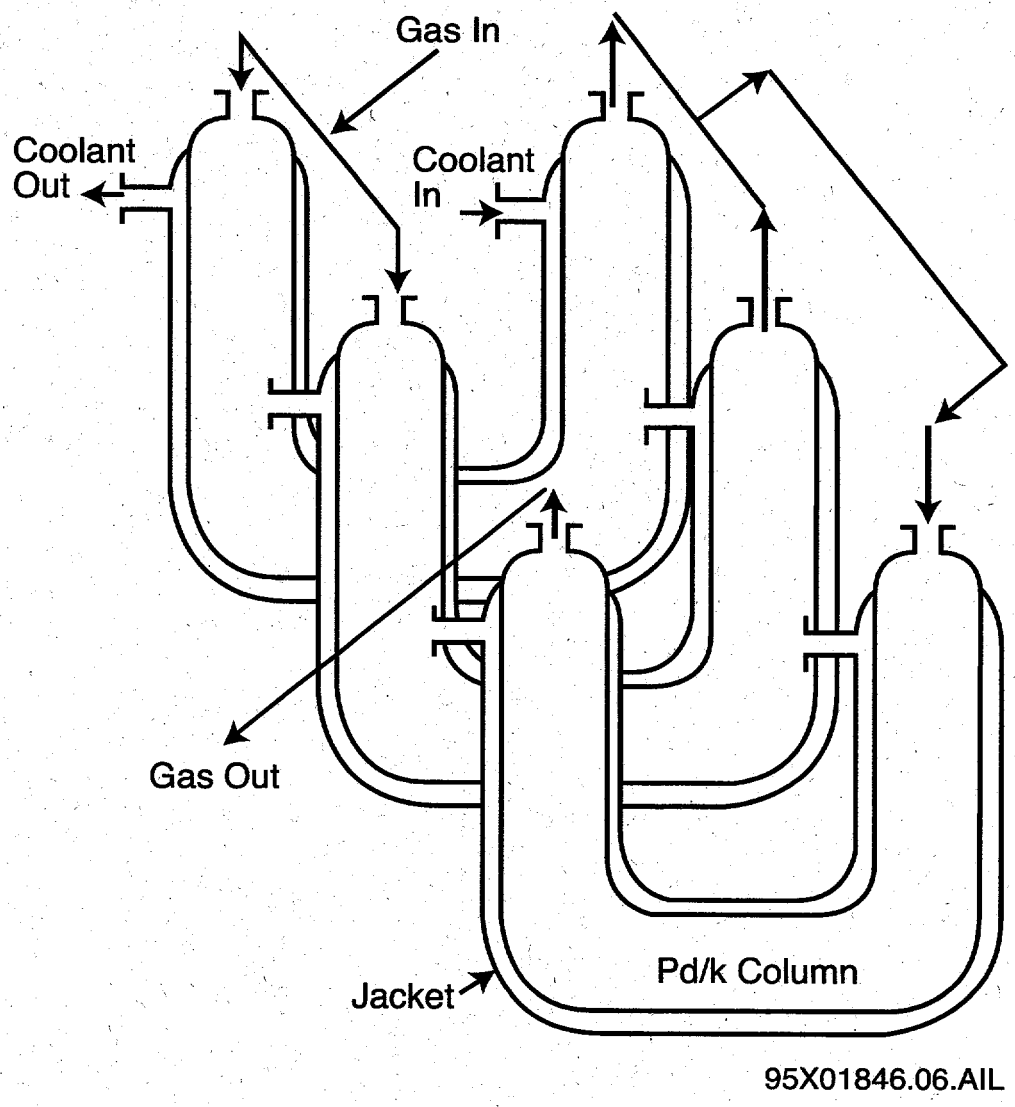

Figure 6. Primary Separator (Flow Through Bed)

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