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## ADVANCED HYDRIDE LABORATORY

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## ADVANCED HYDRIDE LABORATORY

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### Abstract

Metal hydrides have been used at the Savannah River Tritium Facilities since 1984. However, the most extensive application of metal hydride technology at the Savannah River Site is being planned for the Replacement Tritium Facility, a \$140 million facility scheduled for completion in 1990 and startup in 1991. In the new facility, metal hydride technology will be used to store, separate, isotopically purify, pump, and compress hydrogen isotopes.

In support of the Replacement Tritium Facility, a \$3.2 million, "cold", process demonstration facility, the Advanced Hydride Laboratory began operation in November of 1987. The purpose of the Advanced Hydride Laboratory is to demonstrate the Replacement Tritium Facility's metal hydride technology by integrating the various unit operations into an overall process.

This paper will describe the Advanced Hydride Laboratory, its role and its impact on the application of metal hydride technology to tritium handling.

### 1. INTRODUCTION

The Savannah River Site is located on 300 square miles of federally owned land in western South Carolina along the Savannah River. The primary mission of the Savannah River Site is to produce special nuclear materials for national defense and civilian uses. One of the principal nuclear materials produced at the site is tritium.

Tritium processing operations have been performed at SRS since 1955. During this time, improvements in tritium handling and processing have been continually evolving. In 1987 construction began on a new tritium facility, the Replacement Tritium Facility. The Replacement Tritium Facility will replace the current tritium loading and unloading facility with state-of-the-art technology. One of the new technologies that will be incorporated into the Replacement Tritium Facility will be the use of metal hydrides to store, separate, purify, pump, and compress hydrogen isotopes. The Replacement Tritium Facility is scheduled to begin operation in 1991.

In support of the Replacement Tritium Facility, a \$3.2 million, process demonstration facility, the Advanced Hydride Laboratory was constructed and began operation in November of 1987. The purpose of the Advanced Hydride Laboratory is to demonstrate the Replacement Tritium Facility's metal hydride technology by integrating the various unit operation into an overall process.

In this paper the Advanced Hydride Laboratory will be described, its objectives will be discussed, and some experimental results will be reviewed.

## 2. BACKGROUND

### Metal Hydrides

The ability of certain metals and intermetallic compounds to rapidly and reversibly absorb and desorb hydrogen gas at room temperature and atmospheric pressure to form metal hydride compounds represents a technology ideally suited to handling and processing tritium. The hydriding and dehydriding reactions can be used as a basis for the storage, pumping, purification, and separation of hydrogen isotopes. One of the most important advantages of using metal hydride technology is the compact size of metal hydride systems. Metal hydrides have the ability to store hydrogen at densities greater than that of liquid or solid hydrogen. The use of compact hydride vessels in place of conventional process tanks in the Replacement Tritium Facility has led to cost savings of tens of millions of dollars by reducing the size of the facility. Other benefits of metal hydride technology are improved safety by taking advantage of the ability of metal hydrides to store tritium at low pressures (even below atmospheric) and improved reliability by making use of the metal hydrides ability to transport and compress tritium without mechanical parts other than valves [1].

### Advanced Hydride Laboratory Project

While much of the Replacement Tritium Facility's metal hydride technology had been demonstrated in laboratory bench-scale and pilot-scale units prior to 1987, none of the units had been operated together and integrated into an overall process. This was the major objective of the Advance Hydride Laboratory. With construction of the Replacement Tritium Facility nearly underway and startup scheduled just four years away, it was imperative to start up the Advance Hydride Laboratory in time to make an impact on the plant facility.

The Advance Hydride Laboratory was initiated as a "fast track" project. Project Authorization for the Advance Hydride Laboratory was received in December of 1986. The project was

designed in stages so that construction could begin after each stage was completed. Design was essentially completed by the end of April 1987. Construction began in January of 1987 and the facility was physically completed by October 1987. At this time, calibration and startup activities began. On November 10, 1987 the Advanced Hydride Laboratory officially began operation. Overall the Advanced Hydride Laboratory project was completed on schedule and within budget. Excellent cooperation between design and construction groups was essential to the successful completion of this project.

In two years of operation, the Advanced Hydride Laboratory has demonstrated all the metal hydride unit operations to be used in the Replacement Tritium Facility. Metal hydride processes have been integrated, and startup and operating information has been transmitted to the plant. During construction and operation of the Advanced Hydride Laboratory, several key design changes in the Replacement Tritium Facility were made which have already led to substantial savings in startup time and costs. The Advanced Hydride Laboratory continues to generate plant startup and operating data, and is also being used as a training facility for the full-scale plant facility.

### 3. ADVANCED HYDRIDE LABORATORY DESCRIPTION

#### Layout

The Advanced Hydride Laboratory is divided into three main parts: the hydrogen hood, the outside nitrogen circulation system, and the control room. The layout of the Advanced Hydride Laboratory is shown in Figure 1. The hydrogen hood is a 10 by 15 foot, walk-in, process hood which contains the metal hydride applications. Non-radioactive hydrogen and deuterium gases are used in place of tritium in this hood. The hood has been designed to comply with the National Electric Code requirements for a "contained hydrogen handling facility". This basically states that no ignition sources be present. To further mitigate a potential hydrogen explosion, a one room change/minute air flow through the hood has been established for normal operations.

The nitrogen circulation system is the process heating and cooling system for the Advanced Hydride Laboratory. This system is located outside of the main facility and its function is to provide hot and cold nitrogen gas to each of the metal hydride applications in the process hood. The nitrogen system is comprised of: a two stage refrigeration system to cool the gas, a 60 kilowatt electric resistance heater to heat the gas, and two 5000 pounds per hour compressors to circulate the hot and cold nitrogen gas to and from the metal hydride applications.

The third part of the Advanced Hydride Laboratory is the control room. The control room is located next door to the process hood and houses the major components of the control system. The metal hydride processes in the Advanced Hydride Laboratory are sequenced and controlled by a programmable logic controller (PLC). The process can be entirely automated or operated remotely from the control room. In addition to controlling the process, the Advanced Hydride Laboratory's control system is also used to evaluate process control schemes and strategies for the plant facility. The nitrogen heating and cooling system is controlled by a stand alone system using a series of relays from a utility panel located in the control room.

### Hydrogen Process

The Advanced Hydride Laboratory's hydrogen process contains thirteen metal hydride beds (or vessels). A schematic drawing of a typical metal hydride bed is shown in Figure 2. The bed consists of an inner vessel that contains the metal hydride material and an outer annular volume in which the hot or cold nitrogen gas can circulate to raise or lower the temperature of the bed. A stainless steel filter element is located on the inner hydrogen vessel to contain the metal hydride particles within the bed. The metal hydride beds are grouped into three subsystems: Inert Separation, Isotope Separation, and Compressors. A schematic of the Advanced Hydride Laboratory process is shown in Figure 3. The Inert Separation system makes use of a "flow-through" metal hydride bed and a pair of metal hydride "vacuum" beds to separate helium from a mixture of hydrogen isotopes. When the flow-through bed is cold, a mixture of helium gas and hydrogen isotopes is pulled through the bed. The hydrogen isotopes are absorbed onto the metal hydride material in the bed and the helium gas is removed from the outlet of the bed. The separated helium is further purified by two hydrogen diffusers. At high-temperature a palladium tube in the diffusers allows diffusion of the hydrogen isotopes but not the helium gas. To improve the operation of the diffusers, a 20 torr vacuum on the hydrogen side of the first diffuser is maintained by a metal hydride vacuum bed system. Once the hydrogen isotopes and helium gas have been separated, the flow-through bed and the vacuum beds are heated to transport the hydrogen isotopes from the Inert Separation system to the Isotope Separation system.

The Isotope Separation system consists of: a TCAP (Thermal Cyclic Absorption Process) unit, two metal hydride feed beds, and two pairs of product beds. TCAP is a semi-continuous chromatographic separation process developed by the Savannah River Laboratory to separate hydrogen isotopes. The TCAP process, which uses palladium on kieselguhr as a packing material, takes advantage of

palladium's very large isotopic effect. Palladium preferentially absorbs the lighter isotope of hydrogen, protium over the heavier isotopes deuterium and tritium. This effect increases as the temperature decreases. Repeatedly heating and cooling the TCAP column and absorbing and desorbing the hydrogen isotopes in a controlled sequence generates a hydrogen isotope profile in the column. By withdrawing small portions of the column's hydrogen inventory as product from each end of the column during the heating cycle and then supplying an equal amount of feed during the cooling cycle, a semi-continuous isotopic separation process can be created [2]. The separated hydrogen isotopes from TCAP are each removed and stored in a pair of metal hydride beds. A pair of storage beds are used so that one bed is always available to receive gas while the other bed is being recycled.

The final subsystem in the Advanced Hydride Laboratory consists of two metal hydride compressor beds for compressing hydrogen gas to over 10 atmospheres of pressure. The metal hydride compressor system takes advantage of the ability of different metal hydride materials to absorb and desorb hydrogen gas at different temperature and pressure ranges. A first stage metal hydride compressor can be designed to absorb gas at or near atmospheric pressure and then to desorb and deliver gas at an elevated temperature and pressure to a second stage compressor bed. The second stage bed contains a different hydride material that only begins to absorb appreciable amounts of hydrogen at elevated pressures. When the second stage bed is subsequently heated and desorbed it will deliver gas at even higher pressures. While only a two stage metal hydride compressor was demonstrated in the Advanced Hydride Laboratory, a three stage metal hydride compressor has been successfully demonstrated in the Savannah River Laboratory delivering hydrogen gas at over 1000 atmospheres of pressure.

#### Nitrogen Heating and Cooling System

The hydride beds in the Advanced Hydride Laboratory hood are heated and cooled using gaseous nitrogen from a separate nitrogen circulation system. A schematic of the nitrogen heating and cooling system is shown in Figure 4. The nitrogen heating and cooling system is similar to that which is to be used by the Replacement Tritium Facility. Nitrogen is used in the plant to minimize the impact of any tritium release to the heating and cooling system. Any tritium in the heating and cooling system can be removed using the same tritium stripper system that services the nitrogen gloveboxes in the Replacement Tritium Facility. All of the plant's tritium handling equipment is contained in subatmospheric pressure nitrogen gloveboxes. Any process tritium leak will be confined to a glovebox and then captured and returned to the process by the stripper system.

The Advanced Hydride Laboratory's nitrogen heating and cooling system is capable of supplying 5000 pounds per hour of hot or cold gas to the metal hydride beds. Two reciprocating compressors, one on the hot and one on the cold nitrogen loops, provide the needed gas flow. The hot gas loop contains a 60 kilowatt electric resistance heater which is capable of heating the gas by more than 300 °F in a single pass. A small heat exchanger is also installed in the hot nitrogen loop to moderate the temperature of the hot gas returning to the hot gas compressor. The cold loop contains a two stage freon refrigeration system capable of 15.5 tons of cooling at -72 °F. A small cooling tower is also installed as part of the nitrogen system to provide cooling water to the condenser on the refrigeration system and the heat exchanger on the hot gas loop. Both the hot and cold gas loops continually circulate nitrogen gas.

#### Control System

The function of the Advanced Hydride Laboratory's Control System is to provide automatic and manual operation of the metal hydride equipment in the process hood. This basically consists of sequencing both process and nitrogen heating and cooling valves, and monitoring input/output (I/O) sensors. In addition, the control system provides operators with: supervisory control of the process through real time graphic screens, alarm handling, and data acquisition. The major components of the control system are: a programmable logic-controller (PLC), I/O modules, a microcomputer, color graphics monitors, solenoid valves, pump start/stops, and pressure & temperature sensors.

The actual process control is performed by the PLC and the I/O modules. A program is written and stored in the PLC. This program contains the valve sequencing and operating conditions for the metal hydride equipment. Input and output for the PLC is through the I/O base and I/O modules. Data acquisition functions are provided by a special software program operating on a microcomputer. This program collects and stores data from the PLC sensors and can display the stored data in table and graphic formats. The microcomputer also provides real-time supervisory control of the process through graphics displays. In this mode of operation, the operator can change setpoints and operate key process valves from keyboard commands and observe the effect of these changes from the graphic displays.

#### 4. EXPERIMENTAL PROGRAM

##### Objectives

The overall objective of the Advanced Hydride Laboratory is to provide a process demonstration of the integrated metal

hydride technology that is to be used in the plant's Replacement Tritium Facility. While most of the individual metal hydride applications for the Replacement Tritium Facility have been evaluated previously in various laboratory scale facilities, a large-scale, integrated demonstration of the Replacement Tritium Facility metal hydride technology has never been performed. Also, a large-scale nitrogen heating and cooling system to heat and cool metal hydride beds has never been evaluated. In addition to these major objectives, other objectives of the Advanced Hydride Laboratory include: actual plant component and equipment testing, process control and strategy evaluation, and plant startup support and operator training.

### Results

Even before operation of the Advanced Hydrogen Laboratory began, several key accomplishments were made during its construction phase. One of these accomplishments involved the identification of an inadequate nitrogen valve that was being specified throughout the plant facility. Early detection of this inadequate valve has saved the plant several millions of dollars in startup expenses.

In the process area, all three of the Advanced Hydrogen Laboratory's metal hydride subsystems have been demonstrated. In the Inert Separation subsystem, operation of the primary "flow-through" bed in conjunction with both the palladium diffusers and the vacuum bed has been evaluated. Plant requirements on both hydrogen and helium purity have also been demonstrated. In addition, a new temperature measurement technique has been developed to determine the extent of bed fill for the "flow-through" bed. This technique makes use of the large exothermic heat of absorption and the rapid absorption rate between hydrogen and the metal hydride material. As hydrogen is absorbed onto the "flow-through" bed, it has been determined that it is absorbed as a "front" along the depth of the bed. Thermocouples placed at different depths in the bed can record the temperature rise caused by the absorbing hydrogen at each location in the bed. One use of this technique would be to help determine when a "flow-through" bed might be fully absorbed with hydrogen so that either the process can be stopped or another bed can be put on line. Figure 5 shows some experimental data verifying the rapid rate of temperature rise occurring at the end of the "flow-through" bed when it is nearly completely absorbed with hydrogen.

In the Isotope Separation subsystem, the operation of a new TCAP unit was demonstrated. During initial testing of the TCAP unit, a serious operating problem dealing with the gas transport within the unit was identified. Using the Advanced Hydride

Laboratory, the problem was quickly diagnosed and corrected. If the problem had gone unnoticed, critical startup delays would have occurred in the plant facility. Some experimental data from the Advanced Hydride Laboratory's TCAP unit is shown in Figure 6. The two curves in Figure 6 show the degree of isotope separation obtained versus the number of operating cycles when a 50-50 mixture of hydrogen (protium) and deuterium are initially introduced to the unit. Overall, the results show that a high degree of hydrogen and deuterium separation can be achieved.

The final metal hydride subsystem demonstrated in the Advanced Hydride Laboratory is the Compressor subsystem. Figure 7 shows the results from a typical operating run of the compressor system. In this system hydrogen gas is transported from one metal hydride bed to another, increasing the delivery pressure of the hydrogen gas during each transport operation. The first part of Figure 7 shows gas being absorbed by the first stage compressor. The next part of the figure shows gas both being desorbed from the first stage compressor and simultaneously being absorbed onto the higher pressure second stage compressor. The last part of Figure 7 shows gas being desorbed from the second stage compressor and filling two fixed volumes to a given loading pressure.

In addition to evaluating the metal hydride aspects of the Replacement Tritium Facility process, the Advanced Hydride Laboratory has also demonstrated long-term operation of a nitrogen heating and cooling system. Over two years of operating data from the Advanced Hydride Laboratory's nitrogen heating and cooling system have been documented and transmitted to the plant. Information in this area includes both individual component failure data along with overall system performance. Another area where the Advanced Hydride Laboratory has provided plant assistance is in the area of process control. While much of this work is still ongoing, several of the key parameters used to control plant operations have been identified. Modifications to the Advanced Hydride Laboratory are currently in place so that actual plant process control schemes can be further evaluated.

In addition to technical information, the Advanced Hydride Laboratory also provides the plant with early process experience. Since startup, several new plant engineers have been employed in the Advanced Hydride Laboratory. This not only provided valuable manpower to support laboratory activities, but also provided early "hands on" training to future plant operators and engineers. Numerous tours and process demonstrations have also been presented to both plant operations and engineering personnel. These presentations have proven to be an invaluable vehicle for smoothly transferring laboratory-developed technology to the plant.

## 5. SUMMARY AND CONCLUSIONS

The overall objective of the Advanced Hydride Laboratory, to provide a process demonstration of the integrated metal hydride technology for the plant Replacement Tritium Facility, has been accomplished. All of the individual metal hydride applications to be used in the plant have been demonstrated and a large-scale, nitrogen heating and cooling system to heat and cool metal hydride beds has been evaluated. In addition to these major accomplishments, the Advanced Hydride Laboratory has also provided: actual plant component and equipment testing, process control and strategy evaluation, and plant startup support and operator training. Further work in the areas of process optimization and operator training are continuing.

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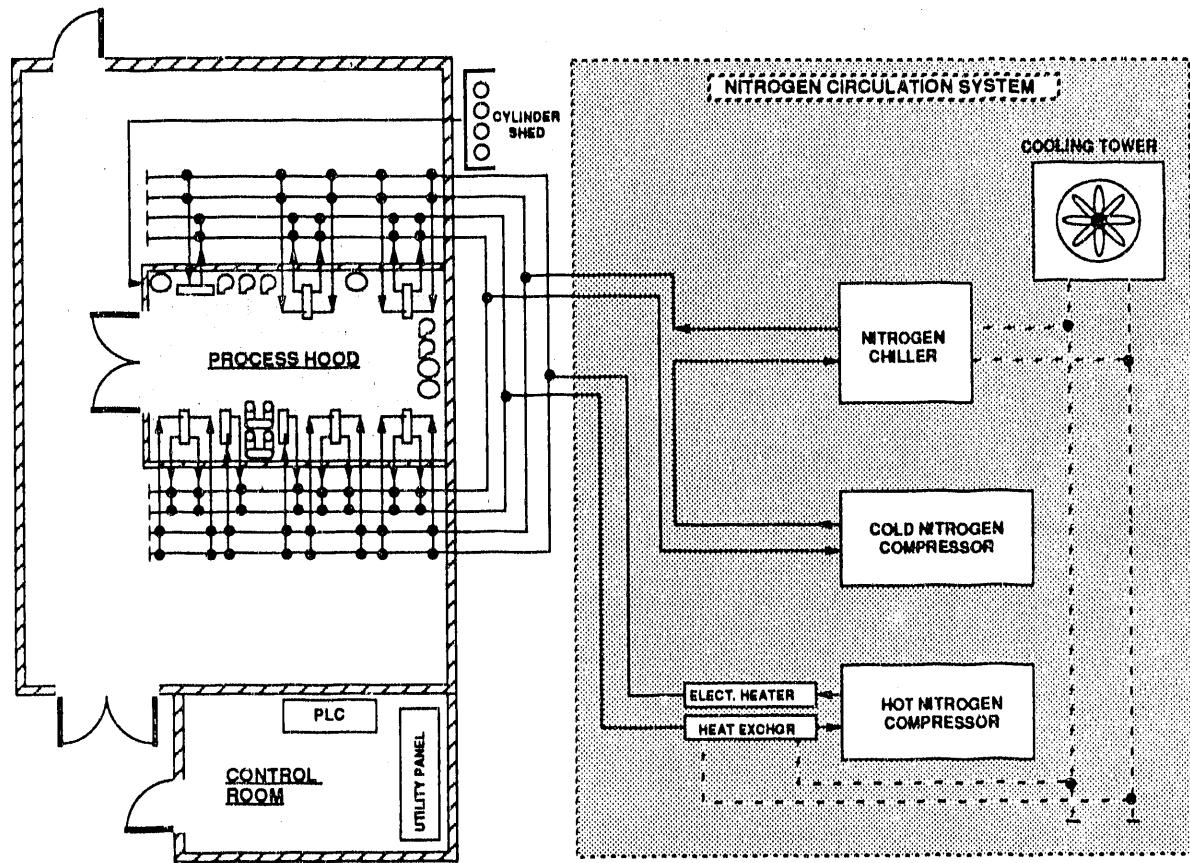


Fig. 1. - Advanced Hydride Laboratory Layout

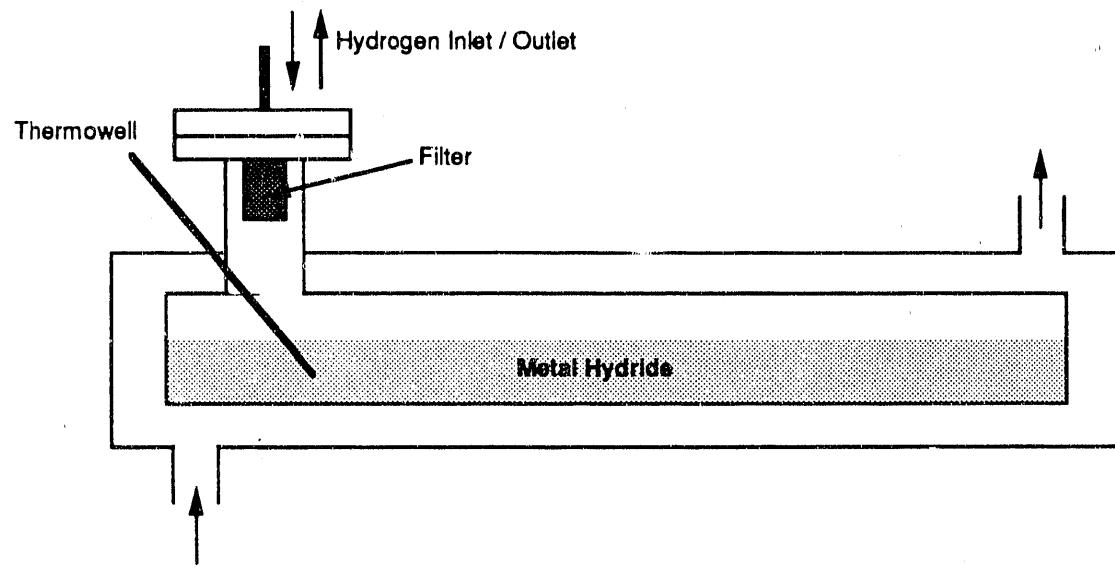


Fig. 2. - Typical Metal Hydride Bed

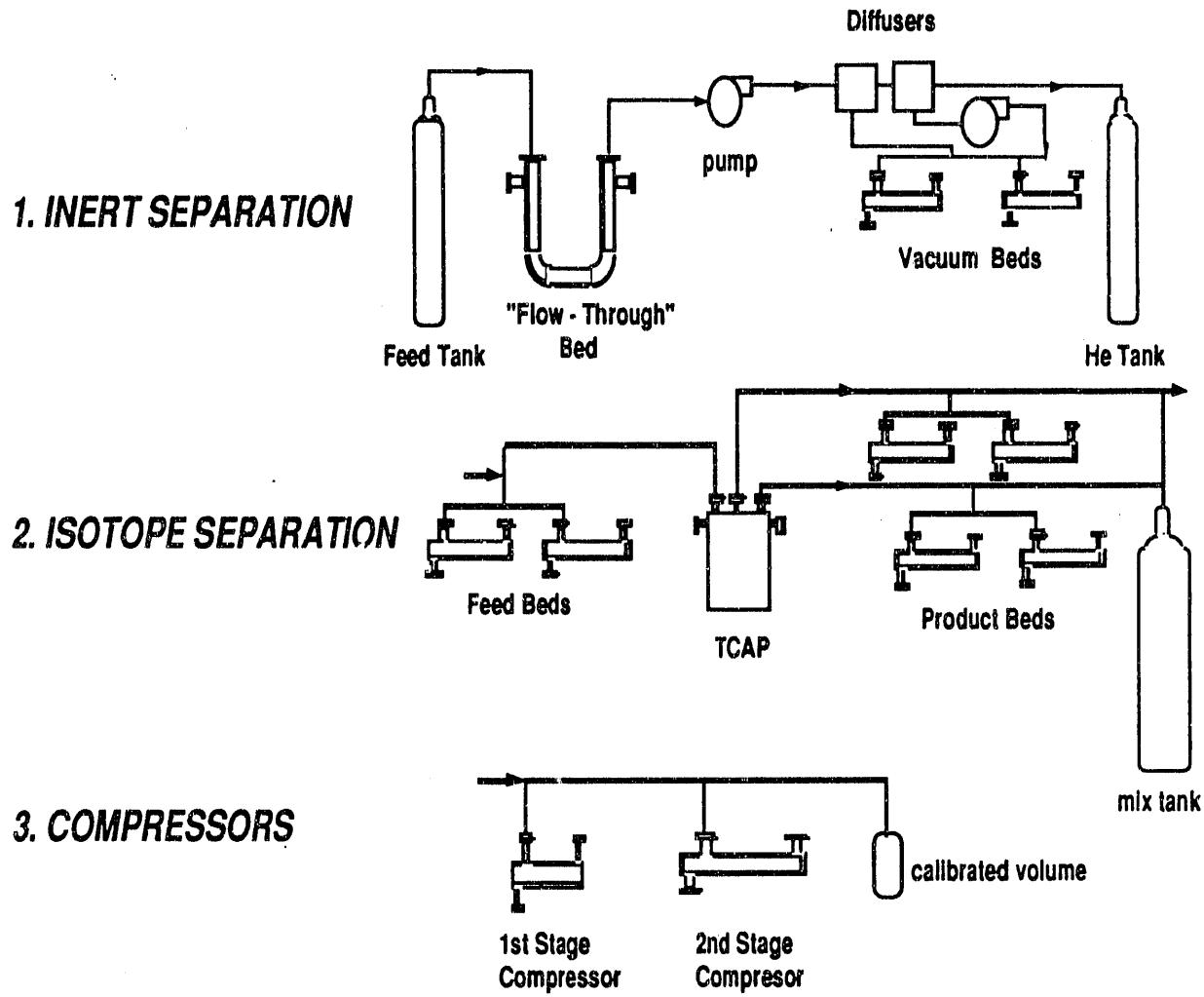


Fig. 3. - Advanced Hydride Laboratory Process Schematic

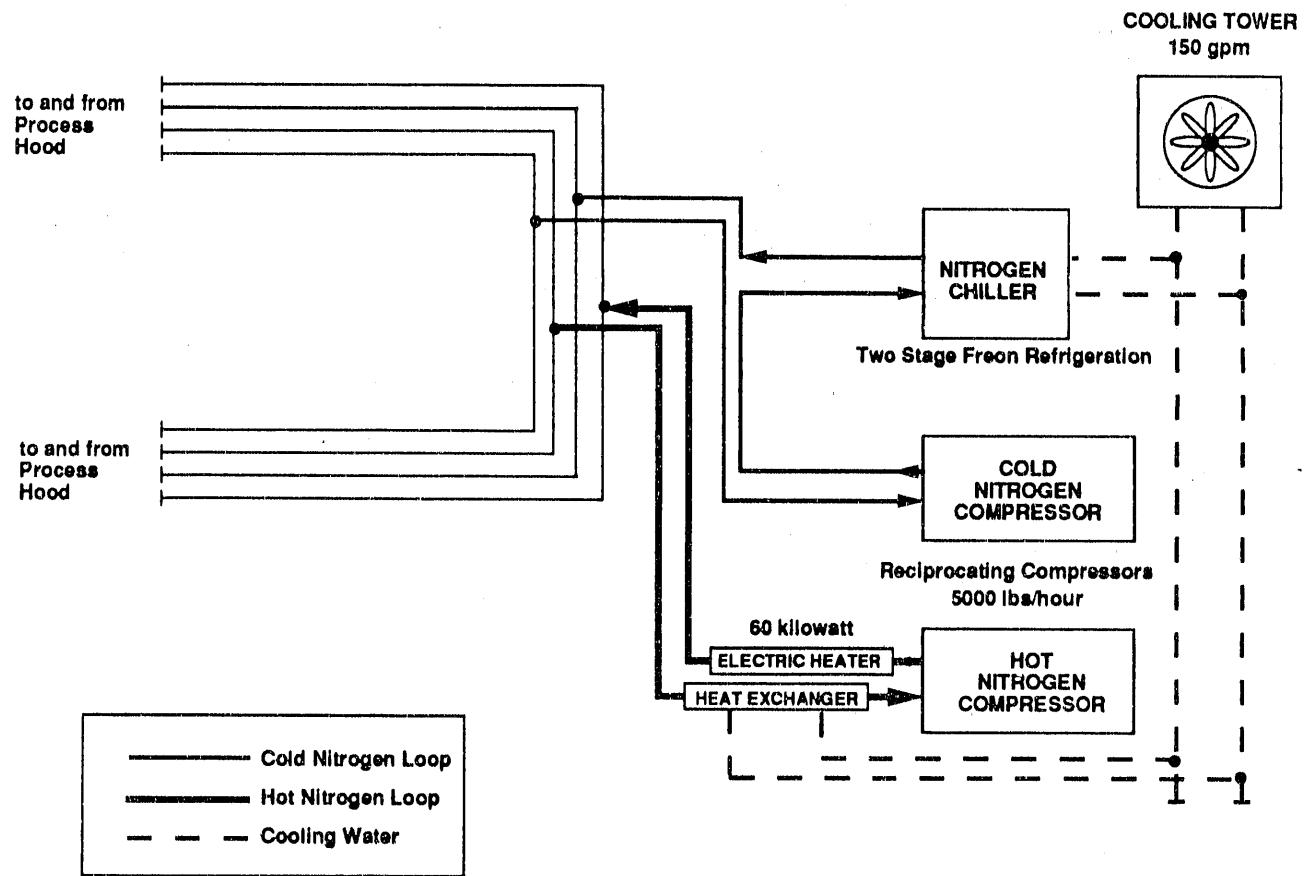


Fig. 4. - Nitrogen Circulation System

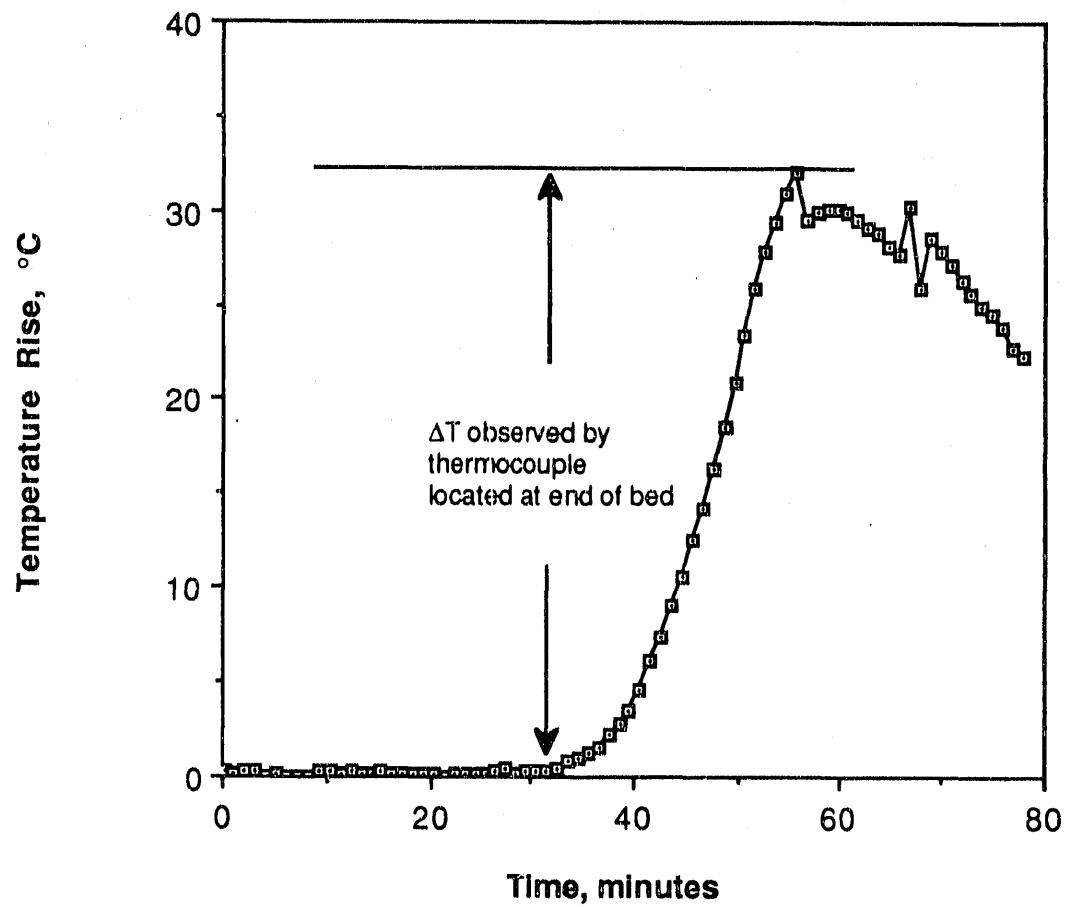


Fig. 5. - Temperature Rise Data for "Flow-Through" Bed

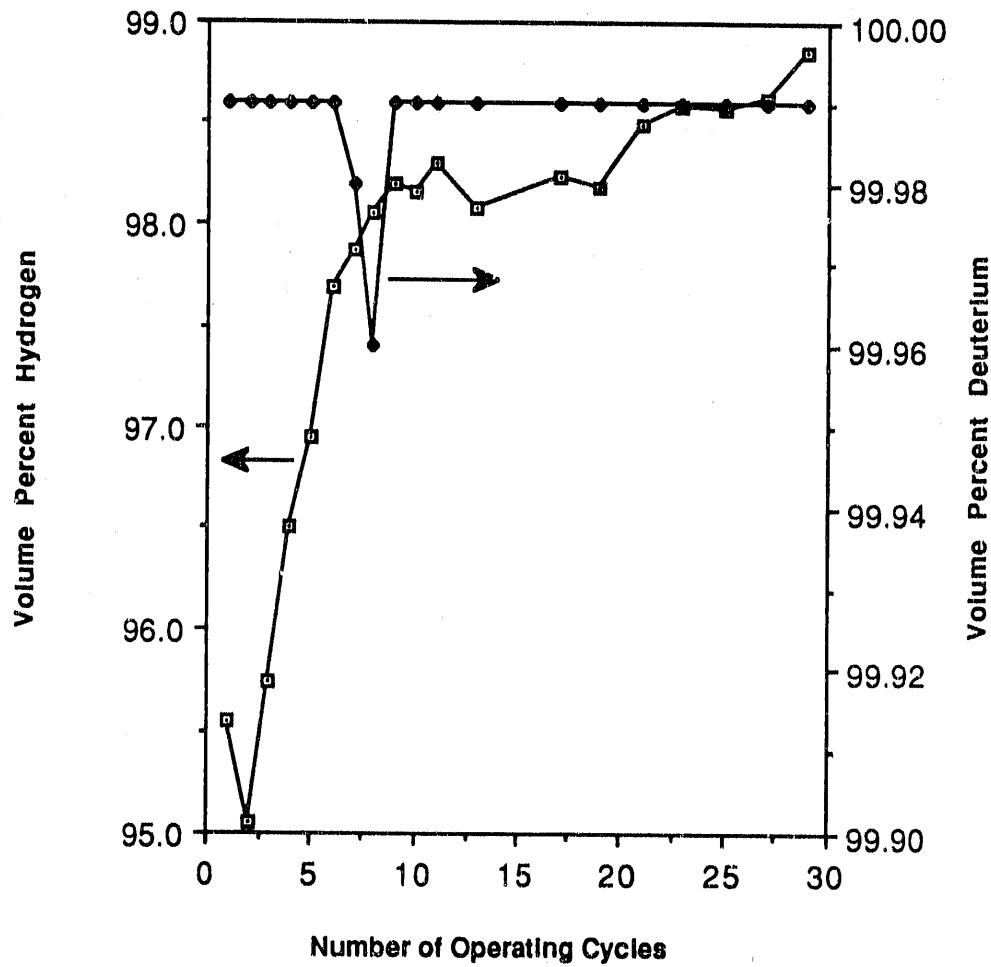


Fig. 6. - Isotope Separation Results

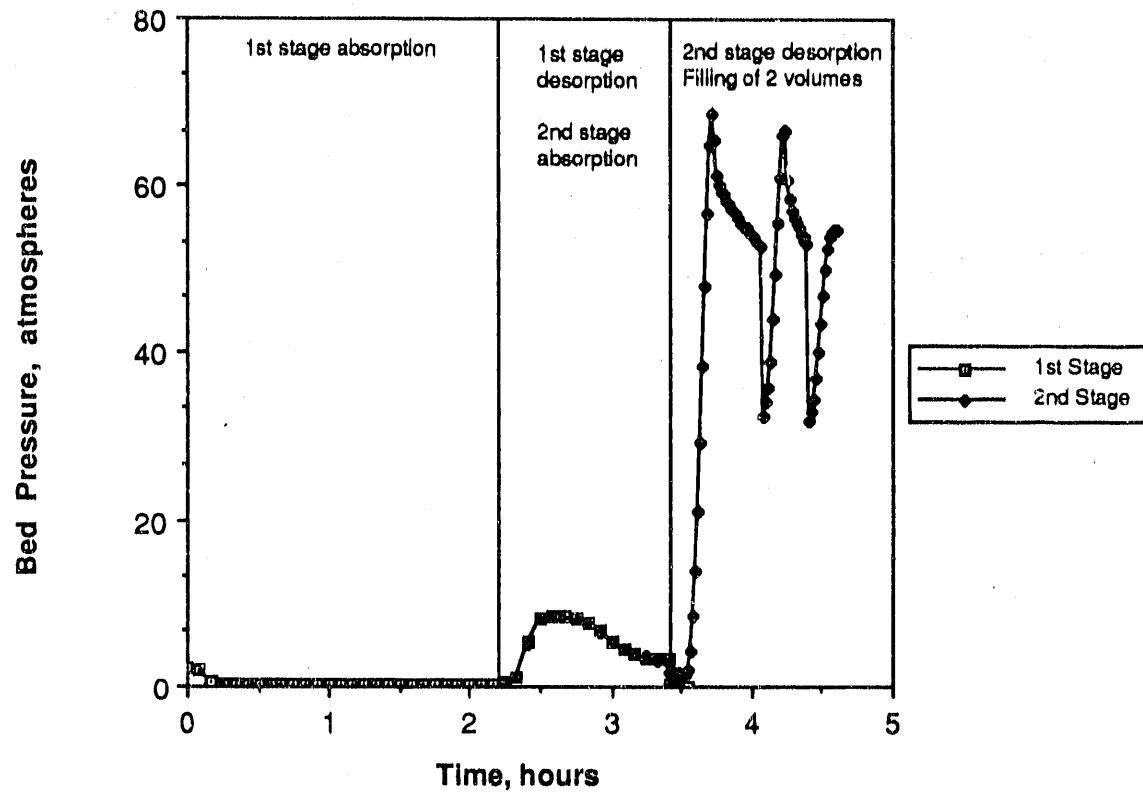


Fig. 7. - Metal Hydride Compressor Results

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