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RARE GAS RECOVERY FACILITY AT THE IDAHO CHEMICAL PROCESSING PLANT

by

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AT THE IDAHO CHEMICAL PROCESSING PLANT

ABSTRACT

Radioactive krypton and xenon from spent nuclear fuel reprocessing operations at the Idaho Chemical Processing Plant are removed from off-gases in a cryogenic recovery unit. The process equipment used and experience from several operating periods are described. A method for processing hydrogen-rich off-gas is also presented.

CONTENTS

CONTENTS (cont'd)

TABLES

FIGURES

SUMMARY

A Rare Gas Plant at the Idaho Chemical Processing Plant uses a cryogenic system for recovering and purifying several thousand curies per day of krypton and a significant volume of xenon from off-gas generated during chemical dissolution of nuclear reactor fuels. Both krypton and xenon are produced in a nuclear reactor and each has potential value for medical and industrial activities. The demonstration of radioactive-krypton recovery may be of particular importance to commercial fuel reprocessors where krypton removal during future years might be required to prevent contamination of the environment.

Presently, the Rare Gas Plant processes up to 20 scfm of hydrogen-lean off-gas evolved from acid dissolution of uranium alloyed fuels. A slight modification to Rare Gas Plant equipment would also allow processing of hydrogen-rich off-gas evolved from the acid dissolution of other types of fuels. With the existing process, the dissolver off-gas is passed first through a catalytic conversion unit which removes N_2O and H_2. Air is added to the process gas to attain the proper stream volume for the equipment and then the total stream is fed to a cryogenic system which is cooled by liquid nitrogen. Here, the krypton and xenon are separated from the gas in a distillation column. Periodically, the bottoms of the distillation column--krypton and xenon dissolved in liquid nitrogen-oxygen--are transferred to a batch still where the krypton and xenon are separated and purified in a fractionation step. Other equipment also exists in the Rare Gas Plant for: (1) transporting and storing dissolver off-gas, (2) removing water vapor and other condensibles from the dissolver off-gas, and (3) facilities for packaging the product.

The Rare Gas Plant has been operated with the present process during several different campaigns. Some problems with equipment and operational procedure were encountered during Rare Gas Plant campaigns, but plant performance improved noticeably toward the end of each campaign as the problems were identified and successfully overcome. Proposed equipment modifications and increased operating proficiency in the Rare Gas Plant will undoubtedly improve its performance during any future operation.

I. INTRODUCTION

Krypton and xenon are both produced in significant quantities during fission of uranium in a nuclear reactor. Because of their inert, gaseous nature, both are of possible value to scientific research. The various isotopes of krypton and xenon formed in reactor fuels are shown in Table I. The isotope Kr-85 has a half-life of 10.8 years, a moderate beta radiation energy, a low gamma radiation level, and is chemically inert. These properties make it useful in self-luminescent light sources, leak detection equipment, thickness gages, gas chromatography, etc. Fission product xenon--which is essentially non-radioactive by the time it is recovered--is used in scintillation devices because it has a higher proportion of heavy isotopes than does naturally occurring xenon. Xenon is also being considered for use in the manufacure of light bulbs having an extra long life.

Krypton-85 is also a potential source of environmental contamination, and recovery and retention of this noble gas may be required in the near future. The nuclear power industry is expanding at a rapid rate and the nuclear fuel reprocessing industry will expand correspondingly. A direct result of this will be an increased production rate of krypton-85 and its possible release to the environment during reprocessing of the nuclear fuels. Although krypton-85 release rates at present reprocessing plants are satisfactorily low, larger plants now being designed may have to have some control over the krypton-85 release rate. The exact recovery required will depend on the fuel processing rate, meteorology, stack height, and site limits.

Table I

THERMAL-NEUTRON FISSION YIELD FROM U-235

Fission Product	Yield %	Half Life
Kr-83	0.544	Stable
Kr-84	1.00	Stable
Kr-85	0.293	10.76 years
Kr-86	2.02	Stable
Xe-131	2.93	Stable
Xe-132	4.38	Stable
Xe-133	6.62	5.3 days
Xe-134	8.06	Stable
Xe-135	6.30	9.2 hours
Xe-136	6.46	Stable

To recover and purify these gases, a Rare Gas Plant was installed at the Idaho Chemical Processing Plant (ICPP). The plant was designed to recover several thousand curies per day of krypton and a significant volume of xenon from off-gas evolving from the acid dissolution of spent nuclear reactor fuel elements. The original process used charcoal absorbers (operating at liquid nitrogen temperatures) for removing radioactive krypton from the dissolver off-gas. When the absorbent was saturated, it was heated to drive off the rare gases which were then collected in a cold trap. However, the product from this early process was impure, and the cooling requirements of the process required more nitrogen than the existing liquid nitrogen plant could produce on a continuous basis.

After it became desirable to operate the fuel dissolvers on a continuous basis, a cryogenic distillation process was installed to reduce the liquid nitrogen requirements and to produce pure products. The cryogenic Rare-Gas-Plant process and equipment, as well as operating experience gained during the campaigns, are described in this report. Although the plant presently can process only hydrogen-lean dissolver off-gas, a modification is described in Appendix A which also would allow processing the hydrogen-rich off-gas.

II. PROCESS DESCRIPTION

The hydrogen-lean gases evolved from the dissolution of irradiated nuclear fuels in nitric acid include hydrogen, water, krypton, xenon, nitrous oxide, nitric oxide, and nitrogen dioxide. At the ICPP, no detectable radioactive iodine is present in the dissolution gases, since the time between fuel element removal from the reactor and dissolution is long enough for the iodine to essentially disappear through radioactive decay. The off-gas also contains air and oxygen--added to the dissolvers during dissolution to minimize nitric acid consumption and to reduce the hydrogen concentration in the off-gas to levels below the explosive limit. The total off-gas from the dissolvers constitutes the feed stream to the Rare Gas Plant.

A flowsheet for rare gas recovery from the hydrogen-lean off-gas generated during the dissolution is shown on Figure 1. The dissolver off-gas is pumped first into 337-ft^3, combination surge and scrubbing vessels by a caustic-sealed Nash Hytor compressor at a pressure of 20 to 40 psig. Nitrogen dioxide is removed during passage of the gas through the caustic-sealed pump; in addition, caustic solution can be sprayed into the surge tank to remove nitrogen dioxide. The collection and scrubbing of the dissolver off-gas with caustic may be conducted, optionally, as either a continuous operation with fresh gas entering while scrubbed gas is withdrawn, or as a consecutive batch-collection and scrubbing operation. Continuous operation is the current practice. The effluent gas after scrubbing is air, with about 10 to 20 percent nitrous oxide and small amounts of hydrogen, nitric oxide, nitrogen dioxide, and the rare gases. From the storage tanks, the gas stream is passed through rhodium-catalyst beds at 1000-1200°F where nitrous oxide is reduced to nitrogen, and the small amount of hydrogen in the gas reacts with oxygen to form water. The efficiency of the catalyst unit is greater than 99 percent. The effluent from the catalytic reduction unit contains nitrogen, oxygen, argon, krypton, xenon, trace amounts of the oxides of nitrogen, and some ammonia. The formation of the ammonia is induced by the rhodium catalyst and elevated temperatures. Most of the water, oxides of nitrogen, and ammonia then are removed in a water-cooled condenser. Air is then added as needed to result in a gas flow of about 20 scfm prior to compression in a second set of Nash-Hytor

compressors. From the caustic-sealed compressor, the gas passes through a demister and a dryer before entering a cryogenic system.

The cryogenic system consists primarily of two regenerators, a distillation unit, and a batch still, and is designed for a feed rate of 20-30 scfm. Feed gas flows alternatively through one of two regenerators[a] (heat exchangers) where it is cooled by the cold packing. The packing is precooled by effluent cold gas from the primary distillation column. The cooled gas leaves the regenerator through a check valve at about -260°F and enters at the tenth of twenty sieve plates in the primary distillation column. Liquid nitrogen introduced into the top of the column flows downward through the sieve plates in the column, condensing and absorbing the higher boiling components[b] in the feed gas such as oxygen, xenon, krypton, and argon. Waste gas (primarily nitrogen) is discharged at the top of the column while the absorbed gases are further concentrated by boiling and rectification in the kettle and lower portion of the column.

The waste gas, after leaving the top of the primary column, cools a batch-still condenser at about 25 psig and -300°F and is then introduced to the second regenerator primarily to cool and to purge the regenerator of any contaminants it collected when the feed gas was flowing through it. Frequent alternating use of the two regenerators to keep their midpoint temperatures near -150°F is an operating requirement of the system. To prevent loss of any residual feed gas contained in the regenerator at the moment of reversal, the initial surge of gas leaving a regenerator is recycled back to the inlet of the compressor. Except for the initial surge, the waste gas leaves the regenerator through a steam jet exhausting to the atmosphere.

Bottoms from the primary column (mostly nitrogen and oxygen), containing the relatively concentrated rare gases, are transferred several

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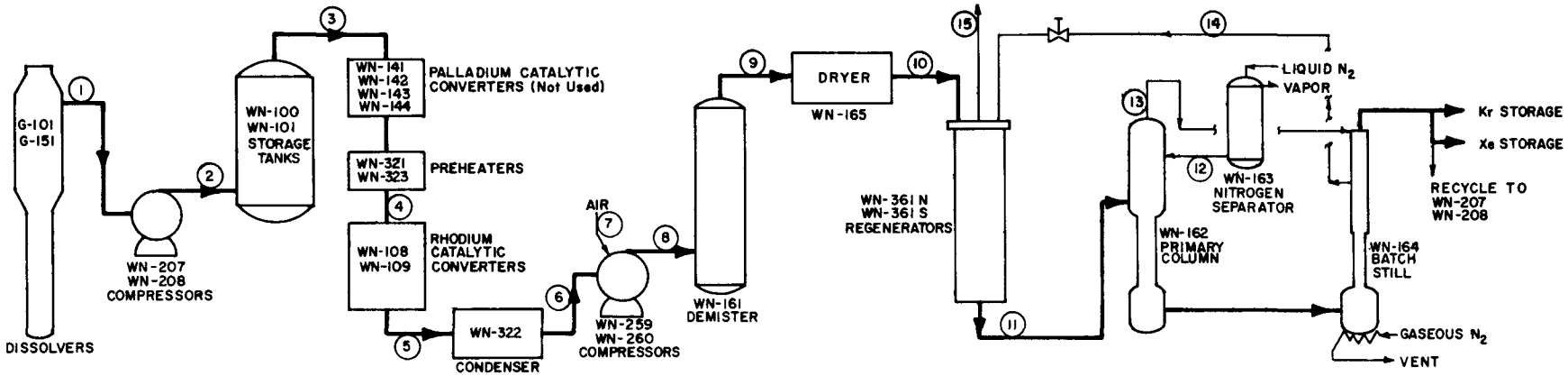
[a] Two regenerators are used in alternate operation in the Rare Gas Plant to cool the gas stream fed to the primary distillation column. The feed stream flows first through one and then the other regenerator--which are essentially packed columns. While the feed stream flows through one, cold effluent gas from the distillation column is flowing through the other regenerator.

[b] See Figure 2 and Table II for boiling and freezing temperatures. Vapor pressure data and freezing temperature for various gas mixtures are given in Appendix B.



STREAM	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
DESCRIPTION	DISSOLVER OFF-GAS	COMPRESSED GAS TO STORAGE	FEED GAS	HEATED FEED GAS	N_2O LEAN FEED GAS	COOLED FEED GAS	MAKEUP AIR	FEED TO PROCESS	FEED TO PROCESS	DRIED FEED TO CRYOGENIC SYSTEM	COOLED FEED GAS TO PRIMARY COLUMN	LIQUID NITROGEN TO PRIMARY COLUMN	PRIMARY COLUMN WASTE GAS	COOLANT GAS FROM BATCH STILL CONDENSER	WASTE GAS TO STACK
Gas Flow, scfm	9	9	9	9	9	9	11	20	20	20	20		30	30	30
Liquid Flow, lb/hr												40			
Pressure, psig	-0.4	20-40	6-8	6-8	6-8	6-8	0	30	29	28	27	30	25	3-5	-1.3
Temperature, °F	90	90	80	330	1000 to 1200	90	70	80	80	80	-260	-310	-300	-300	75
N_2, Vol. %(a)	74.8	74.8	74.9	74.9	89.0	89.0	78.1	83.0	83.0	83.0	83.0	100	100	100	100
O_2, Vol. %(a)	4.6	4.6	4.6	4.6	10.8	10.8	20.9	16.4	16.4	16.4	16.4	0	0	0	0
H_2, Vol. %(a)	2.1	2.1	2.1	2.1	0	0	0	0	0	0	0	0	0	0	0
N_2O, Vol. %(a)	17.5	17.5	17.5	17.5	0.1	0.1	0.1	0.1	0.1	0.1	0	0	0	0	0
Dew pt, °F	90	90	80	80	94	90	58	80	80	-40	-260	-310	-310	-310	-40
NO_2, Vol. %(a)		neg(b)	neg	neg	neg	neg	neg	neg	neg	neg	0	0	0	0	0
CO_2, Vol. %(a)		neg	neg	neg	neg	neg	0.03	neg	neg	neg	0	0	0	0	0
NO, Vol. %(a)	1.0	neg	neg	neg	neg	neg	neg	neg	neg	neg	0	0	0	0	0
N_2O_5, Vol. %(a)		neg	neg	neg	neg	neg	neg	neg	neg	neg	0	0	0	0	0
A, Vol. %(a)		0.9	0.9	0.9	0.9	0.9	0.9	0.9	0.9	0.9	0.9	0	0	0	0
Kr, scfm	5x10-4	5x10-4	5x10-4	5x10-4	5x10-4	5x10-4	5x10-4	5x10-4	5x10-4	5x10-4	5x10-4				
Xe, scfm	3x10-3	3x10-3	3x10-3	3x10-3	3x10-3	3x10-3	3x10-3	3x10-3	3x10-3	3x10-3	3x10-3				

(a) Neglecting water vapor.
(b) Negligible

Figure 1

FLOWSHEET FOR RECOVERY OF KRYPTON AND XENON
FROM HYDROGEN-LEAN DISSOLVER OFF-GAS

Table II

FREEZING POINTS OF COMPONENTS IN
HYDROGEN-LEAN DISSOLVER OFF-GAS STREAM
AT ATMOSPHERIC PRESSURE

Components	Freezing Point, °F
NO_2	11.8
CO_2	-108.8
N_2O	-131.4
Xe	-220.0
NO	-262.5
Kr	-272.2
Ar	-308.6
N_2	-345.6
O_2	-361.1
H_2	-434.4

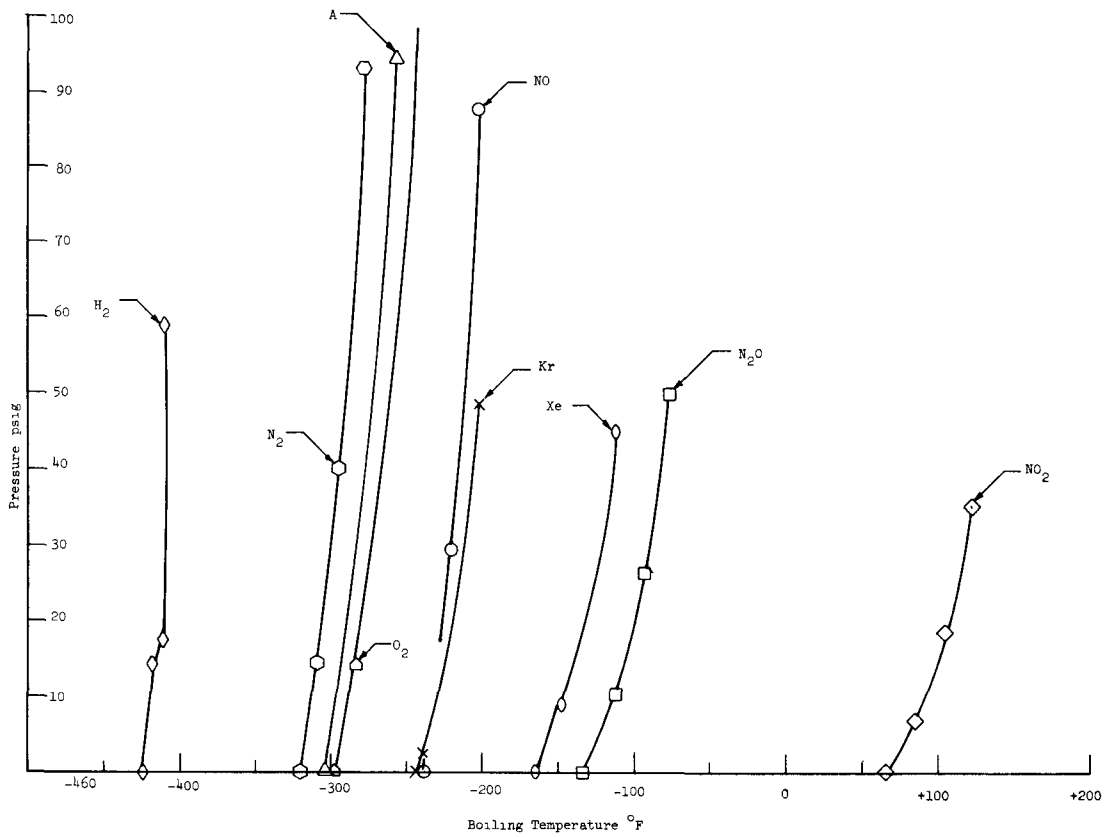


Figure 2

BOILING POINTS OF COMPONENTS
IN THE FEED STOCK TO THE RARE GAS PLANT

times a day to the batch still. The nitrogen and some of the oxygen are distilled off after each transfer, but the rare gas components accumulate until there is sufficient volume to make a run in the still. To distill off the nitrogen and oxygen, heat is supplied by introducing gaseous nitrogen to the heating coil of the batch-still kettle. The batch-still condenser is cooled by the waste gas from the primary column. Nitrogen and oxygen are boiled off at a rate of about 2 cfm at 40 psig column pressure until the temperature indicates that all of the nitrogen and most of the oxygen have been distilled off. (At times other than during the distillation operation, the column is kept cold by passing liquid nitrogen through the coil around the kettle, and kept under pressure to prevent losses of krypton and xenon.) After a sufficient quantity of product is accumulated in the batch still (equivalent to that amount from six or seven liquid transfers), a complete fractionation is performed; heat is supplied by passing nitrogen gas through the heating coil at a rate sufficient to maintain a column pressure of 40 psig and a product take-off rate of about 0.4 to 0.5 cfm. The initial gas fraction, which contains mostly oxygen, is recycled back to the feed-gas compressor to prevent the loss of the small amount of rare gas in the fraction. Krypton is the first fraction taken off, then xenon. The krypton and xenon product fractions are diverted during removal into intermediate storage vessels for later bottling.

III. EQUIPMENT DESCRIPTION

A description of each major piece of equipment and the instrumentation for the Rare Gas Plant is presented in this section. A schematic process flowsheet is shown on Figure 3 and the materials of construction for the equipment are summarized in Table III.

1. COMPRESSORS AND PUMPS

There are five compressors associated with the Rare Gas Plant. One set of two compressors is used to compress off-gas into the storage tanks, another set of two compressors is used to feed the cryogenic system, and a single compressor is used to recycle nitrogen.

1.1 Dissolver Off-Gas Compressors

Two Nash Hytor compressors (WN-207 and WN-208), manufactured by Nash Engineering Company and located in the access corridor of building CPP-604, are available for compressing the dissolver off-gas into the gas storage tanks. One is used while the other is in standby.

1.2 Compressors on the Cryogenic System

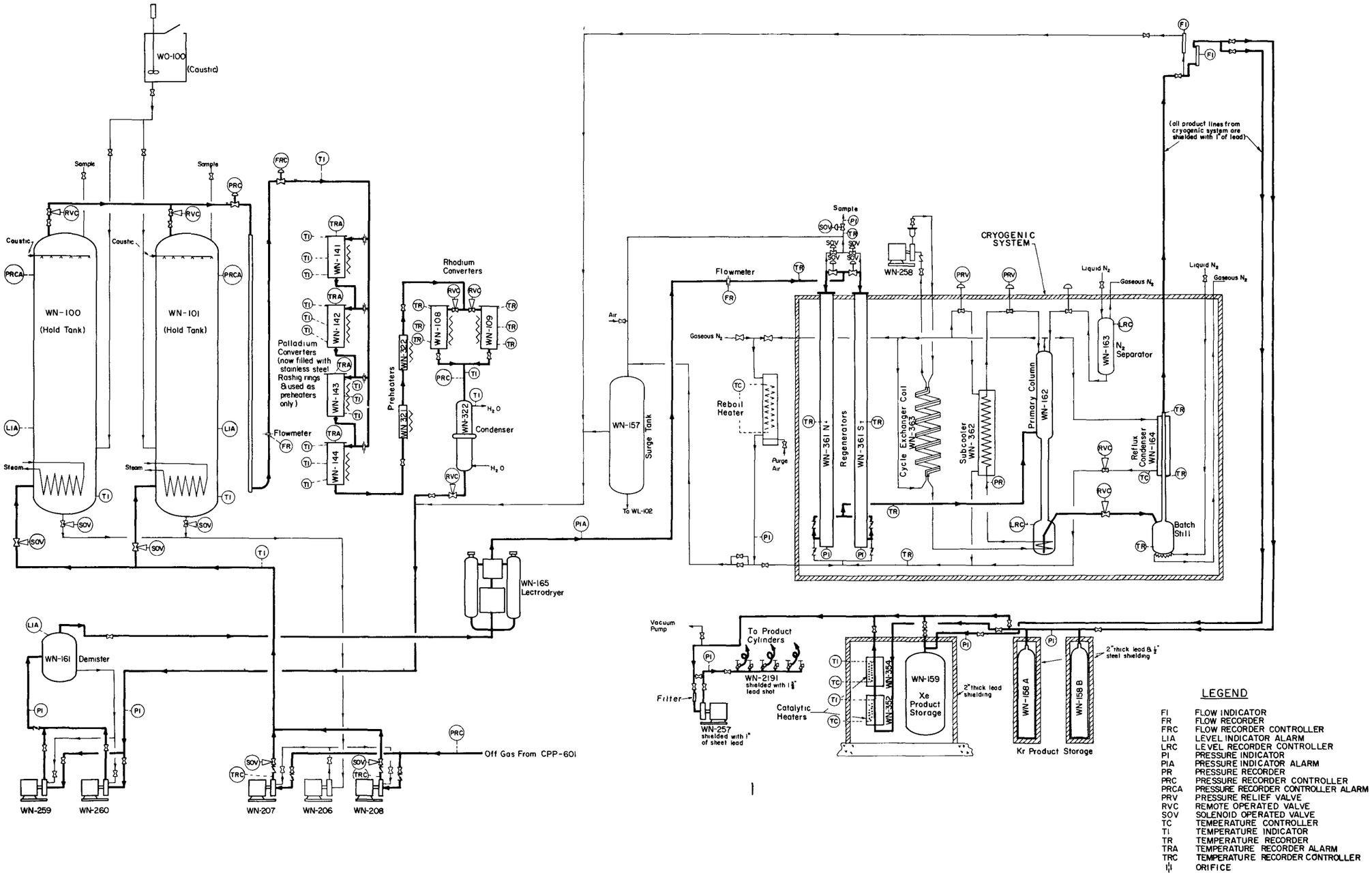
Three compressors are directly associated with the cryogenic system. These include two Nash Hytors (WN-259 and WN-260) manufactured by Nash Engineering Company and one by Pressure Products Industries, Inc. The two Nash compressors, which use a caustic solution as a sealant, are located in the access corridor of building CPP-604, and are used to compress the feed gas to the cryogenic system. One compressor is used while the other one is in standby. The Pressure Products compressor is located in the south gas cell of building CPP-604 and is used to recycle and conserve waste nitrogen. Use of this compressor during normal operation is optional.

1.3 Caustic Pumps for Compressors and Storage Tanks

Three pumps (WN-204, 205, and 206) are available for circulating caustic solution to the spray headers in the dissolver off-gas tanks and to supply lubricant and sealant to the dissolver off-gas compressors. These pumps are single-stage centrifugal types manufactured by Worthington Pump and Machinery Corporation.

1.4 Vacuum Pump

Before conducting a fractionation of product gases contained in the batch still, the product hold tanks are evacuated by a vacuum pump (WN-212) which has two stages and a V-belt drive.



LEGEND

FI	FLOW INDICATOR
FR	FLOW RECORDER
FRC	FLOW RECORDER CONTROLLER
LIA	LEVEL INDICATOR ALARM
LRC	LEVEL RECORDER CONTROLLER
PI	PRESSURE INDICATOR
PIA	PRESSURE INDICATOR ALARM
PR	PRESSURE RECORDER
PRC	PRESSURE RECORDER CONTROLLER
PRCA	PRESSURE RECORDER CONTROLLER ALARM
PRV	PRESSURE RELIEF VALVE
RVC	REMOTE OPERATED VALVE
SOV	SOLENOID OPERATED VALVE
TC	TEMPERATURE CONTROLLER
TI	TEMPERATURE INDICATOR
TR	TEMPERATURE RECORDER
TRA	TEMPERATURE RECORDER ALARM
TRC	TEMPERATURE RECORDER CONTROLLER
⫛	ORIFICE

FIGURE 3

SCHEMATIC DIAGRAM OF RARE GAS PLANT PROCESS

Table III

DESCRIPTION OF MAJOR EQUIPMENT IN THE ICPP RARE GAS PLANT

Name	Description	Material of Construction	Design Pressure	Design Temperature	Capacity
Dissolver Off-Gas Compressors (WN-207*or WN-208)	Nash Hytor compressor	347 SS	Suction: 20"H_2O Discharge: 60 psig	Ambient	40 scfm each
Cryogenic System Compressors (WN-259 or WN-260)	Nash Hytor compressor	347 SS	Suction: 0 psig Discharge: 30 psig	Ambient	30 scfm each
Nitrogen Recycle Compressor (WN-258)	Diaphragm compressor with V-belt drive	--	Suction: 15 psig Discharge: 140 psig	Ambient	5 cfm
Caustic Pump (WN-204, 205 and 206)	Single stage, centrifugal	Carbon steel	Suction: 72 psig Discharge: 101 psig	Ambient	12 gpm of 20% NaOH
Vacuum Pump (WN-212)	Two stage vacuum pump, V-belt drive	--	Suction: 2×10^{-4} mm Hg	Ambient	15.2 cfm at suction pressure
Product Compressor (WN-257)	Diaphragm compressor V-belt drive	--	Suction: 0 psig Discharge: 1000 psig	Ambient	0.35 cfm
Dissolver Off-Gas Storage Tanks (WN-100 or WN-101)	4' OD x 26' vertical, cylindrical with flanged and dished heads	347 SS	100 psig	212°F	337 ft^3
Palladium Catalytic Converters	Vertical, cylindrical with dished heads				
WN-141	3½" OD x 29"	347 SS	100 psig	700°F	150 cfh
WN-142	3" OD x 32"	347 SS	100 psig	700°F	300 cfh
WN-143	3" OD x 32"	347 SS	100 psig	700°F	300 cfh
WN-144	5" OD x 30" Each unit contains two 575 to 667 watt Chromalox electrical heaters	347 SS	100 psig	700°F	500 cfh

*Refers to plant numbering system.

Table III (cont'd)

Name	Description	Material of Construction	Design Pressure	Design Temperature	Capacity
Preheaters (WN-321 & WN-323)	Chromalox steam preheater (1500 watts) 4-5/8" OD x 33-1/4"	347 SS	25 psig	300-350°F	4,100 Btu/hr
Rhodium Catalytic Unit (WN-108 & WN-109)	5" OD x 30" L vertical, cylindrical with dished heads and two Chromalox electrical heaters (662 watts each)	347 SS	100 psig	1200°F	500 cfh
Cooler (WN-322)	6" OD x 9'3/8" vertical, cylindrical with flanged and dished heads 88 Tubes: 3/8" OD x 7'	347 SS	100 psig	shell: 100°F tube: 400°F	25,000 Btu/hr
Demister (WN-161)	24" OD x 48" cylindrical, vertical with dished heads	347 SS	--	--	12½ ft^3
Dryer (WN-165)	BAC-180-SP Lectrodryer molecular sieve desiccant, imbedded electrical heaters	Carbon steel	--	400°C during regeneration	One scfm of water vapor from 30-scfm stream at a temperature of 27°C
Regenerators (WN-361N & 361S)	4" OD x 11'5" with upper and lower grids to support 1/4" ceramic Berl saddles	304 SS	75 psig	-300°F	--
Primary Column (WN-162)	Overall height 86¼" Upper 10 trays Section: 4-1/8" OD x 39-5/8" Lower 10 trays Section: 2-5/8" OD x 30-5/8" Kettle: 6-3/8" OD x 14½" 5 liter capacity with internal heating coil	all copper, silver soldered	75 psig	-300°F	several thousand curies Kr-85 per day

Table III (cont'd)

Name	Description	Material of Construction	Design Pressure	Design Temperature	Capacity
Batch Still (WN-164)	Overall height:59" Kettle: 5-liter capacity, 6-3/8" OD x 14-1/2" Column: 7/8" OD x 36" with 26 staggered baffles Reflux Condenser: 2-5/8" OD x 17" with 39 staggered baffles. Kettle has external heating coil	All copper, silver soldered	75 psig	-300°F	4,000 curies Kr-85 per day
Nitrogen Separator (WN-163)	4-1/8" OD x 26" vertical, cylindrical with dished heads	All copper, silver soldered	75 psig	-300°F	--
Nitrogen Recycle Heat Exchanger (WN-363)	Tube to tube and spiral wound. 13" OD x 36" (one, 1/4" OD x 50' tube between two, 1/4" OD x 50' tubes)	All copper, silver soldered	75 psig	-300°F	--
Sub Cooler (WN-362)	Tube (one 1/4" OD spiral wound) and shell heat exchanger 2-1/8" OD x 44"	All copper, silver soldered	75 psig	-300°F	--
Krypton Product Hold Tanks (WN-158A and B)	9-1/8" OD x 51" cylinder in a 14½" OD x 67½" cask with a removable plug	Carbon steel	2000 psi	70°F	191 cubic feet
Xenon Product Hold Tank (WN-159)	20" OD x 48" right cylindrical vessel dished heads	Carbon steel	--	--	--
Product Shipping Cylinders	Identical to krypton product hold tanks	Carbon steel	2000 psi	70°F	191 cubic feet

1.5 Product Gas Compressor

The product gases are transferred from their respective hold tanks by a diaphragm, V-belt driven compressor (WN-257).

2. STORAGE TANKS

Two tanks (WN-100 and WN-101) with a capacity of 337 cubic feet each are located in the north gas cell of building CPP-604 and are used for storing compressed dissolver off-gas. These tanks also hold caustic solution which is recirculated to spray headers for scrubbing of gases and to the Nash Hytor compressors where it is used as the sealant and lubricant.

3. PALLADIUM CATALYTIC CONVERTERS

Four palladium catalytic converters (WN-141, 142, 143, and 144) are located in the north gas cell of building CPP-604 and are operated in parallel. Originally, they were used for the reduction of oxygen and oxides of nitrogen with hydrogen to form nitrogen and water. Because of their costly and hazardous hydrogen consumption requirements and low capacity they were replaced with a rhodium catalytic converter (see Section 5). These units are still used as preheaters; recently, the palladium catalyst was replaced with stainless steel Raschig rings and the original vessels now are equipped with external rheostat-controlled electrical heaters.

4. PREHEATERS

Two additional preheaters (WN-321 and WN-323) are located between the palladium catalytic converters (now used as preheaters) and the rhodium catalytic converter. They are in the north gas cell of building CPP-604 and are operated in series.

5. RHODIUM CATALYTIC UNIT

The Rare Gas Plant contains a catalytic unit (manufactured by Pure Gas Equipment Company) for converting nitrogen oxides and oxygen in the dissolver off-gas to water and nitrogen. It is located in the north gas cell of building CPP-604 and consists of two catalyst beds (WN-108 and WN-109) operated in parallel. Rhodium on alumina is used as a catalyst. The capacity of the unit is 500 cubic feet of gas per hour, and the rated space velocity is 1800 cubic feet of gas per cubic foot of catalyst per hour. The unit is electrically heated and operating temperatures range

from 1000°F to 1200°F. The vessel is insulated and has external rheostat-controlled heaters.

6. COOLER

A tube-and-shell type cooler (WN-322) is located downstream of the rhodium converters in the north gas cell of building CPP-604 to cool the effluent gas and condense water vapor produced in the catalytic unit.

7. DEMISTER

The demister (WN-161), located in the access corridor of CPP-604, serves as a knockout drum to remove seal solution entrained in the effluent from the feed compressor, and also functions as a reservoir for the seal solution.

8. DRYER

A dryer (WN-165) manufactured by McGraw-Edison Company was installed in the middle gas cell of building CPP-604 during 1968 to remove moisture from the gas feed to the cryogenic system. It is a model BAC-180-SP "LECTRODRYER" with molecular sieve (alumino-silicate) desiccant. The dryer is equipped with embedded electric reactivation heaters to dry the desiccant. The "LECTRODRYER" consists of four beds of desiccant; two beds are on-stream while two beds are being regenerated on an eight hour cycle. Regeneration is accomplished by heating the desiccant to a temperature of about 370°C while purging with air. The desiccant is capable of drying 30 scfm of gas saturated at 27°C down to a dew point of -40°C.

9. THE CRYOGENIC SYSTEM

The cryogenic system equipment[(a)] including regenerators, a primary column, a batch still, a nitrogen separator, nitrogen recycle equipment, a subcooler, and check valves, is enclosed in a cold box approximately 3 feet square by 12 feet high as shown in Figures 4 and 5[(1)]. The box is located in the south gas cell of building CPP-604. The box equipment, excluding the stainless steel regenerators, is fabricated of copper joined with silver solder. The free space in the cold box is filled with an expanded silica insulating material, manufactured by Silbrico Corporation.

(a) Manufactured by the Air Reduction Company.

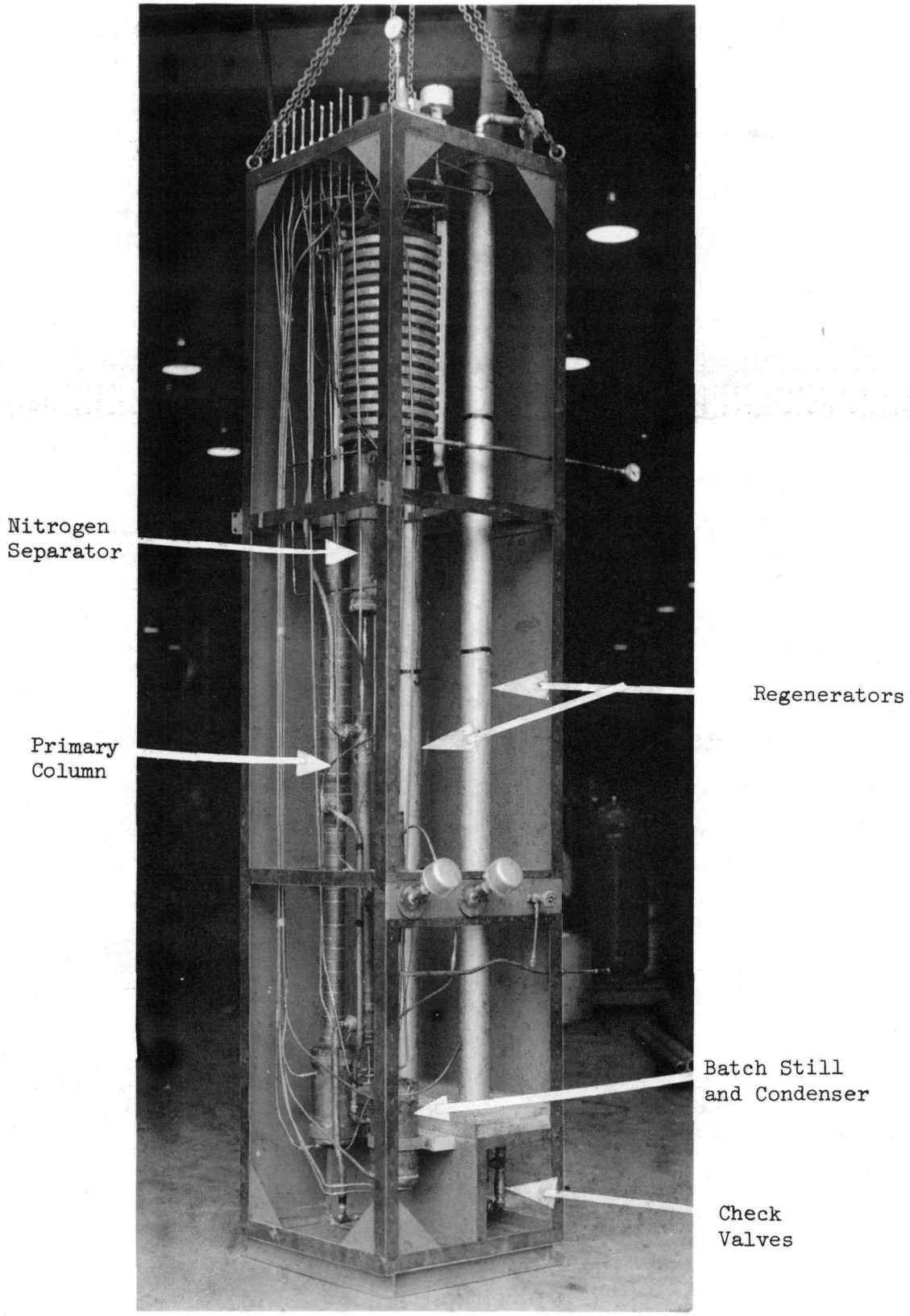


Figure 4

FRONT VIEW PHOTOGRAPH OF CRYOGENIC SYSTEM
USED IN THE RARE GAS PLANT

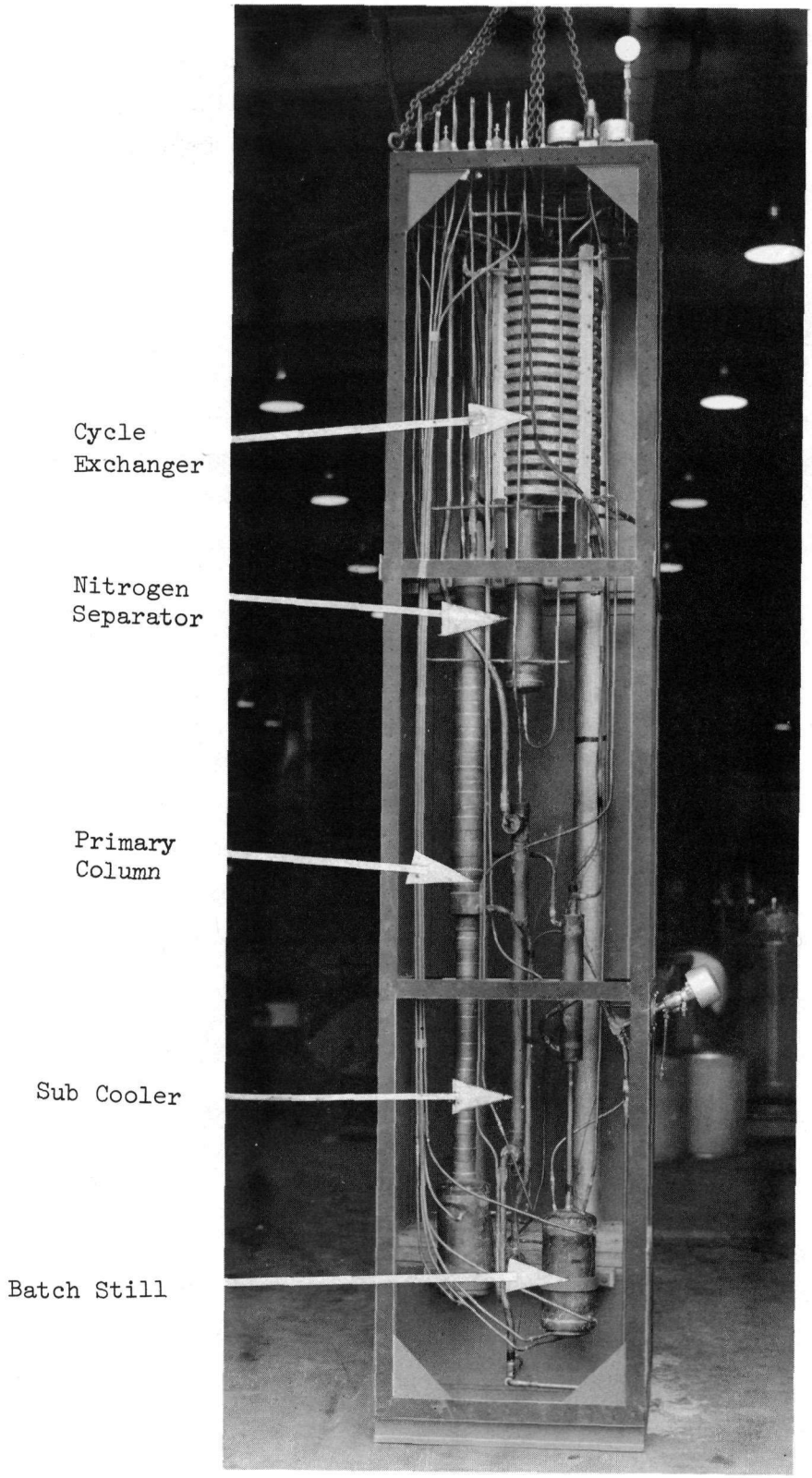


Figure 5

SIDE VIEW PHOTOGRAPH OF CRYOGENIC SYSTEM
USED IN THE RARE GAS PLANT

9.1 Regenerators

The regenerators (WN-361Nand WN-361S) are constructed of stainless steel tubing packed with 1/4-inch ceramic Berl saddles. A check valve assembly connects directly to the bottom of these regenerators and is enclosed in a separate compartment. This permits the use of separate insulation for the check valve assembly so that it is unnecessary to empty the cold box when the valves require attention.

9.2 Primary Column

The primary column (WN-162) shown in Figure 6 is a sieve plate column having ten plates in the upper scrubbing section and ten plates in the lower section; all plates have 3-1/16-inch spacing. Each of the upper plates is perforated with 59 one-eighth-inch diameter holes. The kettle at the bottom of the column has a volume of approximately 5 liters. It is heated by warm gas flowing through two copper coils mounted inside the kettle.

9.3 Batch Still

The batch still (WN-164) shown in Figure 7 has a kettle with a capacity of approximately 5 liters. It is heated by warm gas flowing through a copper coil soldered to the outer surface of the kettle bottom. A 36-inch long copper tube connects to the top of the batch still and forms the inner surface of the condenser. A copper thermowell with semicircular segments is suspended inside the center tube of the condenser and extends below the bottom of the condenser. The baffles on this well cause the gas from the kettle to pursue a tortuous path as it flows through the condenser, thus improving liquid-gas contact and also bringing the gas into contact with the cold inner wall of the condenser. The condenser has an estimated 3 to 5 theoretical stages. Cold waste gases from the top of the primary column pass through a baffled annular space around the condenser tube to provide refrigeration.

9.4 Nitrogen Separator

Liquid nitrogen is supplied at 30 to 60 psig to the cryogenic system through insulated piping from bulk storage. In spite of the insulation, some heat leaks into the transfer pipe causing the nitrogen to evaporate; therefore, the nitrogen is brought into a disengaging vessel (WN-163) before being introduced onto the top plate of the primary column.

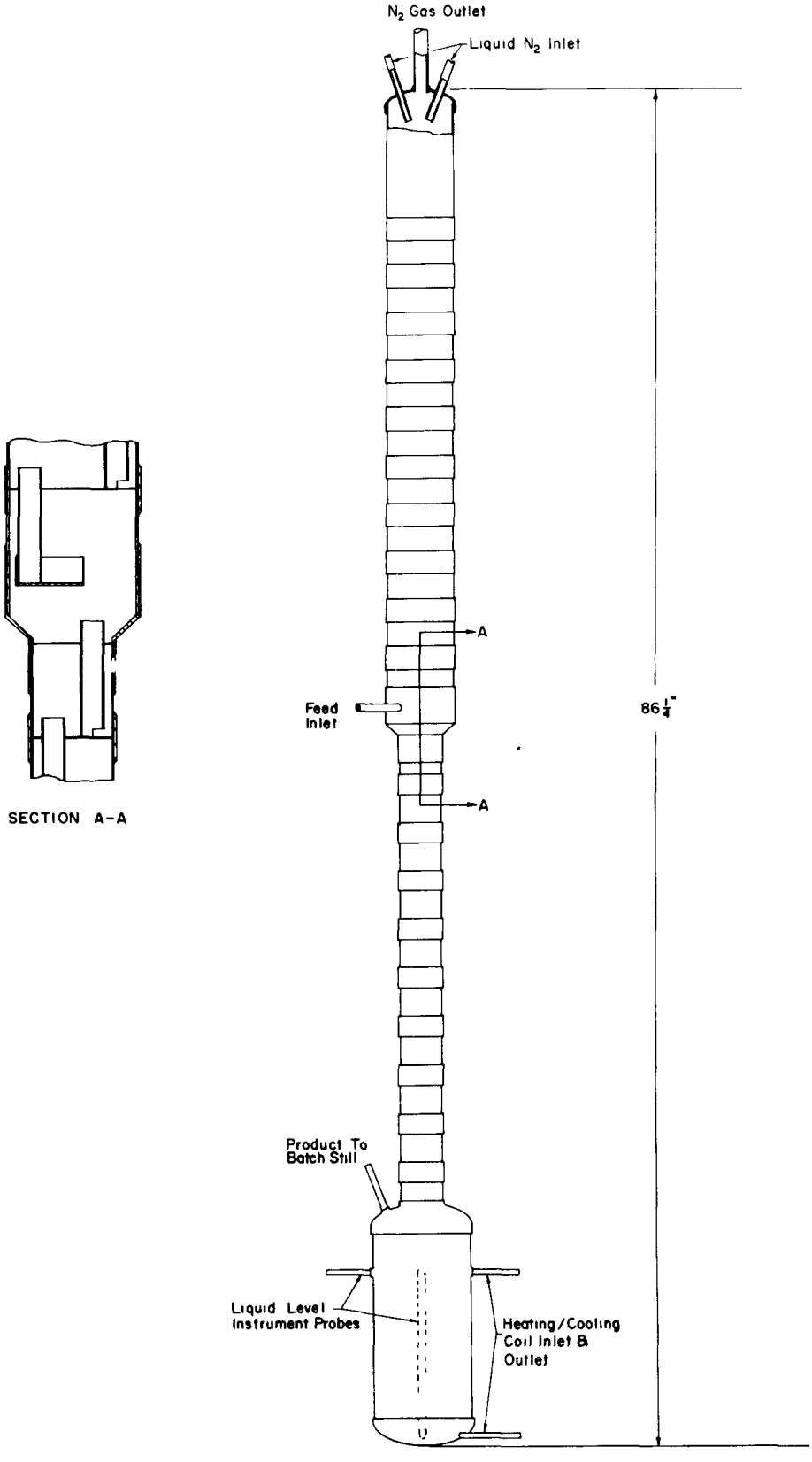


Figure 6

PLAN VIEW OF PRIMARY COLUMN

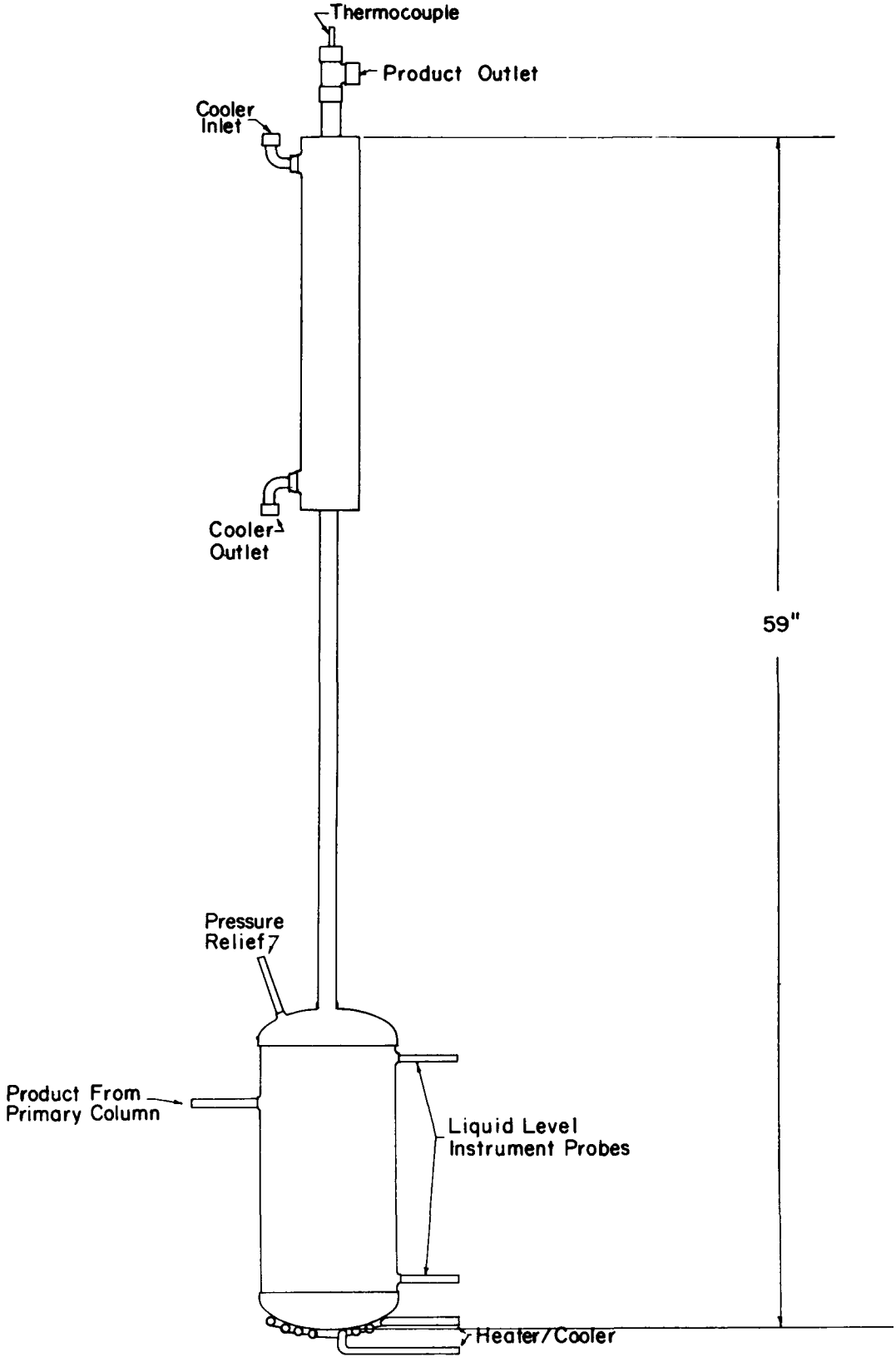


Figure 7

PLAN VIEW OF BATCH STILL

9.5 Nitrogen Recycle System

The nitrogen recycle system is a refrigeration unit designed to conserve liquid nitrogen. Normally, this system is not required, but when in use, part of the waste gas from the top of the primary column is compressed by a diaphragm compressor to approximately 9 atmospheres pressure. This compressed gas gives up its heat of vaporization in the primary column heating coil, and then returns as liquid to the top of the primary column through an expansion valve.

Two heat exchangers are provided for use when the nitrogen recycle system is used. One, the cycle exchanger coil (WN-363), mounted near the top of the unit, is used to provide an exchange of heat between the cold, low-pressure waste gas passing through the two outer coils to the compressor and the hot compressed gas returning from the compressor to the primary column reboil coil.

9.6 Subcooler

The subcooler (WN-362) is a heat exchanger designed to further cool the condensed gas from the primary column heating coil prior to introduction as liquid on the top plate of the primary column. This system also is not required normally. When it is operating, the temperature of the liquid is approximately that of the liquid in the primary column kettle. This liquid passes through the subcooler counter-current to the waste gas going from the top of the primary column to the regenerator and is cooled to near the temperature at the top plate of the primary column.

10. INSTRUMENTATION

A description of the most important instrumentation used in the Rare Gas Plant follows:

10.1 Temperature Measurements

Thermocouples are installed in several locations for measuring temperatures of the following: (1) the feed gas to the cryogenic system, (2) waste gas from the cryogenic system, (3) the mid point of each of the two regenerators, (4) the inlet gas to the primary column, (5) gas from batch still condenser to the regenerators, (6) the top and bottom of the batch still condenser, and (7) the batch still kettle.

10.2 Level Measurements

The liquid levels in the nitrogen separator and the primary column kettle are controlled automatically by recorder-controllers; whereas, the

level in the batch still is only recorded. The level indicators for each of these three vessels consist of differential pressure transmitters connected to two probes, one in the gas space and the other in the liquid.

The level of the liquid in the compressor demister is monitored by pressure probes and indicated by a gage on the panel board. A panel-mounted hand valve is used to add water to the demister as necessary.

10.3 Pressure Measurements

The pressures at the top plate of the primary column, in the batch still, and in the reboil coil of the primary column are recorded by a pressure recorder. These pressures are used for control and in trouble-shooting the operation of the cryogenic system.

Pressure indicators are located on product hold tanks, product gas lines to catalytic units, outlet lines from rhodium converters, discharge side of second stage product gas compressors, outlet of gas dryer, outlet of both regenerators, all product hold tanks, and inlet to product cylinders.

10.4 Timers

Successful operation of the cryogenic system requires uniformity and reproducibility of the regenerator half-cycles. The cycle timer has separately adjustable time switches for each of the half-cycles, permitting individual adjustment of the time to compensate for physical difference between the two regenerators. A third adjustable time switch controls the recycle valves which divert the initial surge of gas from the waste regenerator back to the feed gas compressor.

10.5 Reversing and Recycle Valves

The four regenerator reversing valves and the two recycle valves are solenoid-pilot air-operated valves, constructed so that they open when the air supply fails. The recycle valve in the line to the stack closes when electrically energized; the other five open when electrically energized. Thus, if the timer were turned off, these valves would be closed except for the line to the stack. If the air supply fails, all valves will open regardless of the electrical signal applied. Normal valve operation is to alternately open the pairs of reversing valves each 100 to 150 seconds and to open the valves to recycle the initial surge from each regenerator for 5 to 8 seconds.

10.6 Radiation Monitor

A Jordan remote-radiation monitoring system permits observation and control of the flow of radioactive gases to and from the cryogenic system. Radiation detector heads are installed on the feed line to the system, on the waste gas line from the system, and on the product lines from the batch still. A radiation level indicator is mounted on the panel board. The radiation level variations in the batch-still product stream are detectable and are useful in controlling the separation of the radioactive krypton during fractionation of the oxygen-krypton-xenon mixture in the batch still kettle.

10.7 Unsafe-Practice Alarms

Certain unsafe operating conditions actuate alarms and signal lights which are mounted on the panel board. These include:

(1) High and low demister liquid levels.

(2) High radiation level in either feed or waste gases.

(3) Low pressure in the nitrogen separator.

10.8 Samplers

There are two sample points in the Rare Gas Plant. One is located downstream of the storage tanks and one is located on the waste gas stream from the cryogenic system. They are simple taps where gas can be collected at a slow rate in a plastic bag for an eight-hour integrated sample, or an instantaneous sample can be collected in a gas bomb.

10.9 Flow Measuring Devices

There are two orifices for measuring flows in the Rare Gas Plant. One measures the flowrate of gas just downstream of the storage tanks and the other measures the flowrate of the waste gas from the cryogenic system. The flowmeters are equipped with flow recorders and flow integrators.

11. PRODUCT HANDLING SYSTEM

Product gases issuing from the batch still are placed first in holding tanks and then are transferred to shielded shipping tanks after the batch still fractionation is complete. These tanks and other support equipment for handling Rare Gas Plant product are described in the following paragraphs.

11.1 Krypton Product Hold Tanks

Prior to shipping, krypton gas from the batch still is transferred

into evacuated hold tanks (WN-158A and B) which are located in the south gas cell. The tanks are conventional 2000 psi, 191 cubic-foot gas cylinders contained inside shielded casks Each cask has a removable plug for removing or installing the gas cylinder.

11.2 Xenon Product Hold Tank

Xenon gas from the batch still is transferred into a separate, evacuated hold tank (WN-159) also located in the south gas cell. The xenon hold tank is a carbon steel cylinder surrounded by four-inch thick lead brick for radiation protection.

11.3 Product Shipping Cylinders

When fractionation of the batch still bottoms is complete, the gases collected in the product hold tanks are transferred immediately by a compressor through a manifold to the product shipping cylinders. The product cylinders and shipping casks for both xenon and krypton are identical to the krypton hold tanks discussed in Section 11.1 above. After filling, the cylinders and casks can be removed for shipping by a crane which would lift them up through a hatchway at the top of the cell.

IV. OPERATING EXPERIENCE

Operating experience with the Rare Gas Plant during several campaigns has been generally good and has improved with each succeeding run. During one campaign lasting approximately 1½ months, some 18,000 curies of krypton and 1400 liters of xenon were recovered. The typical analyses of product from one campaign are given in Table IV. Some operational problems with the Rare Gas Plant equipment have occurred and these, as well as a test mode for the presence of acetylene, are discussed in the following sections.

Table IV

TYPICAL KRYPTON AND XENON PRODUCT ANALYSES

Product Collected	Component, Mole Percent[a]						
	Xe	Kr	O_2	N_2	H_2	NO NO_2	A
Krypton-Rich Fraction	2.2	84.3	10.6	1.4	<0.2	<0.05	0.05
	3.5	78.8	9.3	6.9	<0.05	<0.2	0.1
	4.4	84.1	0.4	6.9	<0.05	<0.1	0.11
	3.5	85.7	<0.05	5.3	1.0	0.2	0.05
Xenon-Rich Fraction	92.9	0.4	3.7	0.6	<0.05	<0.05	<0.05
	93.6	0.22	5.1	0.3	<0.05	<0.1	<0.01
	90.7	0.14	5.2	0.4	<0.05	<0.2	<0.01

[a] Components do not total 100% because of analytical accuracy.

1. REGENERATORS

The main problem on early Rare Gas Plant runs involved undesirable freezing of condensibles in the regenerators, causing the regenerators to plug frequently. The required defrosting operation was time-consuming and costly. It is believed that water vapor picked up in the second Nash-Hytor compressor caused the regenerator plugging problems. Subsequently, a dryer (see Section III, 8.) was installed downstream of the compressor to eliminate this problem.

2. INSTRUMENT PROBE FREEZING

Another operating problem involved the freezing of the level probes in the primary column, and the freezing of the transfer line between the

primary column and the batch still. Apparently, these lines froze when the concentrations of krypton and xenon exceeded the solubility limits in the liquid oxygen collected in the bottom of the primary column. The problem was solved by decreasing the time interval between transfers--effectively reducing the product concentration in the primary columns.

3. ACETYLENE TESTS

During one run of the Rare Gas Plant, tests were made to determine if acetylene was present in the dissolver off-gas at concentrations high enough for an explosion hazard. The tests indicated that the total hydrocarbon present in the process gas amounted to less than 10^{-4} percent, far below the lower explosive limit (24 percent) for acetylene in air. It was concluded that there was no explosion hazard; the tests are discussed in Appendix C.

4. RHODIUM CONVERTERS

During the early portion of one campaign, significant quantities of undesirable nitrous oxide, N_2O, in the feed gas were not decomposed to N_2 and O_2 as intended upon passage through a high-temperature converter containing a rhodium-on-alumina catalyst. This problem was identified when instruments for the regeneration columns showed an increasing pressure drop across the column and a decreasing gas flow rate. The N_2O, with a melting point of -131°F, froze on the Raschig rings in the regenerator columns and slowly blocked the gas passageways. Samples of feed gas downstream of the rhodium converters were then analyzed, and results showed that the N_2O decomposition was only 50 percent complete.

Review of operating data for the few days prior to plugging of the regenerating columns indicated that rhodium catalyst in the converters had been briefly exposed to temperatures above 2000°F. This excessive temperature caused partial sintering of the catalyst particles. Channeling of gas flow within the catalyst bed then reduced the overall surface area of the catalyst in contact with N_2O during its passage through the bed. Evidence of the sintering and channeling within the catalyst bed was found when the catalyst was removed from catalytic units. Catalyst at the middle and bottom of the vessels which experienced the highest temperatures had to be removed with a crow bar.

New catalyst was purchased and placed in the unit, and prior to startup, both the old and new catalyst were tested with simulated process gas. The results, as shown in Table V, indicate that greater than 99 percent conversion of the N_2O could be achieved at temperatures above 860°F. After break-up of the chunks of the original catalyst to prevent channeling, the original catalyst had very nearly the same degree of reactivity as did the new catalyst.

Prior to startup, instrumentation was installed to shut off electrical preheaters on the process gas stream whenever the catalyst temperature reached 1200°F. This prevented excessive temperatures and any subsequent damage of the catalyst. Thereafter, no further problems were encountered with N_2O.

5. DRYER

Problems with the dryer have occurred due to contaminants and excessive water in the feed gas. This was caused by malfunction of other Rare Gas Plant equipment which overloaded the demister just upstream of the dryer. Some excess wastes then overflowed from the demister into the dryer, overloaded the desiccant, and passed through to the regenerators. The water froze in the regenerators and severely restricted the gas flow rates. A second demister was installed downstream of the first unit to prevent excess water from reaching the dryer.

Some deterioration of the molecular sieve desiccant was noted during this run. An investigation showed traces of nitric acid were reacting with the desiccant. Nitric acid formed when traces of NO_2 in the feed gas reacted with water vapor absorbed by the desiccant.

6. REGENERATOR CHECK VALVES

Malfunction of check valves located at the base of the regenerators occurred during the early portion of one campaign. When the valves were inspected after an unscheduled shutdown of the Rare Gas Plant, a broken spring was discovered in one valve. All four of the spring-loaded check valves were then removed and replaced with simple swing-type check valves. However, during a later period when excessive amounts of the contaminants were freezing in the regenerators, flow and pressure drop measurements indicated that the swing-type check valves tended to stick. They were removed and replaced with new spring-loaded check valves. Operation thereafter was satisfactory.

Table V

DATA FROM TESTS MADE ON RHODIUM-ALUMINA CATALYST

Test No. / Catalyst	1 original[a]	2 original[a]	3 original[a]	4 new	5 new	6 new	7 new	8 new
Average Operating Temperature, °F	1087	1184	1150	707	860	1020	1100	1155
Space Velocity[b]	500	2010	2010	1950	1970	1980	1090	1990
Linear Gas Velocity[c], ft/sec	0.176	0.74	0.72	0.51	0.60	0.65	0.35	0.72
Feed Gas Composition, Vol%								
N_2O	19.4	11.4	13.60	13.3	16.0	16.1	18.2	15.6
N_2		69.1		67.8	65.7	65.6	63.9	65.9
O_2		18.6		18.2	17.5	17.5	17.1	17.7
A^2		0.8		0.81	0.78	0.79	0.77	0.79
Product Gas Composition, Vol%								
N_2O	0.2	<0.1	<0.1	0.2	0.07	0.07	0.08	0.13
N_2		76.2		76.1	75.5	75.7	75.2	75.5
O_2		23.0		23.0	23.7	23.6	24.0	23.6
A^2		0.7		0.76	0.73	0.73	0.70	0.73
Conversion, %N_2O decomposed	98.9	99.1	98.9	98.5	99.6	99.6	99.6	99.1

(a) Tests made after breakup of sintered chunks into granules.

(b) Standard ft^3 of gas per ft^3 bed per hour.

(c) At process temperature

V. PLANT IMPROVEMENTS FOR FUTURE PROCESSING CAMPAIGNS

Equipment and process modifications that will be made to improve the performance of the Rare Gas Plant during future runs are:

1. Silica gel will be used to replace the molecular sieve desiccant in the"LECTRODRYER" (WN-165) because it is more resistant to nitric acid. In addition, the lower portion of the desiccant will be decrepitation-resistant silica gel to minimize any effects of a sudden surge of water from the demister. During the recent operation, some degradation of the molecular sieve desiccant occurred because trace amounts of NO_2 in the off-gas were also absorbed. The substitution of an acid resistant silica gel will correct this problem, but will require a change in the flow direction of the regeneration gases from parallel flow to counter flow with respect to the absorption flow. This is required to miminize decrepitation of the silica gel due to over saturation with water. Since silica gel has less sorptive capacity for water than molecular sieves, the absorption-regeneration cycle probably will be shortened.
2. An orifice flowmeter is to be installed on the air bleed line to the second set of Nash Hytor compressors (WN-259 and WN-260) to provide additional flow data for accurately computing material balances.
3. Additional gas sampling points are to be installed as follows:
   a. On the dissolver off-gas upstream of the first set of compressors.
   b. On the exit line of the rhodium catalytic converters.
   c. On the process gas inlet line to the cryogenic system.
   d. On the dissolver off-gas line downstream of points from which various pressure relief valves can release krypton into the header.
4. Air leakage into the dissolver off-gas should be minimized in order to maximize total throughput in the Rare Gas Plant. One way to decrease present leakage is to install a redesigned valve for the charging chute of the dissolvers. The decreased air leakage will also allow storage of dissolver off-gas for longer periods of time should the cryogenic unit be inoperative.
5. To reduce the level of NO_2 in the dissolver off-gas being fed to the catalytic unit, the caustic sprays in the top of the hold tanks will be operated continuously. Dissolver off-gas will be compressed into one hold tank with the caustic spray on. Feed gas for the Rare

Gas Plant can be taken from the other hold tank where the caustic spray is off, to minimize carryover of caustic mist.

6. A fast warm-up trace or heating tape will be installed on both regenerators in the cryogenic system. This will allow a much shorter defrosting time for the cryogenic unit, and thus, reduce the amount of dissolver off-gas that must be discharged to the atmosphere after filling of the hold tanks.

VI. REFERENCES

1. Air Reduction Company, "Final Report Recommendation for Improvement of Gas Plant Facility," AIRCO-C-211-1. (SECRET).
2. W. G. Fostowski and J. G. Gurowitsch, Physiochemica USSR Vol. XI, No. 6, 1939.

APPENDIX A

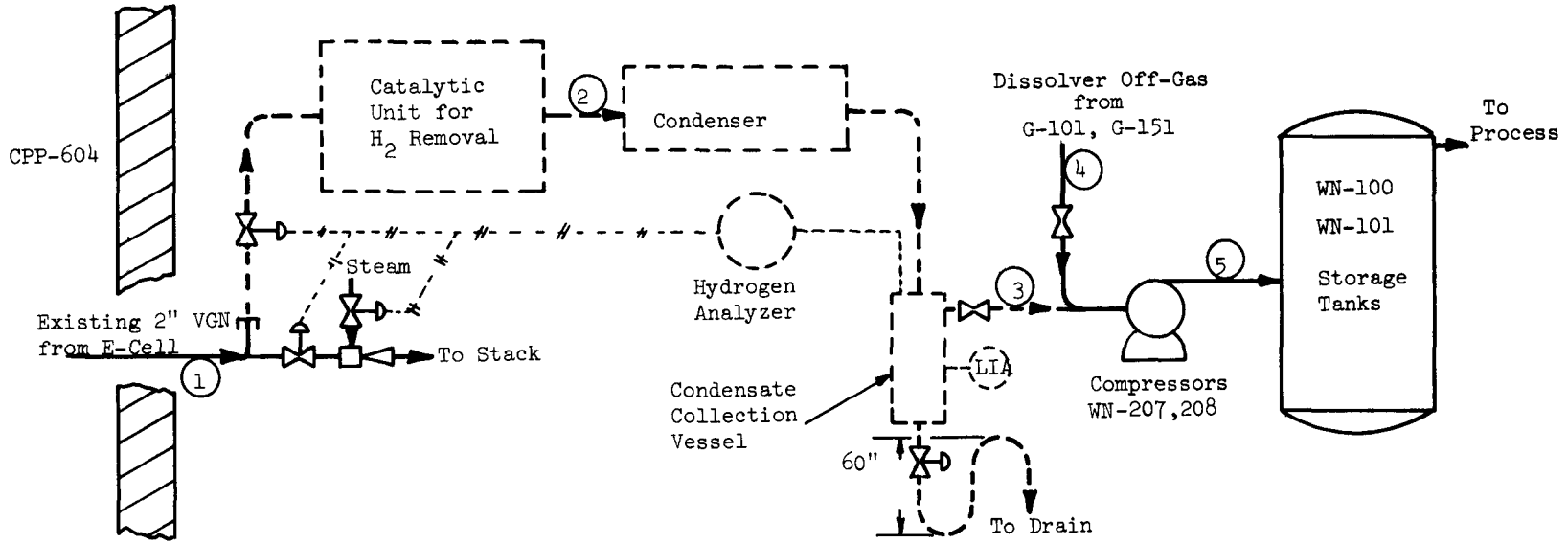
PROCESS DESCRIPTION FOR RECOVERING KRYPTON AND XENON FROM HYDROGEN-RICH DISSOLVER OFF-GAS

If it becomes desirable to recover krypton and xenon from dissolver off-gas at ICPP containing a high hydrogen content, the hydrogen would first be removed from the off-gas catalytically. A condenser and separator pot would be needed to remove water from the discharge stream of the catalytic unit. A hydrogen analyzer would be installed downstream of the catalytic unit to automatically divert the dissolver off-gas to the stack if the hydrogen content became too great.

To permit co-processing of hydrogen-rich and -lean dissolver off-gases at ICPP, the installation would be designed so that off-gas from two different dissolution processes, or both combined, could be processed in the Rare Gas Plant. This could be done by using the piping scheme shown on Figure A-1.

A chemical flowsheet for recovering Kr and Xe from hydrogen-rich dissolver off-gas is shown on Figure A-2. The gas would be processed in the existing Rare Gas Plant after the hydrogen in the gas was catalytically oxidized in a catalyst unit. The hydrogen-rich off-gas flow is intermittent because fuel dissolution is accomplished semi-continuously. During times when the dissolver was being charged with fuel and the system purged with N_2, the dissolver off-gas flow would be diverted to the ICPP stack rather than collected in the hold tanks.

While a dissolution cycle was in progress, the dissolver off-gas flow would vary in rate from about 3 to 50 scfm but would average about 16 scfm. The hydrogen-rich off-gas stream from the dissolver would also contain nitrogen from sparging, some water vapor, and traces of krypton and xenon. The average composition of the off-gas stream would be about 88 percent hydrogen and 12 percent nitrogen. The dissolver off-gas would be passed through the catalytic unit where the hydrogen concentration would be reduced to an average concentration of one percent. Water vapor would be removed from the gaseous effluent in a condenser and separator pot. A compressor (WN-207, or 208) would then compress the gas into storage tanks (WN-100 and WN-101). From the storage tanks, the gas would flow to a compressor (WN-259 or 260) where it would be mixed with air to make up a total flow volume of about 20 scfm. After compression to about 30 psig,



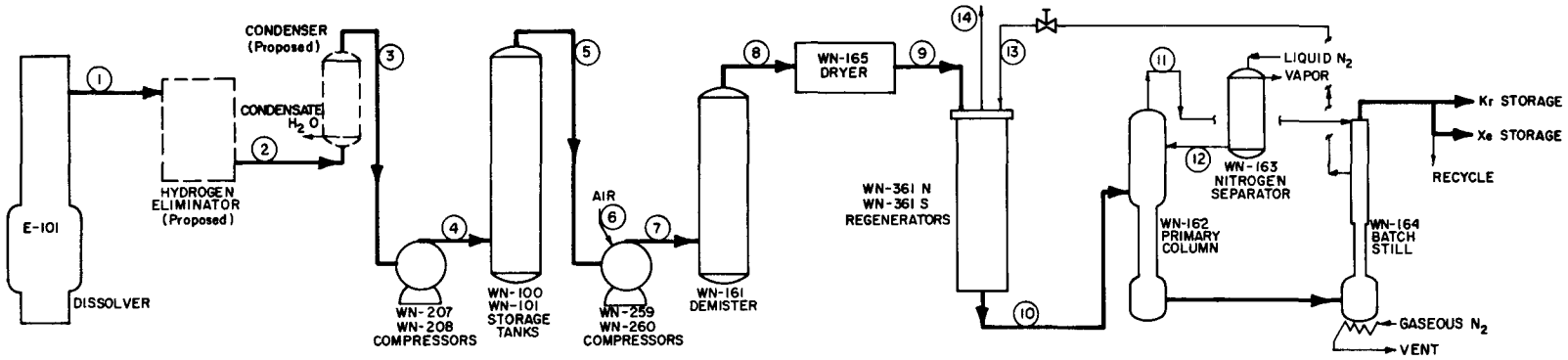
Stream	1	2	3	4	5
Description	1st Dissolver Off-Gas	Hydrogen Lean Off-Gas	Cooled Hydrogen Lean Off-Gas	2nd Dissolver Off-Gas	Combined Off-Gas
Flow, scfm	16	2(a)	2	9	11
Pressure, psig	-(0.4)	-(0.5)	-(0.6)	-(0.6)	20-40
Temp., °F	90	700	90	90	90
H_2, Vol. %	88	1	1	2	2
N_2, Vol. %	12	98	98	75	78
O_2, Vol. %	neg(b)	1	1	4	4
N_2O, Vol. %	neg	neg	neg	19	16
H_2O, lb/hr	neg	42	neg	neg	neg

Legend:
Existing
Proposed

(a) Neglecting water vapor.
(b) Negligible

Figure A-1

FLOWSHEET FOR PROCESSING HYDROGEN-RICH AND HYDROGEN-LEAN OFF-GAS IN THE RARE GAS PLANT



STREAM	1 (a)	2 (a)	3 (a)	4 (a)	5 (c)	6	7	8	9	10	11	12	13	14
DESCRIPTION	DISSOLVER OFF-GAS	HYDROGEN LEAN OFF-GAS	COOLED FEED TO COMPRESSOR	COMPRESSED GAS TO STORAGE	STORED FEED GAS	MAKEUP AIR	FEED TO PROCESS	FEED TO PROCESS	DRIED FEED TO AIRCO UNIT	COOLED FEED GAS TO PRIMARY COLUMN	PRIMARY COLUMN WASTE GAS	LIQUID NITROGEN TO PRIMARY COLUMN	COOLANT GAS FROM BATCH STILL CONDENSER	WASTE GAS TO STACK
Gas Flow, scfm	16.0	2.0 (b)	2.0	2.0	1.3	18.7	20.0	20.0	20.0	20.0	30.0		30.0	30.0
Liquid Flow, lb/hr												40		
Pressure, psig	-0.4	-0.5	-0.6	20-40	20-40	0	30	29	28	27	25	30	3-5	-1.3
Temperature, °F	90	700	90	100	80	70	80	80	80	-260	-310	-310	-300	75
N_2, Vol. % (b)	12.0	98.0	98.0	98.0	98.0	78.1	79.5	79.5	79.5	79.5	>99.9	100	>99.9	>99.9
O_2, Vol. % (b)	neg (d)	1.0	1.0	1.0	1.0	20.9	19.6	19.6	19.6	19.6	0	0	0	0
H_2, Vol. % (b)	88.0	1.0	1.0	1.0	1.0	0	0.1	0.1	0.1	0.1	<0.1	0	< 0.1	<0.1
Dew Pt., °F	90	210	90	90	80	58	64	64	-40	-260	-310	-310	-310	-40
CO_2, Vol. % (b)	0	0	0	0	0	0.03	neg	neg	neg	0	0	0	0	0
A, Vol. % (b)	0	0	0	0	0	0.9	0.8	0.8	0.8	0.8	0	0	0	0
Kr, scfm	$1x10^{-3}$	$1x10^{-3}$	$1x10^{-3}$	$1x10^{-3}$	$6x10^{-4}$	neg	$6x10^{-4}$	$6x10^{-4}$	$6x10^{-4}$	$6x10^{-4}$				
Xe, scfm	$6x10^{-5}$	$6x10^{-5}$	$6x10^{-3}$	$6x10^{-5}$	$4x10^{-3}$	neg	$4x10^{-3}$	$4x10^{-3}$	$4x10^{-3}$	$4x10^{-3}$				
H_2O, lb/hr	neg (e)	42	neg	neg	neg	neg	neg	neg	neg	0	0	0	0	0

(a) Dissolution is a batch process; therefore, flow of these streams is intermittent.

(b) Neglecting water vapor.

(c) Continuous flow on a 24 hour basis.

(d) During dissolution when leakage of air into system will be negligible, during charging operations the off-gas will be vented to the stack.

(e) Negligible.

Figure A-2

CONCEPTUAL FLOWSHEET FOR PROCESSING
HYDROGEN-RICH DISSOLVER OFF-GAS

the gas would be dried during its passage through demister (WN-161) and dryer (WN-165). Then, the gas (N_2, O_2, A Kr, Xe) would flow to the cryogenic system where it would be processed in the same manner as hydrogen-lean dissolver off-gas.

APPENDIX B

VAPOR PRESSURE AND FREEZING TEMPERATURE DATA

This appendix contains vapor pressure data and freezing temperature data for binary systems of krypton, xenon, oxygen and for the individual components.

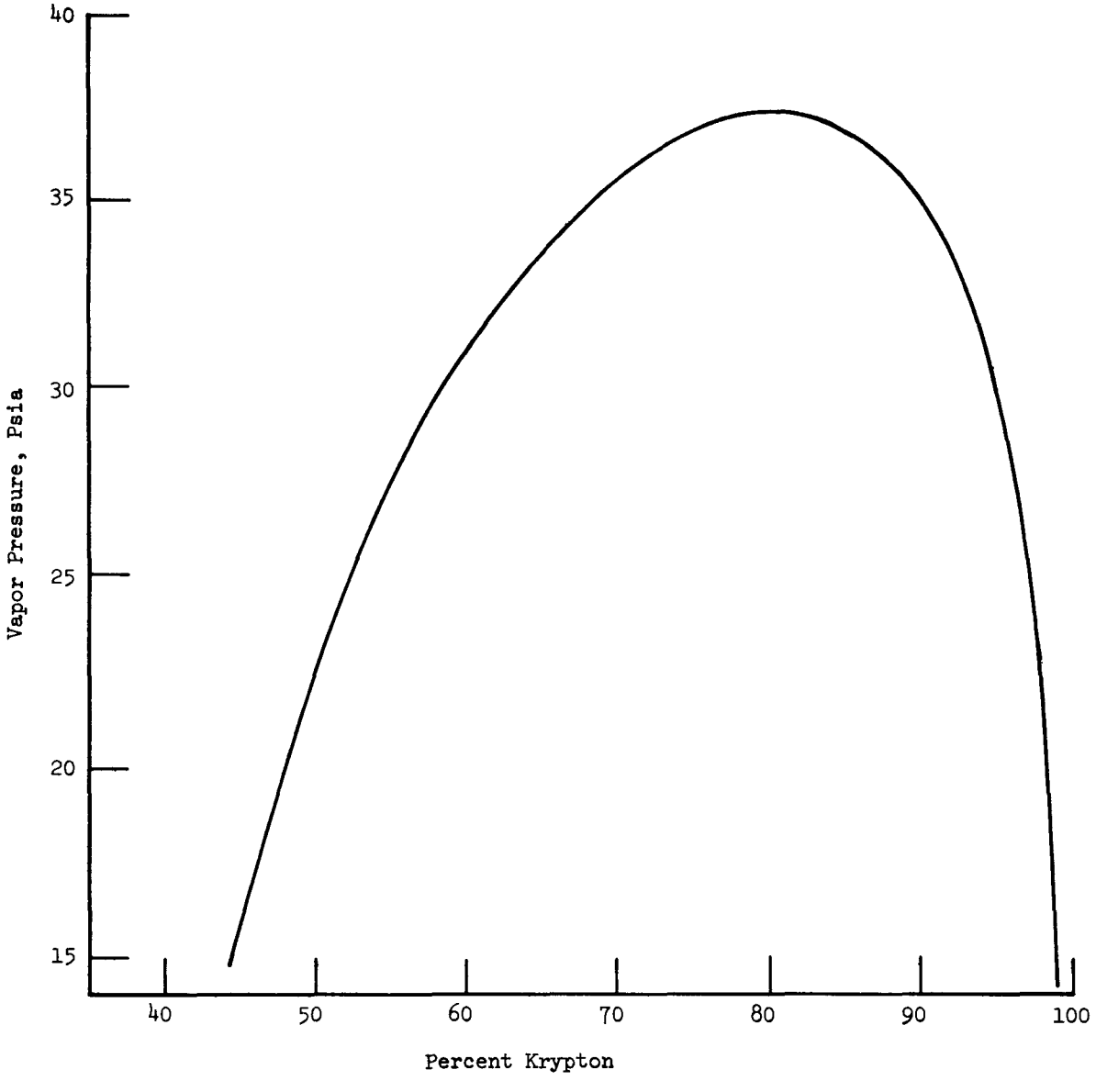


Figure B-1

KRYPTON-OXYGEN BINARY VAPOR PRESSURES OF FREEZING MIXTURES

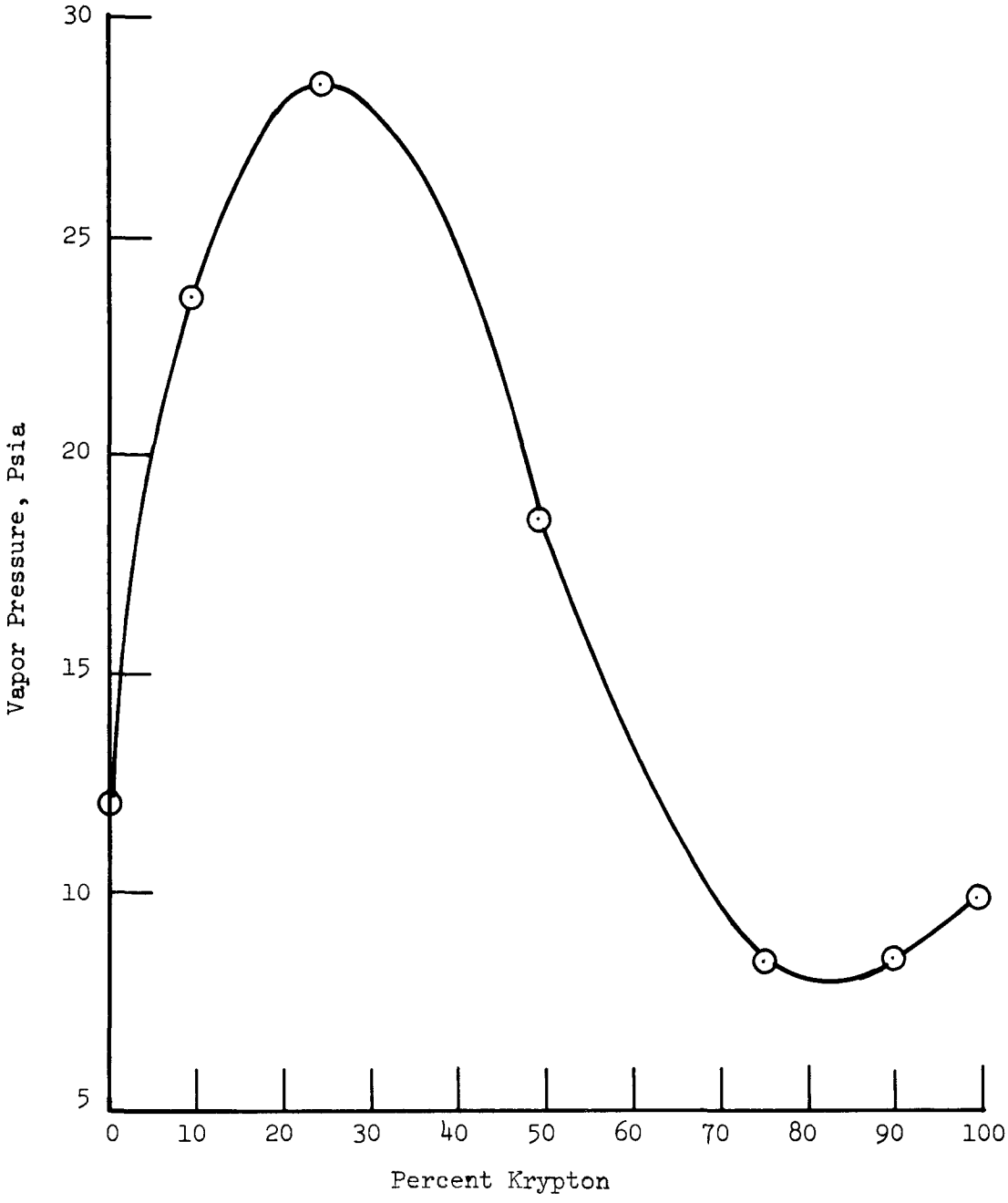


Figure B-2

XENON-KRYPTON BINARY VAPOR PRESSURES OF FREEZING MIXTURES

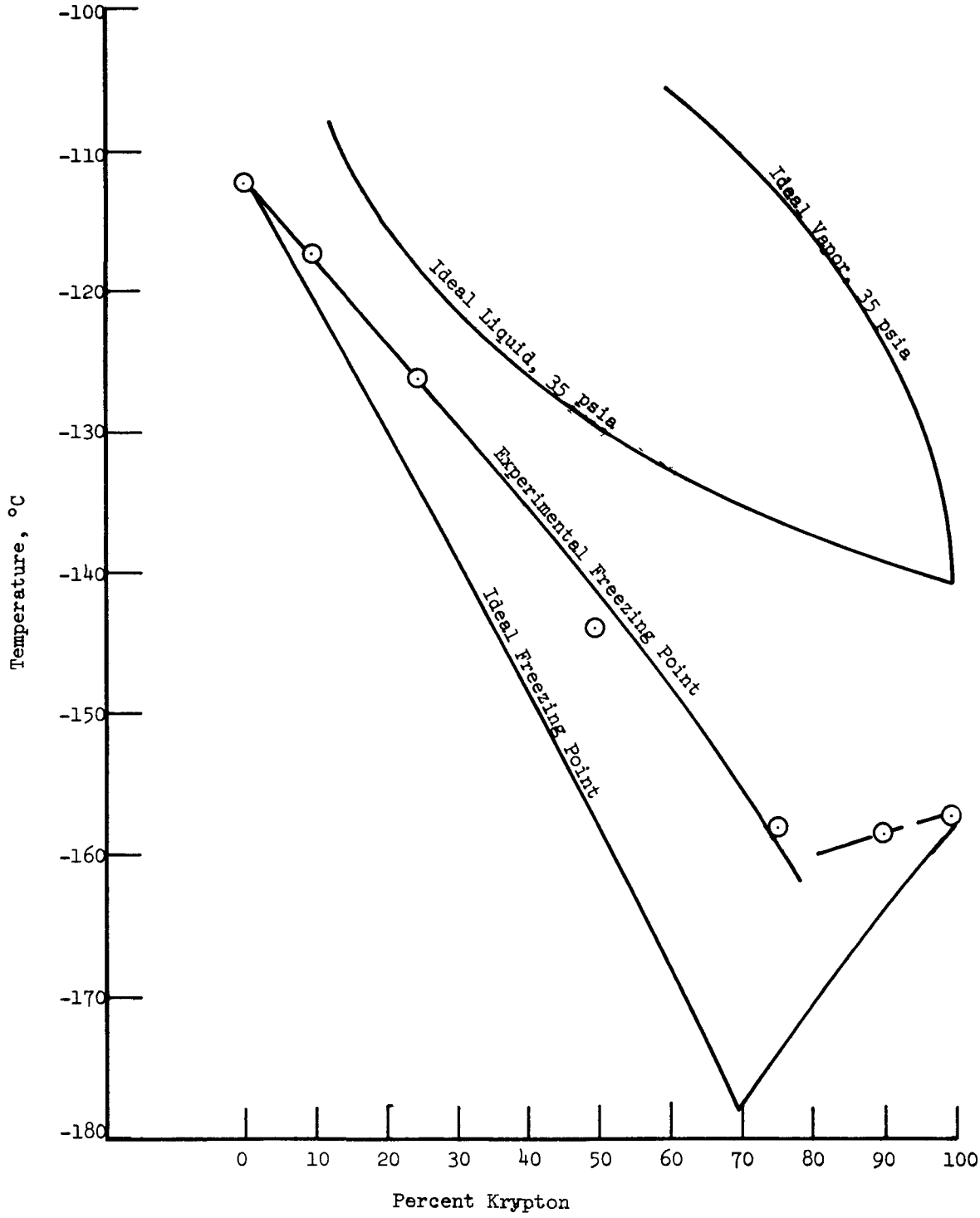


Figure B-3

PHASE DIAGRAM AND FREEZING TEMPERATURES OF KRYPTON-XENON BINARY SYSTEM

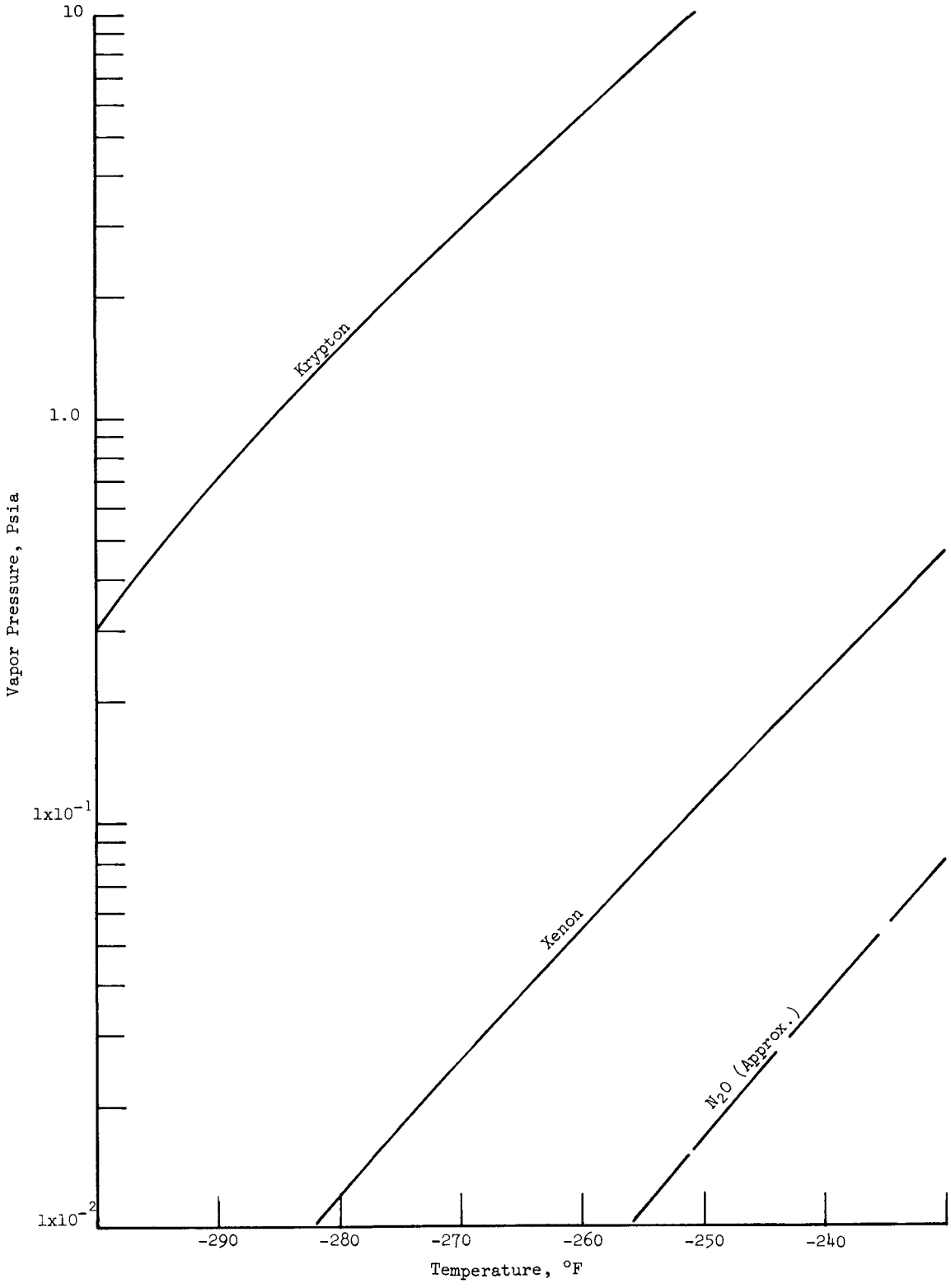


Figure B-4

KRYPTON, XENON, NITROUS OXIDE VAPOR PRESSURE DATA

Table B-I

KRYPTON-OXYGEN EQUILIBRIUM DATA[2]

$-297^{\circ}F$

PSIA	Liq.	Vap.	α_{OK}
14.1	0.02	0.0016	12.7
13.7	0.05	0.0039	13.4
13.1	0.10	0.0078	14.2
12.6	0.15	0.0113	15.3
11.7	0.246	0.0179	17.9
10.7	0.382	0.0242	25.0
10.0	0.501	0.0286	34.0

$-315^{\circ}F$

35.6	0.020	0.0024	8.6
34.7	0.050	0.0058	9.0
33.3	0.100	0.0141	9.6
29.9	0.206	0.0248	10.2
29.0	0.244	0.0293	10.7
27.0	0.316	0.0377	11.8
26.1	0.344	0.0421	11.9
25.2	0.391	0.0466	13.1
24.2	0.426	0.0513	13.7
23.2	0.464	0.0561	14.5

APPENDIX C

ACETYLENE TESTS

Explosions have been known to occur because of the presence of hydrocarbons, especially acetylene, in liquid air plants. Acetylene has a solubility of 5.6 ppm in liquid oxygen. Above this level, solid acetylene separates from and floats in oxygen-acetylene mixtures. Explosions cannot occur when acetylene is in solution in oxygen, but the presence of solid acetylene does create an explosion hazard.

Since some carbon is present in nuclear fuel elements, it was postulated that some acetylene could be present in the dissolver off-gas. To determine if there was an explosion problem, the cryogenic system was operated for extended periods during the first campaign and then the amount of hydrocarbons which had accumulated in the still bottoms and in the primary column was determined. In addition, the hydrocarbon content of the dilution air and dissolver off-gas were determined. Part of the tests were made with a feed gas composed of dissolver off-gas and nitrogen so that the hydrocarbons would be collected in liquid nitrogen. The remainder of the tests were made with an air feed.

The analyses summarized in Table C-1 indicate that the presence of small amounts of acetylene and other heavier hydrocarbons are probably a result of fragmentation of the heavier hydrocarbon components during analysis. Calculations showed that if all the hydrocarbon detected in both the air and dissolver off-gas were acetylene, the Rare Gas Plant could operate several months without exceeding the 5.6 ppm solubility limit in the primary column. Based on this information, explosions in the Rare Gas Plant as a result of acetylene formation are considered to be impossible; nevertheless, administrative control is used which requires removal of the still bottoms once per shift.

Table C-I

HYDROCARBON DEPOSITION IN CRYOGENIC SYSTEM

Days Operation	Feed Gas	Sample From	Total ppm Increase[a]	Average Daily ppm Increase[a]
2	Dissolver Off-Gas in nitrogen	batch still	0.013	
5	Dissolver Off-Gas in nitrogen	batch still	0.15	
4	Dissolver Off-Gas in nitrogen	batch still	0.04	
7	Dissolver Off-Gas in nitrogen	batch still	0.07	
7	Dissolver Off-Gas in nitrogen	batch still	0.34	
25			0.613	0.024
6	Dissolver Off-Gas in nitrogen	primary column	0.018	0.024
3	Air (no Dissolver Off-Gas)	batch still	0.089	
3	Air (no Dissolver Off-Gas)	batch still	0.103	
6			0.192	0.032

[a] Calculated concentration of acetylene in parts per million if all hydrocarbons present as acetylene in four liters of liquid in primary column kettle.