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SELECTIVE ABSORPTION PILOT PLANT FOR
DECONTAMINATION OF FUEL REPROCESSING
PLANT OFF-GAS

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OAK RIDGE GASEOUS DIFFUSION PLANT
OAK RIDGE, TENNESSEE

prepared for the **U.S. ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION**
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Oak Ridge Gaseous Diffusion Plant
Union Carbide Corporation
Oak Ridge, Tennessee

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ABSTRACT

A fluorocarbon-based selective absorption process for removing krypton-85, carbon-14, and radon-222 from the off-gas of conventional light water and advanced reactor fuel reprocessing plants is being developed at the Oak Ridge Gaseous Diffusion Plant in conjunction with fuel recycle work at the Oak Ridge National Laboratory and at the Savannah River Laboratory. The process is characterized by an especially high tolerance for many other reprocessing plant off-gas components. This report presents detailed drawings and descriptions of the second generation development pilot plant as it has evolved after three years of operation. The test facility is designed on the basis of removing 99 percent of the feed gas krypton and 99.9 percent of the carbon and radon, and can handle a nominal 15 scfm (425 slm) of contaminated gas at pressures from 100 to 600 psig (7.0 to 42.2 kg/cm²) and temperatures from minus 45 to plus 25°F (-43 to -4°C). Part of the development program is devoted to identifying flow sheet options and simplifications that lead to an even more economical and reliable process. Two of these applicative flow sheets are discussed.

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SELECTIVE ABSORPTION PILOT PLANT FOR DECONTAMINATION
OF FUEL REPROCESSING PLANT OFF-GAS

INTRODUCTION

Irradiation of nuclear fuels results in the production of substantial quantities of various radioactive isotopes. With the establishment of more stringent environmental emission guidelines, several methods have been proposed for removing these fission products/by-products from contaminated nuclear process off-gases [3,17,29,30,42]. Selective absorption is one of the more versatile processing schemes that has been commercially adapted to the decontamination of different reactor off-gases [5,14,15,39]. Several process solvents, including carbon tetrachloride [23,40], kerosene-base liquids [31], liquid nitrogen [3,4], nitrous oxide [30], liquid carbon dioxide [6,12,13,17,43], dichlorodifluoromethane [24,30,41], and trichlorofluoromethane [25], have been proposed for this and other applications. Considering solvent capacities, separation factors, and thermal and radiation stabilities, as well as overall process safety and economic features, Steinberg [30] suggested in 1959 utilizing an absorption process employing dichlorodifluoromethane (refrigerant-12) for stripping the noble gases from contaminated air streams. Both the solubility data of Steinberg at Brookhaven and Yamamoto and Takeda at the University of Tokyo [46] and the regular solution theory calculations of Merriman at Paducah [27,28] show that krypton and xenon are markedly more soluble in refrigerant-12 than are nitrogen, oxygen, and argon.

Development of the fluorocarbon-based process was initiated in 1967. At that time, a test facility was built at the Oak Ridge Gaseous Diffusion Plant (ORGDP) to establish general process feasibility and to collect krypton absorption data [24]. This initial equipment, while somewhat limited in operating capability, functioned exceptionally well and valuable overall performance information was attained. In 1970, the testing program was oriented to demonstrate specific application of the process to achieve cleanup of the off-gas from light water reactors [32]. Pilot plant tests demonstrated the basic feasibility of the process and showed that better than 99.9% of the krypton and xenon could be removed from various reactor off-gases such as air, nitrogen, argon, helium, and hydrogen, and process concentration factors in excess of 1000 could be achieved. The absorber operation and performance were well defined during these tests. Subsequent data analysis and correlations yielded needed absorber design equations [32,34]. The tests indicated that the intermediate and final stripping operations were complex but the existing facility lacked the provisions to monitor the performance of these two parts of the process well enough to get needed quantitative data. Consequently, only qualitative information was actually obtained on the gas stripping equipment during the earlier work. In 1971, scoping tests showed that the process has a remarkable tolerance for other fuel reprocessing plant off-gas components, such as nitrogen oxides, carbon dioxide, water, iodine, and methyl iodide [26,33,34], but additional work

was not performed in this area, again because of existing pilot plant limitations.

Based on the demonstrated operability, performance, and tolerance that the fluorocarbon process exhibited for typical off-gas components, efforts were initiated to adapt the process to krypton decontamination of LMFBR fuel reprocessing plant off-gas. In concert with the overall LMFBR fuel recycle program at the Oak Ridge National Laboratory (ORNL), a more sophisticated facility was proposed for the expanded program. The new pilot plant was designed in 1972 and put into operation at ORGDP late in 1974 [35,36]. The new pilot plant was built to be especially flexible with the necessary analytical capabilities for overall as well as detailed component analysis. In 1975, it was recognized that unresolved waste management problems were detrimental to the continuing development of commercial LWR fuel cycle facilities. Through an evaluation of available waste management alternatives, specific development needs were identified as necessary in order for LWR reprocessing plants to meet applicable environmental requirements. Specifically, effective and reliable off-gas decontamination equipment was needed for krypton-85, carbon-14, tritium, and nitrogen oxides [1].

Since there were only minor differences between LMFBR and LWR fluorocarbon process applications, a joint LMFBR/LWR fluorocarbon process development program was formulated between ORGDP, ORNL, and the Savannah River Laboratory (SRL) to efficiently meet the needs of both reactor fuel cycles. During this same time, it became obvious that the fluorocarbon-based process being developed for krypton-85 removal could also be used for effective, simultaneous removal of carbon-14 (as carbon dioxide) and various nitrogen oxides. Available data also indicated that the same process might be an effective means for removing iodine, methyl iodide, and water. Especially important was the assessment of the process response to quantities of these other contaminants that might elude upstream off-gas cleanup systems. Consequently, the scope of the fluorocarbon process development effort was broadened to include further definition of this general capability of the process for application to LMFBR and LWR reprocessing plants.

Early in 1977, considerable interest was also expressed in the use of alternate fuel cycles, such as the one based on thorium, and various optional reprocessing schemes that would reduce the world-wide opportunity for plutonium diversion. Consequently, in order to keep the program responsive to the needs of the expanded fuel cycle effort, the fluorocarbon process development program was structured to the newly formed Advanced Fuel Recycle Program at ORNL and the Alternate Fuel Cycle Technologies Program at SRL. Radon-222 has been identified as a potential off-gas problem for the thorium fuel cycle, and therefore, work is scheduled to verify that the fluorocarbon process will also be an effective means for removing this fission product.

The fluorocarbon development program is currently divided into four major areas: (1) process development, (2) process application, (3) solvent chemistry, and (4) reliability analysis. Process development

is being done at the ORGDP in the pilot facility built specifically to study the reprocessing plant application. Process application studies are being performed at both ORGDP and ORNL, while the solvent chemistry work is being performed at ORNL and the University of South Carolina. The Kaman Sciences Corporation (KSC), Colorado Springs, Colorado, is performing process reliability analyses. The main objective of the process development work is to generate all process technology required to completely define the fluorocarbon-based process and all associated peripheral equipment for the reprocessing plant application. Process application studies are providing design models required for process optimization and conceptual plant design. This work is identifying flow sheet options, pointing out the relative effects and importance of individual process elements and operating conditions on the overall system function, and will eventually provide efficient demonstration plant start-up, operating, and shutdown methods and procedures. The solvent chemistry effort is establishing and confirming component solubilities, phase relationships, component interactions, and corrosion characteristics of the fluorocarbon system. The process reliability studies by KSC will evaluate the component and system reliability and recommend necessary flow sheet redundancy and backup support systems to ensure a high process on-line efficiency [45]. The overall effort will culminate in the detailed design and economic evaluation of a demonstration off-gas decontamination facility applicable to commercial reprocessing plants. This is the primary objective of the fluorocarbon process development program.

The second generation development facility has been in operation for nearly three years, and substantial improvements and modifications have been made to the plant equipment and control instrumentation in order to improve the overall performance, operability, and reliability of the process. Quantitative performance data are available in separate process development reports [35,36,37] and ORNL fuel recycle program progress reports [18-22]. The purpose of this report is to present detailed drawings and descriptions of the development pilot plant as it now exists. Part of the development program is devoted to identifying flow sheet options and simplifications that might lead to an even more economical and reliable process. It is important at this time to also briefly describe two of these alternative flow sheets that have evolved from the experimental work. This description will identify the general direction of the process application effort and will show how the process might finally look for the reprocessing plant application.

SUMMARY

EPA Standard 40 CFR Part 190 mandates that approximately 90% of the fission product krypton produced after 1983 be removed from nuclear process off-gas. A corresponding standard for carbon-14 and radon-222 has not been established but is under EPA study. The selective absorption plant is designed on the basis of removing 99% of the krypton and 99.9% of the xenon, radon, and carbon from the feed gas. The pilot plant has added provisions to routinely handle large quantities of other reprocessing

plant off-gas components including tritiated water, various nitrogen oxides, various organics, and iodine.

The test facility can process a nominal 15 scfm of contaminated gas at pressures from 100 to 600 psig and temperatures from minus 45 to plus 25°F. A reprocessing plant off-gas simulation station is capable of mixing a feed gas containing up to 10 components. A solvent recovery subsystem is provided to reduce the fluorocarbon content of the absorber off-gas from 8 to 10 mole percent to less than 1 ppm. Present pilot plant equipment is designed to provide a concentrated gaseous krypton product for storage in standard high pressure gas cylinders and a solid carbon-14 product. The design krypton product concentration factor is 10,000 based on a process inlet concentration of 1 to 10 ppm. Low temperature refrigeration equipment is required to enable process operation at preferred conditions. The pilot plant employs several mechanical evaporative-type refrigeration systems. While this particular system is useful for the pilot plant work, an optional "brine" system has been proposed for the reprocessing plant application.

Process flow sheets based on a combination absorber/fractionator and combination absorber/fractionator/stripper have evolved from the pilot plant work and indicate how the process might be applied to a reprocessing plant. The remaining development program is laid out in such a way as to guarantee the timely evolution of the technology consistent with the needs of the overall fuel recycle effort.

PROCESS REQUIREMENTS AND DESCRIPTION

Existing and proposed environmental emissions are based, in part, on available removal technology. As advanced technologies emerge, emission requirements will probably be altered accordingly, and consequently, what might be considered an adequate decontamination goal now very well might not be acceptable in a few years. EPA Standard 40 CFR Part 190 entitled "Environmental Radiation Protection for Nuclear Power Operations" mandates that the total quantity of radioactive materials entering the general environment from the entire uranium fuel cycle must contain less than 50,000 curies of krypton-85 per gigawatt-year of electrical energy produced after 1983. Assuming that the total burden of krypton retention is placed upon the fuel reprocessor, approximately 90% of the reprocessing plant off-gas krypton will have to be removed [10]. The potential for a long-term impact due to carbon-14 released from fuel cycle operations was not recognized until recently, and consequently, no emission standard exists for carbon-14. The EPA, however, has stated that the development and installation of controls to minimize environmental effects of carbon-14 is an important objective, and they will carefully follow the development of new knowledge concerning both the impact and controllability of this radionuclide [10]. Likewise, no specific emission standard has yet been placed on radon-222.

The fluorocarbon process is capable of removing better than 99.9% of the feed gas krypton and 99.99% of the xenon, carbon (as carbon dioxide),

and radon. The reference or design operating point of the development pilot plant is 99% removal of the fission product krypton. Krypton was chosen as the reference component because it is the least soluble of the radioactive off-gas contaminants. Operating the process at conditions necessary to yield a 99% krypton removal results in a corresponding 99.9% removal of the off-gas xenon, carbon, and radon.

So far, a preferred storage form or concentration limit has not been established for the radionuclides. Higher product concentrations will result in smaller storage requirements; however, the more concentrated fission product activity and decay heat must then be taken into consideration. Present pilot plant equipment is designed to produce a concentrated gaseous krypton product for storage in standard high pressure gas cylinders and a solid carbon-14 product, e.g., calcium or lithium carbonate, for storage in metal drums. Other storage methods can be evaluated as they are identified. The design krypton product concentration factor is 10,000 based on a process inlet concentration of 1 to 10 ppm. In order to achieve the target krypton concentration level, the feed gas carbon dioxide has to be removed from the noble gas. It is optional at this point whether or not the argon or fission product xenon is separated from the krypton. One of the product purification schemes under evaluation does effect a krypton-xenon separation; while another one yields a krypton-argon separation. The carbon released from the irradiated fuel during shearing and dissolution is joined by atmospheric carbon in the reprocessing plant in-leakage via various charging ports and air seals. Consequently, the final purity of the carbon-14 will be established by the amount of other carbon present in the reprocessing plant off-gas. The atmospheric contribution could be sizable and the carbon-14 product grossly diluted.

As currently planned, the krypton-85/carbon-14/radon-222 removal process will be the final step in an integrated chain of processes designed to collectively decontaminate reprocessing plant off-gas. The integrity and reliability of the overall decontamination system will undoubtedly be the subject of considerable interest. Legitimate concern will obviously be expressed not so much about how well the off-gas train will function in normal operation but about the overall consequences of abnormal operation and the capability of the individual processes to meet the challenges imposed by irregular or otherwise uncontrolled feed conditions. In this context, it is imperative to assess what happens in the event that all of the upstream primary removal equipment fails and large amounts of other fission products and chemical contaminants inadvertently pass downstream. For this purpose, the development pilot plant is built to evaluate the behavior and in-house consequences of large amounts of most other reprocessing plant off-gas components, including tritiated water, nitrogen oxides, carbon monoxide, methane, iodine, methyl iodide, and TBP.

Figure 1 is a schematic of the selective absorption process. The process serves to remove volatile radioactive contaminants from reprocessing plant waste gas streams and to subsequently concentrate them, reducing the long-term radioactive waste storage requirements. Absorption, fractionation, and stripping steps must be performed in order to accomplish these process

Figure 1

SCHEMATIC OF THE FASTER PROCESS

objectives. Each separative step exploits the gas-liquid solubility differences that exist between the solvent and the various feed gas constituents. The main separation of radioactive components from the bulk gas is effected in the absorber. The fractionator serves to remove the coabsorbed carrier gas from the solvent, thereby enriching the solvent in the more soluble components. The stripper removes all remaining gas from the process solvent and thereby prepares the solvent for recycle back to the absorber. The absorber consists of only a packed column, while the fractionator and stripper are each composed of a packed column, reboiler, and condenser. In addition, the fractionator equipment also includes a flash drum. Support equipment items for the basic process include a feed gas heat exchanger, process gas compressor, solvent pump, solvent cooler, storage tanks, and several refrigeration compressors. If the feed gas contains significant quantities of high boiling components, i.e., those components with a vapor pressure less than refrigerant-12, a solvent purification still is available as an in-line option to prevent these materials from building up in the recirculating solvent. Final product separation and isolation equipment has also been added to achieve higher product concentrations. Finally, a solvent recovery system is necessary to remove solvent vapor from the absorber off-gas.

The process feed gas is first compressed to the absorber column operating pressure and then cooled to the desired absorption temperature. Amounts of the feed gas water, iodine, and nitrogen dioxide will freeze out in the process gas cooler. The feed gas is then passed into the absorber. Under favorable operating conditions as determined by the absorption temperature, pressure, and process solvent-to-gas flow rate ratio, essentially all of the krypton, xenon, and carbon dioxide, plus a significant quantity of bulk feed gas is dissolved. Essentially all remaining water, iodine, and nitrogen dioxide and feed gas methyl iodide will also be dissolved by the solvent. Typically, the absorber might operate at a pressure of 100 to 300 psig, temperature near -25°F , and solvent-to-gas molar flow ratio of 10 to 15. The decontaminated gas leaving the top of the absorber is then passed on to the fractionator flash drum. The fractionator is normally operated at substantially less pressure than the absorber, e.g., 35 to 50 psig. A large portion of the less soluble gas desorbs upon entering the expansion chamber and passes into the overhead condenser. The loaded solvent containing the remaining gases is then fed into the top of the fractionator column where it is contacted with upflowing solvent vapor from the reboiler which desorbs nearly all of the remaining less soluble dissolved gas. A liquid-to-vapor molar flow rate ratio of approximately 4 to 6 is required for this step. The total concentration of gases dissolved in the solvent at the bottom of the column is generally quite small. Consequently, the fractionator reboiler is operated essentially at the saturation temperature of the solvent, which is 52°F for a pressure of 50 psig. Solvent vapor and a gas mixture consisting predominantly of nitrogen and oxygen pass from the top of the column and into the overhead condenser for solvent removal.

A large fraction of the less soluble absorber feed gases, i.e., nitrogen and oxygen, and a lesser fraction of the more soluble gases, i.e., krypton, xenon, carbon dioxide, and radon, are liberated from the solvent during the fractionation operation. Correspondingly, the remaining dissolved gas becomes further enriched in the more soluble gases. Since a perfect cut cannot be achieved and a measurable amount of radioactive gas is also evolved, the flash unit and column off-gas must be recycled back to the suction of the process compressor.

The solvent is next routed to the stripper section of the process. By operating the stripper column under an even lower pressure than that used in the preceding column, e.g., 15 psig, and with a lower liquid-to-vapor flow ratio, e.g., 2.5 to 3.0, the remainder of the absorbed gas is driven from the solvent and withdrawn from the process as a concentrated gaseous product. A series of physical and chemical traps is available to remove solvent vapor and subsequently isolate the individual radio-nuclides for storage. These traps are relatively small because the product gas flow leaving the top of the stripper varies from less than 0.1 to not more than 1% of the process feed gas flow. The primary process product flow rate is largely established by the amount of carbon dioxide introduced into the plant and the temperature of the stripper condenser.

The less volatile components such as water, iodine, methyl iodide, and nitrogen dioxide, if present, will remain in the solvent during the fractionation and stripping operation and will accumulate in the solvent if not removed. An optional solvent purification still has been added to the pilot plant to remove these feed gas constituents from the recirculating solvent. The still is not an essential part of the process and is bypassed when not needed. Normally, the purification step is conducted at a pressure of 5 to 10 psig. The overhead condenser operates at the dew point of pure refrigerant-12, i.e., around -10 to 0°F; while the reboiler operates at the boiling temperature of the particular reboiler liquid composition being maintained. If, for example, the reboiler contains mostly nitrogen dioxide with only a trace of refrigerant-12, the reboiler temperature would be between 80 and 90°F. A liquid reflux-to-distillate flow rate ratio of 0.5 to 1.5 defines the normal operating range of the pilot plant still. The purified refrigerant flows back to the primary process solvent storage tank prior to being pumped back to the absorber for reuse while the still bottoms product is withdrawn into a waste storage tank.

PILOT PLANT DESIGN

Figure 2 shows a photograph of the ORGDP selective absorption pilot plant as it was being built. Figure 3 shows an overall view of the same equipment. Figure 4 is a photograph of the completed facility with insulation.

PHOTO NO. PH-74-1961
(U)

Figure 2

SELECTIVE ABSORPTION PILOT PLANT DURING CONSTRUCTION

PHOTO NO. PH-73-1450
(U)

Figure 3

OVERVIEW OF THE PILOT PLANT DURING CONSTRUCTION

PHOTO NO. PH-75-370
(u)

Figure 4

OVERVIEW OF THE COMPLETED FACILITY

Figure 5 gives the pilot plant operating flow sheet, showing various gas and liquid process flows, monitoring and control instrumentation, and valve identification. The facility is designed on the basis of handling a nominal 15 scfm of contaminated gas at pressures from 100 to 600 psig and temperatures from minus 45 to plus 25°F. The process solvent flow rate can be varied from 0.5 to over 2.5 gpm. A reprocessing plant off-gas simulation station is provided that is capable of mixing a feed gas containing up to 10 components, including oxygen, argon, nitrogen dioxide, nitric oxide, nitrous oxide, carbon monoxide, methane, water, iodine, and methyl iodide, in addition to the noble gases and carbon dioxide. The solvent recovery subsystem is designed to reduce the fluorocarbon content of the absorber off-gas from 8 to 10 mole percent to less than 1 ppm. The solvent purification still has a maximum throughput of approximately 1.5 gpm. This section describes the pilot plant components in detail.

Absorber

The absorber column is 3 inches in diameter and contains 15 feet of stainless steel, high efficiency, wire mesh Goodloe* Column Packing. The absorber column diameter was determined from design equations provided by the packing manufacturer [16]. Details of the column assembly are given in figures 18 and 19 of Appendix A. The column is constructed from an 18-foot-long piece of 3-inch-diameter, Schedule 40, 304L stainless steel pipe. The column is divided into four sections: each of the bottom three sections contain three feet of packing while the top section contains 6 feet of packing. Gas sample points and thermocouple positions are provided between packing sections. The column is equipped with two solvent feed points that allow operation of either a nine-foot or fifteen-foot column: the top feed point permits use of the total 15 feet of packing while the lower one allows the 9-foot column operation. As recommended by the packing manufacturer [16], subway grating-type packing supports are employed. These supports consist of 1/8-inch-thick by 1-inch-wide stainless steel strips welded on 1-inch centers. Solvent leaving the absorber accumulates in a small reservoir at the bottom of the column. The level of fluid in this reservoir and, hence, the flow of solvent from the absorber are monitored and controlled by a Drexelbrook† Series 408 capacitance probe liquid level control system. The Drexelbrook 700-1-23 capacitance probe has an insertion length of 12 inches and is installed as shown in figure 18 of Appendix A. Pressure taps are provided at the column top and bottom to monitor the total and differential pressure during operation. Efficient column operation normally requires a pressure drop of 0.5 to 1.0 inch of water per foot of column packing.

The absorber column has been approved for a maximum working pressure of 600 psig at 0°F and was hydrostatically tested at 900 psig.

*Packed Column Company, Division of Metex Corporation, Edison, New Jersey.

†Drexelbrook Engineering Company, Glenside, Pennsylvania.

Figure 5

OPERATING FLOW SHEET OF THE SELECTIVE ABSORPTION PILOT PLANT

Fractionator

The fractionator consists of a flash drum, packed column, reboiler, and condenser. The fractionator column is 3 inches in diameter and similar in construction to that used for the absorber except that each of the three packed column sections contains 2.5 feet of Goodloe packing for a total packed height of 7.5 feet. Figures 20 and 21 in Appendix A give the column details. The fractionator reboiler connects directly to the bottom of the column, while the overhead condenser is set off to the side and is joined by a 90-degree elbow. The condenser offset allows external collection and sampling of the overhead condensate. The condenser is mounted 10 degrees to the horizontal.

The fractionator reboiler is 14 inches in diameter and approximately 4 feet long overall. Reboiler details are given in figures 22 and 23 of Appendix A. The reboiler shell was rolled from 0.5-inch-thick 304L stainless steel sheet. Internal heating is provided with a Chromalox* Model TMI-12183 18-kW immersion heater with Incaloy sheath. The heater is flanged through one end of the reboiler shell with 6-inch, 300-pound flanges. The design load on the reboiler is 5 kW, giving the heater a surface heat flux of less than 10 watts per square inch. The heater is controlled by a Loyola† LPAC-3 series, 480-volt, three-phase multipurpose SCR (silicon controlled rectifier) power controller with current and power limiters. The reboiler control variable is the column differential pressure drop. High temperature protection is provided by a Chromalox Model LFB7-7 indicating thermostat. The liquid level is monitored and maintained 4 inches above the heater bundle by a Drexelbrook capacitance probe liquid level control system like that used with the absorber. The insertion length of the probe is 12 inches.

The fractionator overhead condenser is a shell-and-tube-type heat exchanger with process gas in the shell and boiling refrigerant-22 in the tubes. The condenser was designed on the basis of a total condensing duty of 17,000 Btu/hr and overall condensing coefficient of 25 Btu/hr-sq ft-°F. The condenser shell side surface area is 23 square feet. Details of the condenser design are given in figures 24 and 25. The shell is constructed from 6-inch, Schedule 10, 304L stainless steel pipe and contains twelve 1-inch 304L stainless steel tubes. In order to promote better mixing, the tubes are filled with 5/6-inch, 316 stainless steel Pall Rings. At most, only a small portion of the available condensing surface is needed under "normal" operation since the fractionator is routinely fed subcooled liquid relative to the boiling point of the solvent at the prevailing fractionation pressure. Consequently, the bulk of the upflowing reboiler vapor is

*Edwin L. Wiegand Division, Emerson Electric Company, Pittsburgh, Pennsylvania.

†Loyola Industries, Division of Astrophysics Research Corporation, Harbor City, California.

condensed at some internal column point. While this method of operation is unorthodox, it offers substantial operational advantages [22,38]. All or half of the condenser can therefore be removed from service by simply valving out one or both of the refrigerant feed lines.

The fractionator flash drum, shown in figures 20 and 21, is a 6-inch-diameter stainless steel cyclone, approximately 30 inches high and equipped with a tangential inlet, a liquid drain, a vapor discharge pipe, and a mist shield. The expanded bottom section of the unit serves as the fractionator column liquid feed tank.

The fractionator, including the flash unit, packed column, reboiler, condenser, and all interconnecting piping, has been approved for a maximum working pressure of 350 psig at 0°F and was hydrostatically tested at 525 psig.

Stripper

The stripper consists of a packed column, reboiler, primary condenser, and final condenser. Except for size, the design of the stripper is similar to that of the fractionator. The stripper packed column is fabricated from a 6-inch, Schedule 10, 304L stainless steel pipe and contains three packed sections, each containing 4 feet of Goodloe packing, for a total packed height of 12 feet. Column assembly and details are given in figure 26 of Appendix A. Like the absorber and fractionator, the stripper column also contains provisions for pulling internal column gas samples.

Subway grating packing supports are used in each section. A good liquid feed distributor is an important part of every packed column regardless of the packing type and especially for columns with diameters of 6 inches or larger. The stripper column liquid distributor consists of a feed ring welded to a 1-inch feed header. The ring and header are drilled so as to spray liquid uniformly over the top packing surface. With the wire mesh packing installed properly, liquid redistribution between packing sections is not essential but included anyway to guarantee packing efficiency.

The stripper reboiler is constructed just like that of the fractionator except that the stripper unit is 18 inches in diameter and nearly 5 feet long overall. Reboiler assembly and details are given in figures 22 and 23. A Chromalox Model TMI-18403 40 kW immersion heater is used to supply heat. When maintained at 20 to 25 kilowatts, this high surface area heater has a low heat flux of around 10 watts per square inch. This heater is also fed from an SCR power controller and is likewise protected from overheating with a Chromalox Model LFB7-7 indicating thermostat. In this case, the reboiler control variable is the column operating pressure. A Drexel-brook capacitance probe liquid level control system is also employed.

The stripper overhead condenser system consists of two shell and tube heat exchangers. Both are mounted 5 degrees to the horizontal. As shown in figure 4, the final condenser is located directly above the primary unit, which in turn, sets off to the side of the stripper column similar to that

of the fractionator. The primary condenser is cooled with boiling refrigerant-22 on the tube side and the final condenser is cooled with lower temperature boiling refrigerant-502. The primary condenser was designed on the basis of a condensing duty of 77,600 Btu/hr and overall condensing coefficient of 50 Btu/hr-sq ft-°F. The condenser shell side surface area is 52 square feet. The design of the condenser is detailed in figures 27, 28, and 29. The shell is constructed from 10-inch, Schedule 40, 304L stainless steel pipe and contains thirty-two 1-inch 304L stainless steel tubes. The tube sheet is fabricated from 1-inch-thick 304L stainless steel sheet. Like the fractionator condenser, the tubes are also filled with 5/8-inch stainless steel Pall Rings to ensure better tube-side turbulence. The primary condenser is fed from the stripper column through a 6-inch-diameter side inlet. Condenser condensate is first mixed with fresh feed from the fractionator in a 3-ring mixing chamber on the bottom side of the condenser before being refluxed back to the stripper column. The final condenser was designed on the basis of a condensing duty of 10,000 Btu/hr and overall condensing coefficient of 50 Btu/hr-sq ft-°F. The condenser shell side surface area is 15.7 square feet. Design details are given in figures 30 and 31. The shell is constructed from a 4-inch, Schedule 10, 304L stainless steel pipe and contains fourteen 5/8-inch 304L stainless steel tubes.

The stripper assembly is approved for a maximum working pressure of 350 psig at 0°F and was hydrostatically tested at 525 psig.

Solvent Purification Still

The solvent still is built very similar to the stripper and fractionator sections of the plant but, because of the different process piping, operates fundamentally differently: purified solvent is taken from the condenser while solvent impurities are withdrawn from the reboiler. Initially, quantitative recovery of the high boiling feed gas components was not pursued. Rather, the solvent purification still was designed and built to remove only the bulk amount of these components from the circulating solvent [7]. During the past year, however, it became evident that the fluorocarbon process designed for krypton and carbon recovery could act as a valuable backup system to remove other fission products such as iodine and tritiated water and chemical contaminants such as nitrogen dioxide simply by modifying the existing solvent purification equipment to include a rectifying section with automatic reflux control [36]. This work was subsequently performed. A schematic of the modified still and operating flow sheet is given in figure 5. Overall, the control instrumentation of the solvent still is more complex than that required for any of the other sections.

The still column is constructed from a 10-inch, Schedule 10, 304L stainless steel pipe. The column packing is divided into an 8-foot bottom stripping section and a 4-foot top enriching section for a total packed height of 12 feet. Consistent with the rest of the mass transfer equipment, this column is also filled with Goodloe high efficiency wire mesh packing. The column assembly and details are shown in figure 32. Liquid distribution within the still column is accomplished with a feed and reflux ring drilled so as

to spray liquid uniformly over the top surface of each packed section. The packing is supported by subway-type grating constructed out of 1/4-inch-thick by 1-inch-wide 304L stainless steel bar strips. This type of support promotes better gas distribution.

As shown in figure 33, the still reboiler is 10 inches in diameter and approximately 4 feet long. The reboiler is equipped with a Chromalox Model TMI-12303 30 kW immersion heater with Incaloy sheathing. Also regulated by a Loyola silicon-controlled rectifier (SCR) power controller, the heater can supply a surface heat flux of up to 22 watts per square inch. The system is protected from overheating with a Chromalox Model LFB7-7 adjustable differential temperature control. Reboiler liquid level control is achieved utilizing a Drexelbrook capacitance probe.

The overhead condenser is a shell and tube heat exchanger mounted 5 degrees to the horizontal. Cooling is provided on the tube side by boiling refrigerant-22. The condenser was designed on the basis of a condensing duty of 85,250 Btu/hr and an overall condensing coefficient of 50 Btu/hr-sq ft-°F. The condenser shell side surface area is 52 square feet. Construction details, as shown in figure 34, are very similar to that of the stripper primary condenser.

The solvent still assembly and all interconnecting piping have been approved for a maximum working pressure of 150 psig at 0°F and was hydrostatically tested at 225 psig.

Primary Process Auxiliaries

Certain support equipment items are a necessary part of the primary pilot plant to allow the process function. Included in this list is a process gas compressor, solvent pump, gas and liquid heat exchangers, and several storage and surge tanks.

Process Gas Compressor. A Pressure Products* diaphragm compressor system is used to compress the process gas to the operating pressure of the absorber. This package consists of two single-stage diaphragm compressors mounted on a common base and all necessary interstage and afterstage piping and heat exchange equipment. Figure 6 is a photograph of the system. The first stage compressor is a PPI Model 5235. This unit is powered by a 20-hp electric motor and is capable of compressing a nominal 20 scfm of air from atmospheric pressure to 135 psia. The second-stage compressor is a PPI Model 3120. This compressor is powered by a 10-hp motor and is designed to pickup a nominal 20 scfm of air at 110 psig and discharge it at a pressure as high as 575 psig. Both compressors incorporate a triple diaphragm

*Pressure Products Industries, Division of the Duriron Company, Incorporated, Hatboro, Pennsylvania.

Figure 6

PROCESS GAS COMPRESSOR

construction with an integral diaphragm leak detection circuit. As can be seen in figure 6, the compressor heads are bolted to their respective main frame and all process pipes are provided with unions to facilitate easy replacement of the compressor diaphragms. All surfaces in contact with the process gases, including diaphragms, are 300 series stainless steel except for nonmetal seals. These gaskets are fabricated from Teflon and Viton A.

The compressor final discharge pressure is regulated/controlled by an automatic gas recycle circuit that is shown on the operating flow sheet, figure 5, and the pilot plant piping drawing, figure 16. The flow from the compressor to the absorber is set manually by throttling a needle valve in the absorber feed gas line. Both compressor heads and interstage/afterstage heat exchangers are water-cooled. A Brooks* Model 1305-6221 flow alarm rotameter is mounted on the water supply to warn of an inadequate water flow.

Solvent Pump. Three solvent pumps have been used in the course of the development work. The first two were packless, diaphragm-type, positive displacement, controlled volume pumps with hydraulic linkage between drive and diaphragm. Inherent intermediate hydraulic problems encountered at the lower temperature operation necessitated the eventual installation of a Corken† Model F9-101 CORO-FLO regenerative pump. The impeller is the only moving part of this type of pump but a mechanical shaft seal is needed to isolate the process fluid. The unit is powered by an externally coupled 3-hp electric motor and has a discharge capacity in excess of 4 gpm at a maximum differential pressure of 150 psi. Two or more pumps are needed in series to obtain a higher differential pressure. An external pressure and flow control circuit was installed on the pump discharge for precise metering of the process solvent. A mechanical, automatic-priming and differential relief valve is provided in a parallel recycle line to aid in start-up and provide extra running protection.

Feed Gas Heat Exchanger. The feed gas heat exchanger is a standard design, single pass, shell-and-tube-type contactor constructed entirely out of 304L stainless steel. Details of the unit are given in figure 35 of Appendix A. The shell is fabricated from a nominal 6-inch, Schedule 40 pipe. The cooling/condensing surface consists of four 10-foot sections of 3/4-inch O. D. Brown** fintube with sixteen 1/2-inch-high longitudinal fins. The tubes have an external surface area of 1.529 ft²/ft of lineal length, or a total heat transfer surface area of 61 square feet. The heat exchanger is designed on the basis of a heat duty of 1700 Btu/hr and an overall heat

*Brooks Instrument Division, Emerson Electric Company, Hatfield, Pennsylvania.

†Corken Pump Company, Oklahoma City, Oklahoma.

**Brown Fintube Company, Tulsa, Oklahoma.

transfer coefficient of 0.79 Btu/hr-sq ft-°F. Four shell baffles are placed roughly 27 inches apart to assure intimate contacting of the process gas with the extended cold surfaces. The complete assembly is mounted 2-1/2 degrees from the horizontal to facilitate drainage of any condensed liquid into a collection pot. Details of the collection pot are given in figure 36. This container is also fabricated from a section of nominal 6-inch, Schedule 40 stainless steel pipe. The condensate level is monitored and controlled by a capacitance probe system similar to that described earlier. Equally spaced skin thermocouples have been placed one foot apart on the top of the cooler to measure the temperature profile during operation. Internal thermocouples have been installed in the feed and discharge lines to obtain process gas temperatures.

The feed gas heat exchanger has been approved for a maximum working pressure of 600 psig at -25°F and was hydrostatically tested at 900 psig before use.

Solvent Heat Exchanger. The solvent heat exchanger is a standard design shell-and-tube-type heat exchanger with floating head. Construction details are given in figure 37. This unit was designed on the basis of a 10,000 Btu/hr cooling duty and has a cooling surface of 13.5 square feet. The shell is constructed from a 7-foot-long piece of 4-inch, Schedule 10, 304L stainless steel tubes. The actual shell side surface area is 15.7 square feet. Shell baffles are provided to direct fluid flow. Cooling is achieved by expansion and superheat of refrigerant-22 in the exchanger tubes.

The solvent heat exchanger has a maximum working pressure of 350 psig and was hydrostatically tested to 525 psig.

Solvent Storage Tank. The solvent storage tank is constructed from a 1/2-inch-thick 304 stainless steel plate rolled and seam welded to the dimensions of a 16-inch Schedule 40 pipe. The rolled section is 4 feet long and is capped with 1/2-inch-thick 304L stainless steel pipe heads, resulting in a storage volume of 6 cubic feet. Details of the tank construction are given in figures 38 and 39. The tank stands vertically and has an overall height of 6.3 feet, including floor supports. The design cooling load on the coil is 5,000 Btu/hr. Based on an overall heat transfer coefficient of 50 Btu/hr-sq ft-°F, the required external surface area is 5 square feet. The level of solvent, hence the tank inventory, is monitored by a Drexelbrook Model 409 precision level indicator using a Drexelbrook 700-1-23 capacitance probe. The probe is Teflon-coated and has an insertion length of 4 feet. The solvent tank is also provided with a Strahman* Model SVT-4LCF frost-proof liquid level gauge. This gauge provides means for visual inspection of the liquid solvent.

*Strahman Valves, Inc., Florham Park, New Jersey.

The solvent tank has been approved for a maximum operating pressure of 350 psig at 0°F and was hydrostatically tested to 525 psig.

Miscellaneous. Three gas storage tanks are provided to give the pilot plant needed feed gas surge and storage capacity. One tank is part of the feed gas supply; one is installed in the suction line of the gas compressor; and the third is used at the compressor discharge. Figure 40 in Appendix A presents the details of these vessels. All are fabricated from 304L stainless steel. The pressure of each tank is monitored by both a locally mounted mechanical pressure gage and an electronic pressure transmitter that outputs to a panel-mounted recorder. The compressor suction tank has a pressure alarm to warn of low suction pressure, while all tanks are fitted with high pressure relief valves that connect to an outside vent system.

The pilot plant employs a number of different types and sizes of valves. Jamesbury* No. A33-TT 1/4, 1/2, 1, and 1-1/2 inch ball valves for liquid nitrogen service are used for process block valves. This particular valve has a MOP of 2000 psig and is constructed from stainless steel and has Teflon seats and seals. Hoke† 3200 Series, No. 3252F25 and 3252F45 miniature forged stainless steel needle valves and Nupro§ No. SS-4BK and SS-4BMG stainless steel bellows sealed valves are used on gas and liquid sample lines. Kunkle** Model 53 pressure relief valves with separate rupture disc cartridges are used throughout the plant to protect process equipment from overpressure. All pressure relief valves connect to an outside building vent system. With one exception, bellows sealed flow control valves are used exclusively to maintain desired system flows. Each valve has a 1/4- or 1/2-inch forged type 304 stainless steel body, 304 stainless trim, and 300 series stainless steel bellows. Precision Products†† control valves equipped with type 73 bottom load, air-to-open, fail-closed positioners are used on the gas circuits while Dover§§ Uniflow Series control valves equipped with conventional air-to-open topworks without positioner are used to maintain intercolumn solvent flow. A sliding gate Jordan*** control valve has been installed in parallel to the absorber primary liquid level control valve as a backup or reliability measure to ensure continued operation in case the primary valve should become plugged with ice or any other solid material.

*Jamesbury Corporation, Worcester, Massachusetts.

†Hoke, Inc., Cresskill, New Jersey.

§Nuclear Products Company, Cleveland Ohio.

**Kunkle Valve Company, Inc., Fort Wayne, Indiana.

††Precision Products Division, Badger Meter, Inc., Tulsa, Oklahoma.

§§Norris Division, Dover Corporation, Houston, Texas.

***Jordan Valve, Cincinnati, Ohio.

All process equipment operated below ambient temperature is insulated with 3-inch-thick urethane rigid-foam insulation. Interconnecting pipe and refrigeration lines support 2 inches of urethane insulation. The heat conductivity of this material is near 0.019 Btu/hr-ft-°F. Heat loss calculations show that expected ambient heat effects on the pilot plant are negligible compared to overall equipment heat loads.

Product Purification Equipment

The purpose of the product purification subsystem is to remove the accompanying solvent vapor and amounts of various nonradioactive diluent gases such as xenon, oxygen, argon, and nitrogen from the stripper off-gas and thereby isolate and further concentrate the captured krypton-85, carbon-14, and radon-222 in a yet smaller volume for long term storage. This equipment, as shown in figure 5, is located in the stripper off-gas line prior to the process radioactive product collection point. Several schemes incorporating a number of distinct separation and isolation steps are being investigated to identify the relative effects and advantages of the available options. With only a 13X molecular sieve trap to remove the solvent vapor, Kr-85 product concentration factors between 1,000 and 5,000 are obtainable unless large quantities of carbon dioxide are present in the system. In addition, by use of chemical traps for carbon dioxide, oxygen, and nitrogen fixation Kr-85 concentration factors approaching 10,000 can be expected. If a krypton/xenon separation step is also performed, concentration factors in excess of 10,000 are possible. The final choice of product purification equipment, of course, will have to be made on the basis of a cost-benefit analysis.

Figure 41 is a schematic of the pilot plant product purification subsystem designed to produce a relatively pure krypton-argon product, a $C^{14}CO_2$ product, and a xenon product. Figure 7 is a photograph of the equipment as it was being built. The stripper off-gas is first passed into a 13X molecular sieve trap for solvent recovery and then into a batch cold trap maintained at -250°F where all condensable and desublimable components such as carbon dioxide, nitrous oxide, xenon, and water are removed. The remaining gas composed of krypton, argon, oxygen and nitrogen is then passed into a heated copper or manganese oxide trap for oxygen removal and, finally, into a heated titanium or calcium metal trap for nitrogen removal. The resulting product composed of mainly argon and krypton is then pumped into a high pressure gas cylinder for storage. Once the cold trap is loaded, it is valved out of the flow circuit and heated. The desorbed gas is then passed into a 4A molecular sieve trap for carbon dioxide and nitrous oxide removal or hydrated lime reactor for carbon dioxide fixation. In this case the resulting gaseous product will be relatively pure xenon. If radon is present in the feed gas, it will be collected with the xenon. Alternatively, if the cold trap is maintained at -275°F, the krypton can be collected with the xenon instead of with the argon.

A schematic of the pilot plant desublimator is shown in figure 42 with necessary monitoring and control instrumentation described in table II of Appendix B. Figure 43 is an assembly, detail, and section drawing of the unit. The theory of desublimation is not commonly discussed in unit

Figure 7

PRODUCT PURIFICATION EQUIPMENT DURING CONSTRUCTION

operation textbooks but a comprehensive treatment is given in another ORGDP report [8]. An analysis of the pilot plant cold trap will soon be available in a program document [9]. Basically, the cold trap is a single pass shell-and-tube-type heat exchanger. The shell is fabricated from a nominal 2-inch, Schedule 80, 304L stainless steel pipe. A single 3/4-inch O.D. Brown fintube with sixteen 1/2-inch-high longitudinal fins provides the cooling surface. Boiling liquid nitrogen inside the tube provides the required low temperature. The trap is equipped with internal thermocouples every 6 inches along the length of the shell.

Reactive metal traps are widely used in industry to remove oxygen and nitrogen from various inert gases [11,44]. The oxygen trap is fabricated from a nominal 3-inch, Schedule 40, 316 stainless steel pipe, 45 inches long. Both ends are fitted with a 150-pound stainless steel flange. This trap is located in a 7500-watt HEVI DUTY* electric furnace fitted with a Lindberg* Model 59554 temperature controller. A hydrogen regeneration loop is provided to treat the spent copper metal. The nitrogen trap is fabricated from a nominal 1-inch, Schedule 40, 316 stainless steel pipe, 50 inches long. This trap is located in a 3900-watt, Lindberg Model 54371 muffle fitted with a Lindberg Model 59544 temperature controller. Both reactive metal traps have been hydrostatically tested at 100 psig and are approved for a maximum working pressure of 67 psig.

The design of the 13X molecular sieve trap for refrigerant-12 removal is identical to that used for oxygen removal except the 13X trap is only 36 inches long. It is necessary to water-cool the molecular sieve bed because of the large heat of adsorption. Removal tests using a feed gas stream containing 10 mole percent R-12 have shown that loadings near 0.3 lb R-12/lb sieve, and an effluent concentration of less than 1 ppm are obtainable using 13X molecular sieves [36].

Solvent Recovery Traps

Refrigerant-12 has an appreciable vapor pressure at the temperature of the absorber and, consequently, the process off-gas normally contains a significant amount of process solvent. The loss of solvent to the atmosphere is objectionable in a number of ways and cannot generally be tolerated. In any event, development of an effective and economical solvent recovery subsystem is an important part of the overall fluorocarbon process development program. A fluorocarbon discharge level of 1 ppm or less was established arbitrarily as an acceptable performance standard for evaluating various recovery methods. Two recovery schemes are being considered here: low temperature condensation and physical adsorption. In order for a solvent condenser to be effective, it would have to be operated at a temperature below -240°F in order to meet the 1 ppm off-gas specification. Cooling could be achieved with liquid nitrogen.

*Lindberg, Division of Sola Basic Industries, Watertown, Wisconsin.

Alternatively, and perhaps more appealing, the absorber energy of compression could be utilized via an expansion turbine. Devices are currently available that permit the expansion-cooling operation on condensing streams directly, without the use of an intermediate condenser*. Pilot plant testing of an expansion turbine is not planned because it is felt that the technology is well defined and equipment is commercially available. Regenerable solid sorbents are widely used in the chemical industry to recover valuable solvent vapors and clean up various off gas streams. Activated carbon is known to be an effective absorbent for many organic vapors, including refrigerant-12 but is excluded from this application because of the possibility of contact with nitrogen dioxide. Other materials, such as silica gel, molecular sieves, and alumina, hold promise.

In order to evaluate the various sorbent materials for removing refrigerant-12 from the process off-gas, a sorption system was built and installed on the absorber off-gas line. The traps are designed to accommodate many different types of materials and have several regeneration options. Pressure swing, steam, forced air heating, and vacuum regenerating capabilities are provided. A flow schematic of this equipment is given in figure 44. Each trap is constructed from a 4-foot piece of 3-inch, Schedule 40, stainless steel pipe and is flanged to facilitate adsorbent removal. Stainless steel wire screen backed with 1/8-inch-thick perforated stainless steel sheet is used for bed supports. A Miran II infrared analyzer[†] provides in-line analytical capability through inlet and outlet gas sample taps located on each trap. Separate adsorbent trap temperature, pressure, or flow control instrumentation is not necessary for the trap operation because these parameters are established according to the conditions of the absorber. Some separate controls are necessary for the regeneration cycle, however, but must be specified according to the requirements of the particular adsorbent.

Feed Gas Preparation

The feed gas preparation station consists primarily of precision gas flow metering and monitoring instrumentation. Hastings[§] thermal mass flow controllers are used to maintain desired individual feed gas component flows. Each flow control point consists of a Hastings Model FC-1P flow controller with a Model MV-10 motorized valve. The valve is constructed of 316 stainless steel with Teflon packing. Each component flow is displayed on a digital indicator. Two Cole-Parmer** Model 1061 constant temperature electrically heated water baths are used to volatilize high boiling components such as iodine and nitrogen dioxide and a metered nitrogen

*Rotoflow Corporation, Los Angeles, California.

[†]Wilks Scientific Corporation, South Norwalk, Connecticut.

[§]Teledyne Hastings-Raydist, Hampton, Virginia.

**Cole-Parmer, Chicago, Illinois.

entrainment flow is employed to transport the volatilized gas from their respective storage cylinders to one of several pilot plant gas feed points. Water may be introduced into the system by means of a Cole-Parmer Model 7606 Hot Shot electric steam generator or by bubbling a nitrogen pickup flow through a water saturator.

Instrumentation and Control

In order to evaluate process components, it is absolutely essential that tight process control be achievable so that the various parametric dependencies can be accurately determined. Consequently, a considerable effort has been spent developing the pilot plant control capability. Sufficient control is now available to make the pilot plant operation essentially automatic with little or no operator attention except during startup. The remaining step at this time is conversion of the operation to computer control. The pilot plant instrumentation and various control schemes are shown in figure 5, with a description of the individual components given in Appendix B, table I. Figure 8 is a photograph of the pilot plant control panel.

All process pressures are controlled with Taylor* 1300 series electronic differential pressure transmitters and indicating controllers. In most instances, the output signal from the controller is fed to a Moore† Model 77-16 I/P transducer which in turn actuates either a pneumatic-operated gas flow control valve or, in the case of the solvent still, a refrigeration evaporation pressure control element. The stripper pressure, on the other hand, is maintained by a Loyola SCR power controller which, in turn, establishes the reboiler heat load. In order to have the necessary pressure control flexibility and sensitivity, the low side of each differential pressure transmitter is coupled to a variable pressure datum system. The reference pressure is set and maintained for each individual transmitter with a Grove‡ Model 15L pressure reducing and relief regulator which is fed from a common bottled gas supply header. Individual datum pressures are indicated on locally mounted precision mechanical pressure gauges. Pressure drops across column packing are monitored with low range Taylor differential pressure transmitters. On two of the columns, the pressure drop signal is sent only to a recorder. However, the pressure drop signal of the fractionator is used to control the reboiler heat input, while that of the stripper is used to control the overhead condenser refrigeration load. The fractionator differential pressure control loop is a key control point for optimum process operations.

Liquid level control is accomplished in all cases with a Drexelbrook liquid level monitoring system as previously described. A Model 770-1-23

*Taylor Instrument, Process Control Division, Sybron Corp., Rochester, New York.

†Moore Products Company, Spring House, Pennsylvania.

‡Grove Valve and Regulator Company, Oakland, California.

PHOTO NO. PH-77-2980

Figure 8

PILOT PLANT PANEL BOARD

capacitance probe is coupled with a Model 408-1000 transmitter that outputs a 4-20 mA signal to a Taylor controller. In most cases, the controller sends out a control signal to an I/P transducer which, in turn, operates a corresponding liquid flow control valve. The only exception to this scheme is the solvent still reboiler liquid level control scheme which sends the controller signal to a Loyola SCR power controller that maintains the still heat.

Thermal mass flow meters are used to monitor process gas flows. This type of flow meter is affected by fluid temperature and pressure only as the heat capacity of the process gas is dependent upon these parameters. Hastings* Model AHL-25 flow meters with panel readouts measure gas flow to the process, gas flow to the absorber, and gas flow leaving the absorber. Each of these instruments utilizes a Monel laminar flow cell and has a flow range of 0-25 scfm. A Hastings Model AHL-10PG flow meter with a range of 0-10 scfm is used to measure gas flow from the fractionator. Hastings Models ALL-5KPG and ALL-50KPG are used in a parallel arrangement to measure either low or high flow rates from the stripper. These instruments have a range of 0-5,000 sccm and 0-50,000 sccm, respectively.

The process solvent flow to the absorber is controlled using a Brooks[†] Model 3623/5522H transmitting flow meter and an indicating controller coupled to an I/P transducer and a liquid control valve located in the solvent pump discharge line as shown in the process operating flow sheet, figure 5. The process solvent flow rate is recorded on a Leeds and Northrup[§] Speedomax W/L recorder. The flow control system also includes a pressure control loop with integral pump discharge recycle valve. The system for controlling the solvent still product and reflux flow is somewhat more complex. Here, it is necessary to split out a specific fraction of the still condenser distillate stream for column reflux. The distillate and reflux flow rates are measured either by separate Brooks transmitting flow meters or by Wallace and Tiernan** glass-tube-type rotameters. The two transmitting flow meter signals feed into a Taylor series 1330 multiplier-divider network which calculates the operating reflux-to-distillate flow rate ratio. This ratio is used in a liquid flow control loop to maintain a specified still reflux rate relative to a given distillate flow. Each of the three Brooks liquid flow meters has a 0-2.5 gpm flow rate range and is approved for operating pressures as high as 600 psig. The solvent still bottoms product is withdrawn according to the temperature of the reboiler liquid. A Taylor series 1022 E/I thermocouple transducer is used to provide the liquid valve control loop signal.

*Teledyne Hastings-Raydist, Hampton, Virginia.

†Brooks Instrument Division, Emerson Electric Company, Hatfield, Pennsylvania.

§Leeds and Northrup Company, North Wales, Pennsylvania.

**Wallace and Tiernan, Inc., Industrial Products Div., Belleville, New Jersey.

Reboiler heat loads are measured to within 0.1 percent of full scale using Westinghouse* type PXA-10 potential transformers and type CTM-5 current transformers and a Scientific Columbus[†] Digilogic Model Number DL31K5-A2 watt transducer. Each watt transducer outputs a proportional 0-10 volt dc signal to a panel mounted Digitec[§] Model 2780-03 digital dc voltmeter.

Copper-constantan thermocouples are used throughout the pilot plant to measure process temperatures. Relative placement of thermocouples is shown on the operating flow sheet, figure 5. Temperatures are recorded on three Kaye** Digistrip digital recorders and two L & N^{††} Speedomax W/L recorders. Similar Kaye digital voltmeter recorders are utilized to record various system pressures, gas and liquid flow rates, and reboiler heat loads. These recorders provide hard copies of important process parameters on a selectable time interval.

PROCESS REFRIGERATION

Low temperature refrigeration equipment is required to enable process operation at preferred conditions. The pilot plant employs several mechanical evaporative-type refrigeration systems capable of delivering a total of approximately 12 tons of cooling at minus 40°F. This particular arrangement has proven acceptable for the pilot plant work but the overall long-term reliability is questionable for the reprocessing plant application where part or all of the heat exchangers or other parts of the refrigeration circuit might be located in a remote or semiremote maintenance area. For this reason, a recirculating "brine" refrigeration system is being considered for pilot plant evaluation and will probably be recommended for use in a demonstration facility.

Evaporative-Type Refrigeration System

A schematic of the existing pilot plant refrigeration system is shown in figure 45 along with a tabulation of all pilot plant refrigeration expansion valves, heat exchanger backpressure regulators, and other refrigeration parts. Expansion valves and pressure regulators are essential refrigeration control elements [2]. The function of the expansion valve is to keep the particular evaporator nearly full of liquid refrigerant without, at the same time, allowing liquid to return back to the compressor. Too little refrigerant flow will reduce the heat exchanger capacity. On the other hand, compressor damage can result if liquid refrigerant is allowed to flow back to the compressor in the vapor return line. The expansion valves have to be approximately sized for the particular heat load and delivery temperature range. Sporlan^{§§} refrigeration expansion valves are

*Westinghouse Electric Corp., Pittsburgh, Pennsylvania.

[†]Scientific Columbus, Inc., Div. of Esterline Corp., Columbus, Ohio.

[§]United Systems Corporation, Subsidiary of Monsanto, Dayton, Ohio.

**Kaye Instruments, Bedford, Massachusetts.

^{††}Leeds and Northrup Company.

^{§§}Sporlan Valve Company, Saint Louis, Missouri.

used throughout the plant. Alco* Series 722 evaporator pressure regulators are used to control the heat exchanger temperature by maintaining a corresponding refrigerant boiling pressure. The bulk of the pilot plant cooling is accomplished with three refrigerant-22 systems. Each refrigerant-22 compressor-condenser package consists of a Copelametic[†] Model 6RB2-1000 compressor with 10 hp motor and W2WV-1000, two-stage, water-cooled condensing unit and has a capacity of 60,000 Btu/hr at minus 25°F or 42,500 Btu/hr at minus 40°F. The stripper final condenser and the still bottoms product receiver tank are cooled with separate refrigerant-502 systems. Each compressor-condenser package consists of a Copelametic Model NRD1-0310 compressor with a 3-hp motor and W4WL-0300, two-stage, water-cooled condensing unit. Varying process heat loads can result in liquid floodback to the compressor and, consequently, each refrigeration system is protected with a heated suction line liquid accumulator.

Brine-Type Refrigeration System

A brine refrigeration system differs from an evaporative type in that an intermediate heat transfer fluid is used to cool the process equipment instead of a direct boiling or evaporative transfer. Basically, this set-up consists of a refrigerated heat transfer liquid storage tank and cold liquid recirculating pump. The heat transfer fluid can be any liquid with suitable flow and heat transfer characteristics. Local expansion valves and evaporator pressure regulators are not required although a remote flow control element is. In the case of the demonstration facility the brine system would permit location of all mechanical equipment and, equally important, control elements outside the reprocessing plant cell containment. Depending upon the total system load one or more larger refrigeration compressors can be used to cool the brine storage tank. For improved reliability, one or more spare compressors can also be integrated into the system to take over the cooling load in the event one of the regular units fails. In the unlikely event the total mechanical refrigeration system is incapacitated, the thermal inertia of the tank could provide a substantial amount of cooling for a limited duration. If necessary, emergency cooling could be provided via an external liquid nitrogen or compressed carbon dioxide supply. A brine system is being designed for pilot plant testing.

PROCESS ANALYTICAL

The pilot plant has a well-developed process sampling and in-house analytical capability that allows direct determination of component and overall process performance. The sampling system is connected to over 25 different process points and any one of several analytical instruments. Alternatively, gas samples can also be pulled from the process and placed into cylinders

*Alco Controls, Div. of Emerson Electric Co., St. Louis, Missouri.

†Copeland Refrigeration Corporation, Sidney, Ohio.

for mass spectrometer and other laboratory analysis. Gamma scintillation equipment, including a multichannel and several single channel pulse height analyzers, is used for in-line analysis of gas and liquid streams containing gamma emitting isotopes such as Kr-85, Xe-133, Rn-222, and I-131. Additionally, external gamma scanning devices are also available to determine packed column molecular sieve, and cold trap concentration profiles. Figure 9 is a photograph of the sampling manifold and part of the analytical equipment. The sample lines and manifold are constructed from stainless steel. Nupro* Model SS-4BK bellows sealed block valves are used on each of the manifold feed circuits. The manifold is equipped with a precision metering valve, rotameter, sample and manifold pressure gauges, and a vacuum pump connection. Figure 10 is a flow schematic of the gas manifold system.

Gas Analysis

Four different analytical methods are employed locally to determine various component concentrations. Single beam Miran II[†] infrared analyzers are used to obtain carbon dioxide and refrigerant-12 concentrations. The carbon dioxide analyzer operates on a measuring wavelength of 2.77 μ and a reference wavelength of 3.58 μ and has a detection limit of 2 ppm. The refrigerant-12 analyzer operates on an analytical wavelength of 9.09 μ and a reference wavelength of 3.58 μ and has a detection limit of 0.1 ppm. A Miran I infrared analyzer with a variable analytical wavelength is also available to perform other component analysis. All three infrared instruments are fitted with a variable 20 meter path length cell which makes it possible to obtain both low and high concentration levels.

A DuPont[§] Model 411 photometric analyzer is used to determine nitrogen dioxide and nitric oxide process concentrations. This analyzer operates in the visible light region for NO₂ detection, with a measuring wavelength of 436 nm and a reference wavelength of 578 nm, and has a 2 ppm detection limit. The gas cell is 20 inches long. Nitric oxide has no absorbance in the visible range and consequently must be converted to NO₂ prior to the determination. The analyzer has provisions for conducting this reaction inside the gas cell.

Two Panametrics** Model 2000 dew point hydrometers are used to establish gas and liquid stream water contents. Ten aluminum oxide sensors are located throughout the pilot plant. These particular sensors are capable of measuring water vapor dew/frost points of -110 to +20°C over an operating pressure of 5 microns to 5000 psig. This range represents a moisture level of between 0.001 ppm and 20,000 ppm at atmospheric pressure.

*Nuclear Products Company, Cleveland, Ohio.

†Wilks Scientific Corporation, South Norwalk, Connecticut.

§E. I. duPont de Nemours, Wilmington, Delaware.

**Panametrics, Inc., Waltham, Massachusetts.

Figure 9

PILOT PLANT SAMPLING MANIFOLD AND ANALYTICAL EQUIPMENT

Figure 10

SCHEMATIC OF GAS SAMPLING MANIFOLD

Nitrogen, krypton, xenon, carbon dioxide, oxygen, argon, and refrigerant-12 concentrations are determined with a Honeywell* Model 1000 process gas chromatograph. This analyzer has an analysis cycle time of 170 seconds. Composition ranges are as follows: 1) nitrogen, 0-50 percent; 2) krypton, 0-50 percent; 3) xenon, 0-100 percent; 4) carbon dioxide, 0-100 percent; 5) oxygen and argon combined, 0-100 percent; and 6) refrigerant-12, 0-1000 ppm.

Gamma Analysis

Two single channel pulse height analysis systems are used to simultaneously determine the isotopic content of streams entering and leaving various mass transfer equipment. Each system consists of the following ORTEC[†] components: Model 905-3 scintillation detector and photomultiplier tube; Model 266 photomultiplier tube base; Model 113 scintillation preamplifier; Model 452 spectroscopy amplifier; Model 420A timing single channel analyzer; Model 776 timer and counter; Model 775 counter, Model 441 ratemeter; and Model 401A/402A bin and power supply. A Hewlett-Packard[§] Model 6516A high voltage power supply and L and N Speedomax W/L pen recorder are also used. Multicomponent spectral analysis is performed with an ULTIMA II** multichannel analyzer. This computer based system has 1024 channels, a four input mixer/router, and 200 MHz analog-to-digital converter. Spectrum data are displayed on a CRT and thermal printer.

Process gamma counting is performed directly, through the process pipe wall. The location of the available count positions is given on the operating flow sheet, figure 5. Some fifteen positions are available. The process pipe at each counting point is encased in a 1-1/2- to 2-inch-thick lead shield to reduce background radiation to acceptable levels. A hole is bored in one end of each shield to receive the scintillator detector, which is positioned directly against the process pipe. For those cases where the pipe is cold, a small piece of urethane foam insulation is placed against the pipe to protect the sodium iodide crystal. In addition to the stationary shields, several mobile lead shields have been installed in various process locations to obtain the concentration profiles of various pieces of equipment. For column analysis overhead hoists are used to position the devices mounted on vertical tracks parallel to the column faces. Figure 11 is a photograph of the fractionator column scanner. Motor-driven horizontal scanners are used to obtain concentration profiles of the feed gas heat exchanger and the product purification cold trap. The data obtained from the scanners so far have been instrumental to the understanding of the process.

*Honeywell, Inc., Memphis, Tennessee.

†ORTEC, Inc., Oak Ridge, Tennessee.

§Hewlett-Packard, Rockaway, New Jersey.

**INO-TECH, Inc., Madison, Wisconsin.

Figure 11

FRACTIONATOR COLUMN GAMMA SCANNER

FLOW SHEET OPTIONS

It is important to note that the pilot plant is designed to be a versatile development facility that allows investigation of the overall process as well as individual component behavior and performance. Consequently, it should be obvious that the plant is built in a manner and with a number of special features that are not particularly desirable nor well suited to an industrial-type or remote operation. As indicated earlier, part of the overall development effort is, consequently, directed toward identification of flow sheet simplifications that will lead to a more tractable and, equally important, reliable system for the actual reprocessing plant application. Two such options have been identified so far and are presented here so as to indicate, in particular, how the seemingly complex fluorocarbon process might finally be applied. The application flow sheets are well founded upon several thousand hours of pilot plant operation, detailed performance data, and rigorous component modeling studies. From an economic point of view, both of the flow sheet simplifications represent substantial process improvements.

Combination Absorber/Fractionator

Figure 12 is a schematic of the selective absorption process based on a combination absorber/fractionator column. This particular combination has been employed in at least one other application [23]. Of course, a degree of freedom has to be sacrificed with this particular flow sheet: the fractionator pressure and, hence, the reboiler temperature are fixed by the design of the absorber. If the required absorber pressure is high, the consequences of a high reboiler temperature have to be acceptable. Recent pilot plant tests, however, have shown that lower absorber pressures, e.g., 100 to 150 psig, are generally allowable in so far as the performance of the overall process is concerned [20,21]. Operation of the fractionator in this pressure range results in a reboiler temperature of only 90 to 120°F.

The operational and economical advantages of the flow sheet based on the combination absorber/fractionator do look attractive for the reprocessing plant application. First, there will be no external fractionator gas recycle to contend with; which means that the process gas compressor requirements will be substantially reduced. This is particularly true at the lower absorber pressures favored by the absorber/fractionator combination. Also, recycle solvent vapor will not be added to the incoming process gas prior to cold trapping. Consequently, the nitrogen dioxide, water, and iodine removed in the feed gas cooler will be free from cross-contamination making it possible for the nitrogen dioxide and water to be recycled back to the reprocessing plant nitric acid makeup system. Secondly, the fractionator flash unit and condenser can be eliminated. This reduction will lower process equipment costs. Thirdly, the number of process flow controls can be significantly reduced and the reliability of the resulting process improved. Specifically, the modified flow sheet does not require fractionator off-gas instrumentation and controls, condenser refrigeration controls, or an absorber liquid level system. From an operational standpoint, elimination of the liquid control valve between the

Figure 12

SELECTIVE ABSORPTION PROCESS BASED ON COMBINATION
ABSORBER/FRACTIONATOR

absorber and fractionator is desirable since this is the valve that historically tends to plug when large amounts of free water are introduced into the system. Finally, the modified process will require less space.

Combination Absorber/Fractionator/Stripper

Figure 13 is a schematic of an even more simplified process based on a combination absorber/fractionator/stripper. While somewhat radical, this flow sheet is well founded upon existing pilot plant data [22,38]. Unlike its predecessor, the new process contains only a single packed column within which to conduct the necessary process functions of absorption, fractionation, and stripping. A solvent purification still is needed if the feed gas contains significant amounts of high boiling components such as water, nitrogen dioxide, and iodine. Decontaminated off-gas flows from the top of the combination column and regenerated solvent from the bottom while the radioactive gases are collected as a side stream. The top part of the column, i.e., that part above the feed gas point, is the absorption section of the process; the middle section, i.e., that part of the column between the product takeoff and the gas feed point, serves as the fractionator; and the bottom section including the reboiler makes up the stripper. Figure 14 shows a possible instrumentation scheme that might be employed for column control. Like the conventional process, the individual sections of the modified plant operate at different solvent-to-gas flow rate ratios. In fact, in the absence of a pressure gradient, the modified process relies even more heavily upon varying L/G ratios to achieve the three regime operation. The dependency of the equilibrium distribution coefficient with temperature also contributes to the success of the combined operation. An in-line column condenser can be added as part of the combination column between the stripper and fractionator zones as shown in figure 14 to give the process a greater operating flexibility.

This process flow sheet requires even less equipment and control instrumentation than the version based on a combination absorber/fractionator. Because it is a simpler process, it will be even more economical, easier to control, and more reliable.

DEVELOPMENT PROGRAM SCHEDULE

As previously mentioned, the primary objective of the fluorocarbon development program is the generation of all process technology required to complete a comprehensive design and evaluation of a demonstration facility applicable to both conventional and advanced reactor fuel reprocessing plants. A substantial amount of data has already been obtained upon which to base a preliminary process design; but a sizable amount of work still needs to be accomplished before the final design of an off-gas system can be made. The remaining development program is laid out in such a way as to guarantee the timely evolution of the technology consistent with the needs of the various fuel recycle programs.

The major experimental effort of FY 1978 will be directed toward detailed analysis of the pilot plant while simultaneously treating all reprocessing

Figure 13

SELECTIVE ABSORPTION PROCESS BASED ON COMBINATION
ABSORBER/FRACTIONATOR/STRIPPER

DWG. NO. G-77-243
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Figure 14

INSTRUMENTATION SCHEMATIC FOR THE COMBINATION
ABSORBER/FRACTIONATOR/STRIPPER COLUMN

plant off-gas components. In particular, component interactions and multicomponent effects will be evaluated. The capability and performance of the feed gas desublimers and krypton-85/carbon-14 product purification and isolation equipment will be further defined. Methods of dealing with the feed gas desublimates and distillation column bottoms product will be evaluated. Specifically, processing schemes that convert the solution iodine to a nonvolatile solid such as barium iodate and recycle the captured nitrogen dioxide as nitric acid will be pursued. A feed gas catalytic oxidizer will be considered for converting other possible carbon forms such as CO or CH₄ to the preferred dioxide to guarantee a high carbon-14 collection efficiency. Finally, if the additional data gathered continues to support the combination absorber/fractionator or absorber/fractionator/stripper concept another column will be built capable of performing as either the two or three function contacting device and installed in the pilot plant as an option to the existing three column plus solvent still arrangement. In this way, any one of the three flow sheets can be selected for testing simply by valving the simulated feed gas and solvent flow to the appropriate equipment. Process modeling and optimization studies will be continued into FY 1978. Design correlations will be completed for all major process components and rigorous component models derived from pilot plant data will be incorporated into a process model. Flow sheet and equipment modifications and options will be examined in the context of further improving the process efficiency, economics, and reliability for the reprocessing plant application.

It is of interest to point out that at the same time, the solvent chemistry work will be exploring multicomponent solution interactions that might affect the process operability or performance. Solubility limits and solution behavior of all less volatile components such as water, nitrogen dioxide, and iodine will be established and the corrosion characteristics of the system will be better defined. All of the existing phase distribution data for oxygen, nitrogen, and the noble gases in refrigerant-12 were obtained at system temperatures below 35°F. Currently preferred process operating conditions require distribution coefficients at temperatures as high as 100°F. A laboratory effort will also be conducted to determine the light gas solution behavior at higher temperatures. Also, the effects of small quantities, e.g., 1 to 2 percent, of other fluorocarbons, such as refrigerants-113, -114, and -11, on the activity coefficients of the various feed gas components in solution will be examined.

Realizing that system perturbations can occur in actual process application, the effects of various system disturbances on pilot plant operability and performance will be identified in FY 1979. Remedial actions will be formulated in response to various disturbances and component failures, if necessary, to recover plant operation. Remaining work with minor feed gas components such as ruthenium and various organics will be performed. Advanced process control and monitoring schemes not previously considered will be evaluated. Also, final evaluation of the product purification and isolation methods will be made. The final process flow sheet will be decided this year. This decision constitutes a critical milestone in the Process Application Subtask. The design process model will also be completed and optimization techniques will be applied to determine preferred

operating conditions. The reliability analysis model will be modified to reflect the final process flow sheet and those components identified that require redundant or backup elements in order to ensure various levels of process availability. The detailed laboratory analysis of the system solvent chemistry will be continued to identify remaining multicomponent effects. The chemistry program will consider the behavior of remaining feed gas components. Tests designed to verify the integrity of the process solvent will be initiated. In particular, solvent decomposition mechanisms due to radiation, thermal, and chemical forces will be investigated. The preferred materials of construction of the demonstration facility will be identified at the end of FY 1979.

Optimum operating conditions and process reliability will be emphasized in FY 1980 and special application tests, if necessary, will be completed by FY 1981 to answer any remaining questions that were not addressed earlier or simply were not identified until late in the program. The major solvent chemistry program of the R-12 system will be concluded in FY 1980 unless other problems not previously identified become obvious by that time. Process application work will be focused on the demonstration process. Efficient process startup, operating, and shutdown methods and procedures will be formulated that are specifically applicable to the particular fuel recycle facility.

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APPENDIX A

PILOT PLANT LAYOUT AND EQUIPMENT DRAWINGS

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Figure 15
PILOT PLANT PIPING - ELEVATION

Figure 16.

PILOT PLANT PIPING - FLOOR PLAN AND SECTION

Figure 17

PILOT PLANT PIPING - PLATFORM FLOOR PLAN

Figure 18

ABSORBER COLUMN - ASSEMBLY

Figure 19

ABSORBER COLUMN - DETAILS

Figure 20

FRACTIONATOR AND FLASH CHAMBER - ASSEMBLY

COLUMN HEAD SECTION
SCALE: FULL SIZE
SEE DWG. CD-919-E

FLASH CHAMBER
SCALE: 3/8" = 1"
SEE DWG. CD-319-E

DETAIL "B"
SCALE: FULL

NOTES:
1-SEE GENERAL NOTES ON DWG. CD-319-E

FRACTIONATOR AND FLASH CHAMBER ASSEMBLY		CD-319
REFERENCE DRAWINGS		NUMBERS
GAS HEAT NATURAL LABYRINTH OPERATED BY UNION CARBIDE CORPORATION ONE RIDGE, TENNESSEE		
K25 K ₂ -K ₆ REMOVAL PLANT		BUILD NO. K-141
FRACTIONATOR AND FLASH CHAMBER DETAILS		
APPROVED BY DATE	SUBMITTED DATE	RECEIVED DATE
	W. H. H. 12106	CD 320
DATE		1964.5.18

FRACTIONATOR AND FLASH CHAMBER - DETAILS

Figure 22

FRACTIONATOR AND STRIPPER REBOILERS - ASSEMBLY

NOZZLE SCHEDULE		
NOZ	SIZE	SERVICE
A	2" NPS	GAS OUTLET
B	2"	GAS INLET
C	1"	LIQUID OUTLET
D	3"	GAS INLET
E	1/2" BUBE	REFRIGERANT INLET
F	1"	REFRIGERANT INLET
G	1"	REFRIGERANT OUTLET
H	1"	REFRIGERANT OUTLET

NOTES:

- 1- MAXIMUM ALLOWABLE WORKING PRESSURE 300 PSIG AT 6" OUTSIDE TUBES, 200 PSIG, 0°F INSIDE TUBES
- 2- HYDROSTATIC TEST AT 525 PSIG. OUTSIDE TUBES, 300 PSIG INSIDE.
- 3- INSPECT AWC TEST IN ACCORDANCE WITH UCM W-50, SCHEDULE 4 AND PT (UGH W-1)
- 4- WELD IN ACCORDANCE WITH UCM W-502.
- 5- CERTIFICATION OF MATERIAL IS NOT REQUIRED, HOWEVER ONLY PROPERTY STAMPER (30AL) STEELS MATERIAL SHALL BE USED.

△ - INSIDE WALL (30AL 35) FILL RINGS INSIDE FULL LENGTH OF TUBES (1'-0" to 3'-0") FOR THE PURPOSE OF PREVENTING TUBES FROM COLLAPSE UNDER VACUUM. (SEE DRAWING 108-100-7) TO

FRACTIONATOR COND. DETAIL SHEET				CD-3241	
REFERENCE DRAWINGS				NUMBER	
<p>MAX REESE NATIONAL LABORATORY OPERATED BY UNION CARBIDE CORPORATION ONE MOORE, TENNESSEE</p>					
K-25 K-X Removal Pilot Plant				BATCH NO. K-141	
FRACTIONATOR CONDENSER. ASSEMBLY					
DATE	INITIALED	ACCEPTED	REASON	REMARKS	
DATE	M	12106	CD	323	8

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TOLERANCES UNLESS OTHERWISE SPECIFIED:		2 Added Nov. 20th & Nov. 25th		2-10-71		10-11-71	
FRACTIONS ▲		1 As BUILT		DATE		APPROVED	
DECIMALS ▲		NO. REVISIONS		DATE		APPROVED	
ANGLES ▲		DRAWN BY		DATE		APPROVED	
SCALE		DATE		DATE		APPROVED	
		SUBMITTED		DATE		APPROVED	
		APPROVED		DATE		APPROVED	
		APPROVED		DATE		APPROVED	

FRACTIONATOR CONDENSER - ASSEMBLY

Figure 25

FRACTIONATOR CONDENSER - DETAILS

Figure 28

STRIPPER PRIMARY CONDENSER - DETAIL SHEET 1

Figure 29

STRIPPER PRIMARY CONDENSER - DETAIL SHEET 2

Figure 30

STRIPPER FINAL CONDENSER - ASSEMBLY

Figure 31

STRIPPER FINAL CONDENSER - DETAILS

Figure 32

SOLVENT PURIFICATION STILL COLUMN - ASSEMBLY AND DETAILS

Figure 33

SOLVENT STILL REBOILER - ASSEMBLY AND DETAILS

Figure 34

SOLVENT STILL CONDENSER - ASSEMBLY AND DETAILS

Figure 36

GAS HEAT EXCHANGER COLLECTION POT/SOLVENT STILL
CONDENSER SURGE POT

Figure 37

SOLVENT COOLER - ASSEMBLY AND DETAILS

Figure 38

SOLVENT TANK - ASSEMBLY

Figure 39

SOLVENT TANK - DETAILS

FINAL COMPRESSED GAS RESERVOIR

RECYCLE GAS RESERVOIR

Δ

FEED GAS RESERVOIR

A

1. INSPECT AND TEST IN ACCORDANCE WITH UCN W-50 SCHEDULE V AND PT (UCN W-51).
2. WELD IN ACCORDANCE WITH UCN W-302.
3. CERTIFICATION OF MATERIAL IS NOT REQUIRED, HOWEVER ONLY PROPERLY STAMPED (304L) STORES MATERIAL SHOULD BE USED.

FEED AND RECYCLE GAS RESERVOIRS
1- MAXIMUM ALLOWABLE WORKING PRESSURE 350 PSIG
AT 0°F.
2- HYDROSTATIC TEST AT 525 PSIG.

FEED, RECYCLE AND FINAL COMPRESSED GAS RESERVOIRS - DETAILS

TOLERANCES UNLESS OTHERWISE SPECIFIED

FRACTIONS	±	$\frac{1}{64}$
DECIMALS	±	0.005
ANGLES	±	3'

SCALE: $\frac{1}{4}'' = 1' - 0$

NO.	REVISIONS		
DRAWN	DATE	SUBMITTED	DATE

Figure 40

PROCESS GAS STORAGE TANKS

C

- B**

1c

B

F-D-12106-CD-476

LA

T

PRODUCT SEPARATION/PURIFICATION SUBSYSTEM SCHEMATIC

Figure 42

PRODUCT PURIFICATION COLD TRAP SCHEMATIC
AND INSTRUMENTATION

Figure 43

PRODUCT PURIFICATION COLD TRAP - ASSEMBLY,
 DETAILS, AND SECTIONS

Figure 44

SOLVENT RECOVERY EQUIPMENT OPERATING FLOW SHEET

Figure 45

SCHEMATIC OF THE REFRIGERATION SYSTEM

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APPENDIX B

KEY TO INSTRUMENTATION

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TABLE I

INSTRUMENTATION KEY TO OPERATING FLOW SHEET

Symbol	Service	Description	Manufacturer	Model	Input Range	Output Range
PRC-CS	Compressor Suction Pressure Control	Pressure Transmitter	Taylor	1304TD11120	200-800 in. H ₂ O	4-20 mA
		Indicating Controller	Taylor	1304RA10002	0.25-1.25 v	4-20 mA
		I/P Transducer	Moore	77-16	4-20 mA	3-15 psi
PCV-CS	Compressor Suction Pressure Control	Control Valve	Precision Products	$\frac{1}{2}$ in., 0.45 C _v	3-15 psi	Mechanical
PRC-CD	Compressor Discharge Pressure Control	Pressure Transmitter	Taylor	1333TF21221	50-500 psig	4-20 mA
		Indicating Controller	Taylor	1304RA10002	0.25-1.25 v	4-20 mA
		I/P Transducer	Moore	77-16	4-20 mA	3-15 psi
PCV-CD	Compressor Discharge Pressure Control	Control Valve	Precision Products	$\frac{1}{4}$ in., 0.030 C _v	3-15 psi	Mechanical
PR-GR	Gas Reservoir Pressure	Pressure Transmitter	Leeds and Northrup		0-300 psig	0.25-1.25 v
		Recorder	Taylor	1301JA10002	0.25-1.25 v	0-100 div
PRC-PD	Pump Discharge Pressure Control	Pressure Transmitter	Taylor	1333TF11221	50-500 psi	4-20 mA
		Indicating Controller	Taylor	1304RA10002	0.25-1.25 v	4-20 mA
		I/P Transducer	Moore	77-16	4-20 mA	3-15 psi
PCV-PD	Pump Discharge Pressure Control	Control Valve	Dover	$\frac{1}{2}$ in., 0.80 C _v	3-15 psi	Mechanical
PRC-A	Absorber Pressure Control	Pressure Transmitter	Taylor	1304TD11120	200-800 in. H ₂ O	4-20 mA
		Indicating Controller	Taylor	1304RA10002	0.25-1.25 v	4-20 mA
		Recorder	Taylor	1301JA10002	0.25-1.25 v	0-100 div
		I/P Transducer	Moore	77-16	4-20 mA	3-15 psi
PCV-A	Absorber Pressure Control	Control Valve	Precision Products	$\frac{1}{4}$ in., 0.13 C _v	3-15 psi	Mechanical
PdR-A	Absorber Pressure Drop	Δ P Transmitter	Taylor	1303TD11120	20-250 in. H ₂ O	4-20 mA
		Recorder	Taylor	1301JA10002	0.25-1.25 v	0-100 div
PRC-F	Fractionator Pressure Control	Pressure Transmitter	Taylor	1304TD11120	200-800 in. H ₂ O	4-20 mA
		Indicating Controller	Taylor	1304RA10002	0.25-1.25 v	4-20 mA
		Recorder	Taylor	1301JA10002	0.25-1.25 v	0-100 div
		I/P Transducer	Moore	77-16	4-20 mA	3-15 psi
PCV-F	Fractionator Pressure Control	Control Valve	Precision Products	$\frac{1}{4}$ in., 0.08 C _v	3-15 psi	Mechanical
PdRC-F	Fractionator Pressure Drop Control	Δ P Transmitter	Taylor	1302TD11122	5-50 in. H ₂ O	4-20 mA
		Indicating Controller	Taylor	1304RA10002	0.25-1.25 v	4-20 mA
		Recorder	Taylor	1301JA10002	0.25-1.25 v	0-100 div
SCR	Fractionator Pressure Drop Control	Power Controller	Loyola	LPAC-3-480-STD	4-20 mA	0-54 a
		Immersible Heater	Chromalox	TMI-12183	0-22 a	0-18 kW
PRC-S	Stripper Pressure Control	Pressure Transmitter	Taylor	1304TD11120	200-800 in. H ₂ O	4-20 mA
		Indicating Controller	Taylor	1304RA10002	0.25-1.25 v	4-20 mA
		Recorder	Taylor	1301JA10002	0.25-1.25 v	0-100 div
SCR	Stripper Pressure Control	Power Controller	Loyola	LPAC-3-480-STD	4-20 mA	0-54 a
		Immersible Heater	Chromalox	TMI-18403	0-48 a	0-40 kW
PdRC-S	Stripper Pressure Drop Control	Δ P Transmitter	Taylor	1303TD11120	20-250 in. H ₂ O	4-20 mA
		Indicating Controller	Taylor	1304RA10002	0.25-1.25 v	4-20 mA
		Recorder	Taylor	1301JA10002	0.25-1.25 v	0-100 div
		I/P Transducer	Moore	77-16	4-20 mA	3-15 psi
		Refrigeration Control	Alco Controls	724	3-15 psi	2-110 psig

TABLE I (Continued)
INSTRUMENTATION KEY TO OPERATING FLOW SHEET

Symbol	Service	Description	Manufacturer	Model	Input Range	Output Range
PRC-SS	Solvent Still Pressure Control	Pressure Transmitter	Taylor	1304TD21120	200-800 in. H ₂ O	4-20 mA
		Indicating Controller	Taylor	1304RA10002	0.25-1.25 v	4-20 mA
		Recorder	Taylor	1322JA14123	0.25-1.25 v	0-100 div
		I/P Transducer	Moore	77-16	4-20 mA	3-15 psi
PCV-SS	Solvent Still Pressure Control	Refrigeration Control	Alco Controls	724	3-15 psi	2-110 psig
		Control Valve	Dover	$\frac{1}{4}$ in., 0.25 C _v	3-15 psi	Mechanical
PdR-SS	Solvent Still Pressure Drop	Δ P Transmitter	Taylor	1302TD11122	5-50 in. H ₂ O	4-20 mA
		Recorder	Taylor	1322JA14123	0.25-1.25 v	0-100 div
LIC-GX	Feed Gas Cooler Liquid Level Control	Capacitance Probe	Drexelbrook	700-1-23	Liquid Level	0.25-4500 pf
		Transmitter	Drexelbrook	408-1000	0.25-4500 pf	4-20 mA
		Indicating Controller	Taylor	1304RA10002	0.25-1.25 v	4-20 mA
		Indicating Meter	Drexelbrook		4-20 mA	0-100 div
		I/P Transducer	Moore	77-16	4-20 mA	3-15 psi
LCV-GX	Feed Gas Cooler Liquid Level Control	Control Valve	Precision Products	$\frac{1}{4}$ in., 0.2 C _v	3-15 psi	Mechanical
LIC-AB	Absorber Liquid Level Control	Capacitance Probe	Drexelbrook	700-1-23	Liquid Level	0.25-4500 pf
		Transmitter	Drexelbrook	408-1000	0.25-4500 pf	4-20 mA
		Indicating Controller	Taylor	1304RA10002	0.25-1.25 v	4-20 mA
		Indicating Meter	Drexelbrook		4-20 mA	0-100 div
		I/P Transducer	Fairchild	T-5120	4-20 mA	3-15 psi
LCV-AB	Absorber Liquid Level Control	Control Valve	Dover	$\frac{1}{2}$ in., 0.5 C _v	3-15 psi	Mechanical
		Control Valve	Jordan	$\frac{1}{2}$ in., 0.84 C _v	3-15 psi	Mechanical
LIC-FB	Fractionator Liquid Level Control	Capacitance Probe	Drexelbrook	700-1-23	Liquid Level	0.25-4500 pf
		Transmitter	Drexelbrook	408-1000	0.25-4500 pf	4-20 mA
		Indicating Controller	Taylor	1304RA10002	0.25-1.25 v	4-20 mA
		Indicating Meter	Drexelbrook		4-20 mA	0-100 div
		I/P Transducer	Fairchild	T-5120	4-20 mA	3-15 psi
LCV-FB	Fractionator Liquid Level Control	Control Valve	Dover	$\frac{1}{2}$ in., 0.80 C _v	3-15 psi	Mechanical
LIC-SB	Stripper Liquid Level Control	Capacitance Probe	Drexelbrook	700-1-23	Liquid Level	0.25-4500 pf
		Transmitter	Drexelbrook	408-1000	0.25-4500 pf	4-20 mA
		Indicating Controller	Taylor	1304RA10002	0.25-1.25 v	4-20 mA
		Indicating Meter	Drexelbrook		4-20 mA	0-100 div
		I/P Transducer	Fairchild	T-5120	4-20 mA	3-15 psi
LCV-SB	Stripper Liquid Level Control	Control Valve	Dover	$\frac{1}{2}$ in., 1.0 C _v	3-15 psi	Mechanical
		Control Valve	Precision Products	$\frac{1}{2}$ in., 2.0 C _v	3-15 psi	Mechanical
LIC-SST	Still Condenser Liquid Level Control	Capacitance Probe	Drexelbrook	700-1-23	Liquid Level	0.25-4500 pf
		Transmitter	Drexelbrook	408-1000	0.25-4500 pf	4-20 mA
		Indicating Controller	Taylor	1304RA10002	0.25-1.25 v	4-20 mA
		Indicating Meter	Drexelbrook		4-20 mA	0-100 div
		I/P Transducer	Moore	77-16	4-20 mA	3-15 psi
LCV-SST	Still Condenser Liquid Level Control	Control Valve	Precision Products	$\frac{1}{2}$ in., 1.25 C _v	3-15 psi	Mechanical

TABLE I (Continued)
INSTRUMENTATION KEY TO OPERATING FLOW SHEET

Symbol	Service	Description	Manufacturer	Model	Input Range	Output Range
LIC-SSB	Still Reboiler Liquid Level Control	Capacitance Probe Transmitter Indicating Controller Indicating Meter	Drexelbrook Drexelbrook Taylor Drexelbrook	700-1-23 408-1000 1304RA10002	Liquid Level 0.25-4500 pf 0.25-1.25 v 4-20 mA	0.25-4500 pf 4-20 mA 4-20 mA 0-100 div
SCR	Still Reboiler Liquid Level Control	Power Controller Immersible Heater	Loyola Chromalox	LPAC-3-480-STD TMI-12303	4-20 mA 0-36 a	0-54 a 0-30 kW
LI-XV	Solvent Tank Liquid Level	Capacitance Probe Transmitter Indicating Meter	Drexelbrook Drexelbrook Drexelbrook	700-1-23 409-1000	Liquid Level 0.25-4500 pf 4-20 mA	0.25-4500 pf 4-20 mA 0-100 div
SG	Solvent Tank Liquid Level	Liquid Level Sight Glass	Strahman	SVT-4LCF	Liquid Level	Indicator
FI-GR	Process Feed Gas Flow	Mass Flowmeter Transducer Mass Flowmeter Indicator	Hastings-Raydist Hastings-Raydist	H-3M/L-25 AHL-25P	0-25 scfm 0-5 v	0-5 v 0-25 scfm
FI-AB	Absorber Feed Gas Flow	Mass Flowmeter Transducer Mass Flowmeter indicator Recorder	Hastings-Raydist Hastings-Raydist Taylor	H-3M/L-25 AHL-25P 1322JA14123	0-25 scfm 0-5 v 0.25-1.25 v	0-5 v 0-25 scfm 0-100 div
FI-AT	Absorber Off-Gas Flow	Mass Flowmeter Transducer Mass Flowmeter Indicator Recorder	Hastings-Raydist Hastings-Raydist Taylor	H-3M/L-25 AHL-25P 1233JA14123	0-25 scfm 0-5 v 0.25-1.25 v	0-5 v 0-25 scfm 0-100 div
FI-FT	Fractionator Off-Gas Flow	Mass Flowmeter Transducer Mass Flowmeter Indicator	Hastings-Raydist Hastings-Raydist	H-3M/L-10 AHL-10PG	0-10 scfm 0-5 v	0-5 v 0-10 scfm
FIR-ST1	Stripper Off-Gas Flow	Mass Flowmeter Transducer Mass Flowmeter Indicator Recorder	Hastings-Raydist Hastings-Raydist Taylor	H-5KM ALL-5KPG 1322JA14123	0-5000 sccm 0-5 v 0.25-1.25 v	0-5 v 0-5000 sccm 0-100 div
FIR-ST2	Stripper Off-Gas Flow	Mass Flowmeter Transducer Mass Flowmeter Indicator Recorder	Hastings-Raydist Hastings-Raydist Taylor	H-50KM ALL-50KPG 1322JA14123	0-50,000 sccm 0-5 v 0.25-1.25 v	0-5 v 0-50,000 sccm 0-100 div
FIC-ST	Stripper Off-Gas Flow Control	Indicating Controller I/P Transducer	Taylor Moore	1304RA10002 77-16	0.25-1.25 v 4-20 mA	4-20 mA 3-15 psi
FCV-ST1	Stripper Off-Gas Flow Control	Control Valve	Precision Products	$\frac{1}{4}$ in., 0.0022 C _v	3-15 psi	Mechanical
FCV-ST2	Stripper Off-Gas Flow Control	Control Valve	Precision Products	$\frac{1}{4}$ in., 0.019 C _v	3-15 psi	Mechanical
FRC-AX	Absorber Solvent Feed Control	Flowmeter Transmitter Indicating Controller Recorder I/P Transducer	Brooks Taylor Leeds and Northrup Moore	3623/5522H 1304RA10002 Speedomax W/L 77-16	0-2.5 gpm 0.25-1.25 v 0.25-1.25 v 4-20 mA	4-20 mA 4-20 mA 0-100 div 3-15 psi
FCV-AX	Absorber Solvent Feed Control	Control Valve	Dover	$\frac{1}{2}$ in., 0.8 C _v	3-15 psi	Mechanical
FIR-SSP	Solvent Still Distillate Flow	Flowmeter Transmitter Recorder	Brooks Leeds and Northrup	3623/5522H Speedomax W/L	0-2.5 gpm 0.25-1.25 v	4-20 mA 0-100 div
FIR-SSR	Solvent Still Reflux Flow	Flowmeter Transmitter Recorder	Brooks Leeds and Northrup	3623/5522H Speedomax W/L	0-2.5 gpm 0.25-1.25 v	4-20 mA 0-100 div

TABLE I (Continued)
INSTRUMENTATION KEY TO OPERATING FLOW SHEET

Symbol	Service	Description	Manufacturer	Model	Input Range	Output Range
RC-SSR	Solvent Still Reflux Ratio Control	Multiplier/Divider	Taylor	1330NA11000	0.25-1.25 v	4-20 mA
		Indicating Controller	Taylor	1304RA10002	0.25-1.25 v	4-20 mA
		I/P Transducer	Moore	77-16	4-20 mA	3-15 psi
FCV-SSR	Solvent Still Reflux Flow Control	Control Valve	Precision Products	$\frac{1}{2}$ in., 2.0 C _v	3-15 psi	Mechanical
TIC-SSB	Solvent Still Bottoms Flow Control	E/I Transmitter	Taylor	1022TA12140	-20°F - +80°F	4-20 mA
		Indicating Controller	Taylor	1304RA10002	0.25-1.25 v	4-20 mA
		I/P Transducer	Fairchild	T-5120	4-20 mA	3-15 psi
FCV-SSB	Solvent Still Bottoms Flow Control	Control Valve	Dover	$\frac{1}{4}$ in., 0.075 C _v	3-15 psi	Mechanical

TABLE II
INSTRUMENTATION KEY TO PRODUCT PURIFICATION COLD TRAP

Symbol	Service	Description	Manufacturer	Model	Input Range	Output Range
PRC-PT	Product Purification Cold Trap Pressure Control	Pressure Transmitter	Taylor	1332TF11221	10-100 psi	4-20 mA
		Indicating Controller	Taylor	1304RA10002	0.25-1.25 v	4-20 mA
		Recorder	Taylor	1322JA14123	0.25-1.25 v	0-100 div
		I/P Transducer	Moore	77-16	4-20 mA	3-15 psi
PCV-PT	Product Purification Cold Trap Control	Control Valve	Precision Products	$\frac{1}{4}$ in., 0.003 C _v	3-15 psi	Mechanical
PdR-PT	Product Purification Cold Trap Pressure Drop	Δ P Transmitter	Taylor	1301TD11122	0-10 in. H ₂ O	4-20 mA
		Recorder	Taylor	1322JA14123	0.25-1.25 v	0-100 div
TE-LN	Product Purification Liquid Nitrogen Temperature Control	Thermobulb/Thermowell	Taylor	PLCL-9-3A/ 1-10RL-9	-320 to +32°F	19-100 Ω
		Resistance to Current Transmitter	Taylor	1002TA14810	19-43 Ω	4-20 mA
		Indicating Controller	Taylor	1304RA10002	0.25-1.25 v	4-20 mA
		I/P Transducer	Moore	77-16	4-20	3-15 psi
TCV-LN	Product Purification Liquid Nitrogen Control	Control Valve	Precision Products	$\frac{1}{4}$ in., 0.006 C _v	3-15 psi	Mechanical
TE-PT	Product Purification Cold Trap Temperature Control	Thermobulb/Thermowell	Taylor	PLCL-9-3A/ 1-10RL-9	-320 to +32°F	19-100 Ω
		Resistance to Current Transmitter	Taylor	1002TA14810	19-43 Ω	4-20 mA
		Indicating Controller	Taylor	1304RA10002	0.25-1.25 v	4-20 mA
		I/P Transducer	Moore	77-16	4-20 mA	3-15 psi
TCV-PT	Product Purification Cold Trap Temperature Control	Control Valve	Precision Product	$\frac{1}{4}$ in., 0.02 C _v	3-15 psi	Mechanical
FIR-ST	Stripper Product Flow	Thermal Mass Flowmeter Transducer	Tylan	FM-360	0-1 slm	0-5 v
		Thermal Mass Flowmeter Indicator	Tylan	RO-751	0-5 v	0-1 slm
		Recorder	Taylor	1322JA14123	0.25-1.25 v	0-100 div
FIR-PT1	Product Purification Cold Trap Off-Gas Flow	Thermal Mass Flowmeter Transducer	Tylan	FM-360	0-100 sccm	0-5 v
		Thermal Mass Flowmeter Indicator	Tylan	RO-751	0-5 v	0-100 sccm
		Recorder	Taylor	1322JA14123	0.25-1.25 v	0-100 div
FIR-PT2	Product Purification Cold Trap Off-Gas Flow	Thermal Mass Flowmeter Transducer	Tylan	FM-360	0-500 sccm	0-5 v
		Thermal Mass Flowmeter Indicator	Tylan	RO-751	0-5 v	0-500 sccm
		Recorder	Taylor	1322JA14123	0.25-1.25 v	0-100 div

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