

Conf-720823--14

EXPERIMENTAL DEMONSTRATION OF THE SELECTIVE ABSORPTION PROCESS
FOR KRYPTON-XENON REMOVAL*

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Abstract**NOTICE**

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Nuclear reactors and nuclear fuel reprocessing plants present different problems in terms of controlling gaseous activity discharge to the atmosphere. One gas cleaning technique potentially suitable for both cases, however, is the selective absorption process developed at the Oak Ridge Gaseous Diffusion Plant. Fundamental engineering studies, described at the 11th AEC Air Cleaning Conference, have since been followed by more specialized demonstration tests aimed at these two application areas.

I. Introduction

At the 11th AEC Air Cleaning Conference, we described some results of basic studies related to the selective absorption process for removal of krypton and xenon from nuclear process off-gas streams⁽³⁾. Those studies, aimed primarily at the feasibility and basic mass transfer aspects of the process, indicated that selective absorption has considerable potential as a gas cleaning technique and suggested guidelines for further work. During the last two years, therefore, the project has continued but with more emphasis on specific LWR system application than on fundamental characteristics of the process.

It might be recalled from the last conference that the ORGDP program was originally divided into five separate phases, viz:

- PHASE I - Evaluation of absorption process performance, using refrigerant-12 as the solvent, and collection of mass transfer data.
- PHASE II - Same as Phase I, except using refrigerant-11 as the process solvent.
- PHASE III - Determination of the effects on the absorption process of failures in upstream air cleaning systems which result in introduction of various impurities, such as iodine, methyl iodide, and nitrous oxide, to the absorption plant.
- PHASE IV - Investigation of alternative methods for permanent storage of concentrated noble gases.

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*This document is based on work performed at the Oak Ridge Gaseous Diffusion Plant operated by Union Carbide Corporation, Contract W-7405-eng-26 with the United States Atomic Energy Commission.

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PHASE V - Further experimental investigation of special processing situations, possibly including argon-krypton or hydrogen-krypton separations and low concentration tracer work.

The first two phases of the program were described at the previous meeting. Phase IV, dealing with handling of noble gas concentrates prepared by the absorption or some other separation process, was deleted from this project, but related work is under way at other AEC laboratories. During the past two years, then, the program has been focused on process demonstration in two areas which have been characterized recently by increasing interest: use of the absorption process to assist in treating off-gas from reactors and from fuel reprocessing plants.

Reactors and fuel reprocessing plants present very different problems, in terms of noble gas removal. On the one hand, in reactors, the noble gas activity is from both krypton and xenon, and the isotopes are present in very low molar concentrations - a few ppb to 1 or 2 ppm. On the other hand, in chemical plants, krypton-85 is the primary noble gas isotope of concern, and the krypton feed concentration is typically as high as several hundred ppm; what complicates the picture here is the potential presence in the feed of high concentrations of numerous impurities, such as oxides of nitrogen. Therefore, two separate experimental programs were conducted to demonstrate further the potential for applying the absorption technique to these two off-gas cleanup jobs. Both the reactor-oriented tests (Phase VA) and the reprocessing plant-related studies (Phase III) are described below, following a brief discussion of the process. The results of the reactor-oriented tests are presented in sections entitled *Applicability of Design Models*, *Xenon Behavior*, *Process Performance with Different Carrier Gases*, and *Miscellaneous Results*. For the fuel reprocessing plant scoping tests, sections are provided which discuss both *Calculated Impurity Behavior* and *Scoping Experiments*, including the results of laboratory experiments, pilot plant injection tests, and flow tests.

II. Process Description

A schematic flow diagram of the pilot plant is shown in figure 1. Three packed columns comprise the heart of the process. Each column is designed to exploit certain gas-liquid solubility differences that exist between the solvent and the various gas constituents that might be present. The main separation of noble gas from the bulk carrier gas is accomplished in the absorber. Here, the contaminated process gas, after being compressed and cooled, is intimately contacted with the solvent and essentially all of the krypton and xenon, along with a portion of the carrier gas, dissolves. The remaining diluent gas, depleted in contaminants, is vented from the process, while the loaded solvent is passed on to the fractionator. The purpose of the fractionator is to strip the bulk of the coabsorbed carrier gas from the loaded solvent. The fractionator is operated at a much lower pressure than the absorber, and additionally, the gas-laden liquid phase is subjected to the stripping action of counter-flowing solvent vapor. Consequently, the bulk of the less soluble carrier gases readily desorbs. The off-gas from the fractionator must ordinarily be recycled back to the absorption step, however, because it also contains a measurable amount of desorbed krypton and xenon. The enriched solvent stream produced in the fractionator subsequently passes on to the stripper. The purpose of the stripper is to produce a highly concentrated krypton-xenon gas product by driving the remaining dissolved gases from the solvent. The stripper is operated at an even lower pressure than the fractionator and with more liquid-vapor contacting to facilitate desorption of even the most soluble gases. The pure solvent leaving the stripper is pumped back to the absorber for reuse.

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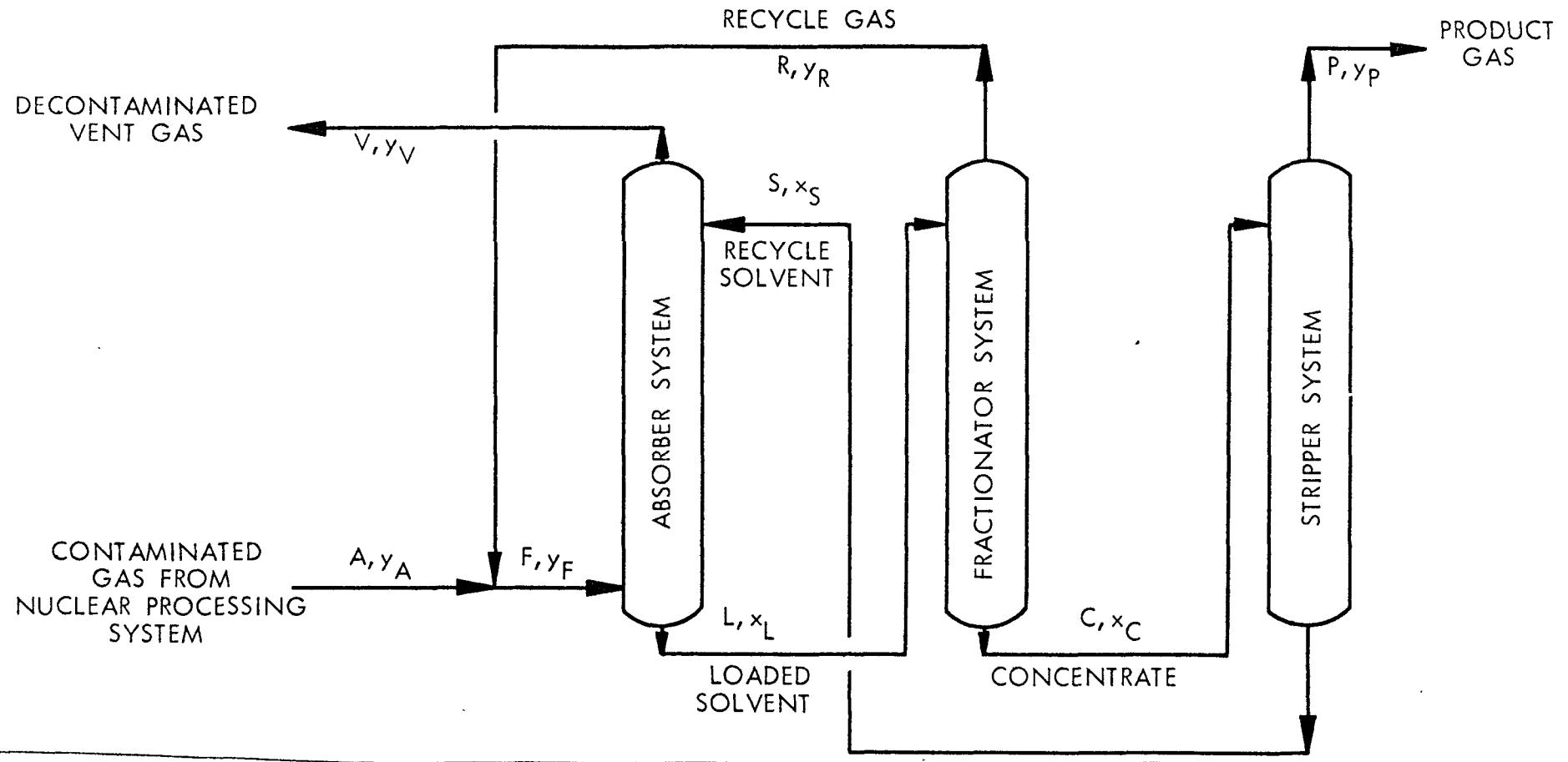


Figure 1
ABSORPTION PROCESS SCHEMATIC FLOW DIAGRAM

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The development pilot plant was designed on the basis of processing a nominal 15 scfm of gas at absorber pressures as high as 600 psig and temperatures as low as minus 90°F. The pilot plant absorber and fractionator columns are 3 inches in diameter and contain 9 feet of column packing. The stripper column is 10 inches in diameter and contains 10 feet of packing.

III. Reactor-Oriented Tests

The primary purpose of reactor-related experiments was to determine how well previously developed performance correlations can be applied to situations where the concentrations of krypton and xenon in the feed gas are very low, as in reactor off-gas treatment. Other goals included experimental verification of predicted xenon behavior and testing with carrier gases other than air. Radioactive krypton and xenon isotopes were used in all tests. The results of these experiments, referred to as program Phase VA, are presented below.

Applicability of Design Models

From an engineering standpoint, particularly when fluorocarbon solvents are used, description of the mass transfer processes occurring in the absorber column is complicated by the combination of high liquid loadings, rapid mass transfer rates, multiple components being transferred, and widely varying liquid-to-gas flow ratios. In the early studies, numerous models and theories were screened, with the goal of developing an overall design package which would not only describe the data adequately but also be reasonably convenient.

Perhaps the simplest to use of the procedures investigated is the one which includes, as the stage model, a conventional film theory description. One of the design packages which we used is based on use of this model, together with an average L/V ratio, adjusted k-values, and an empirical transfer unit height correlation⁽³⁾. This system of equations, while not theoretically sophisticated in this application, is particularly convenient for repetitive calculations and, furthermore, does represent the data well enough for most design purposes. Therefore, it was decided to use this simplified design package as the basis for judging the previous correlations. The design equations from the first experimental campaign of the ORGDP program were based upon tests in which the process feeds contained relatively high levels of krypton, ranging from 40 to 8800 ppm. Although statistical analysis of the early data indicated that the transfer unit height correlations were not dependent upon feed concentrations, the low concentration tests were nevertheless performed because of substantial interest in the reactor case.

For convenience, the design procedure, which was included in our previous paper, is summarized below. Basically the model starts with the representation

$$Z = (N_{OG}) (H_{OG})$$

where Z = required column height, inches;

N_{OG} = number of transfer units required to perform the stated separation; and

H_{OG} = height of the transfer unit, inches.

In the simplified case, the number of transfer units can be given by

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$$N_{OG} = \frac{2.3}{1 - \frac{kG}{L}} \log \left\{ \frac{(y_1 - kx_2)}{(y_2 - kx_1)} \right\} \left(1 - \frac{kG}{L} \right) + \frac{kG}{L}$$

where k = effective gas-liquid equilibrium coefficient;

G/L = ratio of column average molar gas flow-to-liquid flow;

y = gas phase mole fraction of absorbing component;

x = liquid phase mole fraction of absorbing component; and

subscripts 1 and 2 refer to the gas inlet and outlet ends of the column, respectively.

The appropriate value of the effective equilibrium coefficient for the krypton and refrigerant-12 system is given by

$$k = \exp[3.8167 + 0.010197 T]/P_{Total}$$

where T = absorption temperature, °F; and

P_{Total} = total system pressure, atmospheres.

The average L/G molar flow rate ratio for air and refrigerant-12 can be obtained from the known feed L/G molar flow rate ratio by

$$(L/G)_{avg} = 0.443 (L/G)_{Feed}^{1.116} P^{0.318}$$

Finally, H_{OG} values for krypton absorption in refrigerant-12 consistent with the above can be obtained from

$$H_{OG} = \frac{1.36 \times 10^6 G^{1.46}}{\alpha^{0.525} P^{1.74} L^{1.61}}$$

where H_{OG} = transfer unit height, inches;

G = column feed gas flow rate, lb/min-sq ft;

P = absorption pressure, atmospheres;

L = solvent feed flow rate, lb/min-sq ft; and

$\alpha = \exp(2.0394 - 0.008253T)$, with T in °F.

Using the run data from Phase VA krypton-air separation tests, expected krypton removals were calculated using the procedure given above and these were compared with measured values. The results are presented in table I. The measured values agree well with the calculated ones, indicating that the earlier correlations do provide a reliable framework for low concentration designs.

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TABLE I

EXTENSION OF PHASE I ABSORBER CORRELATIONS
TO LOW CONCENTRATION REACTOR CASES

Run	Absorber Feed Gas*		Krypton Removal in Absorber, %	
	Flow, scfm	Krypton Content, ppb	Calculated from Correlations	Measured in Phase VA
1	5.9	150	99.8	99.89 to 99.90
2	6.1	110	99.7	99.88 to 99.89
3	5.9	130	99.7	99.88 to 99.89
4	5.9	130	99.8	99.85 to 99.86
5	5.8	130	99.8	99.91 to 99.92
6	5.9	110	99.8	99.91 to 99.92
7	6.2	70	99.8	99.86 to 99.88
8	5.9	60	99.8	99.87 to 99.89
9	4.8	50	99.8	99.92 to 99.93
10	5.0	60	99.9	99.91 to 99.93
11	5.0	50	99.8	99.90 to 99.92

* For all runs, solvent flow = 1.0 gpm, absorber pressure = 374 psia, absorber temperature = minus 25°F.

Xenon Behavior

Previous experiments involved use of krypton only, since it is the key component in absorber column design. The behavior of xenon, the key component in the stripper in many cases, had been predicted on the basis of krypton data and known solubility differences. As a part of the recent studies, however, it was decided to conduct some experiments with xenon to confirm the predicted disposition.

Eight tests were made at an absorber pressure and temperature of 374 psia and minus 25°F. Simulated process feed gas flows were between 1 and 3 scfm, and the refrigerant-12 solvent flow rate was 1.0 gpm. The carrier gas was air, which had previously been denuded of xenon. Xenon, containing xenon-133, was present in the process feed at concentrations between 3 and 15 ppb. Under these conditions, krypton removals on the order of 99.9% would be expected. Overall process xenon removal efficiencies between 99.95 and 99.99% were measured, confirming that the more soluble xenon can be absorbed at least as readily as krypton and that xenon stripping from the solvent is straightforward.

Performance with Different Carrier Gases

Since the purge or vent gases from various nuclear installations might be comprised of carriers other than air, pilot plant tests were also conducted with nitrogen, argon, helium, and hydrogen gas feeds. These tests, along with the air experiments, are summarized in table II.

It can be seen that krypton can be effectively removed from other carrier gases. As the carrier gas is changed, of course, the sharpness of the separation is affected, since the quantity of gas coabsorbed with the krypton-xenon fraction depends on the solubility of the carrier. As a consequence, changes in fractionator operation from that used in air processing are required as the carrier gas is changed. For example, more rigorous fractionation is required with argon as the carrier than with air to achieve comparable product concentrations, since argon is more soluble than air.

Miscellaneous Results

The use of radioactive isotopes in the Phase VA experiments provided an opportunity for direct measurement of krypton-xenon residence times and inventories in the process. These data are required for assessing the extent of solvent exposure to radiation. For example, the average holdup time for krypton and xenon in contact with solvent is roughly 15 minutes for processing conditions typically employed when refrigerant-12 is used as the solvent. During the Phase VA experiments, the pilot plant activity inventory ranged between 1 and 3 curies; thus the pilot plant inventory was equivalent to that of a system processing between 35,000 and 100,000 curies per year.

IV. Fuel Reprocessing Plant Scoping Tests

For fuel reprocessing plants, the behavior of feed gas impurities is of primary concern, since these not only potentially affect system operability but also determine the overall design and strategy of the krypton removal train. For this part of the program, the behavior of nitric oxide, nitrous oxide, nitrogen dioxide, carbon dioxide, methyl iodide, and iodine in a refrigerant-12 absorption system was evaluated. The evaluation included both calculations and experiments.

Calculated Impurity Behavior

Due to the lack of much definitive experimental data, the Phase III tests were preceded by calculations aimed at providing an initial indication of impurity disposition. In addition to basic physical properties, solubilities of several gaseous impurities in refrigerant-12 and other solvents were estimated using such procedures as: (a) interpolation from plots constructed using measured solubilities and Lennard-Jones constants⁽⁵⁾, (b) a statistical thermodynamics-based estimating model⁽⁴⁾, and (c) a molecular thermodynamics-based procedure⁽⁶⁾.

The results of the statistical mechanics approach were found to be too sensitive to the values of the input parameters to be of consistent utility as a predictive tool, although the equation forms were used to obtain good correlations of experimental solubility data. Comparing known gas solubilities with calculated values, the other two approaches appeared to provide reasonable estimates of solubilities.

TABLE II
OVERALL PROCESS EFFICIENCIES
(Phase VA Only)

Carrier Gas	Noble Gas Contaminant	Number of Pilot Plant Tests	Process Feed*		Processing Conditions			Overall Process Efficiency, %	
			Flow, scfm	Contaminant Concentration, ppb	Absorber Pressure, psia	Absorber Temperature, °F	Solvent Flow, gpm		
Air	Krypton	11	1-3	50 - 200	374	- 25	1.0	99.7	- 99.9
Air	Xenon	8	1-3	3 - 15	374	- 25	1.0	99.95	- 99.99
Nitrogen	Krypton	30	1-6	40 - 1000	314 - 422	- 35 to - 23	0.75-1.0	99.0	- 99.9
Argon	Krypton	6	1-4	10 - 1400	374	- 25	1.0	99.0	- 99.7
Helium	Krypton	9	2-7	100 - 4300	254 - 374	- 40 to - 25	0.75-1.0	99.4	- 99.9
Hydrogen	Krypton	3	1-2	300 - 600	194	- 25	0.75	99.0	- 99.9

* The process feed plus the fractionator recycle make up the absorber feed.

The calculations indicated that the gaseous impurities can generally be grouped into three categories:

1. Very soluble components, such as carbon dioxide and nitrous oxide, that follow krypton and xenon through the process and ultimately concentrate in the stripper off-gas or product stream;
2. Slightly soluble components, such as methane and nitric oxide, that follow nitrogen and oxygen through the process and similarly distribute between the absorber vent and product*; and
3. Relatively insoluble components, such as hydrogen and helium, that pass through the absorber and are subsequently vented.

For the condensed liquids and desublimed solids, i.e., methyl iodide, nitrogen dioxide, and iodine, estimates of behavior were made considering solubilities in similar systems. For example, iodine is known to form solutions with several fluorocarbons⁽¹⁾.

On balance, then, the calculations and available data indicated that the impurities could probably be handled in the absorption process.

Scoping Experiments

Using the above estimates as guides, the scoping tests were next conducted. These tests included a few laboratory screening experiments, pilot plant tests in which various gases were sequentially injected, and runs in which mixtures of impurities were continuously introduced into the system. In several tests, krypton-85 was included so that the process separation performance could be monitored in the presence of impurities.

Laboratory screening tests were conducted with nitric oxide, methyl iodide, and iodine. For nitric oxide, an infrared cell experiment showed that the rate of reaction with oxygen is large enough for nitric oxide to essentially disappear during processing in the absorption system, as expected. For methyl iodide, tests conducted at minus 20°F indicated that the compound is miscible with refrigerant-12. And, in the case of iodine, experiments at minus 20°F showed that iodine forms a violet solution with refrigerant-12, with a solubility on the order of 0.1 mole percent.

For the injection experiments, the pilot plant was operated in a total recycle mode, with the absorber column at 374 psia and minus 25°F. Krypton and xenon were present at concentrations near 20 and 100 ppm, respectively, in the simulated process feed gas. Carbon dioxide, nitrous oxide, nitric oxide, and nitrogen dioxide were sequentially injected into the pilot plant, and the concentrations were gradually built up by repeated additions. The target concentrations were in the high ppm to low percent range in the feed gas. No operational problems were encountered and the overall krypton removal efficiency remained at the initial high level (greater than 99%). Analyses of the gas streams showed that nitrous oxide and carbon dioxide had followed krypton and xenon and that nitric oxide had disappeared to below the limit of detection. Nitrogen dioxide was not found in analyses of the gas streams, indicating that it had remained in the liquid.

* However, based on consideration of kinetic data and the temperatures and pressures prevailing in the absorption system, it was estimated that most nitric oxide in the system would be rapidly converted to nitrogen dioxide in the presence of air.

For the flow tests, the pilot plant was operated, as shown in figure 2, with a continuous air feed, and with the columns vented. In the first run, carbon dioxide, nitrous oxide, nitric oxide, and nitrogen dioxide were metered to the process in amounts giving impurity feed concentrations of several hundred ppm to nearly 1%. This particular mode of plant operation was maintained for two weeks. Carbon dioxide and nitrous oxide, as observed before, absorbed readily in the refrigerant-12 solvent, followed krypton and xenon, and were concentrated in the stripper off-gas. Again, nitric oxide was not detected in either the process off-gas or solvent, indicating essentially complete conversion to nitrogen dioxide. Based on both gas and liquid sample results, nitrogen dioxide, on the other hand, dissolved in the solvent and began accumulating there from the onset of the run. Eventually, corresponding to the buildup in the liquid, small quantities of nitrogen dioxide began appearing in the process off-gas. With 1 to 2 mole percent nitrogen dioxide in the solvent, less than 5 ppm was detected in the absorber off-gas. Later, with 5 to 10 mole percent in the solvent, approximately 200 ppm was detected in the absorber off-gas, and additionally, about 20 ppm was found in the stripper off-gas. As before, no operational difficulties occurred.

A similar pilot plant test was conducted with iodine and methyl iodide. Each material was fed to the plant over a 2-day period. The test data showed that these compounds tend to remain in the solvent and that iodine inadvertently released to the krypton removal system should be contained there and not be further released to the atmosphere.

A summary of the impurity scoping tests is given in table III. Based on these tests, it definitely appears that the krypton absorption process is capable of efficient operation with contaminated feeds of the type likely to be encountered in fuel reprocessing plants in the case of failures of upstream air cleaning devices. Furthermore, it is apparent that, with the proper specification of the process auxiliaries, the absorption system could be designed for sustained operation with a continuous feed containing the above impurities.

V. Plans for Future Work

The ORGDP noble gas absorption program, since its inception in FY 1967, has been oriented mainly toward LWR-related applications - either reactors or chemical plants. With the completion of the work outlined in this paper, we believe it is appropriate to bring the LWR-related portion of our development program to a close. In the framework of what a development program is able to accomplish, many of the basic process questions have been answered, and the technical potential for process application, at least, has been demonstrated. Consequently, no more development work specifically for LWR systems is planned. This decision is supported by the fact that there is now beginning to be some commercial interest in the process⁽²⁾.

A new program involving the selective absorption process, beginning in FY 1973, is being initiated. This new program, which is being conducted jointly with ORNL, has the objective of defining and demonstrating a total krypton removal package for an LMFBR fuel reprocessing plant off-gas treatment system.

A schematic flow diagram is given in figure 3 for a complete krypton absorption plant, reflecting how the process might be applied in this case. The plant includes not only the main columns but also certain auxiliaries. The feed preparation system includes equipment required for drying, filtering, and otherwise conditioning the feed gas. The solvent recovery system removes solvent vapors from the absorber column off-gas stream (process vent). Alternatives being considered here for refrigerant-12 service include cooling-condensing, either directly or using an

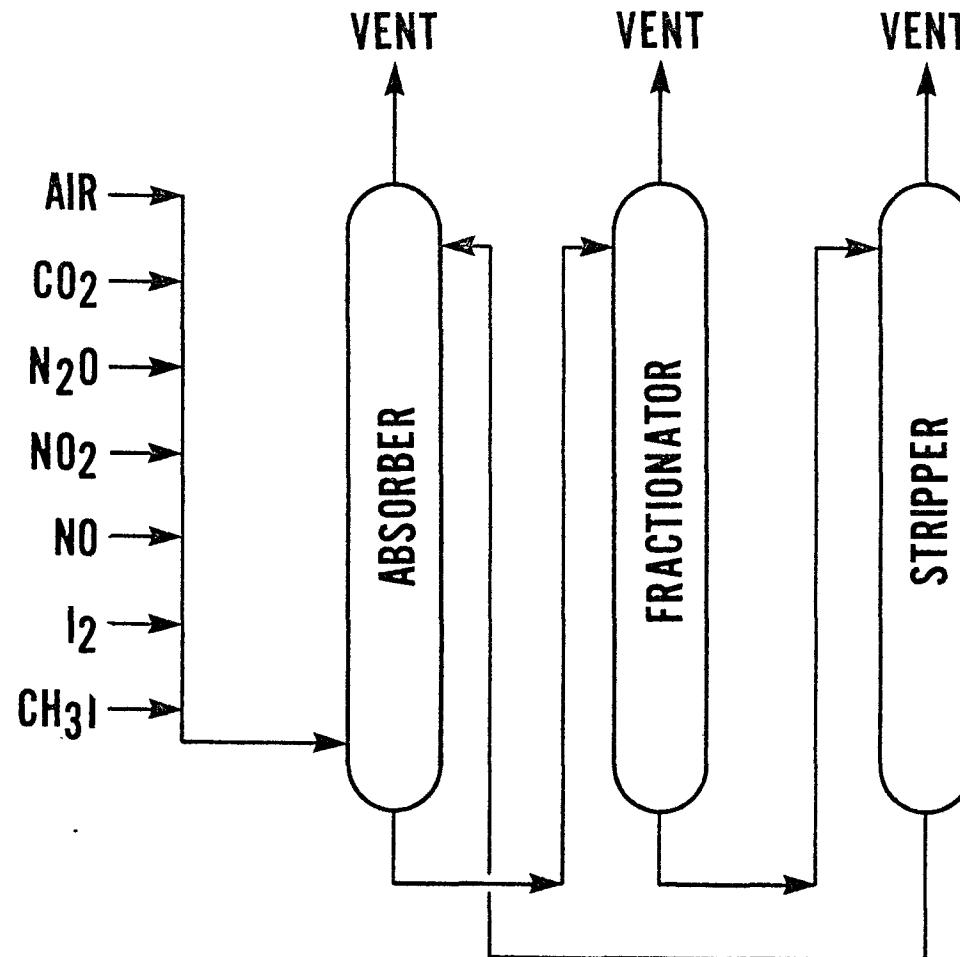


Figure 2
FLOW SCHEMATIC FOR IMPURITIES SCOPING TESTS

TABLE III
SUMMARY OF IMPURITY SCOPING TESTS

<u>Impurity</u>	<u>Concentration In Process Feed, ppm</u>	<u>Disposition in Process</u>	<u>Effect on Plant Operability</u>	<u>Effect on Krypton Removal Efficiency</u>	<u>Influence on Plant Design</u>
Carbon Dioxide	1,000 - 3,500	Follows krypton-xenon	None	None	Carbon dioxide trap in krypton product line
Nitrous Oxide	1,000 - 6,700	Follows krypton-xenon	None	None	Nitrous oxide trap in krypton product line
Nitrogen Dioxide	100 - 600	Accumulates in solvent	None, unless solubility limits are exceeded	None	Solvent purification system
Nitric Oxide	1,000 - 2,000	Converts rapidly to nitrogen dioxide	Same as nitrogen dioxide	Same as nitrogen dioxide	Same as nitrogen dioxide
Iodine	50 - 100	Plates out on surfaces and accumulates in solvent	None, unless solubility limits are exceeded	Not determined	Specially designed gas heat exchanger and solvent purification system
Methyl Iodide	100 - 200	Accumulates in solvent	None	Not determined	Solvent purification system

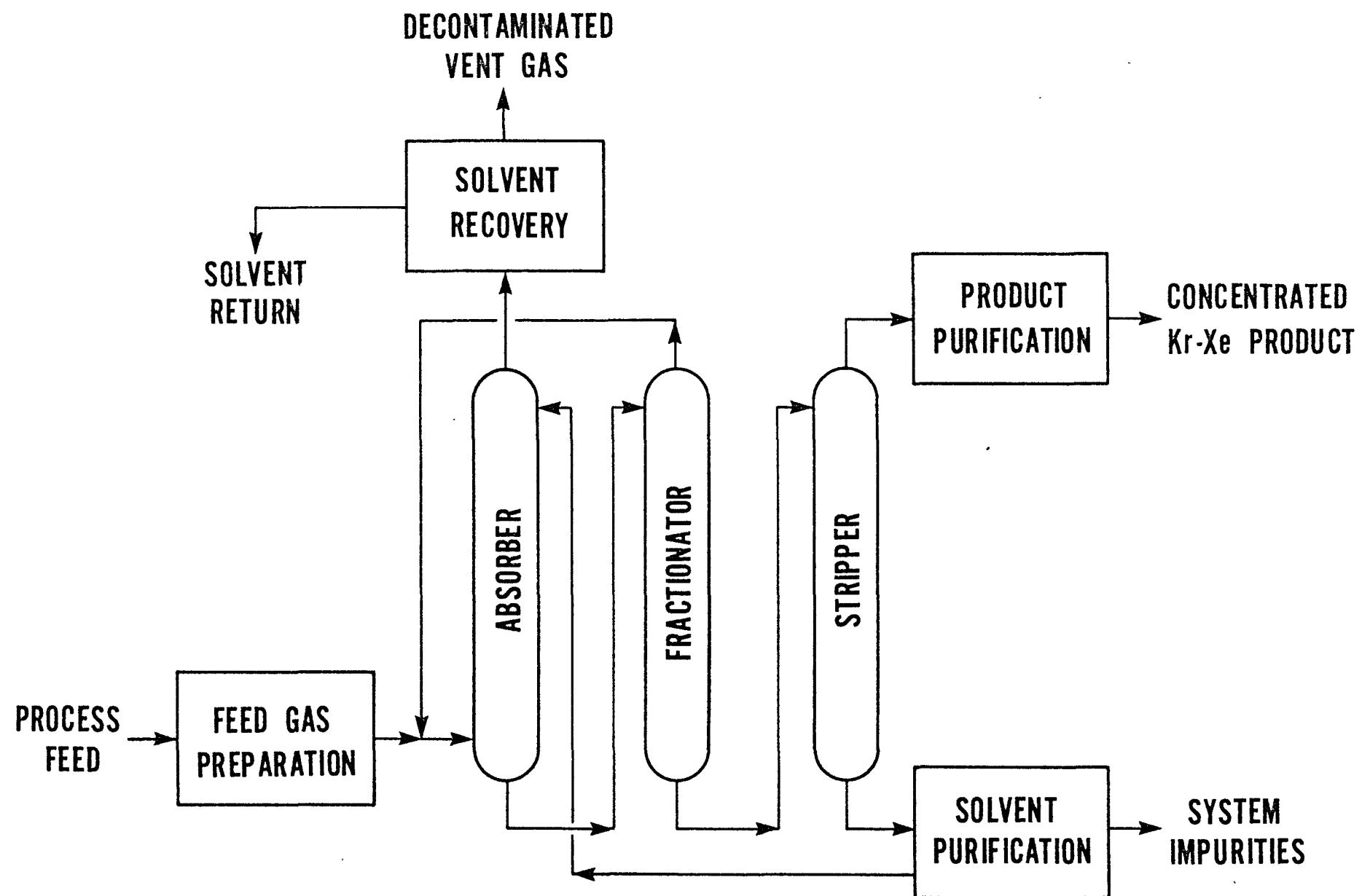


Figure 3
SCHEMATIC FLOW DIAGRAM OF A COMPLETE ABSORPTION PROCESS

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isentropic expansion, adsorption, using 5A molecular sieves, and a combination of these two operations. The solvent purification system is designed to control the accumulation of impurities which remain in the liquid phase. These include iodine, methyl iodide, nitrogen dioxide, heavy hydrocarbons, and the high boiling refrigerant-12 degradation products, refrigerant-114 and refrigerant-113. Finally, the purpose of the product purification system is to scavenge nitrogen, oxygen, nitrous oxide, refrigerant-12, and carbon dioxide from the already low flow rate stripper off-gas, resulting in an essentially pure, highly concentrated noble gas product stream.

During the next two years, these auxiliary systems will be designed, built, and added to the ORGDP pilot plant. The total package will then be operated for extended periods of time. The primary purpose of the work will be to establish the long-term operability and performance of a complete system when processing a feed containing typical IMFBR fuel reprocessing plant concentrations of numerous impurities, such as carbon dioxide, nitrogen oxides, light organics, heavy organics, iodine forms, ozone, particulates, etc. Also, impurity concentration limits above which the process malfunctions will be defined. Based on the scoping tests described today, we are optimistic about the chances of success of the new program.

VI. Conclusion

During the past two years, many of the specific questions raised concerning application of the selective absorption process to reactors and fuel reprocessing plants have been successfully answered, thereby supplementing earlier work which was of a more general nature. For example, the recent data show that the process can be operated efficiently at low concentrations of krypton, as well as at high levels; can remove xenon at least as effectively as krypton; and can separate the noble gases from carrier gases other than air. Also, the process continues to separate krypton efficiently when the feed becomes contaminated with nitrogen oxides and carbon dioxide and, in fact, has a high tolerance for these impurities. Furthermore, it appears that any iodine and methyl iodide passing into the system collect in the solvent and could be removed from the liquid stream, if desired, by treatment of the recycle solvent.

When these development program results are considered along with the earlier results of the program, then the application of the process to a variety of gas cleaning jobs continues to seem very promising.

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12th AEC AIR CLEANING CONFERENCE

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