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**The Experimental Engineering Section
Off-Gas Decontamination Facility's
Fractionator Column:
Installation and Performance**

T. M. Gilliam
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MASTER

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CHEMICAL TECHNOLOGY DIVISION

HTGR FUEL RECYCLE PROGRAM (189a OH045)

Reprocessing Development - Task 300

THE EXPERIMENTAL ENGINEERING SECTION OFF-GAS DECONTAMINATION FACILITY'S
FRACTIONATOR COLUMN: INSTALLATION AND PERFORMANCE

T. M. Gilliam, V. L. Fowler, and D. J. Inman

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FRACTIONATOR COLUMN: INSTALLATION AND PERFORMANCE

T. M. Gilliam, V. L. Fowler, and D. J. Inman

ABSTRACT

A detailed description of the third column recently installed in the Experimental Engineering Section Off-Gas Decontamination Facility (EES-ODF) is presented. The EES-ODF is being used to provide engineering-scale experiments (nominal gas and liquid flows of 5 scfm and 0.5 gpm, respectively) in the development of the Krypton Absorption in Liquid CO₂ (KALC) process.

A detailed discussion of the column's construction is provided. This discussion includes the peripherals associated with the column, such as refrigeration, heat exchangers, instrumentation, etc.

The compressibility of Goodloe packing (the packing in the other columns) and the possible reduced throughput due to this compression have revealed the desirability of a random (i.e., noncompressible) packing. Toward this end, the third column is packed with a new random packing (PRO-PAK). A preliminary comparison between this packing and the woven wire mesh packing (Goodloe) used in the other two columns has been made. Experiments comparing the throughput capacity indicate that the PRO-PAK packing has approximately 60% the capacity of Goodloe for a CO₂ system. When used as a fractionator or stripper with the basic O₂-Kr-CO₂ KALC system, the PRO-PAK column produced HTU values less than or equal to the Goodloe columns under similar operating conditions.

1. INTRODUCTION

As part of the HTGR Fuel Recycle Program,^{1,2} development work is being performed on the KALC (Krypton Absorption in Liquid CO₂) process. The KALC process is being developed as a means of removing and concentrating ⁸⁵Kr from the off-gas generated during the reprocessing of HTGR fuel elements.

In previous experimental campaigns (II and III),^{3,4} mass transfer and fluid dynamics data were collected using a two-column system. The two-column system has been described previously⁵ in great detail and will be discussed only as needed for the clarification of data presented in this report.

The primary objective of Campaign IV (i.e., experiments performed between August 1, 1976, and September 31, 1977) was to obtain data on the effect of minor components on the basic KALC operations. Toward this end, an additional column, packed with a new random packing (PRO-PAK), was installed to give the Experimental Engineering Section Off-Gas Decontamination Facility (EES-ODF) the capability of three-column operations. This report deals with the installation and performance of the new column.

2. DESCRIPTION OF PROCESS COMPONENTS

All of the major pressure vessels described in this report were designed and fabricated to withstand the elevated operating pressures of the facility. Typically, each drawing contains a nozzle identification schedule, a parts list, and notes on fabrication and testing.

The material of construction, 304L stainless steel, was used throughout the process for both vessels and piping (unless otherwise specified). Refrigerant lines external to the process vessels are made of copper. All welded construction is used exclusively except where threaded connections are required for instrumentation.

Packed columns in the EES-ODF are named in accordance with the primary experimental role they will play in the overall KALC process development. The new column is used primarily for the study of fractionating the dissolved light gases from the ^{85}Kr -laden CO_2 solvent. Consequently, it will be referred to as the fractionator.

The fractionator is nominally 2 in. in diam and 9 ft in height. Appendix A provides a detailed description of the fractionator, as well as the peripherals associated with the column, including refrigeration, heat exchangers, instrumentation, etc.

3. FLOODING STUDIES

In the past, the Goodloe wire mesh packing⁶ (Fig. 1) used in the absorber and stripper columns has become distorted^{4,7} to the extent that the gas throughput of the two columns was reduced to ~15% of that predicted by the manufacturer. The compressibility of the packing and the possible reduced throughput due to this compression have revealed the desirability of a random (i.e., noncompressible) packing with comparable throughput and mass transfer characteristics.

Toward this end, the 2-in. fractionator column was filled to a height of 8.9 ft with PRO-PAK packing⁸ (Fig. 2). As with the other

Fig. 1. Goodloe packing.

Fig. 2. PRO-PAK packing.

two columns, the packing was installed in three sections located between the intracolumn samplers (Fig. A-1, Appendix A). A flat wire mesh distributor along with a conical (point upward) wire mesh packing support was installed above and below each section of packing. The PRO-PAK packing is a random packing consisting of thin 0.24-in. by 0.24-in. nickel units. The metal has 1024 holes per square inch and is shaped into half cylinders with corners or edges bent inward to prevent nesting of one piece within another. The holes are not clean cut and have a protrusion (or burr) extending from one side.

The flooding studies were performed using saturated CO_2 with the column operating in total reflux (i.e., no off-gas). The column was considered to be flooded when the pressure drop across the column became greater than 10 in. H_2O . The flooding points were obtained by increasing the boilup rate, at a fixed pump rate, until the desired pressure drop across the column was obtained. A detailed discussion of the flooding point determination is shown in Appendix B.

A summary of the nominal flooding points are presented in Table 1. The experimental flooding data was fit to an equation of the following form:

$$\bar{y} = A + B(\bar{x}) + C(\bar{x})^2, \quad (1)$$

where

$$\bar{y} = \log \left[\frac{U_m^2 \rho_G \mu_L^{0.2}}{g \rho_L} \right],$$

$$\bar{x} = 2.0 + \log \left[\frac{L}{G} \sqrt{\rho_G / \rho_L} \right],$$

Table 1. Summary of PRO-PAK flooding data

Column pressure (atm)	Average column temperature (°C)	$\frac{L}{G} \sqrt{\frac{\rho_G}{\rho_L}}$	$\frac{U_m^2 \rho_G \mu_L^{0.2}}{g \rho_L}$
20	-18.8	1.44	0.0000248
20	-18.8	2.26	0.0000155
20	-18.8	3.91	0.0000075
20	-19.0	3.68	0.0000083
20	-19.1	5.20	0.0000057
20	-18.0	1.84	0.0000194
20	-18.2	2.87	0.0000117
20	-18.2	4.52	0.0000066

$$A = -2.738,$$

$$B = -0.607,$$

$$C = -0.118,$$

$$U_m = \text{vapor velocity, ft/sec,}$$

$$\rho_G = \text{vapor-phase density, lb/ft}^3,$$

$$\mu_L = \text{liquid-phase viscosity, cP,}$$

$$g = \text{constant, } 32.2 \text{ ft/sec}^2,$$

$$\rho_L = \text{liquid-phase density, lb/ft}^3,$$

$$L = \text{liquid rate, lb/hr}\cdot\text{ft}^2,$$

$$G = \text{vapor rate, lb/hr}\cdot\text{ft}^2.$$

Figure 3 presents a comparison between the experimentally determined PRO-PAK and Goodloe⁴ flooding correlations. The figure shows that PRO-PAK has approximately 60% the throughput capacity of the Goodloe packing.

Figure 4 presents a comparison between the flooding data obtained (as well as the resulting empirical correlation) and the predicted correlation of the manufacturer. The discrepancy between the experimental and predicted throughput is not a major concern because the manufacturer's correlation,⁹

$$G = 270(\rho_L)^{0.58} (\rho_G)^{0.42}, \quad (2)$$

where

$$G = \text{mass vapor velocity, lb/hr}\cdot\text{ft}^2,$$

$$\rho_L = \text{density of liquid phase, lb/ft}^3,$$

$$\rho_G = \text{density of vapor phase, lb/ft}^3,$$

Fig. 3. A comparison between PRO-PAK and Goodloe experimental flooding curves.

Fig. 4. A comparison between the experimental and predicted PRO-PAK flooding curves.

disagrees with the accepted correlations of Sherwood¹⁰ and Eckert¹¹ which state that flooding velocity is a function of liquid and vapor rate. This relationship has also been seen experimentally in the KALC facility.⁴ Of some concern, however, is the fact that the experimental throughput for both types of packing is at most 50% of the throughput predicted by the manufacturers. Experimental throughputs approaching the predicted values have been obtained previously for columns packed with Goodloe packing using air-water¹² and air--Freon-12 (see ref. 13) systems. This indicates that perhaps a fundamental physical property is missing from the flooding correlation which would account for this discrepancy. One such variable might be the liquid surface tension (σ) of the system. For the air-water and air-Freon system, as well as the benzene-ethylene dichloride and N-heptane-methyl cyclohexane systems used in the manufacturers' correlation for Goodloe and PRO-PAK packing, respectively, the liquid surface tension was between 10 and 100 dynes/cm;^{14,15} the surface tension of liquid CO₂ is <1.0 dyne/cm.¹⁶ Investigations in this area have been limited. Newton¹⁷ varied the liquid surface tension of an air-water system by the introduction of controlled quantities of surfactants (Sterox SK) and found that the flooding velocity was a function of σ^3 .

Newton determined that flooding velocity decreased with decreasing values for liquid surface tension. This would tend to explain why the CO₂ data (low surface tension) is substantially less than the other systems (high surface tension), indicating that any generalized flooding correlation for PRO-PAK or Goodloe should include liquid surface tension as a variable.

4. MASS TRANSFER STUDIES

Although the addition of the third column gave the EES-ODF the capability of studying the overall performance of the three-column KALC system, the ability to operate a two-column system has been retained. Specifically, the system can be operated in one of three ways: (1) in a three-column mode with the absorber, fractionator, and stripper in series, (2) in a two-column mode with the absorber and fractionator in series, or (3) in a two-column mode with the absorber and stripper in series.

Experiments were performed using a $\text{CO}_2\text{-O}_2\text{-Kr}$ system at conditions similar to those in Campaign III so that a preliminary comparison between the mass transfer characteristics of the PRO-PAK and Goodloe packings could be made. Specifically, experiments were performed to compare the two packings during fractionation and stripping operations. For the mass transfer studies during stripping, the system was operated in a two-column mode consisting of the absorber and fractionator. For mass transfer studies during fractionation, a three-column system was used.

During the operations, the system feed-gas (consisting of >90% CO_2 , <10% O_2 , and tracer levels of ^{85}Kr) was compressed to a pressure slightly higher than the absorber operating pressure and then routed to the bottom of the absorber. As the gas travels up the absorber column, it is contacted with a countercurrent stream of liquid carbon dioxide. The liquid stream absorbs essentially all of the krypton and a large portion of the oxygen, as well. The solute-laden liquid is then routed to the top of the fractionator by an operating pressure differential

(nominally 2 atm) between the two columns. In the fractionator column, the dissolved gas is distilled and leaves the column via the fractionator off-gas. The liquid leaving the bottom of the fractionator is relatively free of solute during stripping studies and is pumped to the top of the absorber as scrub liquid. During fractionator studies, the liquid is routed to the top of the stripper where the remaining dissolved gases are distilled and exit via the stripper off-gas. The liquid leaving the bottom of the stripper is then recycled back to the absorber to act as liquid scrub. In both stripping and fractionation experiments, the column off-gas streams were routed to a compressor where they combine to form the system feed-gas.

As with all mass transfer operations, some operating variables are designated as independent, and others are designated as dependent. Since the analysis was performed during "steady-state," the choice of independent-dependent operating variables became academic. Therefore, during the experiments, the independent variables were chosen on the basis of their ability to be maintained at a desired value.

The variable of primary concern in the operation of the columns was the liquid-to-vapor ratio, the liquid rate being determined by the pump rate. The vapor rate in the absorber column was fixed by controlling the feed-gas rate. The off-gas rate was set manually to complement the feed-gas rate (nominally 80% of feed rate). The absorber pressure and temperature were then allowed to seek their own levels. The fractionator column pressure was set by controlling the amount of refrigerant to the condenser. The fractionator temperature was allowed

to seek its own level. During fractionation studies, the stripper column pressure was set by controlling the amount of refrigerant going to the condenser. The reboiler heat input fixed the vapor rate. The column temperature was allowed to seek its own level. During stripping experiments, the 3-in. column was valved out of the system.

Once these independent variables were set, sufficient time was allowed for the entire system to reach "steady-state." Various temperatures, pressures, liquid holdups, stream concentrations, and flow rates were monitored during the experiments and were used to indicate when "steady-state" had been attained (i.e., no appreciable change in monitored variables for at least 2 hr). As the system approached steady-state operation, the various controllers were fine-tuned to alleviate any controller-induced oscillations. The wide variety of conditions studied caused this to be a time-consuming operation to the extent that controller fine-tuning became a major part of each experiment.

5. MASS TRANSFER DATA

Experiments of this magnitude are, by nature, complex and necessitate certain engineering assumptions in the analysis of the data collected. Although many of these assumptions are based on sound engineering principles, many are based on experience gained from operating the facility. For example, the composition analysis of the gas streams (>1% light-gas content) were found to be more reliable than the analysis of the liquid streams (<1% light-gas content). The

data analysis and the assumptions associated with it are discussed in detail in Appendix C.

5.1 Stripping Studies

The primary function of the stripper column is to "strip" the krypton from the liquid CO_2 and concentrate it in the column's off-gas. Consequently, all mass-transfer-related variables are based on krypton concentrations. A height of a transfer unit (HTU) is calculated based on resistance in the liquid phase using the following equations:¹⁸

$$\text{HTU} = Z/\text{NTU} ,$$

$$\text{NTU} = \frac{x_i - x_o}{(x - x^*)_{\text{lm}}} ,$$

and

$$(x - x^*)_{\text{lm}} = \frac{(x_o - x_o^*) - (x_i - x_i^*)}{\ln \frac{x_o - x_o^*}{x_i - x_i^*}} ,$$

where

Z = height of column packing, ft,

NTU = number of transfer units,

x_i = inlet liquid krypton concentration,

x_o = outlet liquid krypton concentration.

The asterisk quantities denote concentrations in equilibrium with the adjacent phase.

Two parameters, the column and overall decontamination factors, are calculated as a measure of the amount of krypton removed. The column decontamination factor (DF_c) is a measure of the column's efficiency, while the overall decontamination factor (DF_o) indicates the efficiency of the column with its condenser and reboiler. Table 2 presents a summary of the stripping data.

Although six stripper experiments were conducted, only four exhibit a high degree of confidence. Two experiments (C4-206 and C4-214) were suspect due to poor counting statistics. In the analysis of stripper data, the measured-column-vapor-inlet-krypton concentration is of extreme importance because it is the basis for all material balances associated with the column. Thus the reliability of this sample is of the utmost necessity. A statistical analysis of the measured count rate proved to be the most convenient test of reliability. The standard deviation for any sample is defined as¹⁹

$$\sigma_N = \sqrt{\sigma_c^2 + \sigma_\beta^2} ,$$

where

σ_N = standard deviation of the sample,

σ_c = standard deviation of total number of counts registered,

σ_β = standard deviation of the number of background counts registered.

Statistical analysis indicates that there is a 99.7% probability that the true count rate lies within $\pm 3 \sigma_N$ of the measured count rate.¹⁹

Table 2. Stripping mass transfer data based on krypton concentrations

Experiment No.	Pressure (psig)	Average column temperature (°C)	Pump rate (gpm)	L/V (mole/mole)	VK/L	HTU (ft)	DF _c	DF _o
C4-206	250.2	-22.6	0.415	7.8	1.33	0.42	397	1272
C4-214	250.1	-22.9	0.415	8.2	1.27	0.32	817	2873
C4-221	250.0	-22.5	0.415	8.8	1.18	0.27	1069	3529
C4-228	250.0	-22.3	0.415	8.9	1.18	0.21	1865	7127
C4-237	250.0	-22.7	0.415	8.6	1.22	0.28	832	2919
C4-244	249.7	-22.8	0.415	9.5	1.10	0.38	41	124

Thus a somewhat arbitrary decision was made to accept a sample whose count rate was such that the magnitude of $3 \sigma_N$ was $\leq 10\%$ of the actual counts registered (see Table 3).

Figure 5 presents a graphic comparison between the two types of packing. The plot of HTU vs L/V indicates that under similar conditions HTU (PRO-PAK) $\leq$ HTU (Goodloe).

Table 4 presents a more detailed comparison between the 2-in.-fractionator HTU and DF_c and the 3-in.-stripper HTU and DF_c (obtained in Campaign III).⁴ The 2-in.-fractionator HTU values are consistently lower than those for the 3-in. stripper. More importantly, the 2-in.-fractionator DF_c values are consistently higher than the 3-in.-stripper data, indicating that the fractionator has more stages even though it is shorter (8.88 ft vs 11.54 ft). These data indicate that the PRO-PAK packing has a lower HTU value than the Goodloe packing for stripping operations over the range of conditions studied.

5.2 Fractionation Studies

As stated earlier, the primary function of the fractionator is to remove light gases from the liquid. Consequently, its mass transfer parameters will be calculated using oxygen (the least volatile of the light gases of concern) concentrations. As with the stripping studies, the variables of interest are HTUs, DF_c s, and DF_o s. Table 5 presents a summary of the fractionation mass transfer experiments.

No readily available comprehensive method exists to determine the acceptability of an experiment. Consequently, the analysis of the limited data available becomes more difficult. Experiments C4-171,

Table 3. Statistical analysis of stripper
vapor inlet ^{85}Kr concentration

Experiment No.	Standard deviation (counts/min)	Relative standard deviation (%)
C4-206	7.02	5.0
C4-214	3.92	6.6
C4-221	3.33	3.2
C4-228	4.07	1.0
C4-237	6.30	0.8
C4-244	24.90	0.2

Fig. 5. Comparison of PRO-PAK and Goodloe stripping HTUs (based on krypton).

Table 4. Comparison between PRO-PAK and Goodloe krypton stripping data

Experiment No.	PRO-PAK column HTU (ft)	Goodloe column HTU ^a (ft)	PRO-PAK column DF _c	Goodloe column DF _c ^a
C4-221	0.27	0.60	1069	50
C4-228	0.21	0.59	1865	50
C4-237	0.28	0.52	832	80
C4-244	0.38	0.68	41	15

^aTaken from Campaign III data⁴ at similar stripping factors.

Table 5.. Fractionator mass transfer data based on oxygen concentrations

Experiment No.	Pressure (psig)	Average column temperature (°C)	Pump rate (gpm)	L/V (mole/mole)	VK/L	HTU (ft)	DF _c	DF _o
C4-171	280.3	-20.8	0.5533	16.1	1.14	1.19	7	130
C4-181	279.4	-22.8	0.5533	15.5	1.22	0.94	13	45
C4-188	278.8	-23.6	0.5533	15.7	1.19	2.40	4	15
C4-197	280.0	-23.0	0.5533	15.3	1.22	1.33	8	20
C4-251	279.7	-20.2	0.4150	15.4	1.19	0.19	5468	18904
C4-259	281.5	-20.5	0.4150	17.2	1.06	0.68	11	30
C4-266	280.0	-20.8	0.4150	17.0	1.08	0.59	15	47

C4-181, C4-188, and C4-197 were 8-hr runs, and steady-state had apparently not been reached at the time of sampling. As with the Campaign III fractionation studies, the temperature of the fractionator column was continuously changing during each of the 8-hr operations. This, of course, indicates that the oxygen profile is also changing in the column. It should be noted that the determination of nonsteady-state experiments is greatly enhanced by a large population of experiments. This is not the case here; thus it is conceivable, although unlikely, that further experiments would show that these four runs are within acceptable limits. For the purposes of this report, however, it will be assumed that they are unacceptable.

Experiments C4-251, C4-259, and C4-266 were 12-hr runs and showed a greater tendency toward maintaining constant readings for the various monitored variables, especially the fractionator column temperature. Figure 6 presents a comparison between the HTU values obtained for these three experiments using the 2-in. fractionator and those obtained using the 3-in. Goodloe column. As seen in the graph, experiments C4-259 and C4-266 appear to be comparable to the Goodloe data while experiment C4-251 does not. During the analysis of all experiments, the accuracy of the gas concentrations in the various streams is of the utmost importance. An off-gas material balance for experiment C4-251 gives a ^{85}Kr (the most reliably measured component) balance of 0.88 compared with 0.99 and 0.97 for experiments C4-259 and C4-266, respectively (see Table C-7, Appendix C). Consequently, the analysis of experiment C4-251 is suspect. Excluding experiment C4-251, the HTU

Fig. 6. Comparison of PRO-PAK and Goodloe fractionation HTUs (based on oxygen).

values obtained for PRO-PAK appear to fall within the scatter of the Goodloe data, indicating that $HTU (PRO-PAK) \approx HTU (Goodloe)$.

6. CONCLUSIONS AND RECOMMENDATIONS

The installation of the third column (i.e., fractionator) in the KALC facility is complete. The facility now has the capability of being operated in a one-, two-, or three-column mode. Thus the facility is no longer restricted solely to individual column performance experiments but can be used to determine performance data for the overall system, making the facility more versatile and potentially more productive.

In previous campaigns, the Goodloe wire mesh packing used in the absorber and stripper columns had become distorted to the extent that the gas throughput of the two columns was reduced to ~15% of that predicted by the manufacturer. The compressibility of the packing and the possible reduced throughput due to this compression has revealed the desirability of a random (i.e., noncompressible) packing with comparable throughput and mass transfer characteristics. Toward this end, the fractionator was filled with a new random packing known as PRO-PAK. Mass transfer and fluid dynamics data on the new packing have been collected.

Flooding experiments on the 2-in. fractionator with a CO_2 system produced the following flooding curve for PRO-PAK packing:

$$\bar{y} = A + B(\bar{x}) + C(\bar{x})^2 ,$$

where

$$\bar{y} = \log \left[\frac{U_m^2 \rho_G \mu_L^{0.2}}{g \rho_L} \right] ,$$

$$\bar{x} = 2.0 + \log \left[\frac{L}{G} \sqrt{\rho_G / \rho_L} \right] ,$$

$$A = -2.738,$$

$$B = -0.607,$$

$$C = -0.118,$$

U_m = vapor velocity, ft/sec,

ρ_G = vapor-phase density, lb/ft³,

μ_L = liquid-phase viscosity, cP,

g = constant, 32.2 ft/sec²,

ρ_L = liquid-phase density, lb/ft³,

L = liquid rate, lb/hr·ft²,

G = vapor rate, lb/hr·ft².

The empirical flooding curve for PRO-PAK indicated that PRO-PAK has ~60% the throughput capacity of Goodloe.

Experiments comparing the mass transfer characteristics of PRO-PAK and Goodloe packing during fractionation and stripping indicate that HTU (PRO-PAK) $\leq$ HTU (Goodloe). The pertinent operating variables and their ranges explored during the stripping mass transfer studies are the following:

<u>Variable</u>	<u>Range</u>
Pressure, psig	250 $\pm$ 0.5
Temperature, °C	-22.5 $\pm$ 0.4
Liquid-to-vapor ratio, mole/mole	7.8 - 9.5

The pertinent operating variables and their ranges explored during the fractionation mass transfer studies are the following:

<u>Variable</u>	<u>Range</u>
Pressure, psig	280 $\pm$ 2
Temperature, °C	-20.5 - -23.6
Liquid-to-vapor ratio, mole/mole	15.3 - 17.6

It should be noted that the comparison between PRO-PAK and Goodloe mass transfer characteristics is a preliminary one. Consequently, some important operating variables related to mass transfer, such as pressure and pump rate, were varied only slightly or in some cases not at all in the interest of expediency. The limited PRO-PAK mass transfer data restricts the confidence of the comparison, however, the data does indicate that a more extensive campaign to quantify the mass transfer characteristics of PRO-PAK packing is warranted.

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APPENDIX A: DETAILED COMPONENT DESCRIPTION

A.1 Fractionator Column

The fractionator column (see Fig. A-1) is installed in the EES-ODF facility in such a manner that it may be operated alone for specific tests, as a second column for stripping or fractionation studies, or as a fractionation column in a three-column system (see Fig. A-2). In this latter mode, liquid effluent from the absorber level tank enters the top of the fractionation column, where the light gases are removed through the condenser. The liquid, now containing essentially only ^{85}Kr , leaves the column via the reboiler to be fed to the stripper column.

The fractionator column is constructed of three 35-3/4-in.-long segments of 2-in. sched-40 pipe connected by expanded sections (surrounding each column segment). Each expanded section is composed of a 3-in. by 2-in. sched-40 reducer and a 3-1/16-in.-diam sample station (machined from bar stock) for gas and liquid sampling. In addition to sample lines and a fitting for a thermocouple and pressure tap, each expanded section contains a 1/2-in. sched-40 pipe welded through the shoulder of the reducer for solvent inlet.

The three-column segments are packed to a total height of 8.88 ft with commercially available PRO-PAK packing.* Each packed segment contains (1) two thermocouple taps equally spaced along the section, and (2) two pressure taps located 180° from the thermocouple taps. The process volume within the rectification column is 0.25 ft³.

* A product of the Scientific Development Company, State College, Pa.

1. Part No. 7 - BUFF WELD MALE CONNECTOR - SWAGelok
Part No. 200-1-2-3/16 (DRILL THRU FOR 1/8 THRU
EQUALS AFTER WEIDING.)
2. PART NO. 13 - MALE CONNECTOR - SWAGelok Part
No. 100-1-2-3/16 (DRILL THRU FOR 1/8 DIA TUBING)
- △ 3. Part No. 10 - BUTT WELD MALE CONNECTOR - SWAGelok
(CATALOG NO. 400-1-4W-3/16)

DESIGN CRITERIA:

1. MAXIMUM OPERATING PRESSURE - 600 P.S.I.
2. OPERATING TEMPERATURE RANGE - (-20°F TO +100°F)

MATERIALS:

1. MATERIAL SHALL BE STAINLESS STEEL TYPE 304, 304L, 316, 316L OR 307

2. MATERIAL CERT. DOCUMENTS ARE NOT REQUIRED UNLESS STATED OTHERWISE.

3. LAPPING SHALL BE CARVED WITH THE CORNER RADIUS OF 0.25 IN. DEEPS.

ITEM MATERIAL	ASME OR ASTM
(1) PIPE TUBING	ASME OR CCS
(2) BAR STOCK	479
(3) WELDING FITTINGS	403
(4) NUTS	479

TESTING:
1-PRESSURE TEST (HYDROSTATIC) AT 500 PSIG

NOZZLE SCHEDULE		
NOZZLE	SIZE	SERVICE
A	2" DIA	VAPOR INLET
B	2" DIA	VAPOR OUTLET
C	4" DIA	L.IQUID FEED
D	1/4" DIA	GAS SERVICE
E	1/4" DIA	L.IQUID SAMPLE
F	1/4" DIA	PRESSURE TAP
G	1/4" DIA	"
H	1/4" DIA	"
I	1/4" DIA	"
J	1/4" DIA	"
K	1/4" DIA	"
L	1/4" DIA	"
M	1/4" DIA	"
N	1/4" DIA	"
O	1/4" DIA	"
P	1/4" DIA	"
Q	1/4" DIA	"
R	1/4" DIA	"
S	1/4" DIA	"
T	1/4" DIA	"
U	1/4" DIA	"
V	1/4" DIA	"
W	1/4" DIA	"
X	1/4" DIA	"
Y	1/4" DIA	"
Z	1/4" DIA	"
AA	1/4" DIA	"
AB	1/4" DIA	"
AC	1/4" DIA	"
AD	1/4" DIA	"
AE	1/4" DIA	"
AF	1/4" DIA	"
AG	1/4" DIA	"
AH	1/4" DIA	"
AI	1/4" DIA	"
AJ	1/4" DIA	"
AK	1/4" DIA	"
AL	1/4" DIA	"
AM	1/4" DIA	"
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AQ	1/4" DIA	"
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AS	1/4" DIA	"
AT	1/4" DIA	"
AU	1/4" DIA	"
AV	1/4" DIA	"
AW	1/4" DIA	"
AX	1/4" DIA	"
AY	1/4" DIA	"
AZ	1/4" DIA	"
BA	1/4" DIA	"
BB	1/4" DIA	"
BC	1/4" DIA	"
BD	1/4" DIA	"
BE	1/4" DIA	"
BF	1/4" DIA	"
BG	1/4" DIA	"
BH	1/4" DIA	"
BI	1/4" DIA	"
BJ	1/4" DIA	"
BK	1/4" DIA	"
BL	1/4" DIA	"
BM	1/4" DIA	"
BN	1/4" DIA	"
BO	1/4" DIA	"
BP	1/4" DIA	"
BQ	1/4" DIA	"
BR	1/4" DIA	"
BS	1/4" DIA	"
BT	1/4" DIA	"
BU	1/4" DIA	"
BV	1/4" DIA	"
BW	1/4" DIA	"
BX	1/4" DIA	"
BY	1/4" DIA	"
BZ	1/4" DIA	"
CA	1/4" DIA	"
CB	1/4" DIA	"
CC	1/4" DIA	"
CD	1/4" DIA	"
CE	1/4" DIA	"
CF	1/4" DIA	"
CG	1/4" DIA	"
CH	1/4" DIA	"
CI	1/4" DIA	"
CJ	1/4" DIA	"
CK	1/4" DIA	"
CL	1/4" DIA	"
CM	1/4" DIA	"
CN	1/4" DIA	"
CO	1/4" DIA	"
CP	1/4" DIA	"
CQ	1/4" DIA	"
CR	1/4" DIA	"
CS	1/4" DIA	"
CT	1/4" DIA	"
CU	1/4" DIA	"
CV	1/4" DIA	"
CW	1/4" DIA	"
CX	1/4" DIA	"
CY	1/4" DIA	"
CZ	1/4" DIA	"
DA	1/4" DIA	"
DB	1/4" DIA	"
DC	1/4" DIA	"
DD	1/4" DIA	"
DE	1/4" DIA	"
DF	1/4" DIA	"
DG	1/4" DIA	"
DH	1/4" DIA	"
DI	1/4" DIA	"
DJ	1/4" DIA	"
DK	1/4" DIA	"
DL	1/4" DIA	"
DM	1/4" DIA	"
DN	1/4" DIA	"
DO	1/4" DIA	"
DP	1/4" DIA	"
DQ	1/4" DIA	"
DR	1/4" DIA	"
DS	1/4" DIA	"
DT	1/4" DIA	"
DU	1/4" DIA	"
DV	1/4" DIA	"
DW	1/4" DIA	"
DX	1/4" DIA	"
DY	1/4" DIA	"
DZ	1/4" DIA	"

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TOLERANCES UNLESS OTHERWISE SPECIFIED:	NO.		REVISONS				DATE	APPRO	APP
FRACTIONS * $\frac{1}{16}$	DESIGN	DATE	SUBMITTED	DATE	APPROVED	DATE			
DECIMALS * 2.05	FORWARDED $7-2-75$		RECEIVED $7-4-75$						
ANGLES * —	DRAWN	DATE	APPROVED	DATE	APPROVED	DATE			
SCALE: 1:2.5	CHECKED $7-1-75$		APPROVED		DATE	APPROVED	DATE		
	DATE		DATE		DATE	DATE			
	WATERMAN 181275								

REGISTRATION COMPANY - SMALL BUSINESS		CD-100
FEDERAL SERVICE		CD-100
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REFERENCE DRAWINGS		NUMBER
OAK RIDGE NATIONAL LABORATORY OPERATED BY UNION CARBIDE CORPORATION OAK RIDGE, TENNESSEE		
EES	DIVISION OF RESEARCH	BUILDING NO. 1501
REACTOR DESIGN COLUMN		
ASSEMBLY AND DETAILS		
DESIGNED BY <i>W. L. Fanning</i>	CHECKED BY <i>J. B. Fanning</i>	APPROVED BY <i>[Signature]</i>
DATE <i>MAY 1960</i>	CD <i>12106</i>	SHEET <i>8</i>

APPROVED FOR CONSTRUCTION

Fig. A-1. Detailed engineering drawing of fractionator column.

Fig. A-2. The Experimental Engineering Section Off-Gas
Decontamination KALC Facility.

The column is designed for service at pressures up to 600 psig at temperatures from -40 to +100°F and has been hydrostatically pressure-tested at 900 psig. The off-gas line is provided with a 1-in. 550-psig (nominal) rupture disc and a 1-in. 600-psig (nominal) relief valve.

A.2 Fractionator Reboiler

Vapor flow in the fractionator column is provided by vaporization of part of the column liquid bottoms in the reboiler (see Fig. A-3). The unvaporized portion of the liquid bottoms passes from the reboiler to the stripper column (during three-column operations) or to the solvent tank (in one- or two-column operations). The reboiler has a rated heating capacity of 5 kW (electrical immersion heater, General Electric No. 20612) and a process volume of $\sim 0.31 \text{ ft}^3$.

The reboiler is welded directly to the bottom of the fractionator column. Two 3-in. sched-40 tees with ~ 3 in. of 3-in. sched-40 connecting the branches form an "H" as the uppermost portion of the reboiler. One tee is welded to a 3-in. by 2-in. sched-40 concentric reducer which is welded to the fractionator column and forms the upper part of a liquid level reservoir (~ 8 in. of 3-in. sched-40 pipe welds to the other end of the tee and is fitted for a liquid level probe). Thermocouple taps are provided in the capped upper end of the second tee which connects to the 3-in. sched-40 reboiler shell proper. The 3-in. shell houses the immersion heater, which is flanged through the lower end of the shell. Liquid drains from the fractionator column into the level

REMARKS
1- Part No. 16: Immersion Heater - General Electric No. 20813
3 KW, 300psi, Stainless Steel
2- Part No. 18 But: Weld Nuts Connector - Swagelok No. 400-1-4W-316
(No Drill Thru Pipe After Welding.)

NOZZLE SCHEDULE		
NOZZLE	SIZE	SERVICE
A	1/8 NPT	Thermocouple
B	1/2" IPS Sch. 40	Process Fluid Outlet
C	1/2" NPT	Level Probe
D	2" IPS Sch. 40	Rectification Column Interface
E	1/8 NPT	Thermocouple
F	1/2" NPT	Thermocouple
G	1/2" Tubing 0.065" Wall	Pressure Tap
H	1/2" Tubing 0.065" Wall	Pressure Tap
J	3" 6000" Flange	Immersion Heater
K	1/2" Sch 40 Pipe	Blanked Off - Spare

GENERAL NOTES

DESIGN CRITERIA:

- 1-Maximum Operating Pressure : 600 psi
2-Operating Temperature Range : (-40°F) To (100°F)

"MATERIAL:

- MATERIAL
- 1-All Material Shall Be Stainless Steel, Type 304, 304L, 316, 316L, or 307
 - 2-Material Certification Documents Are Not Required Unless Otherwise Stated
 - 3-Material Shall Comply With The Following ASME Or ASTM Specs.

Type Material	ASME	Or ASTM
(a) Pipe Or Tubing	213, 312	Or 269
(b) Bar Stock	479	
(c) Welding Fitting	403	
(d) Plate	240	

WELDING:

- 1-Welds Shall Be Made In Accordance With AWS Welding Symbols
And WPS-302
2-All Welds Shall Be Inspected In Accordance With Mat.WR, 201,
Schedule E
3-Weld No. 1 To 19

TESTING:

- TESTING:
1-Pressure Test (Hydrostatic) At 900 psi

QUALITY ASSURANCE:

- QUALITY ASSURANCE
1-Level 3)

PRESSURE VESSEL REVIEW:

- No. 114

<u>RECTIFICATION COLUMN, ENGR FILM SHEET</u> " " " " " " <u>REFUEL STATION</u> <u>CONDENSER</u> <u>RECTIFICATION COLUMN ASS: 12" DIAM</u>		CD-470-5 CD-469-5 CD-468-5 CD-467-5
REFERENCE DRAWING		NUMBER
Oak Ridge National Laboratory OPERATED BY Union Carbide Corporation OAK RIDGE, TENNESSEE		
U.S. OFF-GAS FACILITY		BUILDING NO. 4521
RECTIFICATION COLUMN REBUILDER ASSEMBLY # DETAILS		
SUBMITTED BY <i>D. H. Hester</i>	RECEIVED <i>4/15/58</i>	APPROVED <i>S. D. Smith</i>
M	12706	CD 467
		FILE

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Fig. A-3. Detailed engineering drawing of fractionator column
reboiler.

reservoir, and finally into the lower part of the heater shell through a 1-1/2-in. sched-40 pipe.

Liquid from the fractionator column and vapor to the column pass through the "H" arrangement; during operations, the liquid level is maintained at approximately the weld junction of the reboiler shell and the 3-in. tee (to ensure that the heaters are totally immersed). Liquid from the lower section exits through a 1/2-in. sched-40 pipe in the side-wall 6 in. above the bottom flange.

The reboiler is designed for operation up to 600 psig over a temperature range of -40 to 100°F. The reboiler has been hydrostatically pressure tested at 900 psig.

A.3 Fractionator Column Reflux Station

Liquid reflux from the fractionator column condenser to the top of the column may be monitored through the fractionator column reflux station (see Fig. A-4). Vapor from the top of the column passes to the condenser, contacting the returning liquid only immediately above the column proper. The reflux rate is determined by the pressure drop produced across an orifice arrangement in the reflux return line, which is monitored with a 0-10 in. H₂O-pressure transmitter.

The reflux station consists of 89 in. of 1/2-in. sched-40 pipe. Liquid enters through a 1/2-in. sched-40 nozzle near the top of the unit and exits through the 3/4-in. x 0.065-in. wall orifice loop in the bottom, returning to the column extension piping. Pressure above the liquid in the station is equalized with the column pressure through a 1/2-in. line near the top of the column extension piping.

Fig. A-4. Detailed engineering drawing of the fractionator column reflux station.

The process volume for the station, including the orifice arrangement, is $\sim 0.01 \text{ ft}^3$. The unit is designed to operate at pressures up to 600 psig and at temperatures ranging from -40 to $+100^\circ\text{F}$ and has been hydrostatically pressure tested at 900 psig.

A.4 Fractionator Column Condenser

A portion of the overhead vapor from the fractionator column is condensed in the fractionator condenser (see Fig. A-5) and returned via the reflux station to the top of the column. The remaining vapor passes from the condenser to a surge tank where it may be used as recycle gas.

The unit consists of a 54-in. length of 8-in. sched-40 pipe as the outer shell. A 54-in. length of 6-in. sched-80 pipe is used as an inner shell to reduce volume and is wound with $\sim 110 \text{ ft}$ (2 coils) of 1/2-in.-OD by 0.065-in. wall-tubing. The two shells are welded together at each end using 8-5/8-in.-OD by 6-5/8-in.-ID by 1-in.-thick plates. These plates are provided with nozzles for process gas and liquid outlets (shell side) and for refrigerant inlet and outlet (tube side). Fittings are also provided for thermocouples. The gas inlet nozzle from the column proper is a 2-in. by 8-in. sched-40 pipe welded into the side of the outer shell 3 in. from the bottom of the condenser. The process volume is $\sim 0.47 \text{ ft}^3$. The anticipated cooling capacity is 12,000 Btu/hr at -40°F .

The fractionator condenser is designed to operate at pressures up to 600 psig at temperatures from -40 to 100°F . The vessel has been hydrostatically pressure tested at 900 psig shell side with 0 psig tube side and 450 psig tube side with 0 psig shell side.

A.5 Refrigeration

Refrigeration for the fractionator condenser is provided by a unit purchased from Tenney Engineering, Inc. (Tenney Job No. 8737). This unit (designated as Unit C) consists of a Copeland No. MRA-2-0500-TFD-460/3/60 compressor assembled with necessary operating and safety hardware as shown on Tenney Dwg.-8737-20-3. It is water-cooled and delivers -10,000 Btu/hr at -40°C through the evaporation and condensation of R-502 (an azeotropic mixture of R-22 and R-115).

A.6 Process Instrumentation and Controls

Installed in the system are 27 copper constantan thermocouples that are constantly monitored with 12-pt. temperature recorders. Fifteen of these thermocouples have been calibrated to $\pm 0.1^\circ\text{C}$ and can be monitored individually with a Leeds and Northrup, Type K-3 Universal Potentiometer (see Table A-1).

The level element for the reboiler is a 12-in. capacitance-type probe (Drexelbrook Model 508, Drexelbrook Eng. Co., Horsham, Penn.), electrically connected to a level indicating transmitter (Drexelbrook Model 408-1000) that has an output of 4 to 20 mA dc. This electrical signal is then converted to a 3-15 psi output through use of an electrical-pneumatic transducer (Conoflow Model T-25, Conoflow Corp. Blackwood, N.J.). This pneumatic signal is then used by a pneumatic controller (three-mode Foxboro Model 48P4, the Foxboro Co., Foxboro, Mass.) to operate the level control valve. This valve (Research Controls, Tulsa, Okla.) is pneumatically operated (3-15 psi) and has a 1/2-in. 316 SST body which contains an equal percentage "trim" ($C_V = 0.8$) and

Table A-1. Thermocouple listing

TR-6-1*	Fractionator column liquid sample, top
6-2*	Fractionator column top, upper section
6-3*	Fractionator column bottom, upper section
6-4*	Fractionator column liquid sample, upper middle
6-5*	Fractionator column top, middle section
6-6*	Fractionator column bottom, middle section
6-7*	Fractionator column liquid sample, lower middle
6-8*	Fractionator column top, lower section
6-9*	Fractionator column bottom, lower section
6-10*	Fractionator column liquid sample, bottom
6-11*	Fractionator reboiler, top
6-12*	Fractionator reboiler, middle
TR-7-1*	Fractionator reboiler, bottom
7-2*	Fractionator column, middle section, skin
7-3*	Fractionator column, bottom sampler, skin
7-4	Fractionator column liquid inlet, V-702
7-5	Fractionator column liquid or gas inlet, V-705
7-6	Fractionator condenser, vapor inlet
7-7	Fractionator condenser, liquid
7-8	Fractionator condenser, liquid outlet
7-9	Fractionator column reflux liquid, inlet
7-10	Fractionator column reflux liquid, outlet
7-11	Fractionator condenser, vapor
7-12	Fractionator condenser, vapor outlet
TR-8-1	Fractionator off-gas, rotameter
8-2	Fractionator condenser refrigerant, inlet
8-3	Fractionator condenser refrigerant, outlet
8-4	Spare
8-5	Spare
8-6	Spare
8-7	Spare
8-8	Spare
8-9	Spare
8-10	Spare
8-11	Spare
8-12	Spare
8-13	Spare
8-14	Spare
8-15	Spare
8-16	Spare

* Also individually monitored with L & N Potentiometer.

a Teflon-packed stem. This valve also contains an integral positioner and is of the air-to-close type.

The off-gas control valve is the same type as the level control valve but contains a "trim" of $C_v = 0.08$. This valve also uses a Foxboro pneumatic controller for operation. Automatic mode controls system pressure via process gas flow rate (with refrigeration control on manual) or manual mode for constant off-gas rate (with refrigeration controller on automatic for pressure control).

The off-gas flow rate is measured with a F & P tube-type rotameter (F & P Model 10A1735, Fisher and Porter Co., Warminster, Pa.) calibrated at the Y-12 Standards Laboratory.

Refrigeration control is also provided by a Foxboro pneumatic controller and an Evaporator Pressure Control valve (EPR-12, 1/2-in. nominal port size, Alco Controls, St. Louis, Mo.) that can be operated in the automatic or manual mode for the purpose described above.

The electrical system for the reboiler immersion heater consists of three "stacked" motor-driven variacs (type 12560, The Superior Electric Co., Bristol, Conn.), with an input of 360 V, and an output of 0 to 480 V and 28 A. The voltage and amperage are measured with two digital multi-meters (Data Precision Model 245, Data Precision Corp., Wakefield, Mass.) using 4:1 potential transformers and 6:1 current transformers (field mounted) so that no more than 120 volts is ever seen in the control room.

One Tigerman "Tele-Alarm" Annunciator (Tigerman Eng. Co., Chicago, Ill.) is employed in the control room to indicate component abnormalities during operations (see Table A-2).

Table A-2. Control-panel alarms (Panel 5)

Position	Description
5-1	Refrigeration unit "C" out
5-2	Fractionator reboiler lo-level
5-3	Fractionator reboiler hi-temperature
5-4	Fractionator column hi-pressure
5-5	Spare
5-6	Facility exhaust fans off

Other related system features and components necessary for operation of the fractionator column have been described previously.¹

A.7 Process Monitoring and Sampling

Krypton-85 and oxygen are continuously monitored in the rectification column off-gas stream. The ^{85}Kr is detected by a beta scintillation detector and the count rate is recorded on a strip chart recorder. A M-S-A Magnetic Oxygen Analyzer Model 802 (Mine Safety Appliances, Pittsburgh, Pa.) is used to measure the oxygen concentration. This analyzer is a paramagnetic wind type oxygen analyzer. The output from the analyzer is recorded on a strip chart recorder. Both krypton and oxygen monitors are used to indicate steady-state operations.

Samples may be taken from eight internal points of the column, the off-gas stream, and the reboiler liquid stream. The samplers are piped to the central sampling station where they can be analyzed for ^{85}Kr and light-gas compositions.^{2,3}

A.8 References for Appendix A

1. R. W. Glass et al., System Features and Component Descriptions for the Unit Operations Off-Gas Decontamination Facility, ORNL/TM-4596 (February 1975).
2. D. M. Levins et al., Monitoring and Analysis of Process Streams in a Kr-85 Off-Gas Decontamination System, ORNL/TM-4923 (July 1975).
3. D. J. Inman, Component Analysis of the Experimental Engineering Section Off-Gas Decontamination System, ORNL/GCR-77/21 (in preparation).

APPENDIX B: FLOODING EXPERIMENTS

B.1 Flooding Point Determination

The fractionator flooding studies were performed with saturated CO_2 . The fractionator column was operated in total reflux (i.e., no off-gas); thus the vapor rate (V) in the column was determined by the boilup rate in the reboiler:

$$V = (KW)(B), \quad (1)$$

where

KW = reboiler heat input, KW,

B = conversion factor at reboiler temperature (nominally 4.2 scfm/kW).

The liquid rate then becomes a function of pump rate and boilup:

$$L = (\text{GPM})A + (kW)(B), \quad (2)$$

where

GPM = pump rate, gal/min,

A = conversion factor at the pump suction temperature (nominally 77.0 scfm/gpm).

The fractionator column was considered to be flooded when the pressure drop across the column became greater than 10 in. H_2O . The flooding points were obtained by increasing the boilup rate, at a fixed pump rate, until the desired pressure drop across the column was obtained. The pressure taps are shown in Appendix A, Fig. A-1.

A summary of the flooding experiments are shown in Table B-1. Flooding points are usually obtained by plotting the log of the pressure drop vs the log of the gas velocity at a constant liquid velocity.¹ The flooding point is then determined as the point at which the pressure drop increases rapidly for a very small change in the gas velocity (or more practically, the point at which the curve undergoes a second slope change). The reliability of this method is greatly reduced by a small number of data points and a lack of visual confirmation of flooding. Consequently, a more convenient method of presenting the flooding point is to use a modified Sherwood, Shipley, and Holloway correlation.² The data are presented (see Fig. B-1) as a plot of

$$\frac{U_m^2 \rho_G \mu_L^{0.2}}{g \rho_L} \quad (\text{as the ordinate value})$$

vs

$$\frac{L}{G} \sqrt{\rho_G / \rho_L} \quad (\text{as the abscissa value}),$$

where

U_m = vapor velocity, ft/sec,

ρ_G = vapor-phase density, lb/ft³,

μ_L = liquid-phase viscosity, cP

g = constant, 32.2 ft/sec²,

ρ_L = liquid-phase density, lb/ft³,

L = liquid rate, lb/hr·ft²,

G = vapor rate, lb/hr·ft².

Table B-1. Summary of flooding data

Liquid pump rate (gpm)	Liquid rate (scfm) ^a	Vapor rate (scfm) ^a	Average column temperature (°C)	ΔP across column (in. H ₂ O)	Flooded (f = yes)	L/V (mole/mole)	$\frac{L}{G} \sqrt{\rho_G / \rho_L}$	$\frac{U_m^2 \rho_G \mu_L^{0.2}}{g \rho_L}$
0.415	32.4	1.27	-19.0	2.3		26.5	6.01	0.0000011
		1.72	-18.9	2.4		19.8	4.49	0.0000020
		2.26	-18.8	2.7		15.3	3.48	0.0000034
		2.46	-18.9	2.9		14.2	3.22	0.0000040
		2.67	-18.8	3.1		13.1	2.97	0.0000048
		2.95	-18.8	3.2		12.0	2.72	0.0000058
		3.65	-18.9	3.7		9.9	2.25	0.0000089
		4.22	-18.8	4.3		8.7	1.97	0.000012
		4.76	-18.8	4.9		7.8	1.77	0.000015
		5.41	-18.8	6.2		7.0	1.59	0.000020
		5.82	-18.8	7.2		6.6	1.50	0.000023
		6.36	-18.8	>10.0	f	6.1	1.38	0.000027
		0.73	-19.0	2.1		45.4	10.30	0.0000004
		0.73	-20.1	2.4		59.9	13.60	0.0000004
0.553	43.0	1.27	-19.3	2.7		34.9	7.90	0.0000011
		1.85	-19.3	3.0		24.2	5.50	0.0000023
		2.34	-18.8	3.5		19.4	4.40	0.0000037
		3.08	-18.8	4.3		15.0	3.40	0.000006
		4.10	-18.8	5.5		11.5	2.61	0.000011
		4.67	-18.8	8.6		10.2	2.32	0.000015
		4.92	-18.8	>10.0	f	9.7	2.20	0.000016
		0.53	-18.8	2.3		82.1	18.63	0.0000002
0.692	53.8	0.98	-19.8	2.6		55.9	12.69	0.0000006
		1.27	-19.4	3.1		43.3	9.83	0.0000011
		0.78	-19.4	2.6		69.6	15.80	0.0000004
		1.93	-18.8	3.8		28.9	6.56	0.0000025
		2.46	-18.8	5.4		22.9	5.20	0.0000064
		3.12	-18.8	7.0		18.2	4.13	0.0000066
		3.53	-18.8	>10.0	f	16.2	3.68	0.0000084

Table B-1 (cont'd)

Liquid pump rate (gpm)	Liquid rate (scfm) ^a	Vapor rate (scfm) ^a	Average column temperature (°C)	ΔP across column (in. H ₂ O)	Flooded (f = yes)	L/V (mole/mole)	$\frac{L}{G} \sqrt{\rho_G / \rho_L}$	$\frac{U_m^2 \rho_G \mu_L^{0.2}}{g \rho_L}$
0.692	53.5	2.67	-19.0	6.0		21.1	4.78	0.0000048
		3.32	-19.0	8.5		17.1	3.89	0.0000074
		3.57	-19.0	>10.0	f	16.0	3.64	0.0000085
		3.49	-19.0	<10.0		16.3	3.71	0.0000081
0.830	64.0	2.30	-19.1	7.2		28.8	6.55	0.000004
		2.71	-19.1	8.5		24.6	5.59	0.000005
		2.99	-19.1	>10.0	f	22.4	5.09	0.000006
		2.87	-19.1	<10.0		23.3	5.30	0.000006
0.484	37.6	4.31	-20.4	6.8		9.7	2.17	0.000013
		5.00	-18.0	9.1		8.5	1.97	0.000016
		5.49	-17.9	>10.0	f	7.9	1.83	0.000020
		5.41	-17.9	<10.0		8.0	1.85	0.000019
0.623	48.0	3.36	-18.9	7.3		15.3	3.48	0.000007
		4.30	-18.2	>10.0	f	12.2	2.83	0.000012
		4.17	-18.2	<10.0		12.5	2.90	0.000011
0.761	58.6	3.35	-18.2	>10.0	f	18.5	4.28	0.000007
		3.01	-18.2	8.8		20.5	4.75	0.000006

^aSTP = 70°F, 1 atm.

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Fig. B-1. Flooding point determination.

This correlation differs from that of Sherwood, Shipley, and Holloway in that the packing factor normally included in the ordinate value has been neglected. The nominal flooding point for each liquid flow rate was determined as the midpoint between the lowest gas flow that caused flooding and the highest gas flow for which the column was not flooded.

Figures B-2 and B-3 present the resulting data as plots of ΔP vs G/ϕ and G/ϕ vs $L\phi/G$, respectively,

where

ΔP = pressure drop, in H_2O /ft packing,

G = vapor rate $lb/hr \cdot ft^2$,

$\phi = \rho_G/0.075$,

ρ_G = gas-phase density, lb/ft^3 ,

L = liquid rate, $lb/hr \cdot ft^2$.

B.2 References for Appendix B

1. A. S. Faust et al., Principles of Unit Operations, pp. 269-70, Wiley, New York, 1970.
2. R. H. Perry and C. H. Chilton, Chemical Engineers Handbook, 5th ed., p. 18-22, McGraw-Hill, New York, 1973.

Fig. B-2. PRO-PAK loading curve.

Table B-2. Data used in Figure B-2

ΔP (in. H_2O /ft)	G/ϕ (lbs/hr·ft ²)	Flooded (f = yes)
0.237	33	
0.253	57	
0.270	76	
0.304	101	
0.327	109	
0.349	120	
0.360	131	
0.417	162	
0.479	189	
0.552	212	
0.698	241	
0.811	259	
>1.13	284	f
0.259	24	
0.270	33	
0.304	56	
0.338	81	
0.394	104	
0.484	138	
0.619	184	
0.969	208	
>1.13	219	f
0.293	35	
0.293	43	
0.349	57	
0.428	86	
0.608	109	
0.788	138	
>1.13	157	f

Fig. B-3. PRO-PAK flooding curve.

Table B-3. Data used
in Figure B-3

G/ϕ (lb/hr·ft ²)	$L\phi/G$
130	152
141	130
148	115
157	107
188	82
214	66
242	53
272	43

APPENDIX C: MASS TRANSFER CALCULATIONS

The data analysis and the assumptions associated with it have been discussed in great detail previously in Campaign III.¹ Consequently, the data-analysis model will be discussed here only as it contributes to the clarity of the data presented.

C.1 Stripping Studies

The analysis of the two-column stripping studies is similar to the analysis of the stripping data in Campaign III except that the 2-in. fractionator has been substituted for the 3-in. stripper. Consequently, only the model highlights will be presented here. To aid in the analysis, a schematic of the two-column system is shown in Fig. C-1. The streams are as follows:

- ① Absorber column liquid feed,
- ② System feed-gas,
- ③ Liquid leaving the absorber packing,
- ④ Vapor entering the absorber packing,
- ⑤ Liquid entering the absorber packing,
- ⑥ Vapor leaving the absorber packing,
- ⑦ Liquid feed to fractionator column,
- ⑧ Absorber column off-gas,
- ⑨ Fractionator column off-gas,
- ⑩ Liquid leaving the fractionator packing,
- ⑪ Vapor entering the fractionator packing,

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Fig. C-1. Schematic of the two-column system used in the stream analysis.

- ⑫ Vapor leaving the fractionator packing, and
- ⑬ Liquid entering the fractionator packing.

During the experiments, only streams 2, 8, and 9 could be identified by both flow rate (via calibrated rotameters) and composition (via sampling and analysis). The resulting component material balance from these streams gave an average of 99 and 97% for oxygen and krypton, respectively. Consequently, the composition normalization used in Campaign III was not used here. The off-gas data and resulting material balances are shown in Table C-2.

The determination of flow rates and compositions of streams 1, 7, 10, 11, 12 and 13 is identical to that in Campaign III. The results of these calculations are shown in Tables C-3 through C-6.

With the major streams defined as to flow rate and composition, the pertinent mass transfer information can now be calculated. The "stripper" column decontamination factor (DF_c) becomes

$$DF_c = \frac{x_{13}}{x_{10}}, \quad (1)$$

where x_{13} and x_{10} are the krypton concentrations of the indicated streams. An overall decontamination factor (DF_o) is calculated to include the effects of the reboiler and column condenser by the following equation:

$$DF_o = \frac{Q_7 x_7}{Q_1 x_1}, \quad (2)$$

Table C-2. Gas stream values for stripper experiments

Experiment No.	Measured ^{85}Kr concentration (ppb)			Measured O_2 concentration (%)			Measured flow rate (scfm) ^a			Balance factor	
	Stream 2	Stream 8	Stream 9	Stream 2	Stream 8	Stream 9	Stream 2	Stream 8	Stream 9	O_2	Kr
C4-206	5.9	0.005	11.6	4.5	2.8	7.9	4.04	2.49	1.47	1.02	0.72
C4-214	5.0	0.005	14.1	4.0	2.7	6.7	4.20	2.72	1.47	1.04	0.99
C4-221	4.6	0.005	12.7	5.6	3.1	9.5	4.19	2.71	1.46	0.96	0.97
C4-228	89.7	0.05	249.7	16.0	9.5	25.0	4.25	2.71	1.49	0.93	0.98
C4-237	68.8	0.14	206.6	12.3	7.6	20.2	4.10	2.58	1.53	1.00	1.12
C4-244	46.9	0.36	138.8	7.7	4.6	12.5	4.13	2.63	1.46	0.96	1.05

^aSTP = 1 atm, 70°F.

Table C-3. Equivalent pump rate (stream 1)
for stripper experiments

Experiment No.	Pump rate (gpm)	Temperature (°C)	Stream 1 (scfm) ^a
C4-206	0.415	-29.4	32.45
C4-214	0.415	-29.4	32.45
C4-221	0.415	-29.3	32.43
C4-228	0.415	-29.3	32.43
C4-237	0.415	-29.0	32.39
C4-244	0.415	-30.5	32.60

^aSTP = 1 atm, 70°F.

Table C-4. Calculated fractionator liquid feed
for stripping experiments

Experiment No.	Q_7 (scfm) ^a	Measured concentrations		Calculated concentrations	
		O ₂ (%)	Krypton (ppb)	O ₂ (%)	Krypton (ppb)
C4-206	33.91	0.27	0.72	0.33	0.70
C4-214	33.92	0.39	0.63	0.27	0.62
C4-221	33.89	0.38	0.59	0.55	0.57
C4-228	33.92	0.39	11.15	1.24	11.23
C4-237	33.92	0.27	8.24	0.91	8.31
C4-244	34.06	0.26	5.66	0.58	5.66

^aSTP = 1 atm, 70°F.

Table C-5. Calculated fractionator bottom information for stripping studies

Experiment No.	Calculated flow rates (scfm) ^a		Measured concentrations			Calculated concentrations	
	Stream 10	Stream 11	Stream 1	Stream 10	Stream 11	Stream 1	Stream 10
			Krypton (ppb)	Krypton (ppb)	Krypton (ppb)	Krypton (ppb)	Krypton (ppb)
C4-206	37.16	4.74	0.002	0.004	0.0062	0.0006	0.0013
C4-214	36.95	4.49	0.002	0.002	0.002	0.0002	0.0005
C4-221	36.59	4.14	0.0005	0.002	0.004	0.0004	0.0008
C4-228	36.54	4.11	0.001	0.003	0.017	0.0016	0.003
C4-237	36.67	4.29	0.006	0.004	0.03	0.0029	0.0061
C4-244	36.41	3.85	0.04	0.08	0.5	0.048	0.096

^aSTP = 1 atm, 70°F.

Table C-6. Calculated fractionator top information for stripping studies

Experiment No.	Calculated flow rate (scfm) ^a		Measured concentration		Calculated concentration
			Stream 12	Stream 13	Stream 13
	Stream 13	Stream 12	Krypton (ppb)	Krypton (ppb)	Krypton (ppb)
C4-206	37.16	4.74	4.05	0.49	0.52
C4-214	36.95	4.45	3.30	0.39	0.40
C4-221	36.59	4.14	3.43	0.36	0.39
C4-228	36.54	4.11	55.97	7.43	6.30
C4-237	36.67	4.29	44.13	5.26	5.16
C4-244	36.41	3.85	36.55	3.65	3.90

^aSTP = 1 atm, 70°F.

where

Q_7 = flow rate of stream 7, scfm,

x_7 = krypton concentration of stream 7, ppb,

Q_1 = flow rate of stream 1, scfm,

x_1 = krypton concentration of stream 1, ppb.

A height of a transfer unit (HTU) value is calculated using:²

$$HTU = Z/NTU \quad (3)$$

$$NTU = \frac{x_i - x_o}{(x - x^*)}_{\ell m} \quad (4)$$

and

$$(x - x^*)_{\ell m} = \frac{(x_o - x_o^*) - (x_i - x_i^*)}{\ln \left[\frac{x_o - x_o^*}{x_i - x_i^*} \right]}, \quad (5)$$

where

Z = height of column packing, ft,

NTU = number of transfer units,

x_i = inlet liquid Kr concentration,

x_o = outlet liquid Kr concentration,

x_i^* = Kr concentration in equilibrium with the inlet gas,

x_o^* = Kr concentration in equilibrium with the outlet gas.

For stripping studies,

Z = 8.88 ft,

x_i = krypton concentration of stream 13, ppb,

- x_o = krypton concentration of stream 10, ppb,
 x_i^* = krypton concentration of stream 12 (ppb) divided by K_i ,
 x_o^* = krypton concentration of stream 11 (ppb) divided by K_o ,
 K_o = krypton equilibrium constant based on pressure and temperature at the bottom of the column,*
 K_i = krypton equilibrium constant based on pressure and temperature at the top of the column.

A column stripping factor (VK/L) is defined by:

$$\frac{VK}{L} = \frac{Q_{11} K_{avg}}{Q_{10}}, \quad (6)$$

where

- Q_{11} = flow rate of stream 11, scfm
 $K_{avg} = (K_i + K_o)/2$,
 Q_{10} = flow rate of stream 10, scfm.

C.2 Fractionation Studies

The data analysis for fractionation studies is similar to that of the stripping experiments, but the system now contains all three columns. To aid in the analysis, a schematic of the three-column system is shown in Fig. C-2. The streams are similar to those in Fig. C-1, with the following additions:

* All equilibrium constants used in this report are calculated using the Glass model.³

Fig. C-2. Schematic of three-column system used in data analysis.

- ⑭ Liquid feed to the stripper
- ⑮ Stripper off-gas.

The off-gas component material balance data (streams 2, 8, 9, 15) are shown in Table C-7. Again, composition normalization was omitted due to the average material balance values of 1.04 and 0.97 for O_2 and Kr, respectively.

The determination of flow rates and compositions of streams 1, 7, 10, 11, 12, and 13 is identical to that in the stripping experiments with the following modification, stream 1 (i.e., pump rate) is no longer equal to the liquid leaving the fractionator reboiler (stream 14). Stream 14 is defined by an overall material balance around the stripper column:

$$Q_{14} = Q_{15} + Q_1 ,$$

where

Q_{15} = stripper off-gas, scfm, and

Q_1 = liquid leaving the stripper reboiler, scfm.

The calculations for the other streams proceed as in the Campaign III analysis substituting Q_{14} for Q_1 whenever the liquid rate leaving the fractionator reboiler is needed. The results of these calculations are shown in Tables C-8 through C-11.

The fractionator column DF_c , HTU, and stripping factor are calculated using Eqs. (1), (3), (4), (5), and (6) with the following modifications:

Table C-7. Gas-stream values for fractionation studies

Experiment No.	Measured ⁸⁵ Kr concentration (ppb)				Measured O ₂ concentration (%)				Measured flow rate (scfm) ^a				Balance factor	
	Stream 2	Stream 8	Stream 9	Stream 15	Stream 2	Stream 8	Stream 9	Stream 15	Stream 2	Stream 8	Stream 9	Stream 15	O ₂	Kr
C4-171	26.10	15.2	--	54.7	4.7	2.6	--	9.5	5.27	3.68	--	1.61	1.00	1.05
C4-181	11.44	1.74	28.92	24.62	11.9	7.3	1.14	25.2	5.97	3.35	0.77	1.69	0.96	1.02
C4-188	10.74	0.11	31.89	19.10	6.2	3.9	2.1	20.0	5.76	3.22	0.78	1.70	1.35	0.93
C4-197	9.80	0.013	29.50	17.50	9.9	6.0	2.4	20.4	5.65	3.02	0.78	1.67	0.96	0.94
C4-251	27.70	0.35	52.50	54.20	6.4	5.4	0.8	10.9	5.33	2.82	0.74	1.67	1.00	0.88
C4-259	22.50	0.13	60.40	42.50	7.4	6.7	1.4	11.6	5.24	2.67	0.74	1.68	0.99	0.99
C4-266	20.70	0.09	56.00	33.70	7.3	6.3	1.5	12.0	5.17	2.59	0.74	1.67	0.99	0.97

^aSTP = 70°F, 1 atm.

Table C-8. Equivalent pump rate
for fractionation experiments

Experiment No.	Pump rate (gpm)	Temperature (°C)	Q ₁ (scfm) ^a
C4-171	0.5533	-27.3	42.87
C4-181	0.5533	-26.3	42.69
C4-188	0.5533	-27.9	42.98
C4-197	0.5533	-24.1	42.28
C4-251	0.4150	-29.8	32.50
C4-259	0.4150	-29.9	32.52
C4-266	0.4150	-29.8	32.50

^a STP = 1 atm, 70°F.

Table C-9. Calculated fractionator liquid feed
for fractionator experiments

Experiment No.	Q ₇ (scfm) ^a	Measured concentrations		Calculated concentrations	
		O ₂ (%)	Krypton (ppb)	O ₂ (%)	Krypton (ppb)
C4-171	44.48	0.06	4.66	0.35	1.84
C4-181	45.15	0.45	1.53	1.05	2.10
C4-188	44.69	0.41	1.34	0.80	1.28
C4-197	44.73	0.38	1.19	0.80	1.17
C4-251	34.91	0.40	3.35	0.54	3.71
C4-259	34.94	0.37	3.55	0.59	3.32
C4-266	33.24	0.31	3.17	0.64	3.12

^aSTP = 1 atm, 70°F.

Table C-10. Calculated bottom information for fractionation studies

Experiment No.	Calculated flow rates (scfm) ^a		Measured concentrations						Calculated concentrations			
	Stream 1C	Stream 11	Stream 14		Stream 10		Stream 11		Stream 14		Stream 10	
			O ₂	Kr	O ₂	Kr	O ₂	Kr	O ₂	Kr	O ₂	Kr
			(%)	(ppb)	(%)	(ppb)	(%)	(ppb)	(%)	(ppb)	(%)	(ppb)
C4-171	45.71	2.84	0.002	1.59	0.006	2.61	0.051	18.03	0.0028	1.94	0.0058	2.94
C4-181	46.46	3.00	0.025	0.60	0.063	1.07	0.435	7.15	0.024	0.77	0.05	1.18
C4-188	48.08	2.51	0.061	0.65	0.11	1.04	0.953	7.3	0.052	0.78	0.107	1.17
C4-197	46.02	2.56	0.05	0.53	0.1	0.90	0.739	6.3	0.041	0.68	0.086	1.04
C4-251	35.49	2.25	0.00005	1.27	0.0002	2.24	0.0005	15.4	0.00003	1.66	0.00006	2.53
C4-259	35.26	2.01	0.015	1.59	0.033	2.3	0.377	17.8	0.021	1.91	0.041	2.82
C4-266	34.53	2.03	0.007	1.37	0.021	2.17	0.252	16.4	0.014	1.76	0.028	2.62

^aSTP = 1 atm, 70°F.

Table C-11. Calculated fractionator top information for fractionator studies

Experiment No.	Calculated flow rate (scfm) ^a		Measured concentration				Calculated concentration	
			Stream 12		Stream 13		Stream 13	
			O ₂	Kr	O ₂	Kr	O ₂	Kr
	Stream 13	Stream 12	(%)	(ppb)	(%)	(ppb)	(%)	(ppb)
C4-171	45.71	2.84	0.64	23.5	0.04	2.74	0.04	3.28
C4-181	46.46	3.00	9.9	10.2	0.23	1.05	0.66	1.38
C4-188	48.08	2.91	5.6	8.7	0.25	1.01	0.39	1.26
C4-197	46.02	2.96	9.8	8.0	0.23	0.91	0.67	1.15
C4-251	35.49	2.25	4.9	21.5	0.23	2.44	0.31	2.92
C4-259	35.26	2.01	7.4	22.2	0.22	2.60	0.44	3.07
C4-266	34.53	2.03	6.7	19.9	0.18	2.30	0.41	2.83

^aSTP = 70°F, 1 atm.

- 1) $x_i = O_2$ concentration of liquid stream i,
- 2) $x_i^* = O_2$ concentration of vapor in equilibrium with x_i ,
- 3) $K_o = O_2$ equilibrium constant based on pressure and temperature at the bottom of the column,
- 4) $K_i = O_2$ equilibrium constant based on pressure and temperature at the top of the column.

The overall DF is calculated using Eq. (2) with the following modifications:

- 1) $x_i = O_2$ concentration of stream i,
- 2) Q_1 is replaced by Q_{14} .

C.3 References for Appendix C

1. T. M. Gilliam, V. L. Fowler, and D. J. Inman, Krypton Absorption in Liquid CO_2 (KALC): Campaign III in the Experimental Engineering Section Off-Gas Decontamination Facility, ORNL/TM-5655 (September 1977).
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