MASTER

K-1653

AEC RESEARCH AND DEVELOPMENT REPORT

APR 19 1966

ENGINEERING-SCALE STUDIES OF BORON ISOTOPE SEPARATION

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Date of Issue: April 15, 1966

Report Number: K-1653

Subject Category: Isotope Separation
TID-4500

ENGINEERING-SCALE STUDIES OF BORON ISOTOPE SEPARATION

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A B S T R A C T

Several disadvantages of boron isotope separation by the dimethyl ether process, which has been used on a production scale in the United States intermittently since 1945, led to engineering-scale studies of a more attractive alternative process based on a gas-liquid chemical exchange between boron trifluoride and a liquid complex formed between boron trifluoride and anisole. An engineering-scale test unit for the anisole process was operated essentially at total reflux, with reflux supplied by complex formation and dissociation in auxiliaries located, respectively, at the boron-11 and boron-10 ends of the separation column. Additionally, an anisole still was employed for continuous solvent purification. The 6-inch diameter isotopic exchange column was packed to a height of 13.5 feet with 5/8-inch Pall rings. During this program, pressures of 20, 26, and 35 psia., anisole flow rates of 1.17, 1.75, and 2.0 gpm., and temperatures from 63 to 105°F. were selected for the nineteen isotope separation tests. Correlations were developed for experimentally determined exchange column stage heights, which were found to range from 22 to 43 inches, depending upon the combination of operating conditions used, and for exchange column pressure drops, which varied between about 0.3 and 1.3 inches of water per foot of packing. One flooding test was performed at 28 psia. and 96°F. with the indicated flooding flow on an anisole basis being 2.7 gpm. Both complex formation and dissociation were carried out with very low boron losses. The total anisole degradation rates during the tests appeared to be equivalent to between 3 and 7% of the anisole inventory in the still and decomposer systems per day. The choice of materials of construction appeared satisfactory on the basis of recycle liquid analyses. Stainless steel 304L was the primary construction material; however, stainless steel 316 and nickel packing materials were employed for the exchange column and for the higher temperature still and decomposer sections, respectively. Successful anisole purification and drying methods, as well as general system operability, were demonstrated.

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ENGINEERING-SCALE STUDIES OF BORON ISOTOPE SEPARATION

Because of the large thermal neutron capture cross-section of boron-10, research on boron isotope separation methods was begun in early 1943 at Columbia University⁽¹⁶⁾. This research led to an equilibrium distillation process, using a gaseous mixture of boron trifluoride and dimethyl ether, which was first applied on a plant scale at facilities operated for the Manhattan District at Whiting, Indiana⁽⁵⁾, and later for the Atomic Energy Commission at Model City, New York⁽¹⁴⁾. Since the complex formed between boron trifluoride and dimethyl ether is volatile, it is not feasible to make a separation between the boron trifluoride and ether with ordinary, thermally refluxed operations. Furthermore, the system exhibits a relatively low effective single-stage separation factor, and flow is limited because of the need to operate at subatmospheric pressures in order to reduce operating temperatures to levels where the rates of ether degradation and solids formation are acceptable. With the dimethyl ether process, it has been found necessary to have a reboiler and condenser for each exchange column, and the single column height and operating throughput are further restricted because of the relatively small available pressure drop.

These failings of the dimethyl ether process led to laboratory searches at the Oak Ridge National Laboratory and elsewhere for a better process solvent. This work^(10,17) showed several advantages of using anisole (methyl phenyl ether) instead of dimethyl ether as the complexing agent. Single-stage separation tests were conducted to obtain basic data, and a small bench-scale recycle system, which was satisfactorily operated for a considerable period of time, yielded reasonable column separation performance data. The separation factor with anisole was found to be such as to allow almost twice the separation per stage. The total plant length required for a specified separation would thus be considerably reduced by using the anisole system, provided reasonable stage lengths are obtained in comparison with the dimethyl ether process when identical packing is used.

Plant operation with the anisole system can be conducted as a simple gas-liquid exchange in cascaded packed columns. Reboilers and condensers are not required for each column, liquid pumps can be used for intercolumn transfer, and at most three gas compressors would suffice to make up the operating pressure drop for a whole cascade. The boron trifluoride-anisole complex can be dissociated in the decomposer system by heating at easily attained temperatures, i.e., below 400°F., to supply both gas reflux to the boron-10 end of the system and anisole for recycle. Liquid complex refluxed to the boron-11 end of the system is formed in the recombiner system at temperatures below 125°F. by contacting the gas leaving the end column and anisole recycled from the decomposer. Cooling is required in the recombiner to remove the heat of reaction and to control

the temperature at about that used in the exchange column. The isotopic exchange columns can operate at temperatures of 70 to 105°F. Preferably, operating pressures are above atmospheric to minimize gas recompression requirements and to reduce the column diameter required for a given throughput. Both operating temperatures and pressures in the exchange columns can be varied over considerable ranges, but generally speaking, as noted later, temperatures between 86 and 104°F. and pressures from 20 to 35 psia. are favored.

Initial larger scale studies of the anisole process performed at the Model City plant were unsuccessful, primarily because of solvent deterioration due, apparently, to water inleakages and failure to purify the solvent continuously⁽⁹⁾. However, the Atomic Energy Commission's continued interest in further production of boron-10 led to authorization of more complete studies of the anisole system at the Oak Ridge Gaseous Diffusion Plant. The main facet of the work was the engineering-scale effort described in this report; however, the studies included laboratory- and bench-scale experiments designed to supply basic engineering data and to evaluate the problems of corrosion and solvent degradation.

The size of the engineering-scale effort was largely dictated by the need to obtain stage separation data on plant-scale packing. Previously made conceptual plant evaluations had led to the recommendation that a high throughput, intermediate efficiency packing, such as the 5/8-inch Pall ring* be tested. This type of packing is considered to be more economical for work on the contemplated production scale than higher efficiency but lower throughput, more expensive, smaller packings. The exchange column section diameter was set at 6 inches, and the packed height was chosen to be 13.5 feet with two intermediate redistribution sections. The test operating temperature range for the exchange column was set at 68 to 104°F. (i.e., 20 to 40°C.), with pressures between 20 and 35 psia. The required circulation rate for flooding tests had been estimated to be about 3.0 gpm. of complex. The auxiliaries were, however, sized for 4 gpm. of complex to assure an adequate flow.

Because of the uncertainties involved in the extrapolation of small-scale test results to full-scale plant design, the following areas were selected for investigation during the pilot plant tests: (a) HTU's and flooding data for this system for plant size packing; (b) solvent degradation in large-scale equipment at various temperatures and pressures; (c) decomposition of the boron trifluoride-anisole complex on a production scale with essentially complete stripping of the gas; (d) recombination of anisole with boron trifluoride tails on a production scale with low boron trifluoride losses; (e) effectiveness of and requirements for anisole drying and purification operations; and (f) suitability of the materials of construction chosen for the various units. The results of these tests are presented after discussions of basic system technology and design.

* Product of the U. S. Stoneware Company.

SUMMARY

Boron isotope separation with the anisole process is accomplished by countercurrent gas-liquid contacting in cascaded, packed columns. Chemical exchange between the boron isotopes in the two phases results in enrichment of the liquid phase in boron-10. In addition to the exchange columns, two auxiliary systems are required. The first is a decomposer, where the complex is dissociated by heating to supply both boron trifluoride gas reflux to the product end of the cascade and anisole for recycle. The stripped anisole, after distillation, is recirculated to the second auxiliary system, the recombiner, where it is reacted with boron trifluoride tails to form the liquid complex that is subsequently refluxed to the boron-11 end of the cascade. Cooling is required in the recombiner to remove the heat of reaction and to control the temperature at about that used in the exchange columns.

The pilot plant utilized all of the systems required for a production plant and the only basic difference in equipment size was, of course, the one arising from the use of a single exchange column rather than cascaded towers. Sizing of the equipment for the engineering-scale test unit was based on the criterion of testing plant-scale packing to the flooding point with exchange column operating temperatures in the range of 68 to 104°F. The packed height of the column was set at 13.5 feet of 5/8-inch Pall rings. When the equipment sizes were initially established, the predicted flooding flow rate was 2.1 gpm. of anisole, or 2.9 gpm. of complex, but accurate data on the viscosity and density of the complex were not available at that time. The auxiliaries were sized for 4 gpm. of complex to assure an adequate circulation rate.

Based on consideration of degradation and corrosion results from both laboratory- and bench-scale studies conducted prior to the pilot plant tests, it appeared that, while degradation rates were somewhat higher with stainless steel, stainless steel was more resistant than nickel to corrosive attack under anhydrous conditions. Thus, stainless steel was used for construction of the engineering-scale equipment and for packing in the low temperature columns. The more costly nickel packing was, however, used in the high temperature columns because it was felt that this would lessen degradation in these units.

Nineteen isotope separation tests were made, and the independent variables used were pressures of about 20, 26, and 35 psia., anisole flow rates of 1.17, 1.75, and 2.0 gpm., and temperatures from 63 to 105°F. The time required for each test was on the order of one day, and during the tests, samples were taken not only to determine the extent of isotope separation but also to assess the performances of the decomposer, the recombiner, and the still as well as the degree of anisole degradation.

The exchange column stage heights observed in these tests fell in the range of 22 to 43 inches. They decreased markedly with increasing temperature and slightly with decreasing flow rate and decreasing pressure. Satisfactory correlations were developed to allow interpolation for

conditions not tested. In an exchange column flooding test conducted at 28 psia. and 96°F., flooding was found to occur at an anisole flow rate of 2.7 gpm. At flow rates below flooding, the observed pressure drops of 0.3 to 1.3 inches of water per foot of packing were generally lower than expected. A correlation for the pressure drop results also was developed.

Both the complex formation and complex dissociation steps were carried out with very low boron losses. The pilot plant reflux ratios, defined as material recycled per unit of that "lost", were on the order of 10^6 at the recombiner end and 10^4 at the decomposer end.

Based on gas-liquid chromatographic analyses of still reboiler samples, the anisole degradation rate appeared to be equivalent to about 3.7% of the high temperature anisole hold-up per day during the first nine tests and about 7% per day during the remaining ten tests. Although this increase during the latter runs seemed to be caused by operating near 36 psia. during runs 10, 11, 12, and 13, it was coincident with an inadvertent release of boron trifluoride from the decomposer to the still which may have altered, at least temporarily, the reactivity of the metal surface in the still, thereby providing an additional site for degradation. Furthermore, it is possible that similar changes could have occurred to the surfaces of the exchange column and exchange column packing, since this unit often reached temperatures as high as 200°F. during test start-ups.

Based on analyses of recycled anisole, corrosion rates appeared to be low in all of the tests and the choice of materials of construction was satisfactory. Preliminary visual examination of the piping and equipment indicated some surface tarnishing but no serious attack. The achievement of tolerably low anisole degradation and corrosion rates is thought to be due largely to the care taken to exclude water from the system. Successful drying, on a routine basis, to less than 50 ppm. water was demonstrated to be feasible using Molecular Sieves*. Additionally, a separate anisole recycle loop was established to provide coolant for condensers, recombiners, and gas coolers. Construction of the system was accomplished carefully employing TIG welding† of equipment and connections and using helium leak detection to locate any leak sources.

The general conclusion from the pilot plant program was that the anisole process should be applicable on a plant scale. Although fund limitations prevented testing of alternative column packings or tower internals arrangements and operation for extended periods of time, sufficient data were obtained to allow reasonably confident plant sizing. Further work was not felt to be needed at the present time.

* Product of Union Carbide Corporation.

† TIG welding is an inert gas-shielded, arc-welding process approved for use with several stainless steel alloys.

TECHNOLOGY AND DESIGN

Since the isotopic exchange system for the separation of boron isotopes is essentially a conventional gas-liquid contacting operation, the calculation of separative potential is done by the same methods used for distillation operations. The exchange between a downflowing liquid complex of boron trifluoride and anisole and upflowing boron trifluoride gas results in the enrichment of the liquid stream in boron-10.

The boron trifluoride gas reflux to the product end of the exchange column is supplied by dissociating the column liquid effluent in an auxiliary called the decomposer system. The stripped anisole from the decomposer is recirculated through a purification still to a second auxiliary, the recombiner system, where it reacts with boron trifluoride tails exiting from the exchange column. The recombiner must be cooled to remove the heat of reaction, and the liquid complex formed in this unit is returned to the boron-11 end of the exchange column.

Since the ratio, in an operating plant, of boron trifluoride withdrawn as product or tails to that returned to the column would be about 1 to 300 at the decomposer end and 1 to 50 at the recombiner end, the decomposer and recombiner must operate smoothly with little fluctuation and with low material losses. For example, the complex fed to the decomposer contains about 1 mole of boron trifluoride per mole of anisole. The product takeoff rate in a production facility would be equivalent to 0.0033 mole of boron trifluoride per mole of anisole. If losses were to be held to about 3% of the product, then the amount of boron trifluoride lost from the decomposer must be held to 0.0001 mole of boron trifluoride per mole of anisole or 100 ppm. boron trifluoride on a mole basis. This corresponds to losses of about 10 ppm. boron on a weight basis. The recombiner efficiency requirement is perhaps a little more difficult to evaluate simply, but with a tails withdrawal rate ten times the product rate on a mole basis, 0.02 mole of boron trifluoride per mole of recirculated anisole would be withdrawn as tails. Assuming no requirement for tails, this material could be vented to a scrubbing system. With respect to the recombiner, the engineering-scale equipment requirements were more stringent, perhaps, than those of a production plant, since operation as close to total reflux as possible at both ends of the pilot plant was needed for data accumulation.

The design of the engineering-scale test unit involved not only the more conventional problems in assessment of mass and energy transfer rates but also the problems of solvent degradation and corrosion. Discussions of the basic systems properties, corrosion, anisole degradation, and systems design are presented subsequently in this section.

SYSTEM PROPERTIES

Isotopic Enrichment

Of the several methods of separation of boron isotopes which have been studied, the gas-liquid chemical exchange method utilizing the boron trifluoride-anisole complex seems to be the most promising. As mentioned previously, the exchange between the boron isotopes in countercurrent streams of gaseous boron trifluoride and liquid boron trifluoride-anisole complex results in the enrichment of boron-10 in the liquid phase:

With a reported half-time of less than 3 seconds, this reaction is suitable for application to continuous operations⁽¹⁸⁾.

The single-stage isotopic separation factor is defined by

$$\alpha = \frac{\text{Anisole} \cdot \text{B}^{10}\text{F}_3(\ell) / \text{Anisole} \cdot \text{B}^{11}\text{F}_3(\ell)}{\text{B}^{10}\text{F}_3(\text{g}) / \text{B}^{11}\text{F}_3(\text{g})} \quad \text{.....(2)}$$

The separation factor has been experimentally determined to vary with temperature according to the following equation⁽⁶⁾:

$$\log_{10} \alpha = \frac{9.87537}{T(^{\circ}\text{K.})} - 0.02005 \quad \text{....(3)*}$$

This equation predicts a decrease in the separation factor with increasing temperature; however, exploratory work by Makarov and Panchenkov⁽¹²⁾ suggests that total separation, considering not only the single-stage separation factor but also the stage length, reaches a maximum at about 105°F.

The number of stages, N, in a separation column operated at total reflux is given by

$$\alpha^N = \frac{R_T}{R_B} \quad \text{.....(4)}$$

* This equation represents a least-squares fit of the unweighed single-stage separation factors obtained at ORNL. Recently, another least-squares correlation, which weighs the data considering the variances, has been developed by Drury⁽⁶⁾. Over the range of conditions studied in the pilot plant, values predicted using the later equation, which will be published in the J. Chem. Phys., do not differ significantly from those obtained from equation (3).

where R_T = the ratio of boron-10 to boron-11 at the isotopic top of the column, and

R_B = the ratio of boron-10 to boron-11 at the isotopic bottom of the column.

The stage length can then be found by dividing the column length by N .

Properties of Boron Trifluoride, Anisole, and
Boron Trifluoride-Anisole Complexes

The physical and chemical properties of anisole and boron trifluoride have been presented by Barber⁽¹⁾, and selected values for these materials are presented in tables I and II. Boron trifluoride is a colorless gas which has a pungent suffocating odor⁽¹³⁾. The gas is irritating to the respiratory tract and is corrosive to the eyes, skin, and mucous membranes. Since the level of detection by smell is about 1.5 ppm., and since boron trifluoride reacts readily either with moisture in ambient air or with ammonia vapors to form visible fumes, there are few problems associated with detection of major leaks; however, it should be noted that a value of 0.3 ppm. has been suggested as a threshold limit value for personnel exposure⁽²⁰⁾.

TABLE I
SELECTED PHYSICAL PROPERTIES OF BORON TRIFLUORIDE*

Property	Value
Molecular Weight	67.81
Melting Point	- 127°C.
Boiling Point (760 mm.)	- 101°C.
Critical Temperature	- 12.25°C.
Critical Pressure	49.2 atm.
Gas Density (0°C.)	3.07666 g./l.
Specific Gravity of Gas (0°C.)	2.38
Density of Liquid (- 101°C.)	1.59 g./cc.
Density of Solid	1.87 g./cc.
Specific Heat, C_p (278°K.)	11.7 cal/degree-mole
Viscosity of Gas:	
- 80°C.	119 micropoise
+ 80°C.	196 micropoise

* Barber, E. J., Physical and Chemical Properties of Some Materials Used in the Anco and Elemental Boron Processes, Union Carbide Corporation, Nuclear Division, Oak Ridge Gaseous Diffusion Plant, October 8, 1962 (K-1548).

TABLE II
SELECTED PHYSICAL PROPERTIES OF ANISOLE*

Property	Value
Molecular Weight	108.13
Melting Point	-37.38°C.
Boiling Point (760 mm.)	153.75°C.
Critical Temperature	384°C.
Critical Pressure	38.8 atm.
Density (20°C.)	0.99402 g./cc.
Specific Heat (24°C.)	0.442 cal/g.-°C.
Heat of Combustion	905.2 Kcal/mole
Heat of Vaporization	8.8 Kcal/mole
Viscosity (20°C.)	1.086 cp.
Surface Tension (20°C.)	35.22 dynes/cm.
Flash Point (Cleveland Open Cup)**	52°C.
Auto Ignition Temperature**	268°C.

* Barber, E. J., Physical and Chemical Properties of Some Materials Used in the Anco and Elemental Boron Processes, Union Carbide Corporation, Nuclear Division, Oak Ridge Gaseous Diffusion Plant, October 8, 1962 (K-1548).

** As determined at the Oak Ridge Gaseous Diffusion Plant.

Pure anisole is a colorless liquid with a vapor pressure of 3.05 mm. Hg at room temperature. The toxicity of anisole is relatively low, and the Cleveland Open Cup flash point is 125°F. (52°C.) An effective solvent for many organics, its behavior as a Lewis base also accounts for the formation of the complex with boron trifluoride, which is a Lewis acid.

Some physical properties of the boron trifluoride-anisole complex are shown in table III. Additional properties, including vapor pressure data and the variations of complex density and viscosity with mole ratio and temperature are presented by Barber⁽¹⁾ and by Barber and Madix⁽²⁾. These data provided the information needed for the pilot plant equipment design calculations.

TABLE III
SELECTED PHYSICAL PROPERTIES OF
BORON TRIFLUORIDE-ANISOLE COMPLEXES*

Property	Value
Molecular Weight of 1:1 Complex	175.94
Melting Point**	3.0°C.
Density of 1:1 Complex (20°C.)**	1.2350 g./ml.
Viscosity of 1:1 Complex (20°C.)**	5.41 cp.
Heat of Formation	-12.3 ± 0.001 Kcal/mole

* Barber, E. J., Physical and Chemical Properties of Some Materials Used in the Anco and Elemental Boron Processes, Union Carbide Corporation, Nuclear Division, Oak Ridge Gaseous Diffusion Plant, October 8, 1962 (K-1548).

** As determined at the Oak Ridge Gaseous Diffusion Plant.

CORROSION

The choice of appropriate materials of construction for the equipment used in the pilot plant was based upon a reconciliation of the problems presented by the corrosive actions of the chemicals involved in the process and those caused by the participation of various metals in the solvent degradation process.

The Hooker Electrochemical Company, in an investigation⁽⁸⁾ of several of the problems associated with the anisole process, made corrosion tests on Hastelloy-C*, copper, Monel, stainless steel 316, and red brass. These tests were made under operating conditions simulating those expected in a proposed pilot plant⁽⁹⁾. The results, presented in table IV, indicate that, where there is a possibility of water entering the system, Monel and Hastelloy-C were superior to the other materials tested; however, stainless steel 316 was better than Monel when low water concentrations were maintained. Corrosion of Hastelloy-C was negligible in either case.

* Product of Union Carbide Corporation.

TABLE IV

SUMMARY OF HOOKER ELECTROCHEMICAL COMPANY CORROSION TEST RESULTS*

<u>Material</u>	<u>Corrosion Rate, mils/yr.</u>
<u>Series I: 0.007% Initial Water Content, 32-Day Test</u>	
Hastelloy-C	0.02
Copper	0.24
Monel	0.21
Stainless Steel 316	0.03
Red Brass	0.33
<u>Series II: 1.0% Initial Water Content, 18.7-Day Test</u>	
Hastelloy-C	0.02
Copper	2.75
Monel	0.51
Stainless Steel 316	12.42
Red Brass	1.60

* Hambrock, K. O., Boron Isotope Separation with Anisole-Boron Trifluoride Complex, Hooker Electrochemical Company, August 26, 1957 (HEC-80).

C. G. Jones, of the Oak Ridge Gaseous Diffusion Plant, conducted a preliminary study designed to screen various construction materials on the basis of corrosion resistance. In this experiment, several metal strips were placed in an autoclave in which boron trifluoride-anisole complex containing 15 to 23 ppm. water was held at 295°F. for 20 days to effect degradation. The results of this experiment, shown in table V, indicate that stainless steel 316, Kanigen-plated mild steel 1020, A-nickel, and Monel resisted corrosion considerably better than stainless steel 347, stainless steel 304, and mild steel 1020. The low system water content was again seen to account for the durability of stainless steel 316.

TABLE V

SUMMARY OF ORGDP AUTOCLAVE CORROSION TEST RESULTS*

Material	Corrosion Rate, mils/yr.
Stainless Steel 316	0.26
Kanigen-Plated Mild Steel 1020	0.50
A-nickel	1.31
Monel	1.17
Stainless Steel 347	2.97
Stainless Steel 304	9.90
Mild Steel 1020	43.6

* C. G. Jones, Union Carbide Corporation, Nuclear Division, Oak Ridge Gaseous Diffusion Plant, unpublished data.

Extensive laboratory-scale corrosion studies performed at the ORGDP in glass columns containing sections to simulate the behavior of an anisole reboiler, a complex decomposer, and a recombiner have been described elsewhere⁽³⁾. However, the results of these studies are summarized in table VI. System water contents were low in all tests, but both nickel and Monel samples located near the recombiner section of the apparatus developed a generalized surface roughening during the tests. Stainless steel alloys were found to be more resistant to corrosion, showing no specific type of corrosive attack. Significantly, the protection offered to stainless steel samples by a passivating oxide film was demonstrated in a test with stainless steel Pall rings depassivated with hydrofluoric acid. Immediately after start-up, a viscous, orange liquid formed on the surface of the rings in the region of the column characterized by a sharp temperature gradient. In a concurrent test in identical apparatus, however, no such liquid was formed on the surface of Pall rings which had not been depassivated. Thus, the immediate effect of depassivation of the stainless steel was apparent, but the fact that these materials may have become repassivated as the test progressed was suggested by the similarity in appearance of the rings used in the two tests after the first few hours of exposure.

In these tests, it was noted that the slight corrosion of the various stainless steel alloys depended more on the sample location than on the specific alloy tested, with the worst attack occurring in the sharp temperature gradient region between the recombiner and decomposer sections. These results were confirmed in bench-scale anisole degradation tests, where no significant corrosive attack of stainless steel was observed in contrast to a definite intergranular attack found when nickel was used as the material of construction of the test loop. In the bench-scale tests, which are described in the following section, the most active site

TABLE VI

SUMMARY OF ORGDP LABORATORY-SCALE CORROSION TEST RESULTS

Material Tested	Exposure Time, hr.	Corrosion Rate, mils/year				
		Sample Location				
		Recombiner	Upper Decomposer	Lower Decomposer	Reboiler, Interface	Reboiler, Immersed
Nickel 200	744	11 ^a	< 1	< 1 ^b	< 1 ^b	< 1
Nickel, Pall Ring ^c	1,944	4 ^a	-	-	-	-
Monel	1,200	3 ^a	< 1 ^b	< 1 ^b	< 1 ^{b,d}	< 1 ^d
Stainless Steel ^e	1,032	1 ^a	< 1	< 1	< 1	< 1
Stainless Steel, welded ^f	984	< 1	< 1	< 1	< 1	< 1
Stainless Steel 316, Pall Rings						
Depassivated	100	1 ^a	2 ^a	1	-	-
As-Received	100	< 1 ^a	< 1 ^a	< 1	-	-

a. Sample was coated with black deposits by end of test.

b. The sample was partially coated with a red deposit by end of test.

c. This Pall ring was exposed during both the nickel and Monel tests.

d. Sample was slightly tarnished by end of test.

e. Alloys 304L, 310, and 316 were tested.

f. Alloys 304L, 316, 321, and 347 were tested.

for corrosion again appeared to be the transition region between the cold recombiner and the hot decomposer. On the basis of these tests, stainless steel 304L was chosen as the primary material of construction for the engineering-scale test units, with recognition of the fact that its use required that anhydrous conditions be maintained to prevent the formation of acids which would destroy the passive surface. The use of nickel packing in some of the equipment items resulted from information gained in anisole degradation tests rather than from the results of the corrosion tests.

ANISOLE DEGRADATION

The important problem of anisole degradation was investigated on both a laboratory- and bench-scale at the ORGDP(3). In the laboratory tests, summarized in table VII, the rate of solvent degradation, determined during the previously mentioned corrosion tests, was found to be influenced by the water content of the system, the metal surface area exposed, and not only the particular metal used but also the nature of its surface. When only a small metal area was exposed, the degradation rate was extremely low for all materials; however, the rate with stainless steels appeared to be about twice that for nickel and Monel. With much larger surface areas exposed, the degradation rate for depassivated stainless steel 316 was found to be about four times that observed for as-received, i.e., oxide-passivated, metal.

A bench-scale, continuously operated, all-metal test loop containing a recombiner, decomposer, distillation system, and liquid pumps also was used in the degradation tests. The distillation system was used to fractionate the entire decomposer-stripped solvent stream in order to remove degradation products before the anisole was returned to the recombiner. Operation of the unit at decomposer pressures up to 40 psig., as shown in table VIII, indicated the following:

1. The solvent degradation rate is higher with stainless steel than with nickel.
2. The degradation rate increases with operating pressure and temperature, but does not decrease at the same rate after operation at higher temperatures and pressures. Thus, operation at high temperatures and pressures apparently causes irreversible changes in the reactivity of the metal surfaces.
3. Only a small amount of water inleakage is sufficient to cause severe degradation; however, the exclusion of water from the system to the practical limit of about 100 ppm. is only a necessary, not a sufficient, condition for inhibiting degradation.
4. Complete distillation of the solvent can be used effectively to remove degradation products from recycled liquid.

TABLE VII
ANISOLE DEGRADATION RATES DURING RUNS MADE
IN LABORATORY GLASS COLUMNS

<u>Materials Under Study</u>	<u>Time After Start-Up, hr.</u>	<u>Average Anisole Degradation Rate*, % Charge Per Day</u>	<u>Estimated Exposed Metal Surface, sq.cm.</u>
Nickel	24	0.024	175
	168	0.004	
	336	0.009	
	528	0.007	
	744	0.012	
Monel†	912	0.010	175
	1,080	0.006	
	1,944	0.013	
	1,944	0.008‡	
Stainless Steel	168	0.043	250
	1,032	0.017	
	1,032	0.012§	
Stainless Steel, TIG-Welded	480	0.031	525
Stainless Steel, As-Received Pall Rings	100	0.08	5,875
Stainless Steel, Depassivated Pall Rings	100	0.36	5,875

* 90 ml. sample distillation except as noted.

† Monel coupons substituted for nickel using same anisole.

‡ 717 ml. distillation.

§ 897 ml. distillation.

TABLE VIII
ANISOLE DEGRADATION RATES DURING BENCH-SCALE TESTS

Test No.	Environment	Decomposer		Duration, hr.	Water Content, ppm.	Still Bottoms	Degradation
		Pressure, psig.	Temperature, °F.			Withdrawal Rate, %/Day of High Temperature Anisole Hold-Up	Rate, %/Day of High Temperature Anisole Hold-Up
1	Nickel	40	410	720	95	9	0.45
2	Stainless Steel	40	410	195	*	6	†
3	Stainless Steel	40	410	165	105	4	3
4	Stainless Steel	40	410	126	100	8	(6 to 9‡)
5a	Stainless Steel	20	370	450	60	10	1 to 2
5b	Stainless Steel	40	410	281	70	3	1 to 2§
6a	Stainless Steel	20	370	732	100	2 - 5	0.4
6b	Stainless Steel	40	410	397	110	5 - 7	4.8
6c	Stainless Steel	20	370	439	85	3 - 5	1.2

* Increased from 67 to 1,420 ppm. shortly before end of test.

† Because of the water inleakage which occurred during this test, no attempt was made to determine this value.

‡ These results are based on only two analyses and therefore must be considered less certain than the rates given for the other tests. For this reason, the values are bracketed. Analysis of the final still pot residue provided the lower value. Other samples were taken but were inadvertently discarded in the laboratory.

§ At the end of the test, degradation products were being formed in the decomposer at a rate of about 10% of the anisole passed through the decomposer.

Laboratory work identified the degradation products in the solvents from the various bench-scale tests. The degradation products formed during two of the tests are listed in tables IX and X. From these data, the mechanisms presented in table XI were formulated to account for the formation of the various compounds. The principal mechanism apparently involves (1) a boron trifluoride-catalyzed Friedel-Crafts type of alkylation of the aromatic ring to give o- and p-methyl anisoles and m-cresol, and (2) a dealkylation of the anisole and substituted anisoles to form phenol and cresols. These processes involve the cyclic utilization and regeneration of methyl fluoride and hydrogen fluoride. It was demonstrated that hydrogen fluoride forms an acidic complex with anisole and boron trifluoride which probably is the active intermediate for the listed reactions as well as a vehicle for recycling hydrogen fluoride between the exchange column and the decomposer system, thereby tending to propagate the degradation process once it is begun.

TABLE IX

ANISOLE DEGRADATION PRODUCTS OBSERVED IN
BENCH-SCALE TEST 1

<u>Component</u>	<u>Composition</u>	<u>% of Total Degradation Products</u>
1	o-Methyl Anisole (Methyl o-Tolyl Ether*) Azeotrope† with Phenol	24.7
2	o-, m-, p-Cresols; Mixed	23.1
3	p-Methyl Anisole (Methyl p-Tolyl Ether*)	19.9
4	Phenol	15.9
5	Mixed Cresols and Phenol	8.6
6	o-Methyl Anisole, Anisole; Mixed	5.6
7	o-Methyl Anisole, p-Methyl Anisole, Phenol; Mixed	1.4
8	Oils and Solids	<u>0.8</u>
		100.0

* Alternative names for compounds.

† Azeotrope contained about 70 mole percent o-methyl anisole.

TABLE X
ANISOLE DEGRADATION PRODUCTS OBSERVED IN
BENCH-SCALE TEST 3

<u>Component</u>	<u>Composition</u>	<u>% of Total Degradation Products</u>
1	o-Methyl Anisole	24.0
2	o-, m-, p-Cresols; Mixed*	22.0
3	p-Methyl Anisole	21.3
4	Phenol	21.2
5	p-Methyl Anisole, Phenol; Mixed	5.4
6	Anisole, o-Methyl Anisole; Mixed	3.1
7	o-Methyl Anisole, p-Methyl Anisole; Mixed	2.4
8	Miscellaneous Solids	<u>0.6</u>
		100.0

* The meta substitution appeared to predominate in the mixture.

In summary, the degradation rate at 20 psig. with stainless steel equipment appeared acceptable. Even the rate at 40 psig. did not appear so high as to rule out operation at that condition; however, the differences in the behavior of nickel and stainless steel in the degradation tests did suggest the use of nickel in areas where corrosion would not be a significant factor. Thus nickel was chosen as the packing material for the high temperature columns in the engineering-scale equipment.

ENGINEERING-SCALE EQUIPMENT DESIGN

Before proceeding with the presentation of the pilot plant test results, listed in the next section, brief design summaries giving the results of calculations and stating the assumptions made for each of the major equipment items are presented. More detailed specifications for the various units and flow schemes are included in the appendix.

Sizing of the equipment for the pilot plant was based on the criterion of testing plant-scale packing to the flooding point with exchange column temperatures between 68 and 104°F. Six-inch diameter pipe was used for

TABLE XI
PROBABLE REACTIONS IN THE DEGRADATION OF ANISOLE

* Alternative names are methyl-o-tolyl ether and methyl-p-tolyl ether.

the exchange column, resulting in a tower-diameter-to-packing-size ratio of about 9.6 for 5/8-inch Pall rings. The flooding point for the 6-inch column was preliminarily estimated to correspond to a complex flow near 3 gpm.; however, at the time the design and fabrication of the auxiliaries were being performed, data for estimation of the flooding flow, particularly complex viscosity data, were lacking. Thus, to provide assurance of adequate circulation, auxiliary equipment, such as the recombiner, decomposer, and still, was sized to accommodate complex flows up to 4 gpm. This flow rate corresponds to about 3.2 gpm. of anisole and is near that required for a production facility with a product rate of 5 kg. of enriched material per day. In this respect, the pilot plant exchange column diameter is about that expected for a plant operating at 2.5 kg. production per day.

Exchange Column

As mentioned above, the exchange column diameter was fixed at 6 inches by the overall goals of the engineering-scale study. The height of the packed section was chosen to be 13.5 feet, with liquid redistribution provided at 4.5-foot intervals. Several factors influenced the selection of the 5/8-inch Pall rings from the many types of commercial and laboratory-scale tower packing materials. First, the available information concerning relative mass transfer efficiencies, hold-up and pressure drop characteristics, and allowable throughputs, as well as the availability of various packings in the required materials of construction, seemed to indicate that Pall rings were generally superior to the other types of rings and saddles commonly used in large-scale industrial operations. Furthermore, although high efficiency, laboratory-scale packing, such as protruded packing, did offer the potential advantage over Pall rings of having low stage lengths (i.e., near 6 inches), it was felt that this advantage was outweighed, especially in a plant of the size envisioned for a production facility, by the significantly lower allowable throughput per unit area, a greater tendency to plug, and, most important, the much higher unit cost. In short, it was desired to gain engineering data on the type of packing most likely to be selected for use in a production plant.

Two items of major interest in the column design were the expected boron-10 enrichment of the liquid stream at total reflux and corresponding stage lengths for the 6-inch column. Using a separation factor of 1.03 and assuming, for design purposes, a transfer unit height of 18 inches, a value typical of many gas-liquid systems using 5/8-inch Pall rings, the expected isotopic gradient at total reflux was from about 17% boron-10 at the recombiner to about 22% boron-10 at the decomposer, as compared with a normal assay near 18.7% boron-10. Mass spectrographic methods were available to provide the analytical accuracy and precision required to allow evaluation of stage lengths. Of additional interest were the maximum flow rates allowable, since it was necessary to be able to flood the column. Table XII lists some anisole flow rates at which flooding could be expected for various operating pressures. These estimates were based on the use of 5/8-inch Pall rings and operation at the temperatures required to maintain a 1:1 mole ratio complex.

TABLE XII
CALCULATED EXCHANGE COLUMN FLOODING FLOWS

<u>Pressure,</u> <u>psia.</u>	<u>Flooding Flow,</u> <u>gal./min. (anisole)*</u>
14.7	1.95
34.7	2.55
54.7	2.82

* Based on a 1:1 complex at the required equilibrium temperature being contacted by a corresponding boron trifluoride flow.

Since both stage length and column pressure drop are highly dependent on tower internals, such as liquid distributors and packing support plates, as well as on the type of packing used, a series of tests was made prior to the pilot plant experiments to determine the performance of the commercially available internals suggested for use in the 6-inch column. These tests, using air and water, were performed in a 6-inch diameter column, 4 feet long and packed with 5/8-inch Pall rings. The results showed that the suggested support plates caused excessive pressure drop, limiting the throughput to about 65% of the predicted values, while over the range of flows tested, the distributors tended to supply almost all of the liquid to the center of the bed. For these reasons, the internals were redesigned, as discussed in detail in the appendix. In order to reduce the pressure drop caused by the support plates and thereby allow the expected throughput, a corrugated screen support plate with a large area available for gas flow was employed. The addition of notched, liquid return tubes to the distributor served to provide for an even distribution of liquid over the packed bed.

Decomposer

The decomposer's function is the dissociation of the boron trifluoride-anisole complex and the subsequent regeneration of gaseous boron trifluoride and liquid anisole. Additionally, to meet the specifications imposed on this process, the dissociation must be effected with less than 10 ppm., on a weight basis, of boron remaining in the anisole effluent. The decomposer was designed as two units, each having a column and reboiler, to be operated in series and at two temperature levels. This arrangement allowed approximately 80% of the design heat requirement to be supplied by 100 psig. steam, with the remaining load borne by electrical heating. Also, this method reduced the hold-up of anisole at the higher temperature required for complete complex dissociation, thereby probably minimizing degradation.

Anisole with less than 3 weight percent boron trifluoride can be obtained with temperatures near 260°F. in the low temperature section of the decomposer. Because of the ease of separation, a majority of the dissociation was predicted to occur in the reboiler, with probably less than 7% of the complex breaking up in the column. The heat load for the low temperature decomposition step varies somewhat with operating pressure, but is near 400,000 Btu./hr. at the 4 gpm. complex flow rate that served as the design basis. With direct steam heating, the calculated required heat transfer surface was about 40 square feet, based on an overall heat transfer coefficient of 145 Btu./hr.-sq.ft.-°F. derived by conventional means. Excess area was provided because of uncertainties in the fouling tendencies of the process streams and lack of data concerning boiling coefficients. The excess area also allowed reduction of the boron trifluoride content of the liquor fed to the high temperature decomposer from the assumed design value of 3 weight percent, thus easing the load on that unit.

The low temperature decomposer stripping column diameter was set at 12 inches to allow operation below a nominal 65% of flooding. About five feet of 1-inch Pall ring packing was used to give an adequate number of mass transfer units for the range of operating conditions considered. Liquid hold-up in the low temperature section of the decomposer was estimated at 500 pounds. Of this hold-up, 478 pounds is retained in the reboiler. The boron trifluoride hold-up was estimated to be less than 15 pounds.

In the high temperature section, the liquid is heated to approximately 375°F. and boiled, at which point less than 10 ppm. of boron remains in the liquid. A maximum heating load of 90,000 Btu./hr., supplied electrically, was expected for the high temperature decomposer. Based on a heat flux of 21 watts per square inch, 8.7 square feet of surface was required. The actual surface area provided for heat transfer was 11 square feet. A 6-inch diameter high temperature decomposer stripper, packed to a height of 3 feet with 5/8-inch Pall ring packing, also was employed. Liquid hold-up in the high temperature section was estimated to be approximately 100 pounds, with 95 pounds in the reboiler section. The boron trifluoride hold-up in this section is negligible.

Before the boron trifluoride is returned to the exchange column, it is cooled to the exchange column operating temperature in a boron trifluoride cooler located at the top of the low temperature decomposer column. Also, a small amount of complex probably is refluxed at this point. The cooler design consisted of two coils of finned tubing having about 25 square feet of fin surface area. Refrigerated anisole was chosen as the coolant for the gas, which enters the unit at temperatures near 100°F.

Recombiner

The boron trifluoride-anisole complex used in the isotopic exchange column is formed in the recombiner. The complex formers, which flow counter-currently inside the tubes of the two shell and tube heat exchangers which serve as the basic parts of the recombiner, are the isotopically depleted

boron trifluoride stream from the top of the exchange column and the anisole stream from the purification still product tank.

The major design consideration for this unit was the removal of the heat of formation of the complex. Formation of the entire complex at 68°F. gives rise to a refrigeration load approaching 30 tons, while recombination at higher temperatures generates the problem of formation of complexes with boron trifluoride-to-anisole mole ratios lower than desired as long as reasonable pressures are maintained. Thus, the use of two temperature levels in the recombiner represented a balance among the requirements of refrigeration and complex composition as well as the necessary heat transfer area for the various cases.

Operating at an average temperature near 110°F., the first heat exchanger was designed to provide for about 80% of the complex formation. At a complex production rate of 4 gpm., the estimated cooling load for this section was 250,000 Btu./hr. This section of the recombiner can be cooled with approximately 35 gpm. of anisole coolant entering at room temperature. The second heat exchanger, which operates near the exchange column temperature, is the site for the cooling of the complex, as well as for the rest of the complex formation. The coolant chosen for this section, approximately 23 gpm. of refrigerated anisole, removes 106,000 Btu./hr. Anisole was selected as the primary coolant instead of water in order to eliminate the possibility of a water leakage into the system, which would greatly increase solvent degradation and corrosion. Based on an estimated overall heat transfer coefficient of 70 Btu./hr.-sq.ft.-°F., the required heat transfer areas for the 110 and 68°F. sections were 238 square feet and 71 square feet, respectively. Slight excesses in these areas were provided to allow greater operational flexibility.

The estimated numbers of transfer units required for mass transfer were 2.9 for the 110°F. section and 1.8 for the 68°F. section. To provide for more intimate contacting between phases, 5/8-inch Pall rings were placed inside the tubes of the two exchangers. The HTU expected for both sections was about 1.5 feet, an intermediate value between the HTU's calculated for wetted-wall operation and those estimated for a completely packed, packed-bed absorption operation accompanied by a chemical reaction. The equipment design was, however, controlled by the heat transfer requirements, and the lengths of the 110 and 68°F. sections allowed 3.4 feet and 2.2 feet per transfer unit. A back-up complexing section for final stripping of boron trifluoride from the vent gas, consisting of a cooled coil of finned tubing, was designed for installation above the 110°F. section.

Anisole Still

Purification of the process anisole mainly is required to remove high boiling degradation products, such as phenol, cresol, and the methyl-substituted anisoles. Condensable materials more volatile than anisole would tend to accumulate in the system between the decomposer and recombiner and therefore would not be expected to reach the still.

Because the heat load for the still is rather high for the use of electricity, steam was selected to provide the reboil heat. Since the maximum available temperature differential with the 100 psig. plant steam was not considered adequate for reboiler operation with impure anisole, vacuum distillation was indicated. Furthermore, vacuum distillation seemed advantageous because the low temperatures involved reduced the possibility of further solvent degradation in the purification still. The nominal operating pressure of the still was chosen to be 300 mm. of mercury.

At a total boilup rate of about 6 gpm., the estimated heat loads for the reboiler and condenser were about 370,000 Btu./hr., neglecting the quantity of reboiler feed which flashes because of the operating pressure difference between the decomposer and the still. Using a heat transfer coefficient of 100 Btu./hr.-sq.ft.-°F., the calculated reboiler area was 74 sq.ft. The 84 square feet of area actually provided allowed use of the same design used for the low temperature decomposer reboiler. The heat transfer coefficient and the required area estimated for the anisole-cooled condenser were 70 Btu./hr.-sq.ft.-°F. and 28 square feet, respectively. The still column, 16 inches in diameter, was packed to a height of 7 feet with 1-inch Pall rings. Liquid hold-up for the still system was estimated to be 550 pounds with 478 pounds in the reboiler. Hold-up in the condenser was assumed negligible.

Miscellaneous

Other process equipment provided for the engineering-scale test unit included a double-pipe heat exchanger, located between the still and the still product tank, designed to remove approximately 200,000 Btu./hr. while cooling the anisole product to about 100°F. Additional still product cooling was provided for by external coils on the product tank. Also, three $\frac{1}{4}$ gpm. diaphragm metering pumps were selected to feed the recombiner, the exchange column, and the decomposer.

Finally, coolant supply recirculation systems furnishing approximately 100 gpm. of anisole at room temperature and 40 gpm. of anisole at 30°F. were required. In the first system, water was chosen to cool the recycled anisole, while in the second loop a 12-ton refrigeration unit was incorporated to reach the required 30°F.

PILOT PLANT OPERATING SCHEMES AND TEST RESULTS

Following preliminary drying and shakedown runs, nineteen isotope separation tests and one flooding test were made in the pilot plant equipment. In the separation tests, pressures of about 20, 26, and 35 psia.; anisole flow rates of 1.17, 1.75, and 2.0 gpm.; and temperatures from 63 to 105°F. were employed, while the flooding test was carried out at 28 psia. and 96°F. The time required for each test was on the order of one day, and during the tests, samples were taken not only to determine the degree of isotope separation but also to assess the performances

of the decomposer, the recombiner, and the still as well as the extent of solvent degradation.

OPERATING SCHEMES

Initial Start-Up

During installation of the process equipment, all of the columns were wet-packed using water. Although the water was drained from the system after the tower packing operations were completed, sufficient moisture remained to require that the first pilot plant operation be the drying of the surfaces of the units. For this reason, before any tests with anisole and boron trifluoride were begun, each equipment item and all lines were purged with dry nitrogen until the exit dew point of the gas was below minus 50°F. Next, the performances of the still and the various instruments were checked, and some further drying was accomplished, by completing a shakedown procedure generally consisting of the following steps or operations:

1. The coolant loops were filled with anisole and tested.
2. The still reboiler and still product tank were filled with dry anisole (water content less than 50 ppm.).
3. The still system was evacuated until the pressure was 300 mm. of mercury. Also, the remaining units were pressurized to about 10 psig. with nitrogen.
4. The still was operated at total reflux while pressure drops, allowable boilup rates, and the required steam pressures were established.
5. Still product take-off was begun and, simultaneously, the recombiner feed pump was started.
6. When enough anisole had been circulated to the recombiner to fill its reservoir partially, the exchange column feed pump was started. Then, similarly, when the exchange column reservoir began to fill, the decomposer feed pump was started.
7. The decomposer reboilers were allowed to fill to their normal operating levels and then were heated. The subsequent opening of a valve between the high temperature decomposer reboiler and the still reboiler completed the loop.
8. Anisole was circulated through the entire system while the following checks were made:
 - a. All instruments were tested. For example, temperature indicators were checked with a potentiometer, and temporary level upsets were imposed in order to determine the responses of the level controllers.

- b. Each sample point was tested to see if samples could be withdrawn easily.
- c. Vent lines, the scrubber, and the pressure relief systems were checked.
- d. Periodic samples were analyzed for water buildup. When the anisole contained more than 500 ppm. water, the system was shut down and drained, and the anisole was dried and then returned to the system for additional circulation. This cycle was repeated until all instruments were working as designed and the last traces of moisture were removed, as indicated by anisole sample water contents below 50 ppm.

Routine Start-Up

After the preliminary operations had been completed, the isotope separation experiments were begun using start-up techniques very similar to the procedure outlined above. However, for each test, boron trifluoride at the desired operating pressure was used rather than nitrogen to pressurize the system, temperatures were fixed at the predetermined values by adjusting the heating and cooling rates, and the system recycle flow rate was established by selecting the appropriate setting on the recombiner feed pump control.

Sampling

During the tests, periodic sampling was required to monitor the performances of the various units. The specific reasons and generalized procedures for obtaining these samples are listed below:

1. Exchange Column Samples

During each test, concurrent gas samples were withdrawn at about 4-hour intervals from both the top and bottom of the exchange column; these samples were analyzed for the ratio of boron-10 to boron-11. Sampling was accomplished by twice filling the small sample cylinders with boron trifluoride and evacuating using a salt-soda lime trap for gas disposal. After the two rinses, the tubes were filled a third time to provide the sample. Sampling was continued until it was clear from the deviations of the results that both steady-state and isotopic equilibrium conditions had been attained. The averages of the ratios were then used with equation (4) to determine the stage height for the given experiment.

2. Decomposer Samples

The most important decomposer samples were taken from the high temperature decomposer liquid effluent. These samples were analyzed for boron, to indicate the amount of enriched product being lost to the still and, additionally, were intermittently checked for iron, nickel, and chromium concentrations, in order to aid in revealing

qualitative evidence of any major corrosive attack. Also, a few samples of the liquid flowing from the low temperature reboiler into the high temperature stripping column were analyzed for boron. These results allowed a determination of the effectiveness of the low temperature decomposer section in dissociating the complex.

3. Recombiner Samples

Analyses of samples of the recombiner vent stream were used not only to reveal the amount of boron trifluoride being lost through this unit, but also to provide semiquantitative estimates of the severity of anisole degradation, since the gaseous degradation product, methyl fluoride, tended to accumulate in the recombiner. Additionally, although the density of the recombiner exit liquid stream was continuously measured and recorded by a gamma ray attenuation device, independent density determinations were made for periodic liquid samples taken from this stream in order to verify the performance of the instrument, since the density was used to establish the mole ratio of boron trifluoride to anisole in the complex. In order to ensure representative complex samples, the samples were obtained from a continuous flow-through sample loop connected across the high and low pressure sides of the exchange column feed pump. Finally, as a matter of interest, the individual performance of each of the two recombiner units was assessed by intermittent density analyses of liquid complex flowing between the 110 and 68°F. sections.

4. Purification Still Samples

The concentrations of degradation products and of iron, nickel, and chromium in samples of the still feed, waste, and product streams were used to calculate the anisole degradation rate, to help indicate any major corrosive attack, and to evaluate the efficiency of the still. Also, the still product stream's water content was determined frequently to verify the existence of anhydrous conditions in the system.

Routine and Final Shutdown

Weekend and other temporary shutdowns were effected by turning off the main process pumps, reducing the heat input to the decomposer, and, in most instances, shutting down the still. Final shutdown procedures were equally straightforward, but were oriented toward removing all boron trifluoride from the system. This was accomplished by:

1. Valving the exit gas lines from the decomposer and the exchange column to the scrubber system, and subsequently reducing the system pressure to atmospheric.
2. Stopping the recombiner feed pump.
3. Transferring all remaining complex from the recombiner and exchange column to the decomposer for dissociation.

Using this procedure, all boron trifluoride was forced to the scrubber, and all anisole was routed from the decomposer through the still and, finally, to the still product tank. The still reboiler was drained and the system was then purged to remove the remaining anisole vapor.

EXCHANGE COLUMN PERFORMANCE

Isotope Separation and Stage Heights

The conditions for and results of the separation tests are listed in table XIII. As can be seen, the stage heights were observed to decrease markedly with increasing temperature and slightly with decreasing flow rate and decreasing pressure. For example, at an anisole flow rate of 2.0 gpm., the largest stage height found was 40.1 inches at a pressure and temperature of 35.9 psia. and 74.6°F.; the smallest HTU at this flow was 23.3 inches at 20.1 psia. and 103.7°F. The data from all tests are represented very closely in the range studied by either of the following equations:

$$HTU = 32.629 \left(\frac{P}{14.7} \right)^{0.130} (G')^{0.180} \left(\frac{\alpha \mu_L}{\rho_L} \right)^{0.732} \quad \dots (5)$$

$$HTU = 27.498 \left(\frac{P}{14.7} \right)^{0.138} (L')^{0.185} \left(\frac{\alpha \mu_L}{\rho_L} \right)^{0.758} \quad \dots (6)*$$

where HTU = stage height, inches,

P = system pressure, psia.,

G' = boron trifluoride flow rate, lb./hr.-sq.ft.,

L' = complex flow rate, lb./hr.-sq.ft.,

α = single-stage separation factor,

μ_L = complex viscosity, lb./ft.-hr., and

ρ_L = complex density, lb./cu.ft.

The groupings of variables used in equations (5) and (6) were suggested by the Murch equation, described by Perry⁽¹⁹⁾. The addition of the pressure term was felt necessary to extend the correlation to non-atmospheric operation. A comparison of actual HTU's and HTU's calculated using equation (5) is shown in figure 1.

* It is interesting to note that experimental data concerning Pall rings presented in U. S. Stoneware Company data sheet GR-242 show the mass transfer coefficient to vary with the 0.19 power of liquid rate in the range of interest.

TABLE XIII
EXCHANGE COLUMN OPERATING CONDITIONS AND RESULTS

Run	Temperature, °F.	Pressure, psia.	Anisole Flow, gpm.	Isotope Ratio*	Separation Factor†	Stage Height, in.	Complex Mole Ratio, BF ₃ /Anisole‡	Complex Viscosity, cp.	Pressure Drop, in. of Water ft. of packing
1	62.8	25.0	1.17	1.1270	1.0320	42.7	1.0	5.9	0.30
2	87.1	25.5	1.75	1.1600	1.0285	30.6	0.95	3.7	0.59
3	86.8	33.4	1.75	1.1500	1.0292	33.3	1.0	4.0	0.57
4	92.4	25.5	1.75	1.1742	1.0284	28.2	0.92	3.3	0.59
5	98.0	25.8	1.75	1.1870	1.0276	25.7	0.90	3.0	0.62
6	104.8	25.1	1.75	1.1979	1.0267	23.6	0.85	2.6	0.52
7	101.2	25.2	1.17	1.2050	1.0272	23.3	0.90	2.9	0.29
8	104.2	26.6	1.17	1.2110	1.0268	22.4	0.87	2.7	0.27
9	84.9	26.4	1.17	1.1707	1.0294	29.8	0.98	4.0	0.29
10	74.6	35.9	2.0	1.1310	1.0309	40.1	1.0	4.8	0.72
11	83.4	35.4	2.0	1.1415	1.0296	35.8	1.0	4.2	0.90
12	89.5	34.9	2.0	1.1540	1.0288	32.1	0.99	3.7	0.92
13	104.2	35.2	2.0	1.1752	1.0268	26.5	0.92	2.8	0.83
14	77.3	26.4	2.0	1.1394	1.0305	37.3	1.0	4.6	0.93
15	94.0	26.2	2.0	1.1528	1.0282	29.8	0.90	3.2	0.84
16	104.8	26.5	2.0	1.1750	1.0267	26.5	0.86	2.6	0.82
17	75.8	20.1	2.0	1.1521	1.0307	34.6	0.98	4.6	1.34
18	94.7	20.1	2.0	1.1868	1.0281	26.2	0.88	3.1	1.11
19	103.7	20.1	2.0	1.2024	1.0268	23.3	0.84	2.6	1.01

* $(B^{10}/B^{11} \text{ in Product})/(B^{10}/B^{11} \text{ in Tails})$.

† Predicted from equation (3).

‡ Average of mole ratios predicted by on-line density meter, laboratory analyses, and pressure and temperature conditions when these data were available.

Figure 1

STAGE LENGTHS ESTIMATED USING
A CORRELATION DEVELOPED FROM PILOT PLANT DATA

Exchange Column Pressure Drops

In the exchange column flooding test, flooding was found to occur at an anisole flow rate of about 2.7 gpm., a value which agrees well with the flooding rate predicted for the specific test conditions using the generalized pressure drop correlation presented by the U. S. Stoneware Company⁽⁴⁾. This correlation consists of a family of curves, with pressure drop as the parameter, based on two groupings of variables:

$$X = \frac{L}{G} \left(\frac{\rho_g}{\rho_L} \right)^{1/2} \quad \text{.....(7)}$$

and

$$Y = \frac{G^2 F \psi \mu^{0.2}}{\rho_g \rho_L g_c} \quad \text{.....(8)}$$

where

L, G = liquid and gas flow rates, lb./sec.-sq.ft.,

ρ_L, ρ_g = liquid and gas densities, lb./cu.ft.,

F = packing factor = 71 for 5/8-inch Pall rings,

μ = liquid viscosity, cp.,

ψ = water density/liquid density, and

g_c = gravitational constant = 32.2 (lb.mass/lb.force)(ft./sq.sec.).

Although this correlation seemed valid for estimation of the flooding rate, agreement between the actual pressure drops and values predicted by this correlation was poor for many of the separation tests, as can be seen in figure 2. Efforts to modify the correlation by the inclusion of a surface tension term in equation (7) and by changes in the packing factor used in equation (8) were largely unsatisfactory.

Better results, shown in figure 3, were obtained when a modified version of the Kozeny-Carman relation presented by Morton and associates⁽¹⁵⁾ was used to correlate the pressure drop data:

$$f = \frac{A}{Re} + B \left(\frac{1}{Re} \right)^{0.1} \quad \text{.....(9)}$$

Figure 2

EXCHANGE COLUMN PRESSURE DROPS PREDICTED
BY THE GENERALIZED PRESSURE DROP CORRELATION

Figure 3

EXCHANGE COLUMN PRESSURE DROPS PREDICTED
BY A MODIFIED FRICTION FACTOR CORRELATION

where

$$f = \text{friction factor} = (\Delta P g E^3) / (Z \rho u^2 S),$$

$$Re = \text{Reynolds number} = (\rho u) / (\mu_g S),$$

$$\Delta P = \text{pressure drop, lb. force/sq.ft.,}$$

$$g = \text{gravitational constant} = 32.2 (\text{lb.mass/lb.force})(\text{ft./sq.sec.}),$$

$$E = \text{porosity, cu.ft./cu.ft.} = \text{void fraction of dry packing minus liquid hold-up,}$$

$$Z = \text{bed length, feet,}$$

$$\rho = \text{effective gas density, lb./cu.ft.,}$$

$$u = \text{superficial gas velocity, ft./sec.,}$$

$$S = \text{surface area of packing, sq.ft./cu.ft.,}$$

$$\mu_g = \text{gas viscosity, lb./ft.-sec., and}$$

$$A, B = \text{constants related to the type of packing used.}$$

This approach includes modification of the gas density in the loading region by introducing an "effective" gas density to account for liquid entrainment. The values suggested by Morton for the constants, A and B, resulted in pressure drop predictions that were about twice the actual values. For this reason, two least-squares analyses were carried out to determine the values of these constants which would be predicted by the actual data. In the first case, least-squares values for both A and B were solved for and found to be minus 8.40 and plus 0.547, respectively. The second case involved setting the coefficient of the laminar term, the constant A, to 16 and subsequently finding the least-squares value for B. In this instance, B was found to be 0.461 and, furthermore, it was found that the variance was extremely close to that encountered in the first case, thus illustrating the significance of the turbulent term. In both cases, the hold-up was estimated using the method of Jesser and Elgin⁽¹¹⁾.

The uncertainties and rather tedious procedures involved in estimating hold-ups and effective gas densities led to a simpler empirical correlation, involving only flow rates and densities, similar to the one used by Treybal⁽⁷⁾. The equation arrived at was:

$$\Delta P = A (10)^{(19.86L)/\rho_L} \left(\frac{G}{\rho_g} \right)^{0.6436} - B \quad \dots (10)$$

where

ΔP = pressure drop, in. H_2O /ft.,

L, G = liquid and gas flow rates, lb./sec.-sq.ft., and

ρ_L, ρ_g = liquid and gas densities, lb./cu.ft.

For values of G^2/ρ less than 1, A and B were found to be 0.1543 and 0, respectively. For other cases, values determined for A and B were 0.225 and 0.367.

The overall agreement between actual and predicted pressure drops is slightly better with equation (10) than with equation (9), but although equation (10) does fit the data well in the range of conditions studied in the pilot plant tests, as shown in figure 4, it is strictly an empirical equation. For this reason, equation (9) would probably be preferred for estimation of pressure drops under conditions which do not fall in the 20 to 35 psia., 63 to 105°F., and 1.17 to 2.0 gpm. of antisolvent ranges, but extrapolation in either case should be exercised with caution.

DECOMPOSER PERFORMANCE

The operating conditions for the decomposer are shown in table XIV. Temperatures of the low temperature and high temperature reboilers were near 295 and 350°F., respectively, and the heat loads, varying somewhat with pressures and flows, were between about 190,000 and 330,000 Btu./hr. for the low temperature unit and between about 14 and 20 kw. for the high temperature reboiler. The heat load for the boron trifluoride cooler was small, ranging from 800 to 4,700 Btu./hr. The estimated overall heat transfer coefficient for the low temperature reboiler decreased from 600 to 250 Btu./hr.-sq.ft.-°F. after the first few runs, while the average estimated coefficient for the boron trifluoride gas cooler was near 2 Btu./hr.-sq.ft.-°F. Pressure drops through the low temperature stripper were near 0.35 inch of water per foot for most of the tests; although the average pressure drop across the high temperature column was higher, i.e., 0.72 inch of water per foot; both of the columns appeared to operate smoothly and below the flooding point.

Samples indicated that the boron trifluoride content of the low temperature decomposer liquid effluent was less than 0.1 weight percent, a value well below the design limit of 3%. Also, for most of the runs, boron lost through the high temperature decomposer to the still as a trimer of boron oxyfluoride, $(BOF)_3$, was also within the design limits of the pilot plant as far as separation data collection at essentially total reflux was concerned. In these runs, the boron content of the still feed stream was below 10 ppm., corresponding to decomposer reflux ratios of 10^4 and estimated production facility product losses, based on a 300 to 1 ratio of reflux to product, of about 1 to 2%. However,

Figure 4

EXCHANGE COLUMN PRESSURE DROPS ESTIMATED
USING AN ALTERED FORM OF TREYBAL'S EQUATION

TABLE XIV
DECOMPOSER OPERATING CONDITIONS AND PERFORMANCE

Run	Column Pressure Drop, inches of water foot of packing		Temperature, °F.		Heat Loads			Amount of Boron in Decomposer Effluent Liquid, ppm.	Decomposer Reflux Ratio	Estimated Product Loss*, %	Estimated Heat Transfer Coefficients, Btu./hr.-sq.ft.-°F.	
	Low Temperature Stripper	High Temperature Stripper	Low Temperature Reboiler	High Temperature Reboiler	Low Temperature Reboiler, Btu./hr.	High Temperature Reboiler, kw.	HF ₃ Cooler, Btu./hr.				HF ₃ Cooler	U-Tube Reboiler
1	0.30	0.73	293.0	345.6	193,000	19.9	2,000	3.5	2.9 × 10 ⁴	1.0	1.8	511
2	0.32	0.67	299.6	345.0	283,000	18.7	2,100	3.0	3.3 × 10 ⁴	0.9	1.7	-
3	0.33	0.74	299.0	368.0	288,000	20.4	2,400	2.0	5.0 × 10 ⁴	0.6	2.1	575
4	0.33	0.74	300.0	345.0	286,000	19.1	1,000	4.0	2.5 × 10 ⁴	1.2	1.1	654
5	0.33	0.74	300.0	346.0	284,000	19.4	1,600	-	-	-	1.3	681
6	0.31	0.74	299.9	345.4	280,000	19.2	1,500	4.0	2.5 × 10 ⁴	1.2	1.2	600
7	0.28	0.74	302.0	347.0	191,000	18.6	800	-	-	-	0.6	661
8	0.26	0.75	295.2	347.8	188,000	19.2	1,000	6.0	1.7 × 10 ⁴	1.8	0.7	219
9	0.28	0.70	295.2	349.3	193,000	18.6	1,600	6.0	1.7 × 10 ⁴	1.8	1.4	225
10	0.35	0.75	291.0	373.0	328,000	21.1	2,700	-	-	-	2.4	289
11	0.39	0.69	287.0	375.0	325,000	16.2	1,700	-	-	-	-	221
12	0.36	0.73	287.4	374.0	324,000	17.4	1,600	43	2.3 × 10 ³	12.9	5.4	221
13	0.36	0.43	291.4	374.3	318,000	17.4	4,700	-	-	-	5.1	271
14	0.36	0.56	290.1	350.0	327,000	14.6	2,300	46	2.2 × 10 ³	13.8	5.4	256
15	0.34	0.55	286.9	349.9	315,000	14.5	1,300	-	-	-	2.2	203
16	0.32	0.50	290.6	352.1	314,000	14.8	900	-	-	-	1.2	252
17	0.46	1.20	301.8	332.5	333,000	19.9	2,200	-	-	-	4.3	267
18	0.44	0.88	299.0	334.0	321,000	17.2	1,400	8	1.2 × 10 ⁴	2.4	2.0	290
19	0.45	0.93	299.2	331.8	317,000	17.5	800	8	1.2 × 10 ⁴	2.4	0.9	294

* Estimated value based on a production facility operating at a reflux ratio of 300 to 1.

during runs 12 and 14, the boron content of the still feed increased to about 45 ppm., reducing the decomposer reflux ratio to 10^3 and increasing the estimated product loss to about 13%. It is not known if this increased boron loss was due to increased anisole degradation or to some undetected operating fluctuation of the high temperature unit, but in subsequent tests, the boron losses did return to below 10 ppm.

RECOMBINER PERFORMANCE

Since the recombiner operating conditions varied considerably with the exchange column requirements for the various tests, and since temperature profiles of the two recombiner units were not obtained, a quantitative assessment of the heat transfer performance of the recombiner was not possible; however, the fact that the units appeared to operate both smoothly to produce complex with the desired composition and efficiently with negligible boron losses implies that the design procedure and chosen parameters discussed previously were at least adequately correct. Samples indicated that about 80% of the complex was produced in the higher temperature unit, with the remaining complex formation and any necessary cooling taking place in the smaller, low temperature unit. Boron losses through venting from the recombiner were extremely low throughout all the tests and, in many instances, the boron trifluoride content of the vent stream was below the limit of detection (less than 0.1%). The losses corresponded to reflux ratios at the recombiner end of the pilot plant of 10^6 or greater.

PURIFICATION STILL PERFORMANCE

The purification still operating conditions are summarized in table XV. At a pressure of about 6 psia., still reboiler temperatures varied from about 260 to 290°F., depending upon the concentration of degradation products in the reboiler. Temperatures just below the condenser were near 260°F. for all runs. Heat loads for the condenser varied from 238,000 Btu./hr. to 430,000 Btu./hr. On the other hand, heat loads for the reboiler were less, appearing to be between 180,000 and 389,000 Btu./hr., because from 15 to 40% of the hot feed flashed as it went from the pressurized decomposer to the vacuum-operated still. The average estimated heat transfer coefficient for the reboiler was near 120 Btu./hr.-sq.ft.-°F. The ratio of liquid boiled up to that removed as product was between 1.6 and 3.6, depending on the system circulation rate. The anisole concentration in the still feed was from 84 to 93 weight percent with the methylated anisoles constituting the major impurity; the anisole content of the still product was generally above 97%. The anisole concentration in the product stream did decrease to about 85% in the last few tests because of a large concentration of reboiler impurities, especially the methylated anisoles which have boiling points near that of anisole. This situation could be remedied easily in a production plant by adding more stages to the still column and was not felt to hurt the pilot plant separation data since these compounds

TABLE XV
PURIFICATION STILL OPERATING CONDITIONS AND PERFORMANCE

Run	Pressure, psia.	Temperature, °F.		Heat Loads, Btu./hr.		Estimated Reboiler Heat Transfer Coefficient, Btu./hr.-sq.ft.-°F.	Estimated Boilup Rate, lb./hr.	Product Rate, lb./hr.
		Reboiler	Top of Column	Condenser	Reboiler			
1	6.00	258.4	258.4	312,000	284,000	118	2,050	575
2	6.00	260.6	258.9	430,000	389,000	162	2,830	860
3	6.00	263.0	262.0	272,000	221,000	93	1,790	860
4	5.85	264.0	260.8	342,000	303,000	142	2,250	860
5	6.00	263.0	259.5	345,000	305,000	143	2,270	860
6	5.91	261.2	258.0	240,000	200,000	116	1,580	860
7	5.90	261.3	260.0	251,000	223,000	103	1,650	575
8	5.80	262.2	248.2	227,000	199,000	105	1,490	575
9	5.70	262.4	249.8	253,000	225,000	121	1,660	575
10	5.82	272.2	255.2	271,000	216,000	112	1,780	980
11	6.00	269.5	258.5	254,000	196,000	94	1,670	980
12	6.00	269.6	259.6	238,000	180,000	89	1,560	980
13	6.08	272.3	259.1	260,000	204,000	105	1,710	980
14	5.89	274.1	255.2	351,000	310,000	166	2,310	980
15	5.93	274.9	257.1	364,000	323,000	166	2,390	980
16	5.82	280.1	259.3	273,000	234,000	112	1,800	980
17	6.00	290.0	261.0	276,000	253,000	106	1,820	980
18	6.00	288.0	260.0	293,000	268,000	108	1,930	980
19	5.90	288.1	260.0	334,000	311,000	120	2,200	980

form complexes with boron trifluoride having separation factors similar to those for anisole.

During the tests, still bottoms were withdrawn at rates varying between 1 and 7% of the high temperature anisole hold-up per day and at anisole compositions ranging from 30 to 50%. Finally, although the pressure drops for the packed still column section alone were not available, the column appeared to operate below the flooding point.

ANISOLE DEGRADATION

Chromatographic analyses of still reboiler samples provided the primary source of information concerning anisole degradation. The degradation products observed were essentially the same as those listed in tables IX and X, but small amounts of one additional compound, m-methyl anisole, were also found. Based on these analyses, the anisole degradation rate appeared to be about 3.7% of the high temperature anisole hold-up per day during the first 9 runs and about 7% per day during the remaining tests. Although this increase apparently was caused by operating at about 36 psia. during runs 10, 11, 12, and 13, it was coincident with an inadvertent release of boron trifluoride from the decomposer to the still during a start-up. This release may have temporarily changed the reactivity of the metal surface in the still, thereby providing an additional site for degradation. Other possible factors are mentioned later. Both the increase in degradation rate with increasing pressure and the failure of the degradation rate to return to the value observed at lower pressures were noticed in the bench-scale tests mentioned previously. It is interesting to note that degradation rates based on boron losses to the still and rates based on the quantity of methyl fluoride found in the recombiner vent were on the order of 1% or less of the high temperature anisole hold-up per day during the first 9 runs and from 2 to 4% per day during runs 10 through 19; however, the methods used in these determinations are felt to be less accurate than the method based on actual still reboiler sample analyses. These numbers do have a significance, though, in that they allow at least a qualitative verification of the different degradation mechanisms, as discussed by Barber and Madix⁽²⁾.

The question of anisole degradation at room temperatures and at temperatures near the exchange column operating temperatures remained essentially unanswered by these studies because it was impossible to distinguish between degradation in the auxiliary units and degradation in the column. However, analysis of one boron trifluoride-anisole complex sample allowed to stand at room temperature in a nickel sample tube for 133 days did indicate an anisole degradation rate near 0.5% per day under these conditions. On this basis and in the absence of further information, it was felt that reasonable degradation rates for a production facility could be estimated by applying the 0.5% per day rate to column inventory and summing with the results of using the 3.7 to 7.0% per day value for still and decomposer inventories.

CORROSION

Since the pilot plant units were not inspected metallurgically following the tests, no actual corrosion rates and mechanisms are available. A limited visual examination did not reveal any serious attack. Also, although the metallo-organic compounds resulting from corrosive attack generally are rather insoluble, samples of the stripped anisole stream exiting from the decomposer were periodically analyzed for iron, nickel, and chromium concentrations in order to provide an indication of major corrosive attack. In all cases, including the runs during which degradation seemed most severe, the concentrations of these elements were found to be below the lower limit of detection (less than 0.5 ppm.). Thus, it appeared that no serious corrosion was occurring. Based on analyses of still bottoms, essentially all of the metallic impurities fed to the still remained in the reboiler. This was expected because of bench-scale test results.

MISCELLANEOUS RESULTS

With only a few minor exceptions during the initial shakedown tests, all auxiliary equipment, such as metering pumps and instruments, functioned well. Molecular Sieve traps effectively reduced the water content of all anisole charged to the pilot plant system to below 50 ppm., and the system water content remained near this value during all tests.

Finally, the isotopic equilibrium time was less than 5 hours and may have been less than 2 hours. The rapidity with which isotopic equilibrium was reached reflected the relatively small product hold-up in the decomposer and exchange column as compared with the hold-up which would be present in a production facility with many columns.

CONCLUSION

The pilot plant goals previously outlined were attained with the collection of engineering-scale data concerning stage heights, exchange column flooding and pressure drop characteristics, solvent degradation, complex dissociation and recombination, anisole drying and purification, and the performance of selected materials of construction.

Stage heights with 5/8-inch Pall rings were found to be larger than originally assumed, but not so high as to rule out the use of this packing in a production facility. In fact, although a detailed economic analysis was not made, it appears that the cost per stage still would be lower for Pall rings than for high efficiency protruded packing, at the higher flows, primarily because of the higher cost and the smaller allowable flows characteristic of the latter. Additionally, at least some compensation for the increased stage heights is offered by the column pressure drop results. Although the maximum allowable or flooding flow rate was essentially the same as had been estimated originally,

the pressure drops below the flooding point were significantly lower than initially anticipated. This means that stable operation of a production facility now appears to be possible at a higher percentage of flooding than was previously expected, thereby either increasing the maximum practical operating throughput for a given column diameter or reducing the column diameter required for a specified flow rate. In short, the combined experimental HTU and pressure drop results served both to lengthen and to narrow the previously envisioned production plant.

The anisole degradation rates both before and after the high pressure separation tests were greater than the rates suggested by earlier bench-scale degradation test results; however, it should be mentioned that the turnover time for the pilot plant was on the order of 1 hour, whereas that for the small-scale system was approximately 1 day. Furthermore, degradation in the pilot plant was increased, perhaps, not only by the release of boron trifluoride to the still as previously indicated, but also by the high temperatures (near 200°F.) encountered in the exchange column during start-ups of the tests, when the complex formation reaction zone moved through the uncooled column to the recombiner. For these reasons, it seems possible that the surfaces of both the still and the exchange column could have been altered sufficiently to allow these units to act, at least temporarily, as degradation sites. In this respect, operation of a production facility would be more similar to the small-scale tests, since turnover times would be increased and since numerous start-ups and changes of operating conditions would not be necessary. On this basis, the anisole degradation rates observed in the pilot plant tests could possibly be considered to be maximum values for the ends of the process, with an even further reduction in degradation possible by operating the decomposer at a lower pressure and temperature and then compressing the gas to reach the pressure desired in the first exchange column. Compressor costs, of course, would strongly influence their location in the process. In any event, anisole degradation at the lower temperatures expected in the exchange columns seems to require some further study.

With the use of two temperature levels in the dissociation and the recombination steps, boron losses at both ends of the plant were encouragingly low. It should be mentioned that, even if the decomposer operation were less efficient, laboratory work showed that enriched material lost to the still could be recovered from the still bottoms as boric acid. Continuous distillation of the anisole proved to be effective in isolating anisole from its degradation products as long as still reboiler impurities remained below about 50%. Additionally, adsorption on Molecular Sieves was sufficient to reduce to tolerable limits the water content of all anisole charged to the system.

During the tests, there was no evidence to indicate any severe corrosion; therefore, the choice of materials of construction seems satisfactory for this process, with the possible exception that further degradation tests might result in the substitution of nickel packing for stainless steel packing in the exchange columns.

Finally, system operation was generally smooth, and none of the problems of surges, pressure excursions, and other instabilities reported in earlier studies of this process⁽⁹⁾ were encountered. In short, no apparent technological barriers to the application of the anisole process on a production scale were found.

ACKNOWLEDGEMENT

Throughout the design, construction, and operation of the engineering-scale facility described herein, several groups at the Oak Ridge Gaseous Diffusion Plant provided valuable assistance. The authors would like to express their gratitude for this extensive participation.

Other members of the Engineering Development Department (to which the authors belong) made very substantial contributions to the pilot plant effort. S. H. Smiley, the Department Superintendent, provided much of the overall project direction and made significant technical suggestions, while D. C. Brater supervised operation of the facility. Also contributing was D. L. Breton, who made many of the design calculations.

Members of the Chemical Development Department had a marked influence on the project. E. J. Barber and J. L. Madix made many helpful suggestions and performed laboratory experiments which provided valuable information on corrosion, solvent degradation, and the physical and chemical properties of the process materials. Their helpful review of this report is also appreciated. Additionally, C. G. Jones supplied autoclave corrosion data which were employed in making the initial material of construction selections.

Based on their cost studies for possible plant systems, S. A. Levin, M. Hanig, and S. C. Jacobs, of the Operations Analysis Division, established ranges of temperatures, pressures, and flow rates to be included in the pilot plant studies. Additionally, they reviewed the completion report and related pilot plant results to cascade design.

The Works Laboratory made a major contribution to the project. J. C. Horton established reliable procedures for boron isotopic analysis; J. G. Million developed gas-liquid chromatographic techniques for analyzing contaminated anisole samples; R. J. Wertz provided, on a routine basis, density analyses of hard-to-handle boron trifluoride-anisole complex samples; and J. T. Mottern determined the concentrations of iron, nickel, chromium, boron, and water in samples from various system streams.

The Plant Engineering Division furnished active project support, particularly during the design phase. J. A. Iacovino, S. W. Fiveash, and others were responsible for significant contributions regarding design details and for preparation of the engineering drawings and flow sheets.

The Maintenance Division's careful fabrication of specialized, leak-free equipment was a major factor in the successful exclusion of water from the pilot plant system and thus was an important contribution to the ease of operation.

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APPENDIX

PILOT PLANT EQUIPMENT AND PROCESS DESCRIPTION

The boron isotope separation pilot plant consists of an isotopic exchange column, decomposer and recombiner units, a purification system, two coolant loops, auxiliary equipment such as pumps, and, of course, the required instrumentation. In this appendix, each of these items is discussed in detail with respect to both its role in the system flow scheme and the relevant construction details. Before proceeding with the specific explanations, however, a summary of the flow pattern is presented.

The process flow sheet is figure 5. As can be seen from this drawing, the boron trifluoride-anisole complex leaving the exchange column is pumped to the feed plate of the low temperature stripping column. Complex flowing down through this packed column is stripped by contact with hot anisole and boron trifluoride rising from the steam heated low temperature decomposer reboiler and the high temperature decomposer column. The low temperature decomposer column contains a combination off-gas cooler and reflux condenser section mounted above the feed plate. Stripped complex from the low temperature reboiler flows by gravity to the packed high temperature decomposer column, where it is further stripped by contact with vapors rising from the electrically heated high temperature decomposer reboiler. Liquid anisole is withdrawn from the reboiler and flows by system pressure differential to the vacuum-operated anisole purification still.

Continuous measurement of the liquid level in the reservoir at the bottom of the exchange column is required to hold a constant inventory at this point and to prevent the diaphragm pump from either entraining column gas or allowing accumulation of liquid in the column. The pump is set so as to pump slightly more fluid than that which is required to hold a constant inventory, and the level measurement instrumentation is used to actuate a small control valve which allows recycle of material around the pump as needed to hold the desired level. Similarly, a control is required to maintain the liquid in the high temperature reboiler at a constant level thereby preventing release of product gas to the distillation column.

The anisole product from the purification still is removed from a flow splitter, which is mounted between the still column and condenser, and flows from there into the still product tank. The rate of product removal is adjusted automatically so that a constant level can be maintained in the still reboiler. Still bottoms are periodically withdrawn as dictated by analyses of degradation product concentrations in the reboiler. Since the still operates at a controlled vacuum, product is withdrawn into an evacuated storage tank which is vented to the still equipment. From this tank, the purified anisole is metered to the recombiner. This automatically controlled anisole flow establishes the

overall system recycle rate; therefore, a highly accurate diaphragm metering pump is utilized. Venting of the recombiner, which is necessary for removal of inerts and volatile degradation products, is controlled from the recombiner head pressure while the addition of boron trifluoride to the system is accomplished manually whenever it is necessary to adjust the complex composition as indicated by the continuous density measurement of the recombiner liquid exit stream. Completing the circuit, the complex formed in the recombiner is then pumped from a small surge volume into the exchange column. The liquid level in the recombiner surge volume is monitored continuously and is held constant by recycling around the pump in the manner described previously for the exchange column. It should be noted that in the pilot plant the recombiner and decomposer operate at essentially the same pressure as the exchange column, since neither a gas compressor for pumping from the decomposer nor a throttling valve for reducing the pressure between the exchange column and the recombiner was provided.

The coolant interchange flow sheet is figure 6. Two interchange loops are used, one of which supplies refrigerated anisole at about 30°F. to the low temperature portion of the recombiner and to the boron trifluoride cooler; the other furnishes anisole cooled with water to about 73°F. to the purification still condenser and the higher temperature recombiner unit.

The refrigerated anisole loop consists of a hold tank, a pump, and a refrigeration unit connected in series. For operation at higher temperatures, a portion of the anisole by-passes the refrigeration system and then blends with the effluent from that system in order to maintain the desired coolant temperature. The water-cooled anisole loop employs a hold tank, a pump, and a water-cooled shell and tube heat exchanger in series. Flows in both loops are varied by manually adjusting valves.

In summary, the four major systems of the pilot plant, i.e., the isotopic exchange system, the decomposer system, the recombiner system, and the anisole purifications system, have approximately 700 square feet of heat exchange surface area and 42 feet of packed column height. These process equipment sizes, coupled with the numerous valves, instruments, pumps, and other auxiliaries, show that this unit is indeed comparable in size, except for the exchange column length, to production equipment. The various equipment items are described in the following sections.

EXCHANGE SYSTEM

Boron trifluoride-anisole complex leaves the recombiner reservoir through 1-inch stainless steel pipe and flows through a cooling bath into the suction side of the exchange column feed pump. To prevent flashing in the pump, the manufacturer recommended that the suction pressure be maintained at least 5 psi. above the vapor pressure of the liquid being pumped. Since space limitations prevented installing the recombiner equipment high enough over the pump to provide a liquid head sufficient to increase the suction pressure by the required amount, a cooling bath

designed to reduce the temperature and therefore the vapor pressure of the suction stream was necessitated. The exchange column feed pump effluent is fed through a 1-inch line containing a pulsation dampener to the top liquid distributor plate of the column, from which it flows countercurrently to a gaseous stream of boron trifluoride sent to the bottom of the column through 2-inch pipe from the decomposer. Gas exiting from the top of the column is returned to the recombiner through a 2-inch line, while any make-up gas required is fed to the lower or middle section of the column through either of two 3/4-inch lines.

Equipment Description

1. Exchange Column, B-100, Figure 7

Purpose: To provide a site for the isotopic exchange between the boron isotopes in countercurrent streams of gaseous boron trifluoride and liquid boron trifluoride-anisole complex, with the resulting enrichment of boron-10 in the liquid phase.

Design Basis: Operating Temperature - 68 to 104°F.

Operating Pressure - 5 to 35 psig.

Diameter - 6 inches

Total Height - 17 feet 2-1/2 inches

Packed Height - 13 feet 6 inches

Packing Support Plates - 3

Liquid Distributors - 3

Hold-Down Plates - none

Packing - 5/8-inch Pall rings

Insulation - none

Materials of Construction -

Column: Stainless Steel 304L

Packing: Stainless Steel 316

The isotopic exchange column consists of four sections of 6-inch diameter, schedule 40S, stainless steel 304L pipe, flanged and seal welded. The upper section of the column, 13 inches long, is the site of liquid entrance and gas exit. Liquid flows into this section through a 1-inch nozzle located on the side and is directed downward toward the distributor by a 1-inch standard 90° ell. The

gas exit line is 2 inches in diameter and is connected to the top of the 6-inch standard weight weld cap used to form the top head of the column. This line contains a manually operated, 1-inch stainless steel gate valve for use in connecting the unit to the scrubber system if required.

The three remaining column sections, each 5 feet 4-1/2 inches long, contain the 13.5 feet of 5/8-inch, stainless steel 316 Pall rings. Each section contains 4.5 feet of the packing, as well as a packing support plate and a liquid distributor. The internals were modified after preliminary air-water tests had proven them to be unsatisfactory, and the modifications are shown in figure 8. The bases of the new support plates were fabricated in half-sections from 1-inch lengths of 5-inch pipe. Packing support which did not restrict gas flow then was provided by attaching corrugated, 2 by 2-mesh, stainless steel screen (with corrugations 1 inch deep and 1/2 inch apart) to the bases. The liquid distributors were modified to provide for a more even distribution of liquid to the bed. This was accomplished by drilling additional 1/4-inch diameter holes in the bottoms of the U. S. Stoneware Company Figure 845 "Orifice" distributors and then inserting 1/4-inch stainless steel tubing through each hole. The tubing, which was extended 1 inch below the plates, also was slotted at the top. Sections of stainless steel welding rod were tacked across the gas risers to prevent sweeping any packing by the distributor plate during the flooding tests, thereby eliminating the need for a hold-down plate.

The principal gas feed to the column is through a 2-inch nozzle located below the support plate in the bottom section of the column; however, boron trifluoride make-up lines, 3/4 inch in diameter, are similarly attached to the remaining column sections. Finally, three coils of 5/8-inch copper tubing are wound around the three main column sections to furnish either heating or cooling, but these lines were not required during the tests.

2. Exchange Column Reservoir, B-101, figure 7

Purpose: To provide a location for liquid level control instrumentation and to hold the contents of the column during temporary shutdowns.

Design Basis: Diameter - 6 inches

Height - 2 feet 3 inches

Capacity - about 3.5 gallons

Cooling Provided - none

Insulation - none

Material of Construction - stainless steel 304L

The exchange column reservoir is a section of 6-inch diameter, schedule 40S, stainless steel 304L pipe, integrally connected to the bottom of the column by flanging and seal welding. Bubbler-type liquid level instrumentation is located in this unit, and a 1-inch complex exit line extends from the bottom of the 6-inch standard weight weld cap used to form the bottom head of the reservoir.

DECOMPOSER SYSTEM

Boron trifluoride-anisole complex, enriched in boron-10, exits from the exchange column reservoir through the previously mentioned low temperature bath and flows to the suction side of the decomposer feed pump. Using a diaphragm metering pump, the complex then is delivered through 1-inch pipe to the top of the low temperature decomposer stripper. Flowing downward through this packed column, the complex is contacted with boron trifluoride gas and anisole vapors rising from the other decomposer sections. The partially stripped liquid stream then passes into the shell of the low temperature reboiler, which contains a steam-heated U-tube bundle. Most of the complex dissociation occurs in this unit. An overflow leg maintains a constant liquid level in the low temperature reboiler, and the liquid drains downward from the leg through a high temperature stripper column and into the electrically heated, high temperature reboiler for final stripping of the boron trifluoride. Liquid and gas lines connecting the various decomposer units are made of 1-inch and 4-inch stainless steel 304L pipe, respectively, except for a 2-inch gas line connecting the high temperature stripper and low temperature reboiler.

A constant level is maintained in the high temperature reboiler, and the exit anisole stream flows through a seal leg and control valve into the still reboiler. After passing over a finned-tube cooler, the gas stream leaving the decomposer system is returned to the bottom of the exchange column through 2-inch stainless steel pipe.

Equipment Description

1. High Temperature Reboiler, B-200, Figure 9

Purpose: To provide the heat necessary to raise the stripped liquid stream to 340°F. and to complete the complex dissociation.

Design Basis: Operating Temperature - 340 to 375°F.

Operating Pressure - 5 to 35 psig.

Description of Unit - electrically heated heat exchanger

Shell Diameter - 8 inches

Total Length - 10 feet 4 inches

Heat Transfer Area - 11 square feet

Heating Required - 90,000 Btu./hr.

Heat Transfer Medium - electrical

Insulation - 4-inch thick magnesia

Material of Construction - stainless steel 304L

The high temperature decomposer reboiler is a horizontally mounted vessel made from 8-inch, Schedule 40, stainless steel 304L pipe, enclosed at each end by 8-inch standard weight weld caps. Four-inch and 1-inch nozzles, located on the top of the reboiler, provide for vapor exit and liquid inlet, respectively. Also located on the top of the reboiler is a 4-inch inspection port, while a 1-inch line for anisole exit is positioned at the bottom. Electrical heaters cover the bottom half of the unit, and the entire reboiler is lagged with 4 inches of magnesia. A constant liquid level is maintained in the reboiler by instrumentation located in an adjacent, 3-inch diameter, 27-inch long seal leg.

2. High Temperature Stripper, B-201, Figure 10

Purpose: To provide a site for the partial dissociation of the boron trifluoride-anisole complex.

Design Basis: Operating Temperature - 260 to 375°F.

Operating Pressure - 5 to 35 psig.

Diameter - 6 inches

Total Height - 5 feet 8-3/8 inches

Packed Height - 2 feet 10 inches

Packing Support Plates - 1

Liquid Distributors - 1

Hold-Down Plates - none

Packing - 5/8-inch Pall rings

Insulation - 2-inch thick magnesia

Materials of Construction -

Column: stainless steel 304L

Packing: nickel

The 5-foot 3-3/8-inch long high temperature stripper is a section of 6-inch diameter, Schedule 40S, stainless steel 304L pipe enclosed at both ends with 6-inch standard weight weld caps. A 2-inch nozzle located in the top allows gas return to the low temperature stripper. Liquid drains into the column from the low temperature reboiler through 1-inch pipe and is directed downward to the distributor by a 1-inch, 90-degree ell. The packed height is 2 feet 10 inches of 5/8-inch Pall rings, and the packing support and liquid distributor plates are identical to those used in the exchange column and shown in figure 8. Four-inch and 1-inch nozzles located in the bottom of the column connect with vapor and liquid lines leading to the high temperature reboiler. Two-inch thick magnesia insulation is used.

3. Low Temperature Reboiler, B-202, Figure 11

Purpose: To provide the heat required for most of the complex dissociation.

Design Basis: Operating Temperature - near 300°F.

Operating Pressure - 5 to 35 psig.

Description of Unit - U-tube bundle, shell and tube heat exchanger

Shell Diameter - 20 inches

Tube Diameter - 1 inch

Total Length - 10 feet 3-9/16 inches

Tube Length - 16 feet before bending

Number of Tubes - 20

Heat Transfer Area - 84 square feet

Heating Required - 400,000 Btu./hr.

Heat Transfer Medium - steam (up to 100 psig.) on tube side

Insulation - 2-inch thick magnesia

Material of Construction -

Steam Head: steel

Other: stainless steel 304L

The low temperature decomposer reboiler is a shell and tube heat exchanger containing 20 U-tubes; heat is supplied by steam on the

tube side. The reboiler shell is fabricated from 20-inch, schedule 10, stainless steel 304L pipe with an overall length of 10 feet 3-9/16 inches, while the U-tubes are 1-inch diameter, 0.083-inch wall, stainless steel 304L tubing. The tubes are welded to a 1-1/2-inch thick stainless steel 304L tube sheet which, in turn, is welded to the shell. The steam side head is a 20-inch diameter flanged and dished steel head, 3/8 inch thick, welded to a 7-5/16-inch length of 20-inch diameter steel piping containing two 1-1/2-inch diameter steam nozzles. The steam head is flanged to the tube sheet and sealed with a compressed asbestos gasket.

Liquid enters the top of the reboiler through 1-inch pipe, while gas exits through a nearby 4-inch line. Also at the top is a 4-inch inspection port. A 1-inch drain line extends from the bottom of the unit to the bottom of an overflow leg, 3 inches in diameter and 26-1/2 inches high, which is designed to keep the reboiler liquid level constant and above the tubes. A 1-inch line extending from the overflow device carries liquid to the high temperature stripper.

4. Low Temperature Stripper, B-203, Figure 12

Purpose: To provide a site for partial dissociation of the boron trifluoride-anisole complex.

Design Basis: Operating Temperature - 100 to 260°F.

Operating Pressure - 5 to 35 psig.

Diameter - 12 inches

Total Height - 6 feet 8 inches

Packed Height - 4-1/2 feet

Packing Support Plates - 1

Liquid Distributors - 1

Hold-Down Plates - 1

Packing - 1-inch Pall rings

Insulation - 2-inch thick magnesia

Materials of Construction -

Packing: nickel

Other: stainless steel 304L

2

The low temperature stripper, made of schedule 20, stainless steel 304L pipe, has a diameter of 12 inches and an overall length of 6 feet 8 inches. The unit, which is sealed at the bottom by a 12-inch standard weight weld cap, contains 4-1/2 feet of 1-inch nickel Pall rings supported by a 12-inch, U. S. Stoneware Company Figure 796H "Gas-Injection" support plate. The top of the bed is provided with both a hold-down plate and a packing support plate. The hold-down plate consists of a ribbed stainless steel ring, 11 inches in diameter and 1/2 inch high with two-by-two mesh stainless steel screen welded to the ring. The entire assembly is then integrally attached to a 12-inch, U. S. Stoneware Company Figure 798 "Weirflow" liquid distributor by three 1/4-inch stainless steel rods, 7 inches long. The 4-inch gas line from the low temperature reboiler enters the column immediately below the support plate and the liquid drains from the bottom to the reboiler through 1-inch pipe. Gas leaves the column through the boron trifluoride cooler.

5. Boron Trifluoride Cooler, B-204, Figure 12

Purpose: To cool the boron trifluoride exiting from the decomposer and to reflux small amounts of complex to the low temperature stripper.

Design Basis: Operating Temperature - 70 to 105°F.

Operating Pressure - 5 to 35 psig.

Description of Unit - finned tube heat exchanger

Shell Diameter - 12 inches

Tube Diameter - 1 inch

Total Height - 2 feet 8 inches

Tube Length - 39 feet 3 inches (total for 2 coiled tubes)

Heat Transfer Area - 26 square feet

Cooling Required - less than 12,000 Btu./hr.

Coolant - anisole

Coolant Flow - 10 gpm.

Coolant Temperature - 30°F.

Insulation - 2-inch thick magnesia

Material of Construction -

Tubes: stainless steel 316

Other: stainless steel 304L

The boron trifluoride cooler is a continuation of the low temperature decomposer stripper and is joined to that vessel by lap-joint flanges with a seal weld. The shell of this cooler is built in the same manner as the shell for the low temperature column and is similarly capped. Coolant for this section is delivered through two tubes coiled in the annular space between the shell and a 16-inch section of 6-inch, schedule 40, stainless steel pipe concentrically located inside the shell. The tubes are 1-inch, integrally finned, stainless steel 316 tubing with a wall thickness of 0.083 inch. Two 1-1/2 inch nozzles provide for coolant entrance and exit. A 1-inch line located below the cooling coils delivers complex to the low temperature column, and a 2-inch nozzle at the top of the cooler allows for boron trifluoride exit to the exchange column.

RECOMBINER SYSTEM

Boron trifluoride from the top of the exchange column and anisole from the still product tank are reacted in the recombiner units to form complex. The smaller of the two primary units is used to saturate fully and to cool the complex, while the larger unit is the site of most of the complex formation and, for this reason, bears most of the cooling load. The larger unit also contains a cooling coil to provide a site for complexing with fresh anisole any boron trifluoride remaining in the exit gas stream. Boron trifluoride enters the bottom of the smaller recombiner unit through 2-inch stainless steel pipe, while anisole is pumped from the still product storage tank through a 1-inch stainless steel line across the cooling coils of the larger unit. A diaphragm metering pump is used to supply a steady liquid stream, and the liquid feed line is equipped with a pulsation dampener. Liquid flows by gravity from the larger unit into the smaller recombiner section, in which complex saturation is completed. The two recombiner sections are connected by a 2-inch gas line and a 1-1/2-inch liquid line; unabsorbed gases are removed through a 2-inch vent line from the top of the recombiner system at a rate controlled so as to keep the system pressure at the desired value. A diaphragm-operated control valve is employed.

Complex leaves the recombiner system through a 1-inch line and enters a reservoir, where a constant liquid level is maintained. A radiation-type density meter provides a continuous check of the composition of the complex in the reservoir.

Equipment Description1. Recombiner, B-300, Figure 13

Purpose: To provide a site for about 20% of the complex formation while cooling the complex to the desired operating temperature.

Design Basis: Operating Temperature - 68 to 104°F.

Operating Pressure - 5 to 35 psig.

Description of Unit - shell and tube heat exchanger

Shell Diameter - 16 inches

Tube Diameter - 1 inch

Total Height - 7 feet 1 inch

Tube Length - 4 feet

Number of Tubes - 109

Heat Transfer Area - 115 sq.ft.

Cooling Required - about 106,000 Btu./hr.

Coolant - anisole

Coolant Flow - 25 gpm.

Coolant Temperature - 30°F.

Insulation - 2 inches of hair felt covered with aluminum sheet

Materials of Construction -

Shell: steel

Other: stainless steel 304L

This section of the recombiner, which is the smaller of the two primary recombiner units, is a shell and tube heat exchanger with tubes filled with 5/8-inch stainless steel Pall rings in order to present a greater contact surface for mass transfer. The main body of the shell is 4 feet long and is fabricated from 16-inch, schedule 30, steel pipe. The 16-inch flanged and dished stainless steel 304L heads are welded to 7-1/2-inch lengths of 16-inch, schedule 10, stainless steel 304L pipe and then flanged and seal welded to the outside of the tube sheets. Two 23-1/2-inch diameter, stainless

steel 304L tube sheets, 1-1/2 inches thick, are welded to the shell. Each tube sheet is drilled to accommodate one hundred nine 1-inch, stainless steel 304L tubes on a 1-1/4-inch triangular pitch. The tubes, each 4 feet long with a wall thickness of 0.083 inch, are welded into the tube sheets at both the top and bottom of the unit. The tops of the tubes are extended 3/8 inch above the tube sheet to allow for two 1/4-inch deep V-notches, 180 degrees apart, in each tube. The notches function as flow distribution weirs to provide the same liquid flow rate to each tube. There are three segmental baffles, spaced 1 foot apart, in the shell, and two 2-inch shell nozzles are used for coolant entrance and exit. The shell is insulated with 2 inches of hair felt covered with aluminum sheet.

The upper head of this unit contains a 2-inch gas line, as well as a 1-1/2-inch line for delivery of complex from the other recombiner unit. This liquid feed line, after entering the head, is bent in a 15-1/4-inch circle and is sealed at one end. Twenty-one 1/8-inch holes are drilled on the outside of the torus to force liquid to discharge against the side of the head rather than to allow it to flow directly into some of the tubes, thereby starving others. The bottom head contains a 2-inch boron trifluoride entry nozzle and a 1-inch complex drain line leading to the recombiner reservoir.

2. Recombiner, B-301, Figure 14

Purpose: To provide a site for 80% of the formation of the anisole-boron trifluoride complex.

Design Basis: Operating Temperature - 110°F. (average)

Operating Pressure - 5 to 35 psig.

Description of Unit - shell and tube heat exchanger

Shell Diameter - 16 inches

Tube Diameter - 1 inch

Total Height - 11 feet 6-7/8 inches

Tube Length - 10 feet

Number of Tubes - 109

Heat Transfer Area - 288 sq.ft.

Cooling Required - about 250,000 Btu./hr.

Coolant - anisole

Coolant Flow - 35 gpm.

Coolant Temperature - 70°F.

Insulation - 2 inches of hair felt covered with
aluminum sheet

Materials of Construction -

Shell: steel

Other: stainless steel 304L

Except for three differences, this section of the recombiner is identical to the smaller unit described previously. First, the shell and the stainless steel 304L tubes are 10 feet long, thereby necessitating nine shell baffles. Second, three shell nozzles allow various coolant flow patterns; and third, the upper head contains a cooler, B-302, which is described next.

3. Recombiner, B-302, Figure 14

Purpose: To precool the anisole inlet stream to the recombiner and to remove, if necessary during final stripping, any reaction heat.

Design Basis: Operating Temperature - 70°F.

Operating Pressure - 5 to 35 psig.

Description of Unit - finned tube heat exchanger

Shell Diameter - 16 inches

Tube Diameter - 1 inch

Total Height - 21-7/8 inches

Tube Length - 32 feet 3 inches (total for 2 coiled tubes)

Heat Transfer Area - 22 sq.ft.

Cooling Required - less than 20,000 Btu./hr.

Coolant - anisole

Coolant Flow - 5 gpm.

Coolant Temperature - 30°F.

Insulation - none

Materials of Construction -

Tubes: stainless steel 316

Other: stainless steel 304L

This section of the recombiner is integrally associated with the larger recombiner unit and is joined to that vessel with a flange and seal-weld arrangement. The shell of this section is 16-inch, stainless steel 304L pipe, and the top consists of a 16-inch, flanged and dished, stainless steel 304L head. Coolant is distributed through two tubes coiled in the annular space between the shell and the 9-1/2-inch diameter sleeve enclosed by the shell. The tubes are 1-inch, integrally finned, stainless steel 316 tubing, and are attached to two 1-1/2-inch nozzles for coolant supply and discharge. Unabsorbed gas is vented through a 2-inch line located at the top of the unit.

Anisole enters the recombiner through a 1-inch line which, after penetrating the shell, is bent on a 15-1/2-inch diameter and is sealed at the end. This line is perforated to deliver anisole to the cooling coils. As the chilled anisole flows from the coils, it is deflected to the outside of the shell by a 14-7/8-inch outside diameter, 2-3/4-inch high truncated cone fastened to the bottom of the sleeve. This arrangement prevents preferential delivery of the anisole to any of the tubes of the larger recombiner section.

4. Recombiner Reservoir

Purpose: To provide a site for liquid level and density instrumentation and to hold the contents of the recombiner during temporary shutdowns.

Design Basis: Diameter - 6 inches

Height - 41-1/2 inches

Capacity - about 5 gallons

Cooling Provided - none

Insulation - none

Material of Construction - stainless steel 304L

The recombiner reservoir is fabricated from 6-inch, schedule 40, stainless steel 304L pipe, 41-1/2 inches high. The flat, stainless steel end plates are 1/4 inch thick. This unit is equipped with both liquid level and density instrumentation. Complex inlet and outlet nozzles are 1-1/2-inch pipe.

PURIFICATION STILL SYSTEM

Anisole from the seal leg used at the high temperature decomposer reboiler flows to the still reboiler through a 1-inch line. An air jet ejector maintains the still system pressure at about 6 psia., so no pumps are required for the transfer. A 4-inch vapor line and a 1-inch

liquid return line connect the reboiler to the packed still column, and in turn, a 3-inch vapor line connects the column to the condenser. Non-condensables are removed from the condenser through the jet, and a constant nitrogen purge is used at this point to minimize pressure cycling and to provide a means for pressure recovery should the jet pull the system down too far. A check valve in the jet suction line prevents backflow of air into the condenser. Condensed liquid drains to a flow splitter, from which a part is routed through a double pipe heat exchanger to a water-cooled product tank. The product rate is automatically set so as to maintain a constant level in the reboiler. A diaphragm control valve is used to throttle the flow to the product tank with the remaining condensate being refluxed through 1-inch pipe and a rotameter. The flow splitter assures that at least half of the boilup is returned to the column as reflux. At rates determined by analytical results, still bottoms are withdrawn from the reboiler through a 1-inch line connected to an evacuated waste drum.

Equipment Description

1. Still Reboiler, B-400, Figure 15

Purpose: To provide the heat necessary for the continuous distillation of the process anisole.

Design Basis: Operating Temperature - 250 to 300°F.

Operating Pressure - 6 psia.

Description of Unit - U-tube heat exchanger

Shell Diameter - 20 inches

Tube Diameter - 1 inch

Total Length - 10 feet 3-9/16 inches

Tube Length - 16 feet before bending

Number of Tubes - 20

Heat Transfer Area - 84 sq.ft.

Heating Required - about 400,000 Btu./hr.

Heat Transfer Medium - steam (up to 100 psig.)

Insulation - 2-inch thick magnesia

Materials of Construction -

Steam Head: steel

Other: stainless steel 304L

Except for a 1-1/2-inch rather than a 1-inch liquid entrance nozzle on the top of the reboiler, the specifications for this unit are the same as those discussed previously for the low temperature decomposer reboiler.

2. Still Column, B-401, Figures 16 and 17

Purpose: To separate anisole from its high boiling degradation products.

Design Basis: Operating Temperature - 260 to 290°F.

Operating Pressure - 6 psia.

Diameter - 16 inches

Total Height - 10 feet 5-7/8 inches

Packed Height - 6 feet 9 inches

Packing Support Plates - 1

Liquid Distributors - 1

Hold-Down Plates - 1

Packing - 1-inch Pall rings

Insulation - 2-inch thick magnesia

Materials of Construction -

Column: stainless steel 304L

Packing: nickel

The purification still column is fabricated from 16-inch, schedule 10, stainless steel 304L pipe and is 10 feet 5-7/8 inches long. The 6-foot 9-inch deep bed of 1-inch nickel Pall rings is supported at the bottom by a U. S. Stoneware Company Figure 796H "Gas-Injection" support plate, while liquid distribution is effected at the top of the bed using a U. S. Stoneware Company Figure 798 "Weirflow" distributor. A hold-down plate, illustrated in figure 8, is attached to the distributor in the same manner described for the low temperature decomposer stripper. The ends of the column are enclosed by 16-inch diameter flanged and dished heads which are flanged and seal welded to the column. Liquid return and vapor feed to the condenser are provided for by 1-inch and 3-inch lines connected to the top of the column, and 1-inch and 4-inch liquid and gas nozzles at the bottom of the column connect it to the reboiler.

3. Still Condenser, B-402, Figure 18

Purpose: To reflux distilled anisole to the flow splitter.

Design Basis: Operating Temperature - 260°F.

Operating Pressure - 6 psia.

Description of Unit - shell and tube heat exchanger

Shell Diameter - 8 inches

Tube Diameter - 1 inch

Total Length - 9 feet 7-1/8 inches

Tube Length - 8 feet

Number of Tubes - 31

Heat Transfer Area - 65 sq.ft.

Cooling Required - less than 425,000 Btu./hr.

Coolant - anisole

Coolant Flow - 70 gpm.

Coolant Temperature - 70°F.

Insulation - 2-inch thick magnesia

Materials of Construction -

Shell: steel

Other: stainless steel 304L

The anisole purification still condenser is a shell and tube heat exchanger which is mounted at an angle of 30 degrees from horizontal. The main part of the shell consists of an 8-foot long section of 8-inch, schedule 40 steel pipe. The stainless steel heads, made using 8-inch standard weight weld caps, are connected to the shell by flanges, and the joints are seal welded. Two 1-1/2-inch shell nozzles allow coolant entrance and exit. Liquid and vapor recycle to the still column are provided for by 3-inch and 1-inch nozzles in the lower head. Thirty-one 1-inch, stainless steel 304L tubes, arranged on a 1-1/4-inch triangular pitch, are welded to 1-1/2-inch thick tube sheets. Segmental baffles are spaced 2 feet apart.

4. Still Product Cooler

Purpose: To cool the still product anisole from 260°F. to near the desired operating temperature.

Design Basis: Operating Temperature - 150 to 260°F.

Operating Pressure - 6 psia.

Description of Unit - double pipe heat exchanger

Anisole Line Diameter - 1 inch

Jacket Diameter - 2 inches

Jacketed Length - about 50 feet

Heat Transfer Area - 20 sq.ft.

Cooling Required - less than 200,000 Btu./hr.

Coolant - water

Coolant Flow - 5 gpm.

Coolant Temperature - 70°F.

Insulation - 1-inch thick anti-sweat

Materials of Construction -

Anisole Side: Monel

Coolant Side: steel

The stainless steel anisole product line from the flow splitter is joined to a 1-inch diameter Monel line. The line is then jacketed for about 50 feet in four sections with a steel pipe whose diameter is 2 inches. Water coolant flows countercurrently in the annulus. The outlet from this double pipe heat exchanger is, in turn, connected to the still product tank, and the entire assembly is lagged with 1-inch anti-sweat insulation.

5. Still Product Tank, B-403

Purpose: To provide a reservoir for the purified anisole used to form complex in the recombiner as well as to perform additional product cooling if required.

Design Basis: Diameter - 30 inches

Height - 62 inches

Capacity - 200 gallons

Heat Transfer Area - 10 sq.ft.

Cooling Provided - less than 25,000 Btu./hr., as required

Coolant - water

Coolant Flow - 2 gpm.

Coolant Temperature - 70°F.

Insulation - 2-inch thick hair felt covered with aluminum sheet

Materials of Construction - stainless steel 316

The stainless steel 316 still product tank, 30 inches in diameter and 62 inches high, is equipped with an external cooling coil of 1-inch stainless steel tubing which is welded to the sides of the tank. Anisole enters the top of the tank through a 1-inch line which can be valved, as desired, either to the still product cooler (during normal operation), to the high temperature decomposer reboiler (if it is necessary for some reason to by-pass the still), or to a Molecular Sieve trap (during charging operations). The tank is insulated with 2-inch thick hair felt and covered with aluminum sheet. A sight glass is used to determine the liquid level.

COOLANT SUPPLY SYSTEMS

A flow of approximately 100 gallons per minute of anisole at 70°F. is supplied to the still condenser and to the large recombiner unit by one of the two primary coolant loops. Using a seal-less centrifugal (canned-motor) pump, the anisole is pumped from a 60-gallon, stainless steel 316 storage tank to the tube side of a water-cooled, shell and tube heat exchanger. From this unit, the anisole is delivered through 1-1/2-inch and 2-inch lines to the process equipment items before being returned to the sight-glass equipped storage tank. The lines are equipped with gate valves to allow adjustment of the flow rates as necessitated by changes in process conditions.

The refrigerated anisole supply loop is similar, except that 40 gallons per minute of anisole at 30°F. is directed to the smaller recombiner, the boron trifluoride cooler, and the anisole chiller located atop the larger recombiner. Again, a seal-less centrifugal pump delivers anisole from an identical storage tank to a shell and tube heat exchanger, but the coolant used in this exchanger is evaporating refrigerant-22 supplied by a 20 hp. water-cooled condensing unit. Anisole flow rates from the evaporator to the process vessels are adjusted using gate valves, and additionally, the temperature of the coolant stream is maintained automatically at the desired level by instrumentation which causes the refrigeration

system to be by-passed either partially or completely if the temperature decreases below the set point.

A third, smaller coolant loop is required, as mentioned before, to cool the liquid streams entering the suction sides of the exchange column and decomposer feed pumps. Refrigerant-22 is furnished by a 1-ton condensing unit and is delivered through copper tubing to a glycol bath, where it evaporates, cooling the bath as well as the complex flowing through two Monel coils located in the bath and connected to the pump suction ports.

The refrigeration units used in the refrigerated anisole supply system and in connection with the glycol bath were Copelametic* models W2WM-2000 and KSL-100C, respectively. The storage tanks and heat exchangers were obtained from plant surplus and thus were not designed for this specific application. No attempt was made to collect data from the heat exchanger units, since they were undoubtedly oversized, and for this reason, detailed descriptions would serve no useful purpose here.

AUXILIARY EQUIPMENT

The principal auxiliary equipment items were the three diaphragm metering pumps used to feed the recombiner, exchange column, and the decomposer and the seal-less centrifugal pumps used in the coolant interchange systems. However, other items including pulsation dampeners, an entrainment separator, and Molecular Sieve traps were utilized and are discussed here.

Equipment Description

1. Anisole Pump, B-500

Purpose: To deliver a known steady stream of anisole to the recombiner.

Design Basis: Operating Temperature - 65 to 100°F.

Process Fluid - anisole

Description of Unit - diaphragm metering pump

Capacity - 4 gpm.

Suction Pressure - 5 psi. above vapor pressure

Discharge Pressure - 50 psig.

Motor Horsepower - 1

* Product of the Copeland Refrigeration Corporation, Sidney, Ohio.

Materials of Construction -

Diaphragm: stainless steel 316

Reagent Head: stainless steel 316

Valve Housings: stainless steel 316

Valve Seats: stainless steel 316

Gaskets: Teflon*

Valves: Hastelloy-C† ball checks

The recombiner feed pump is a Lapp Pulsafeeder‡ positive displacement diaphragm metering pump rated to deliver a constant flow of liquid at flows from 0 to 4 gpm. and at discharge pressures at least as high as 50 psig. Suction and discharge ports are 1-1/2 inches in diameter, and the pump is driven by a 1-hp., 1800-rpm., totally enclosed motor wired for operation in a 3-phase, 60-cycle, 220/440-volt, ac. circuit. Ball check valves are employed in the reagent head, and internal pressure relief is provided. Adjustment of the stroke establishes the desired anisole flow rate and thereby sets the system recycle rate.

2. Complex Pumps, B-501 and B-502

Purpose: To deliver a steady flow of boron trifluoride-anisole complex to the exchange column and decomposer.

Design Basis: Operating Temperature - 65 to 100°F.

Process Fluid - boron trifluoride-anisole complex

Description of Unit - diaphragm metering pump

Capacity - 4 gpm.

Suction Pressure - 5 psig. above vapor pressure

Discharge Pressure - 50 psig.

Motor Horsepower - 1

Materials of Construction -

Same as for anisole pump, B-500

* Product of E. I. du Pont de Nemours and Company, Incorporated.

† Product of Union Carbide Corporation.

‡ Product of the Lapp Insulator Company, Incorporated.

The exchange column and decomposer feed pumps are identical to the recombiner feed pump but are installed differently. In order to ensure the maintenance of constant liquid levels in the recombiner and exchange column reservoirs, each of these pumps is connected in parallel with a by-pass line containing a diaphragm-operated control valve with a coefficient of 2.5. The pumps are set to operate at a capacity higher than that necessitated by the recombiner feed pump setting. The control valves are activated by changes in the reservoir levels to allow either increases or decreases in the amount of liquid recycled around the pumps, as required.

3. Coolant Supply System Pumps, B-503 and B-504

Purpose: To supply anisole coolant to various process equipment items.

Design Basis: Operating Temperature - up to 200°F.

Process Fluid - anisole

Description of Unit - seal-less centrifugal (canned-motor) pump

Capacity - 150 gpm. (B-503) and 60 gpm. (B-504)

Suction Pressure - flooded

Discharge Pressure - 50 psig.

Motor Horsepower - 1-1/2

Materials of Construction -

Wetted parts are stainless steel 316, except for Viton-A gaskets and graphite bearings

Both coolant system supply pumps are Series G Chempump* seal-less centrifugal pumps. All wetted parts are made of stainless steel 316 with the exception of Viton-A gaskets and graphite bearings. These pumps are identical except for slight differences in impeller diameters due to the different capacities; both pumps are driven by totally enclosed motors operated in 3-phase, 60-cycle, 220/440-volt ac. circuits.

4. Pressure Relief Systems

Pressure relief systems were provided for the exchange column, the decomposer, the recombiner, and the purification still. Each relief system consists of a 50 psig. Monel rupture disc, a pressure gauge,

* Product of Chempump Division, Crane Company. The 60 and 150 gpm. pumps were Models GA and GC, respectively.

and a 60 psig. pressure relief valve installed in series with the rupture disc on the process side. This arrangement allowed design safety criteria to be satisfied and also protected the system from possible air inleakages through the valve. The pressure gauge was included to indicate penetration of the rupture disc.

5. Pulsation Dampeners

Since complex is discharged from the recombiner and exchange column feed pumps in pulses, dampeners were installed on the discharge lines from these units. The dampeners were fabricated from 6-inch diameter stainless steel pipe, 2-1/2 feet long, with 1/4-inch thick stainless steel end plates. These units are pressured with nitrogen so as to provide, after pressure equalization with the system, compressible gas volumes above the liquid levels to dampen out the surges. A dampener was not installed on the decomposer feed line, since it was not felt that a surging flow would detract from its efficiency.

6. Entrainment Separator

A knock-out drum type entrainment separator was installed on the discharge line from the purification still condenser to allow return of liquid forced to the top of the condenser. This device is a stainless steel chamber, made from 6-inch diameter pipe and 1-1/2 feet long, with nozzles at the middle for vapor entrance, at the top for vapor exit, and at the bottom for liquid return to the still.

7. Molecular Sieve Traps

The shells of the Molecular Sieve traps, used to dry anisole before charging it to the system, are 6-foot long sections of 6-inch steel pipe. Two units, each containing about 50 pounds of Linde Type 4A Molecular Sieves, were required to allow use of one while regenerating the other. Flow rates were 3 gpm. for rough drying to below about 300 ppm. water and 1 gpm. during final water removal to below 50 ppm. The beds were regenerated first by steaming to remove anisole and other organics and then by heating and purging with dry nitrogen to remove the water.

INSTRUMENTATION

The pilot plant instrument diagram is figure 19, with explanations of the various symbols used and descriptions of the instruments given in table XVI. Also, the instrumentation used in the coolant loops is illustrated in figure 6. Although there were exceptions, in general the instrumentation scheme called for recording system temperatures, flows, column pressure drops, and complex density, while both recording and controlling pressures and liquid levels.

Temperature Measurement and Control

Iron-Constantan thermocouple assemblies connected to multipoint temperature recorders were used to sense and to record the temperatures at the following points in the system:

Exchange Column	- 3 internal and 4 external (wall) points
High Temperature Decomposer Reboiler	- 1 internal point and 1 external point
High Temperature Decomposer Stripper	- 2 internal points and 1 external point
Low Temperature Decomposer Reboiler	- 1 internal point
Low Temperature Decomposer Stripper	- 3 internal points
Recombiner Reservoir	- 1 internal point
Still Reboiler	- 1 internal point
Still Column	- 3 internal points
Anisole Line from Still Product Tank to Recombiner	- 1 external point
Boron Trifluoride Line from Decomposer to Exchange Column	- 1 external point
Boron Trifluoride Line from Exchange Column to Recombiner	- 1 external point

Additionally, dial-type thermometers were used to indicate the temperature of the coolant streams, and single-point controller-recorders were used to control the temperatures of the glycol bath and the refrigerated anisole stream exiting from the coolant system, as has been discussed previously.

Flow Measurement and Control

Since the pilot plant operated at essentially total reflux, liquid and gas flows through each unit were controlled by setting the recombiner feed metering pump to deliver the desired amount of anisole and subsequently maintaining constant liquid levels throughout the system. However, in order to provide a check on steady operation, orifice assemblies connected to differential pressure transmitters and, in turn, to pneumatic recorders, were installed in the liquid line between the still product tank and the recombiner and in the gas line between the decomposer and the exchange column. Also, a 1.5 to 15 gpm. liquid rotameter indicated the amount of anisole refluxed to the still column. Two-tenths

to 2.0 std. cfh. gas rotameters were used in the purge streams to the many instrument taps and in the nitrogen line connected to the inlet side of the still system ejector.

Pressure Drop Measurement

Pressure drops across the exchange column, high and low temperature decomposer strippers, and the still column were measured and recorded by connecting differential pressure transmitters coupled with pneumatic recorders to taps at both the tops and bottoms of these columns. An additional pressure drop gauge was used intermittently to confirm the exchange column values.

Density Measurement

The density of the boron trifluoride-anisole complex was measured using an Ohmart Corporation Model PG-100-6-090150 density meter. This instrument contained a 150-millicurie Cesium-137 source which emitted a 0.66 MEV gamma from a shielded holder affixed to the liquid line between the recombiner and exchange column. Directly across from the source holder, on the other side of the line, was an electronic measuring cell which, according to the intensity of the impinging gamma, generated a signal for an associated electronic recorder. Since the extent of gamma-ray attenuation was proportional to the density of the complex filling the line, changes in this density, and thus in the mole ratio of boron trifluoride to anisole, could be easily detected. Analytical results were used to correlate chart readings with the approximate specific gravity, but the chief value of the unit lay in its ability to allow a continuous indication of changes in complex composition and, therefore, of system performance.

Pressure Measurement and Control

The operating pressure in the exchange column, decomposer, and recombiner sections of the system was controlled by venting accumulated inerts from the top of the recombiner whenever the pressure exceeded the set point. This was accomplished by using the output of a pneumatic controller-recorder, which was connected to a pressure transmitter at the top of the recombiner system, to open a diaphragm-operated control valve on the vent line. Pressures in the exchange column and in the decomposer were similarly measured, but no control was necessary at those points.

The pressure in the purification still was controlled by using an absolute pressure transmitter, pneumatic controller-recorder arrangement to provide an operating signal for a control valve located on the air line feeding the air-jet ejector. All remaining pressures of interest, such as steam pressures and coolant loop pump discharge pressures, were indicated using gauges.

Liquid Level Measurement and Control

Liquid levels in the still reboiler, recombiner reservoir, exchange column reservoir, and high temperature decomposer reboiler were held constant during system operation by using "bubbler" arrangements. These units were assembled by locating purged pressure taps in the reservoirs above and below the desired liquid level. Lines from these taps were connected to differential pressure transmitters, since the pressure differences were proportional to the liquid heads above the lower taps. Transmitter outputs were fed to pneumatic controller-recorders connected to various control valves. For the recombiner and exchange column systems, the control valves were placed on by-pass lines installed in parallel with the complex pumps which were located immediately below the reservoirs. Decreases in levels caused these valves to open; thereby increasing the amount of liquid recycled as explained in a previous section.

The level control valve for the still reboiler, on the other hand, was located on the still product line so that decreases in reboiler level could be met by increasing the amount of liquid returned to the still as reflux. Finally, the high temperature decomposer level control valve was located so as to stop liquid removal in the event that the high temperature decomposer reboiler level fell below the set point. This was important not only because the electrical heaters made it imperative to maintain liquid in the reboiler at all times, but also because this unit provided the seal between the pressurized portion of the system, which contained boron trifluoride, and the evacuated part, which did not contain boron trifluoride. For this reason, a float-type level controller was installed in the high temperature decomposer reservoir and was connected to the main control valve through a solenoid arrangement in order to serve as a back-up for the primary level control unit.

TABLE XVI
KEY TO INSTRUMENTATION

<u>Symbol</u>	<u>Service</u>	<u>Description</u>	<u>Input Range</u>	<u>Output Range</u>
PT-101	Still Column Pressure Control	Absolute Pressure Transmitter	0-20 psia.	3-18 psig.
PFC-102	Still Column Pressure Control	Pneumatic Recording Controller	3-18 psig.	3-18 psig.
PCV-103	Still Column Pressure Control	Diaphragm Operated Control Valve with CV = 2.5	3-18 psig.	
PI-104	Still Column Vent Pressure Indication	Pressure Gauge	30 in. vac. to 100 psig.	30-0-100
FI-105	Nitrogen Flow to Reflux Condenser Vent	Purge Rotameter with Constant Differential Regulator	0.2 to 2.0 std. cfh.	
LT-106	Level Control in Still Reboiler	Differential Pressure Transmitter	0-1 psig.	3-18 psig.
LRC-107	Level Control in Still Reboiler	Pneumatic Recording Controller	3-18 psig.	3-18 psig.
LCV-108	Level Control in Still Reboiler	Diaphragm Operated Control Valve with CV = 2.5	3-18 psig.	
FIC-109	Bubble System, High Side of LT-106	Purge Rotameter with Constant Differential Regulator	0.2 to 2.0 std. cfh.	
FIC-110	Bubble System, Low Side of LT-106	Purge Rotameter with Constant Differential Regulator	0.2 to 2.0 std. cfh.	
CI-111	Liquid Flow from Reflux Condenser	Liquid Rotameter	1.5 to 15 gpm.	
TE-112	Still Reboiler Internal Temperature	Thermocouple Assembly (I/C)		
TE-113	Still Column Lower Section Internal Temperature	Thermocouple Assembly (I/C)		
TE-114	Still Column Middle Section Internal Temperature	Thermocouple Assembly (I/C)		

TABLE XVI
(Continued)

KEY TO INSTRUMENTATION

<u>Symbol</u>	<u>Service</u>	<u>Description</u>	<u>Input Range</u>	<u>Output Range</u>
TE-115	Still Column Upper Section Internal Temperature	Thermocouple Assembly (I/C)		
FE-116	Purified Anisole Flow to Recombiner	Orifice Assembly		
FT-117	Purified Anisole Flow to Recombiner	Differential Pressure Transmitter	0-72 in. water	3-18 psig.
FR-118	Purified Anisole Flow to Recombiner	Pneumatic Pressure Recorder	3-18 psig.	
TE-119	Anisole Pump Discharge Temperature	Thermocouple Assembly (I/C)		
DT-120	Complex Feed Density Measurement	Source and Detector Section, Radiation-Type Density Meter		
DT-121	Complex Feed Density Measurement	Indicator Section, Radiation-Type Density Meter	0-10 mv.	
DR-122	Complex Feed Density Measurement	Density Recorder	0-10 mv.	
LT-123	Complex Feed Level Control	Differential Pressure Transmitter	0-1 psig.	3-18 psig.
LRC-124	Complex Feed Level Control	Pneumatic Recording Controller	3-18 psig.	18-3 psig.
LCV-125	Complex Feed Level Control	Diaphragm Operated Control Valve with CV = 2.5		
FIC-126	Bubble System, Low Side of LT-123	Purge Rotameter with Constant Differential Regulator	0.2 to 2.0 std. cfh.	
FIC-127	Bubble System, High Side of LT-123	Purge Rotameter with Constant Differential Regulator	0.2 to 2.0 std. cfh.	
PT-128	Recombiner Pressure Control	Pressure Transmitter	0-65 psia.	3-18 psig.

TABLE XVI
(Continued)
KEY TO INSTRUMENTATION

<u>Symbol</u>	<u>Service</u>	<u>Description</u>	<u>Input Range</u>	<u>Output Range</u>
PRC-129	Recombiner Pressure Control	Pneumatic Pressure Recording Controller	3-18 psig.	18-3 psig.
PCV-130	Recombiner Pressure Control	Diaphragm Operated Control Valve with CV = 2.5	3-18 psig.	
TI-131	Recombiner Coolant Outlet Temperature	Temperature Indicator, Dial Type	0-100°F.	
TI-132	Recombiner Coolant Outlet Temperature	Temperature Indicator, Dial Type	0-200°F.	
TI-133	Still Product Tank Internal Temperature	Temperature Indicator, Dial Type	0-200°F.	
TI-134	Recombiner Coolant Outlet Temperature	Temperature Indicator, Dial Type	0-200°F.	
TE-135	Recombiner Reservoir Internal Temperature	Thermocouple Assembly (I/C)		
TI-136	Recombiner Outlet Liquid Temperature	Temperature Indicator, Dial Type	0-200°F.	
PI-137	Boron Trifluoride to Recombiner Pressure	Pressure Gauge	0-100 psig.	
PI-138	Exchange Column Relief Pressure Indication	Pressure Gauge	0-100 psig.	
PI-139	Recombiner Relief Pressure Indication	Pressure Gauge	0-100 psig.	
PdT-140	Differential Pressure Across Exchange Column	Differential Pressure Transmitter	0-1.5 psig.	3-18 psig.

TABLE XVI
(Continued)

KEY TO INSTRUMENTATION

Symbol	Service	Description	Input Range	Output Range
PdR-141	Differential Pressure Across Exchange Column	Pneumatic Pressure Recorder	3-18 psig.	
LT-142	Exchange Column Reservoir Level Control	Differential Pressure Transmitter	0-1 psig.	3-18 psig.
LRC-143	Exchange Column Reservoir Level Control	Pneumatic Pressure Recording Controller	3-18 psig.	18-3 psig.
LCV-144	Exchange Column Reservoir Level Control	Diaphragm Operated Control Valve with CV = 2.5	3-18 psig.	
FIC-145	Bubble System, Low Side of LT-142	Purge Rotameter with Constant Differential Regulator	0.2 to 2.0 std. cfh.	∞
FIC-146	Bubble System, High Side of LT-142	Purge Rotameter with Constant Differential Regulator	0.2 to 2.0 std. cfh.	
PT-147	Pressure, Bottom of Exchange Column	Pressure Transmitter	0-65 psia.	3-18 psig.
PR-148	Pressure, Bottom of Exchange Column	Pneumatic Pressure Recorder	3-18 psig.	
FIC-149	Purge System, PT-147	Purge Rotameter with Constant Differential Regulator	0.2 to 2.0 std. cfh.	
TE-150	Boron Trifluoride Temperature at Exchange Column Outlet	Thermocouple Assembly (I/C)		
TE-151	Skin Temperature, Top Section of Exchange Column	Thermocouple Assembly (I/C)		
TE-152	Internal Temperature, Top Section of Exchange Column	Thermocouple Assembly (I/C)		

TABLE XVI
(Continued)

KEY TO INSTRUMENTATION

<u>Symbol</u>	<u>Service</u>	<u>Description</u>	<u>Input Range</u>	<u>Output Range</u>
TE-153	Skin Temperature, Middle Section of Exchange Column	Thermocouple Assembly (I/C)		
TE-154	Internal Temperature, Middle Section of Exchange Column	Thermocouple Assembly (I/C)		
TE-155	Skin Temperature, Lower Section of Exchange Column	Thermocouple Assembly (I/C)		
TE-156	Skin Temperature, Lower Section of Exchange Column	Thermocouple Assembly (I/C)		
TE-157	Internal Temperature, Lower Section of Exchange Column	Thermocouple Assembly (I/C)		
TE-158	Boron Trifluoride Temperature at Exchange Column Inlet	Thermocouple Assembly (I/C)		
PI-159	Low Temperature Stripper Column Relief Pressure Indication	Pressure Gauge	0-100 psig.	
FE-160	Boron Trifluoride Flow to Exchange Column	Orifice Assembly		
FT-161	Boron Trifluoride Flow to Exchange Column	Differential Pressure Transmitter	0-2 psig.	3-18 psig.
FR-162	Boron Trifluoride Flow to Exchange Column	Pneumatic Pressure Recorder	3-18 psig.	
PT-163	Boron Trifluoride Pressure to Exchange Column	Pressure Transmitter	0-65 psia.	3-18 psig.
PR-164	Boron Trifluoride Pressure to Exchange Column	Pneumatic Pressure Recorder	3-18 psig.	

TABLE XVI
(Continued)

KEY TO INSTRUMENTATION

Symbol	Service	Description	Input Range	Output Range
LT-165	Level Control, High Temperature Reboiler	Differential Pressure Transmitter	0-1 psig.	3-18 psig.
LRC-166	Level Control, High Temperature Reboiler	Differential Pressure Controller	3-18 psig.	3-18 psig.
LCV-167	Level Control, High Temperature Reboiler	Diaphragm Operated Control Valve with CV = 2.5	3-18 psig.	
FIC-168	Bubble System, High Side of LT-165	Purge Rotameter with Constant Differential Regulator	0.2 to 2.0 std. cfh.	
FIC-169	Bubble System, Low Side of LT-165	Purge Rotameter with Constant Differential Regulator	0.2 to 2.0 std. cfh.	
TE-170	Skin Temperature, High Temperature Reboiler	Thermocouple Assembly (I/C)		
TE-171	Internal Temperature, High Temperature Reboiler	Thermocouple Assembly (I/C)		
TE-172	Internal Temperature, High Temperature Stripper Column	Thermocouple Assembly (I/C)		
TE-173	Skin Temperature, High Temperature Stripper Column	Thermocouple Assembly (I/C)		
TE-174	Internal Temperature, High Temperature Stripper Column	Thermocouple Assembly (I/C)		
TE-175	Internal Temperature, Low Temperature Reboiler	Thermocouple Assembly (I/C)		
TE-176	Internal Temperature, Low Temperature Stripper Column	Thermocouple Assembly (I/C)		

TABLE XVI
(Continued)

KEY TO INSTRUMENTATION

<u>Symbol</u>	<u>Service</u>	<u>Description</u>	<u>Input Range</u>	<u>Output Range</u>
TE-177	Internal Temperature, Low Temperature Stripper Column	Thermocouple Assembly (I/C)		
TE-178	Internal Temperature, Low Temperature Stripper Column	Thermocouple Assembly (I/C)		
PCV-179	Boron Trifluoride Makeup Pressure Control	Cylinder Regulator		
PI-180	Boron Trifluoride Cylinder Pressure	Pressure Gauge		
PI-181	Boron Trifluoride Cylinder Reduced Pressure	Pressure Gauge		
PT-182	Low Temperature Stripper Column Pressure	Absolute Pressure Transmitter	0-65 psia.	3-18 psig.
PR-183	Low Temperature Stripper Column Pressure	Pneumatic Pressure Recorder	3-18 psig.	
PI-184	Anisole Pump Surge Tank Pressure	Pressure Gauge	0-100 psig.	
PI-185	Complexed Feed Pump Surge Tank Pressure	Pressure Gauge	0-100 psig.	
PCV-186	Steam Pressure Reduction to Still Reboiler	Self-Contained Pressure Regulator		
PI-187	Steam to Still Reboiler Pressure Indication	Pressure Gauge	0-100 psig.	
PCV-188	Steam Pressure Reduction to Decomposer Reboiler	Self-Contained Pressure Regulator		
PI-189	Steam to Decomposer Reboiler Pressure Indication	Pressure Gauge	0-100 psig.	

TABLE XVI
(Continued)

KEY TO INSTRUMENTATION

<u>Symbol</u>	<u>Service</u>	<u>Description</u>	<u>Input Range</u>	<u>Output Range</u>
TR-190	System Temperatures	20-Point L and N Speedomax for I/C Couples	100 to 600°F.	
TR-191	System Temperatures	12-Point L and N Micromax for I/C Couples	0 to 350°F.	
LS-192	High Temperature Reboiler Level Control Override	Level Switch		
LCV-193	High Temperature Reboiler Level Control Override	Three-Way Solenoid Valve		
TI-194	Condenser Coolant Outlet Temperature	Temperature Indicator, Dial Type	0-300°F.	
TI-195	Boron Trifluoride Cooler Coolant Outlet Temperature	Temperature Indicator, Dial Type	0-200°F.	
TI-196	Water-Cooled Anisole Coolant Hold Tank Inlet Temperature	Temperature Indicator, Dial Type	0-300°F.	
TI-197	Water-Cooled Anisole Coolant Temperature	Temperature Indicator, Dial Type	0-200°F.	
TI-198	Water Coolant Inlet Temperature	Temperature Indicator, Dial Type	0-200°F.	
TI-199	Water Coolant Outlet Temperature	Temperature Indicator, Dial Type	0-200°F.	
TI-200	Refrigerated Anisole Coolant Outlet Temperature	Temperature Indicator, Dial Type	-40 to 120°F.	
TI-201	Refrigerated Anisole Coolant Inlet Temperature	Temperature Indicator, Dial Type	0-200°F.	

TABLE XVI
(Continued)

KEY TO INSTRUMENTATION

<u>Symbol</u>	<u>Service</u>	<u>Description</u>	<u>Input Range</u>	<u>Output Range</u>
TE-202	Refrigerated Anisole Coolant Outlet Temperature	Temperature Element - Gas Filled Bulb		
TRC-203	Refrigerated Anisole Coolant Outlet Temperature	Temperature Recorder Controller Taylor Fulscope	0-300°F.	3-18 psig.
TCV-204	Refrigerated Anisole Coolant Outlet Temperature	Diaphragm Operated Control Valve	3-18 psig.	
PI-205	Coolant Pump Discharge Pressure	Pressure Gauge	0-100 psig.	
PI-206	Coolant Pump Discharge Pressure	Pressure Gauge	0-100 psig.	
PdT-207	Differential Pressure Across Still Equipment	Differential Pressure Transmitter	0-3 psig.	3-18 psig.
PdR-208	Differential Pressure Across Still Equipment	Pneumatic Pressure Recorder	3-18 psig.	
TE-209	Decomposer Feed Exit Temperature from Glycol Bath	Thermocouple Assembly (I/C)		
TRC-210	Decomposer Feed Exit Temperature from Glycol Bath	Single Point L and M Micromax Recorder Controller for I/C Couple	0-100°F.	
PdT-211	Differential Pressure Across High Temperature Stripper	Differential Pressure Transmitter	0-1 psig.	3-18 psig.
PdT-212	Differential Pressure Across Low Temperature Stripper	Differential Pressure Transmitter	0-1 psig.	3-18 psig.
XX-213	Pressure Selection, Output of PdT-211 or PdT-212	Three-Way Switching Cock		

TABLE XVI
(Continued)

KEY TO INSTRUMENTATION

<u>Symbol</u>	<u>Service</u>	<u>Description</u>	<u>Input Range</u>	<u>Output Range</u>
PdR-214	Differential Pressure Across High or Low Temperature Stripper	Pneumatic Pressure Recorder	3-18 psig.	
FIC-215	Purge System, PdT-140	Purge Rotameter with Constant Differential Regulator	0.2 to 2.0 std. cfh.	
FIC-216	Purge System, PdT-212	Purge Rotameter with Constant Differential Regulator	0.2 to 2.0 std. cfh.	
FIC-217	Purge System, PdT-212	Purge Rotameter with Constant Differential Regulator	0.2 to 2.0 std. cfh.	
FIC-218	Purge System, PdT-211	Purge Rotameter with Constant Differential Regulator	0.2 to 2.0 std. cfh.	
FIC-219	Purge System, PdT-211	Purge Rotameter with Constant Differential Regulator	0.2 to 2.0 std. cfh.

Figure 5
BORON ISOTOPE SEPARATION
PROCESS FLOW SHEET
Reference Drawing E-P-13019A

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Figure 6
COOLANT INTERCHANGE SYSTEM
FLOW SHEET

Reference Drawing E-P-13019AC

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PARTS LIST				
PART NO.	DWG. NO.	QTY. REQD.	DESCRIPTION (NAME, SIZE, ETC.)	MATERIAL
1	E-P1013Y	2	1" SCH. 40S PIPE 4 3/8" LG.	STAINLESS STEEL
2	E-P1013Y	2	6" STD. WT. WELD CAP	STAINLESS STEEL
3	E-P1013Y	1	1" SCH. 40S PIPE - 7 1/2" LG.	STAINLESS STEEL
4	E-P1013Y	1	1" STD. WT. 90° ELL. 3-W.	STAINLESS STEEL
5	E-P1013Y	8	6" 150° LAP JOINT FLANGE	STAINLESS STEEL
6	E-P1013Y	1	6" SCH. 40S PIPE - 5 1/2" LG.	STAINLESS STEEL
7	E-P1013Y	3	3/8" THK. PL. 1 7/8" WIDE X 26 3/8" LG.	STAINLESS STEEL
8	E-P1013Y	9	1/8" THK. PL. 7" WIDE X 2 1/2" LG.	STAINLESS STEEL
9	E-P1013Y	3	LIQUID DISTRIBUTOR	STAINLESS STEEL
10	E-P1013Y	3	6" SCH. 40S PIPE - 5' - 3 3/8" LG.	STAINLESS STEEL
11	E-P1013Y	1	5/8" BALL RINGS - 2.81 CU. FT.	STAINLESS STEEL
12	E-P1013Y	3	PACKING SUPPORT PLATE	STAINLESS STEEL
13	E-P1013Y	3	1/4" THK. PL. 1 1/2" WIDE X 16 7/8" LG.	STAINLESS STEEL
14	E-P1013Y	3	1" SCH. 40S PIPE 4 3/8" LG.	STAINLESS STEEL
15	E-P1013Y	1	6" SCH. 40S PIPE 3 1/2" LG.	STAINLESS STEEL
16	E-P1013Y	2	3/8" THK. PL. 3" X 2 1/4" X 5/8"	STAINLESS STEEL
17	E-P1013Y	2	1/2" THK. PL. 3 1/2" WIDE X 6 1/2" LG.	STAINLESS STEEL
18	E-P1013Y	1	2" SCH. 40S PIPE 6 7/8" LG.	STAINLESS STEEL
19	E-P1013Y	1	6" SCH. 40S PIPE 1' - 7 1/8" LG.	STAINLESS STEEL
20	E-P1013Y	1	1" SCH. 40S PIPE 4 3/8" LG.	STAINLESS STEEL
21	E-P1013Y	32	3/4" DIA. X 4 1/2" LG. STUD BOLTS W/HEX NUTS	ASTM A 307
22	E-P1013Y	2	1/4" THK. PL. 1 1/2" DIA.	STAINLESS STEEL
23	E-P1013Y	2	1" SCH. 40S PIPE 1 1/2" LONG	STAINLESS STEEL

Figure 7
ISOTOPIC EXCHANGE COLUMN AND
RESERVOIR, B-100 AND B-101
Reference Drawing E-P-13019Y

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Figure 8
PACKED COLUMN DISTRIBUTORS AND
SUPPORT PLATES
Reference Drawing E-P-13019AD

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Figure 9
HIGH TEMPERATURE DECOMPOSER
REBOILER, B-200
Reference Drawing E-P-13019AB

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Figure 10
HIGH TEMPERATURE DECOMPOSER
STRIPPER, B-201

Reference Drawing E-P-13019AA

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PARTS LIST			
PART NO.	DWG. NO.	QTY. REQD.	DESCRIPTION (NAME, SIZE, ETC.)
1	E-P-13019P	1	FLG. DISK HD. 2" X 2" X 1/4" DIA. 1/4" DIA. 1/4" DIA. 1/4" DIA.
2	E-P-13019P	1	20" SCH. 10 PIPE 2" DIA. 1/4" DIA. 1/4" DIA.
3	E-P-13019P	1	1/2" THK. PLATE x 1/2" WIDE x 6'-0" LONG
4	E-P-13019P	1	1" PIPE, SCH. 40S, 9' LONG
5	E-P-13019P	1	1" PIPE, SCH. 40S, 6' 1/2" LONG
6	E-P-13019P	1	4" PIPE, SCH. 40S, 6' 1/2" LONG
7	E-P-13019P	1	4" PIPE, SCH. 40S, 8' 1/2" LONG
8	E-P-13019P	1	4" - 120" LAP JOINT FLG.
9	E-P-13019P	1	1/2" THK. PLATE x 6" DIA.
10	E-P-13019P	1	GASKET, 4" DIA. 6" DIA. FLEXITALLIC "CGIL"
11	E-P-13019P	1	4" - 120" BLIND FLG., 1/4" R.R.
12	E-P-13019P	2	1/2" THK. PLATE x 6" WIDE x 14' 1/2" LONG
13	E-P-13019P	2	1/2" THK. PLATE x 12" WIDE x 13' 1/2" LONG
14	E-P-13019P	2	1/2" THK. PLATE x 8" WIDE x (21') LONG
15	E-P-13019P	2	20" - 150" LAP JOINT FLG.
16	E-P-13019P	1	20" - 150" WELD NECK FLG., 1/4" R.R.
17	E-P-13019P	1	20" PIPE, SCH. 20, 7' 1/2" LONG
18	E-P-13019P	2	1 1/2" - 150" WELD NECK FLG., 1/4" R.R.
19	E-P-13019P	2	1 1/2" PIPE, SCH. 40, 4' 1/2" LONG
20	E-P-13019P	1	1/2" THK. PLATE x 10 1/2" WIDE x 13' 1/2" LONG
21	E-P-13019P	1	W. GASKET x 1/4" DIA. 1/4" DIA. 1/4" DIA.
22	E-P-13019P	1	1 1/2" THK. PLATE x 25" DIA.
24	E-P-13019P	2	1/2" THK. PLATE x 14' 1/2" LONG x 12' 1/2" WIDE
26	E-P-13019P	5	1" O.D. TUBING, .083 WALL THK., 12' - 1/2" LONG
27	E-P-13019P	5	1" O.D. TUBING, .083 WALL THK., 12' - 1/2" LONG
28	E-P-13019P	6	1" O.D. TUBING, .083 WALL THK., 12' - 1/2" LONG
29	E-P-13019P	6	1" O.D. TUBING, .083 WALL THK., 12' - 1/2" LONG
30	E-P-13019P	20	1 1/2" DIA. x 8' 1/2" LONG STUD BOLTS W/HEN NUTS
31	E-P-13019P	15	3/8" DIA. x 1/4" LG. BOLTS W/HEN NUTS
32	E-P-13019P	4	1/2" DIA. ROD x 7' - 4 1/2" LONG
33	E-P-13019P	1	1/2" THK. SHEET x 3' 1/2" DIA.
34	E-P-13019P	1	1" PIPE, SCH. 40, 25' LONG
35	E-P-13019P	1	FLG. DISK HD. 2" X 2" X 1/4" DIA. 1/4" DIA. 1/4" DIA.
36	E-P-13019P	4	1/2" THK. PLATE x 6" WIDE x 12' LONG
37	E-P-13019P	2	1 1/2" DIA. ROD x 15' LONG
38	E-P-13019P	1	1/2" THK. PLATE x 6 1/2" DIA.

Figure 11
LOW TEMPERATURE DECOMPOSER
REBOILER, B-202
Reference Drawing E-P-13019P

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PARTS LIST

PART NO.	DWG. NO.	NO. REQD.	DESCRIPTION (NAME, SIZE, ETC.)	MATERIAL
1	E-P-150192	1	2" SCH. 40S PIPE - 3 1/2" LG.	STEAINLESS ASTM A-312 S30400
2	E-P-150192	2	12" STD. WT. WELD CAP	STEAINLESS ASTM A-312 S30400
3	E-P-150192	2	1 1/2" SCH. 40S PIPE - 8 1/4" LG.	STEAINLESS ASTM A-312 S30400
4	E-P-150192	6	6" SCH. 40S PIPE - 1' 4" LG.	DITTO
5	E-P-150192	2	3/8" THK. PL. - 6" O.D.	STEAINLESS ASTM A-312 S30400
6	E-P-150192	1	2" SCH. 40S PIPE - 3 1/4" LG.	STEAINLESS ASTM A-312 S30400
7	E-P-150192	1	1" SCH. 40S PIPE - 7 1/2" LG.	DITTO
8	E-P-150192	1	1" STD. WT. 90° ELL. W.R.	STEAINLESS ASTM A-312 S30400
9	E-P-150192	2	3/8" THK. PL. - 1 1/2" W X 48" LG.	STEAINLESS ASTM A-312 S30400
10	E-P-150192	2	12" ISO. LAP JOINT FLANGE	STEEL FORGED ASTM A-181 F155
11	E-P-150192	1	12" SCH. 20 PIPE - 250" WALL X 1-10 7/8" LG.	DITTO
12	E-P-150192	1	12" SCH. 20 PIPE - 250" WALL X 5'-9 3/8"	STEAINLESS ASTM A-312 S30400
13	E-P-150192	1	1" SCH. 40S PIPE - 4 3/8" LG.	STEAINLESS ASTM A-312 S30400
14	E-P-150192	1	4" SCH. 40S PIPE - 6" LG.	DITTO
15	E-P-150192	1	1/4" THK. PL. - 3'-2 1/2" LG. X 1" WIDE	STEAINLESS ASTM A-312 S30400
16	E-P-150192	3	40# THK. PL. - 1/8" WIDE X 2 3/8" LG.	DITTO
17	E-P-150192	2	1/2" THK. PL. - 3 1/2" WIDE X 6 1/4" LG.	STEAINLESS ASTM A-312 S30400
18	E-P-150192	2	3/8" THK. PL. - 6 1/4" SQ	STEAINLESS ASTM A-312 S30400
19	E-P-150192	1	1" TUBE, WOLVERINE "TRUFIN" TYPE S T 1.03 WALL - 1' 0" LG.	STEAINLESS ASTM A-312 S30400
20	E-P-150192	1	1" TUBE, WOLVERINE "TRUFIN" TYPE S T 1.03 WALL - 22" S LG.	DITTO
21	E-P-150192	1	3/16" THK. PL. - 2 3/4" S	STEAINLESS ASTM A-312 S30400
22	E-P-150192	1	12" GAS INJECTION SUPPORT PLATE FIG. 736H US STONEWALL CO.	STEAINLESS ASTM A-312 S30400
23	E-P-150192	1	1" PALL RINGS, 3.0" CU. FT.	NICKEL ASTM B-562
24	E-P-150192	3	1 1/8" PIPE - SCH. 40S 3 1/2" W LG.	STEAINLESS ASTM A-312 S30400
25	E-P-150192	3	1/16" THK. PL. - 1 3/2" O.D.	STEAINLESS ASTM A-312 S30400
26	E-P-150192	12	3/8" DIA. X 3 1/2" LG. STUD BOLTS W/HEX NUTS	STEAINLESS ASTM A-312 S30400
27	E-P-150192	1	12" WEIRFLOW DISTRIBUTOR, FIG. 736 US STONEWALL CO.	STEAINLESS ASTM A-312 S30400
28	E-P-1501940	1	PACKING HOLD DOWN PLATE, N#1	STEAINLESS ASTM A-312 S30400

Figure 12
LOW TEMPERATURE DECOMPOSER STRIPPER AND
BF₃ COOLER, B-203 AND B-204
Reference Drawing E-P-13019R

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PARTS LIST				
PART NO.	DWG. NO.	NO. REQD.	DESCRIPTION (NAME, SIZE, ETC.)	MATERIAL
1	E-1010-T	2	FLG. & DISHED HEAD .035" COODS 1/8", O.D. 1/8" GAUGE, O.A. 1.51 IN. I.D. A. & W.	STAINLESS ASTM A-286
2	E-1010-T	2	1/2" SCH 10 PIPE 72" LONG	ASTM A-106 ASTM A-286
3	E-1010-T	2	1 1/2" 150# LAP JOINT FLANGE	ASTM A-106 ASTM A-286
4	E-1010-T	2	3/8" THK. PLATE 1/2" WIDE X 57" LONG	STAINLESS ASTM A-286
5	E-1010-T	2	1/4" THK. PLATE 2 1/2" DIA	ASTM A-286
6	E-1010-T	2	1/8" O.D. SCH. SCH. 30 3/8" LONG	STL GRADE ASTM A-286
7	E-1010-T	3	1/4" THK. PLATE 1 1/2" WIDE X 18 1/2" LONG	STL GRADE ASTM A-286
8	E-1010-T	1	2" SCH. SCH. PIPE 7 3/8" LONG	STAINLESS ASTM A-286
9	E-1010-T	1	2" SCH. SCH. PIPE 12" LONG	DITTO
10	E-1010-T	109	1/4" O.D. TUBING .035" WALL THK	STAINLESS ASTM A-286
11	E-1010-T	1	1/2" SCH. SCH. PIPE 22" LONG	ASTM A-286
12	E-1010-T	1	1/8" THK. PLATE 5" DIA	ASTM A-286
13	E-1010-T	2	1/4" THK. PLATE 3 1/2" WIDE X 17" LONG	STL GRADE ASTM A-286
14	E-1010-T	4	3/8" THK. PLATE 6" WIDE X 6 1/2" LONG	STL GRADE ASTM A-286
15	E-1010-T	1	1/2" SCH. SCH. PIPE 6 1/2" LONG	STAINLESS ASTM A-286
16	E-1010-T	32	1" DIA. X 5 3/4" LONG	STL GRADE ASTM A-286
17	E-1010-T	2	2" SCH. SCH. PIPE WITH HEX NUTS	STAINLESS ASTM A-286
18	E-1010-T	2	7 3/8" LONG	ASTM A-286
19	E-1010-T	3	90" PALL RINGS 1.7 CUBIC FT.	ASTM A-286 STAINLESS ASTM A-286

Figure 13
RECOMBINER, B-300
Reference Drawing E-P-13019T

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Figure 14
RECOMBINER, B-301 AND B-302
Reference Drawing E-P-13019S

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PARTS LIST				
PART NO.	DWG. NO.	NO. REQD.	DESCRIPTION (NAME, SIZE, ETC.)	MATERIAL
1	E-P-10019U	1	FLGD. & DISHED HOD, ASME CODE, 20" O.D., 16" GAUGE, 1/2" SCH. 40, 2 1/2" DIA.	STEEL ASTM A 213
2	E-P-10019U	1	20" SCH. 10, PIPE, 150" HALL, 8 1/4" H ₁ LONG	STEEL ASTM A 213
3	E-P-10019U	1	3/8" THK. PLATE x 1 1/2" WIDE x 6' 0" L ₁ G.	STEEL ASTM A 213
4	E-P-10019U	1	18" PIPE, SCH. 40S, 9' LONG	STEEL ASTM A 213
5	E-P-10019U	1	1" PIPE, SCH. 40S, 6 3/8" LONG	STEEL ASTM A 213
6	E-P-10019U	1	8" PIPE, SCH. 40S, 6 3/8" LONG	STEEL ASTM A 213
7	E-P-10019U	1	6" PIPE, SCH. 40S, 5 1/4" LONG	STEEL ASTM A 213
8	E-P-10019U	1	4" - 130" LAP JOINT FLG.	STEEL ASTM A 213
9	E-P-10019U	1	3/8" THK. PLATE x 6 1/2" DIA.	STEEL ASTM A 213
10	E-P-10019U	1	GASKET, 4 1/2" DIA. x 6 1/2" O.D. FLEXITALLIC CG 11	ASTM FILLER ASTM A 213
11	E-P-10019U	1	4" - 150" BLIND FLG., 1/2" R.R.	FORGED STEEL ASTM A 213
12	E-P-10019U	2	1/2" THK. PLATE x 8" WIDE x 14 1/2" LONG	STEEL ASTM A 213
13	E-P-10019U	2	3/8" THK. PLATE x 12 1/2" WIDE x 13 1/2" LONG	STEEL ASTM A 213
14	E-P-10019U	2	3/8" INK. PLATE x 8" WIDE x 21" LONG	STEEL ASTM A 213
15	E-P-10019U	1	20" - 150" LAP JOINT FLG.	STEEL ASTM A 213
16	E-P-10019U	1	20" - 150" WELD NECK FLG., 1/2" R.R.	FORGED STEEL ASTM A 213
17	E-P-10019U	1	20" PIPE, SCH. 20, 1 1/2" LONG	STEEL ASTM A 213
18	E-P-10019U	2	1 1/2" - 150" WELD NECK FLG., 1/2" R.R.	FORGED STEEL ASTM A 213
19	E-P-10019U	2	1 1/2" PIPE, SCH. 40, 4 1/2" LONG	STEEL ASTM A 213
20	E-P-10019U	1	3/8" THK. PLATE x 18 1/2" WIDE x 19 1/2" LONG	STEEL ASTM A 213
21	E-P-10019U	1	1/2" THK. GASKET x 3 1/2" DIA. 1/2" G. SUBC. 8" H ₁ IN. H ₁ H ₁	COMPRESSED ASTM A 213
22	E-P-10019U	1	1 1/2" THK. PLATE x 23" DIA.	STEEL ASTM A 213
24	E-P-10019U	2	3/8" THK. PLATE x 19 1/2" LONG x 12 1/2" WIDE	STEEL ASTM A 213
26	E-P-10019U	1	1" O.D. TUBING, .035 WALL THK., 14 1/2" H ₁ LONG	STEEL ASTM A 213
27	E-P-10019U	5	1" O.D. TUBING, .035 WALL THK., 17 1/2" H ₁ LONG	STEEL ASTM A 213
28	E-P-10019U	6	1" O.D. TUBING, .035 WALL THK., 16 1/2" H ₁ LONG	STEEL ASTM A 213
29	E-P-10019U	6	1" O.D. TUBING, .035 WALL THK., 16 1/2" H ₁ LONG	STEEL ASTM A 213
30	E-P-10019U	20	1 1/2" DIA. x 8 1/2" LONG STUD BOLTS N/HX NUTS	STEEL ASTM A 213
31	E-P-10019U	8	3/8" DIA. x 3 1/2" LG. BOLTS N/HX NUTS	STEEL ASTM A 213
32	E-P-10019U	4	3/8" DIA. ROD x 2 1/2" H ₁ LONG	STEEL ASTM A 213
33	E-P-10019U	1	1/2" THK. SHEET x 13 1/2" DIA.	STEEL ASTM A 213
34	E-P-10019U	1	8" PIPE, SCH. 40S, 29" LONG	STEEL ASTM A 213
35	E-P-10019U	1	FLGD. & DISHED HOD, ASME CODE, 20" O.D., 16" GAUGE, 1/2" SCH. 40, 2 1/2" DIA.	STEEL ASTM A 213
36	E-P-10019U	4	3/8" THK. PLATE x 6" WIDE x 12" LONG	STEEL ASTM A 213
37	E-P-10019U	2	3/8" DIA. ROD x 1 1/2" LONG	STEEL ASTM A 213
38	E-P-10019U	1	3/8" THK. PLATE x 6 1/2" DIA.	STEEL ASTM A 213

Figure 15
PURIFICATION STILL REBOILER, B-400
Reference Drawing E-P-13019U

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Figure 16
PURIFICATION STILL COLUMN,
B-401, SHEET 1
Reference Drawing E-P-13019V

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Figure 17
 PURIFICATION STILL COLUMN,
 B-401, SHEET 2
 Reference Drawing E-P-13019W

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PARTS LIST				
PART NO.	DWG. NO.	NO. REQD.	DESCRIPTION (NAME, SIZE, ETC.)	MATERIAL
1	E-P-1019X	1	8" SCH. 40 PIPE 5' 10" LONG	STL. GRADE A
2	E-P-1019X	2	1/2" THK. PLATE 18" DIA.	STL. GRADE A
3	E-P-1019X	2	1/2" SCH. 40 PIPE 8" LONG	STL. GRADE A
4	E-P-1019X	3	1/2" THK. PLATE 18" WIDE X 8" LONG	STL. GRADE A
5	E-P-1019X	3	1/2" DIA. ROD 6'-0" LONG	STL. GRADE A
6	E-P-1019X	1	1/4" DIA. ROD 6'-0" LONG	STL. GRADE A
7	E-P-1019X	2	3/8" THK. PLATE 8" WIDE X 10 1/2" LONG	STL. GRADE A
8	E-P-1019X	2	3/8" THK. PLATE 18" WIDE X 6" LONG	STL. GRADE A
9	E-P-1019X	2	3/8" THK. PLATE 17 1/2" WIDE X 7 1/2" LONG	STL. GRADE A
10	E-P-1019X	4	3/8" THK. PLATE 2" WIDE X 9" LONG	STL. GRADE A
11	E-P-1019X	2	1/2" WIDE X 3 1/2" LONG	STL. GRADE A
12	E-P-1019X	2	8" WIDE LAP JOINT PLANK	STL. GRADE A
13	E-P-1019X	2	8" STD. WRIGHT WELD CAP	STL. GRADE A
14	E-P-1019X	1	8" SCH. 40S PIPE 5' 10" LONG	STL. GRADE A
15	E-P-1019X	1	3" SCH. 40S PIPE 6' 10" LONG	STL. GRADE A
16	E-P-1019X	1	1" SCH. 40S PIPE 18'-0" LONG	STL. GRADE A
17	E-P-1019X	1	1" SCH. 40S PIPE 6'-0" LONG	STL. GRADE A
18	E-P-1019X	31	1/8" DIA. TUBING, 3/8" WALL THK.	STL. GRADE A
19	E-P-1019X	16	3/4" DIA. X 3 1/2" LONG STD. BOLTS WITH HEX NUTS	STL. GRADE A

Figure 18
PURIFICATION STILL CONDENSER,
B-402
Reference Drawing E-P-13019X

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Figure 19
INSTRUMENT DIAGRAM
Reference Drawing E-I-13019A