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ENGINEERING-SCALE STUDIES OF BORON ISOTOPE SEPARATION USING THE BORON TRIFLUORIDE-ANISOLE COMPLEX*

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The advantages of using volatile fluorine compounds in isotope separation processes are well known. It was natural, then, to turn to boron trifluoride when a need was seen for material enriched in the high cross-section isotope, boron-10. Initial thermal diffusion and distillation experiments were begun in 1943 at Columbia University(11), and this research led to an equilibrium distillation process employing a gaseous mixture of boron trifluoride and dimethyl ether. This process has been applied intermittently on a plant scale at facilities operated for the Manhattan District at Whiting, Indiana(4), and later for the Atomic Energy Commission at Model City, New York(10).

The dimethyl ether process, selected to satisfy United States requirements for boron-10, has its problems. Since both the boron trifluoride and the ether complex are volatile, it is not feasible to make a separation between the two components with ordinary thermal refluxing. For this reason, the cascade product stream contains boron trifluoride and the ether, as well as a certain amount of ether degradation products.

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* This document is based on work performed at the Oak Ridge Gaseous Diffusion Plant operated by Union Carbide Corporation for the United States Atomic Energy Commission.

Furthermore, as a result of the volatility and dissociation of the complex, the system exhibits a relatively low effective single-stage separation factor, and flow is limited because of the need to operate at subatmospheric pressures 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 both the single column height and the operating throughput are restricted because of the relatively small available pressure drop.

These shortcomings of the dimethyl ether process led to laboratory searches at the Oak Ridge National Laboratory and elsewhere for a better process solvent. The most attractive possibility uncovered during the course of this work(8,12) involved the use of anisole (methyl phenly 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 operated satisfactorily for a considerable period of time, yielded good column separation performance data. In fact, the separation factor with anisole was found to be such as to allow almost twice the separation per stage as with dimethyl ether. The total plant length required for a specified separation would thus be considerably reduced by using the anisole system, provided stage lengths were reasonable in comparison with the dimethyl ether process (using identical packing). The anisole system's potential advantages, which will be discussed in more detail later, were almost immediately overshadowed by the results of early tests(6,7) which indicated severe operating limitations because of corrosion and anisole degradation; however, a second look at the anisole separation system appeared to be justified; therefore, the AEC authorized the Technical Division at the Oak Ridge Gaseous Diffusion Plant to make the more complete studies of the anisole system described herein.

The main facet of our program was the engineering-scale effort discussed in this paper; however, the studies included extensive laboratory- and bench-scale experiments designed to supply basic engineering data and to evaluate the problems of corrosion and solvent degradation. In this work, which is described elsewhere(1,2), boron trifluoride-anisole complex vapor pressures, viscosities, densities, and other properties were measured. Complex densities and viscosities at test conditions were near 75 pounds per cubic foot and between 2 and 5 cp., respectively. Also, the anisole degradation products were identified, and a degradation mechanism was postulated*. Additionally, various materials were screened for possible service in the engineering-scale unit. It was established that exclusion of water from the system is the key to minimizing corrosion and solvent degradation. Also, 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 chosen for construction of the engineering-scale equipment and for packing in the low temperature columns; however, the most costly nickel

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* The principal degradation mechanism apparently involves (a) a boron trifluoride-catalyzed Friedal-Crafts type of alkylation of the aromatic ring to give methylated anisoles and m-cresol, and (b) a dealkylation of the anisole and substituted anisoles to form phenol and cresols. These processes involve the cyclic utilization and regeneration of hydrogen fluoride, which was found to react with anisole and boron trifluoride to form an acidic complex believed to be the vehicle for recycling hydrogen fluoride.

packing was used in the high temperature columns to minimize degradation in these units.

ENGINEERING STUDIES

System Design

The isotopic exchange system for the separation of boron isotopes is a conventional gas-liquid contacting operation, and 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 thermally dissociating the column liquid effluent in the so-called decomposer system. The stripped anisole from the two-stage 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 two-stage 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.

A summary of pertinent equipment design information is given in table I. Other auxiliaries were provided, including intermediate coolant interchange systems used to reduce the chances for water inleakage. Coolant anisole was supplied at two temperature levels to the various condensers and coolers.

TABLE I

ENGINEERING-SCALE TEST UNIT PROCESS EQUIPMENT DESIGN SUMMARY

COLUMNS

Name	Exchange Column	Low Temperature Decomposer Stripper	High Temperature Decomposer Stripper	Purification Still Column
Function	To contact counter-current streams of gaseous boron trifluoride and liquid anisole-boron trifluoride complex for isotope separation	To separate boron trifluoride and anisole by rectification	To separate boron trifluoride and anisole by rectification	To separate anisole from its high boiling degradation products
Diameter	6 inches	12 inches	6 inches	16 inches
Height	17 feet 2-1/2 inches	6 feet 8 inches	5 feet 8-3/8 inches	10 feet 5-7/8 inches
Packing Height	13 feet 6 inches	4 feet 6 inches	2 feet 10 inches	6 feet 9 inches
Packing Size	5/8 inch	1 inch	5/8 inch	1 inch
Material of Construction:				
Packing	Stainless Steel 316	Nickel	Nickel	Nickel
Other Exposed Surfaces	Stainless Steel 304 L	Stainless Steel 304 L	Stainless Steel 304 L	Stainless Steel 304 L

TABLE I (Contd.)

ENGINEERING-SCALE TEST UNIT
PROCESS EQUIPMENT DESIGN SUMMARY

HEAT EXCHANGERS

Name	Low Temperature Decomposer-Reboiler	High Temperature Decomposer-Reboiler	Recombiner, Unit 1	Recombiner, Unit 2	Purification Still Reboiler	Purification Still Condenser
Function	To provide heat necessary for most of complex dissociation	To provide heat necessary to complete complex dissociation	To provide site for formation of about 80% of required complex	To provide site for formation of remaining complex and for any necessary cooling	To provide heat necessary for continuous distillation of process anisole	To reflux distilled anisole
Type	U-Tube	Electrically Heated	Shell and Tube	Shell and Tube	U-Tube	Shell and Tube
Surface Area	84 square feet	11 square feet	288 square feet	115 square feet	84 square feet	65 square feet
Number of Tubes	20	None	109	109	20	31
Tube Diameter	1 inch	-	1 inch	1 inch	1 inch	1 inch
Tube Length	16 feet before bending	-	10 feet	4 feet	16 feet before bending	8 feet
Shell Side Fluid	Anisole and Complex	Anisole	Anisole Coolant	Anisole Coolant	Anisole	Anisole Coolant
Tube Side Fluid	Steam	-	Complex	Complex	Steam	Anisole
Material of Construction:						
Packing	None	None	5/8-inch stainless steel 316	5/8-inch stainless steel 316	None	None
Other Exposed Surfaces	Stainless Steel 304 L	Stainless Steel 304 L	Stainless Steel 304 L	Stainless Steel 304 L	Stainless Steel 304 L	Stainless Steel 304 L

The size of the engineering-scale effort was largely dictated by the need to obtain stage separation data on packing considered suitable for plant-scale use. Previous 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. 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 gallons per minute of complex; however, the auxiliaries were sized for 4 gallons per minute of complex to assure an adequate flow.

To obtain data which would allow confident sizing of a production cascade, the pilot-plant tests were aimed at providing engineering-scale information on (a) stage lengths and flooding characteristics of plant-size packing; (b) solvent degradation in large-scale equipment at various temperatures and pressures; (c) complex dissociation and recombination on a production scale, with emphasis on achieving low boron losses; (d) anisole purification and drying; and (e) suitability of the materials of construction.

Test Results

Following preliminary drying and shakedown runs, nineteen isotope separation tests and one flooding test were made in the pilot-plant equipment.

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* Product of the U. S. Stoneware Company.

In the separation tests, pressures of about 20, 26, and 35 psia.; anisole flow rates of 1.17, 1.75, and 2.0 gallons per minute; and temperatures from 63 to 105°F. were employed, while the flooding test was carried out at 28 psia. and 96°F. 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. Both the test conditions and the results are described in more detail in the project completion report(9).

Exchange Column Performance. As shown in table II, stage heights for isotope separation 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 gallons per minute, 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:

$$\text{HTU} = 32.629 \left(\frac{P}{14.7}\right)^{0.130} (G')^{0.180} \left(\frac{\alpha\mu_L}{\rho_L}\right)^{0.732} \qquad (1)$$

$$\text{HTU} = 27.498 \left(\frac{P}{14.7}\right)^{0.138} (L')^{0.185} \left(\frac{\alpha\mu_L}{\rho_L}\right)^{0.758} \qquad (2)*$$

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* 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 II

EXCHANGE COLUMN OPERATING CONDITIONS AND RESULTS

Run Number	Temperature, °F.	Pressure, psia.	Anisole Flow, gpm.	Stage Height, in.	Pressure Drop, in. of water / ft. of packing
1	62.8	25.0	1.17	42.7	0.30
2	87.1	25.5	1.75	30.6	0.59
3	86.8	33.4	1.75	33.3	0.57
4	92.4	25.5	1.75	28.2	0.59
5	98.0	25.8	1.75	25.7	0.62
6	104.8	25.1	1.75	23.6	0.52
7	101.2	25.2	1.17	23.3	0.29
8	104.2	26.6	1.17	22.4	0.27
9	84.9	26.4	1.17	29.8	0.29
10	74.6	35.9	2.0	40.1	0.72
11	83.4	35.4	2.0	35.8	0.90
12	89.5	34.9	2.0	32.1	0.92
13	104.2	35.2	2.0	26.5	0.83
14	77.3	26.4	2.0	37.3	0.93
15	94.0	26.2	2.0	29.8	0.84
16	104.8	26.5	2.0	26.5	0.82
17	75.8	20.1	2.0	34.6	1.34
18	94.7	20.1	2.0	26.2	1.11
19	103.7	20.1	2.0	23.3	1.01

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 (1) and (2) were suggested by the Murch equation, described by Perry(13). The addition of the pressure term was felt necessary to extend the correlation to nonatmospheric operation. A comparison of actual HTU's and HTU's calculated using equation (2) is shown in figure 1.

In the exchange column flooding test, flooding was found to occur at a complex flow rate equivalent to about 2.7 gallons per minute of anisole. This value 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(3). The generalized correlation consists of a family of curves, with pressure drop as the parameter, based on two groups of variables:

$$X = \frac{L}{G} \left(\frac{\rho_g}{\rho_L}\right)^{1/2} \qquad (3)$$

and

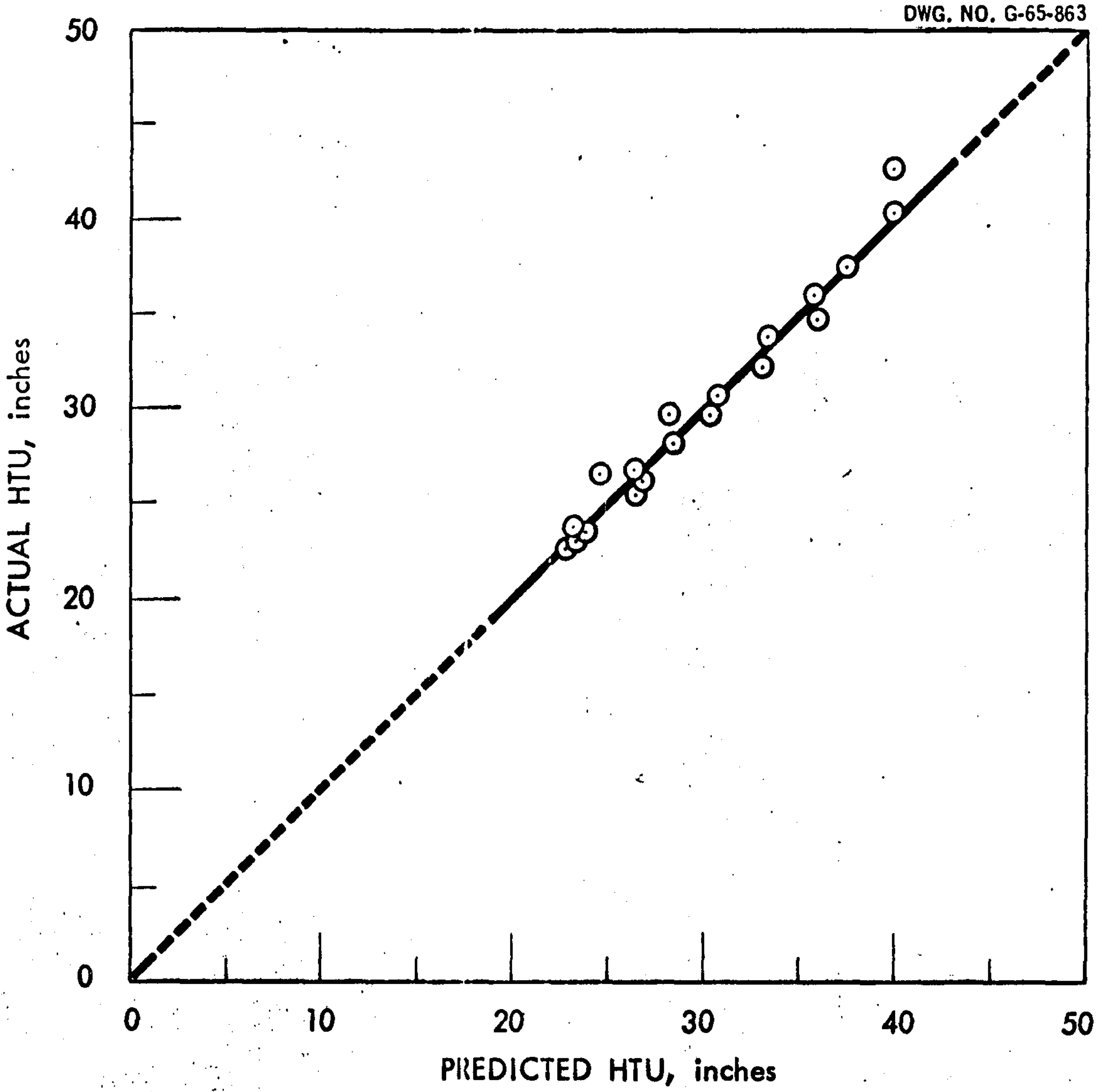

Figure 1

STAGE LENGTHS ESTIMATED USING
A CORRELATION DEVELOPED FROM PILOT PLANT DATA

$$Y = \frac{G^2 F \psi \mu^{0.2}}{\rho_g \; \rho_L \; g_c} \qquad (4)$$

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 shown in table II 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 (3) and by changes in the packing factor used in equation (4) were largely unsatisfactory.

A simple empirical correlation involving only flow rates and densities and similar to an equation used by Treybal$^{(5)}$ was finally chosen to represent the pressure drop data. The equation arrived at was

$$\Delta P = A \, (10)^{(19.86L)/\rho_L} \left(\frac{G^2}{\rho_g} \right)^{0.6436} - B \qquad (5)$$

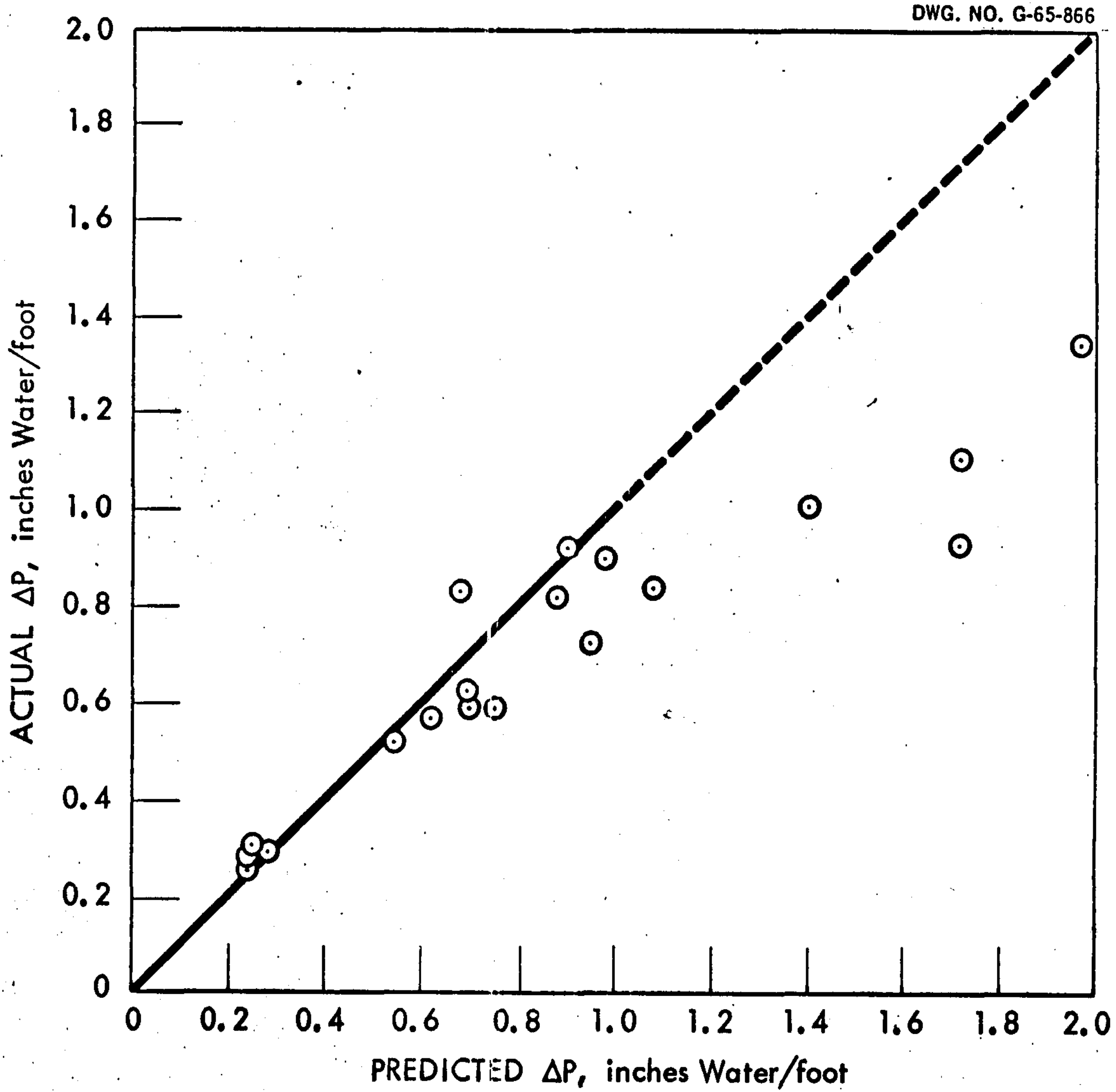

Figure 2

EXCHANGE COLUMN PRESSURE DROPS PREDICTED
BY THE GENERALIZED PRESSURE DROP CORRELATION

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_g 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. Although this equation represents the pilot-plant data fairly well in the range studied, as shown in figure 3, extrapolation outside this range should be viewed with caution.

Complex Dissociation and Recombination. During the separation tests, both complex dissociation and recombination were accomplished with low material losses. The low and high temperature decomposer reboilers used to supply dissociation heat were operated near 295 and 350°F., respectively; the strippers associated with these units 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 within the design limit set for the unit. Also, for most of the runs, the amount of boron lost through the high temperature decomposer to the still, parimarily as a trimer of boron oxyfluoride, $(BOF)_3$, was sufficiently small that the pilot plant could be assumed to be at total reflux as far as separation data collection was concerned. In these runs, the boron content of the still feed stream was below 10 ppm., corresponding

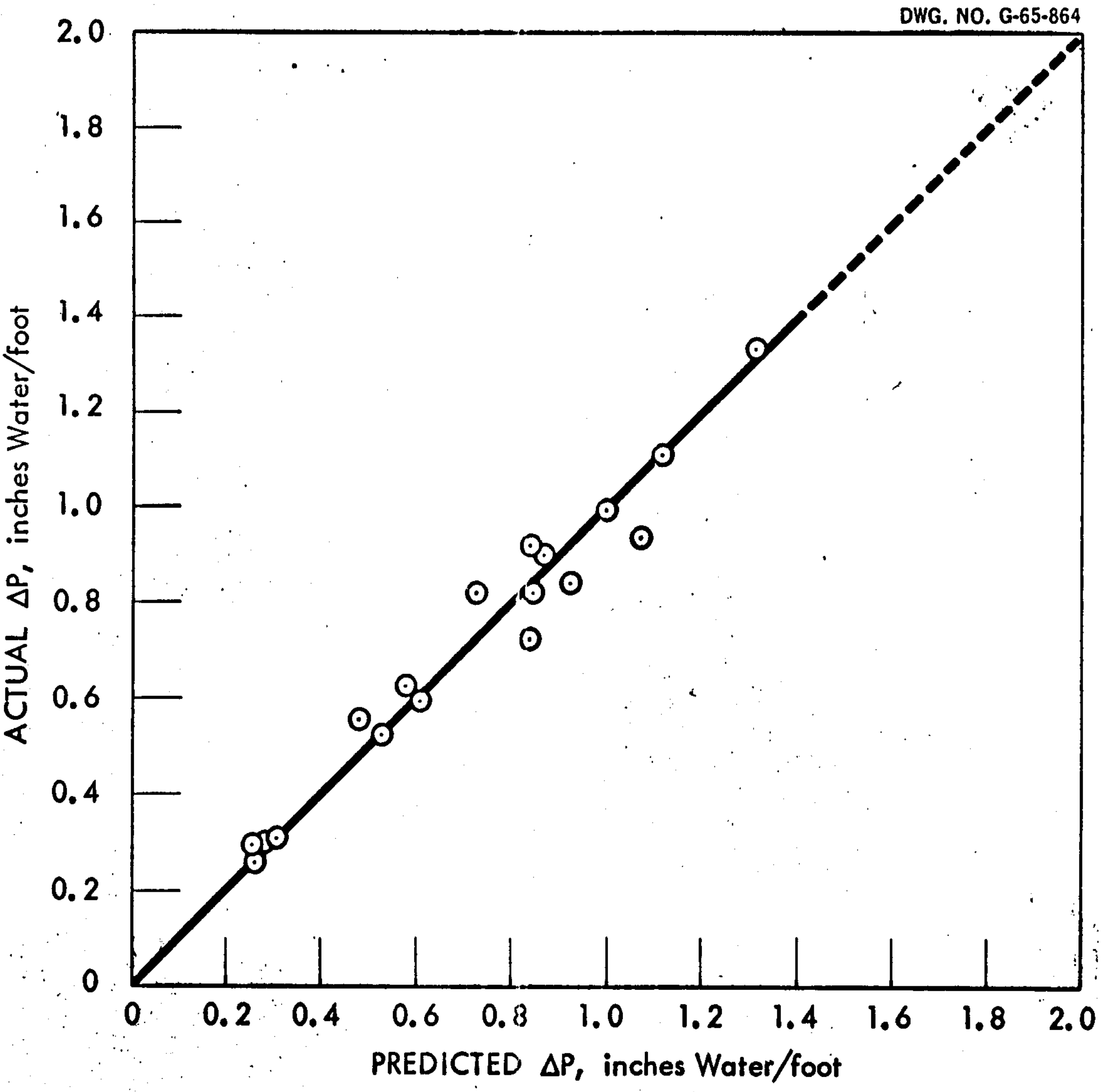

Figure 3

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

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 smoothly to produce complex with the desired composition and efficiently with negligible boron losses implies that the design procedure and parameters were at least adequate.

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 and of the pilot plant of 10^6 or greater.

Anisole Purification and Drying. Some of the most significant pilot-plant information concerns the measures taken for anisole purification and drying. 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. Similarly, the purification still was generally effective in isolating degradation products from the recycle stream. The vacuum still was operated at a pressure of 6 psia., and reboiler temperatures varied from 260 to 290°F. Condenser effluent temperatures were near 260°F. for all runs. The anisole concentration in the still feed was from 84 to 93 weight percent with 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, in fact, was not felt to hurt the pilot-plant separation data, since the methylated anisoles should form complexes with boron trifluoride having separation factors similar to those for anisole.

Anisole Degradation. Chromatographic analyses of still reboiler samples provided the primary source of information concerning anisole degradation. Based on these analyses, the anisole degradation rate appeared to be about 3.7% of the high temperature anisole holdup per day during the first nine 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 startup. This release

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* Expressed in terms of total inventory, these numbers would, of course, be considerably lower.

may have temporarily changed the reactivity of the metal surface in the still, thereby providing an additional site for degradation. Other possible factors will be 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 had been noticed also in the bench-scale tests.

The question of anisole degradation at room temperature 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 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 the 3.7 to 7.0% per day value to the high temperature anisole holdup.

In general, 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 indicated previously, but also by the high temperatures (near 200°F.) encountered in the exchange column during startups 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 startups and changes of operating conditions would not be necessary. On this basis, the anisole degradation rates observed in the pilot-plant tests could probably be considered to be maximum values for the ends of the process.

CONCLUSIONS

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. In fact, little or no extrapolation of data would be required for production plant design.

Stage heights with 5/8-inch Pall rings were found to be larger than originally assumed, but certainly 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, at the higher flows, the cost per stage would be lower for Pall rings than for high efficiency, laboratory packing, primarily because of the higher cost and the smaller allowable flows characteristic of the latter. Additionally, considerable 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.

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 the water content of all anisole charged to the system to tolerable limits.

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. System operation was generally smooth, and none of the problems of surges, pressure excursions, and other instabilities reported in earlier studies of this process(2) were encountered. In short, no apparent technological barriers to the application of the anisole process on a production scale were found.

It now appears that the anisole system does offer several advantages over the dimethyl ether process for boron isotope separation. As a final thought, therefore, it seems appropriate to summarize the most significant differences between the two systems in terms of cascade design:

1. Cascade Length. The larger effective separation factor with anisole, which has already been noted, is perhaps its most important advantage, since because of this, an anisole cascade would be much shorter than a comparable dimethyl ether plant. Stage heights, of course, also influence the total plant column length, but there is no information to indicate that either process would have an advantage in HTU values if the same packing is considered.

2. Cascade Width. Even if the same packing is employed in the two systems, throughput in the dimethyl ether plant is limited because of the need to operate below atmospheric pressure. (As stated earlier, low pressures are required to allow use of the lower temperatures needed to hold the solvent degradation rate within acceptable limits.)

Consequently, columns for the anisole process, which is not restricted in this manner, can be somewhat smaller in diameter than those required by a dimethyl ether facility designed for the same product rate.

3. Equipment Requirements. Equipment requirements are generally simpler with anisole, since reflux streams are supplied by units (perhaps with spares) located only at the ends of the cascade. Intercolumn transfer of the boron trifluoride-anisole complex is achieved directly with liquid pumps, and at most, three gas compressors are utilized to make up the operating pressure drop for the entire cascade. In contrast, the dimethyl ether process, as has been mentioned, requires refluxers for each column, and if sparing of reflux equipment is deemed necessary to ensure against product losses or backmixing, spare units must also be provided for each exchange column.

4. Solvent Degradation. The degradation rate with anisole (considered in terms of total plant inventory) does not appear to be greater than that with dimethyl ether. The primary difference, aside from solvent replacement costs, is that the anisole degradation products do not interfere either with the isotope separation process or with the subsequent boron recovery steps.

5. Operating Conditions. The anisole process is operated at temperatures considerably lower than those used in the dimethyl ether process and, as stated before, at pressures above one atmosphere. Because of these and other differences in physical properties, solvent handling, both in

and out of the cascade, should be safer and easier to control with the anisole process.

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