WSRC-TR-97-0360
Rev. 0

PILOT-SCALE BENZENE RETENTION AND RELEASE DEMONSTRATION (U)

SAFETY • RESPONSIBILITY • SECURITY
SRS
SAVANNAH RIVER SITE

DECEMBER 1997

Westinghouse Savannah River Company
Savannah River Site
Aiken, SC 29808

PREPARED FOR THE U.S. DEPARTMENT OF ENERGY UNDER CONTRACT NO. DE-AC09-96SR18500

This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-96SR18500 with the U. S. Department of Energy.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

This report has been reproduced directly from the best available copy.

Available for sale to the public, in paper, from: U.S. Department of Commerce, National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161,
phone: (800) 553-6847,
fax: (703) 605-6900
email: orders@ntis.fedworld.gov
online ordering: http://www.ntis.gov/help/index.asp

Available electronically at http://www.osti.gov/bridge
Available for a processing fee to U.S. Department of Energy and its contractors, in paper, from: U.S. Department of Energy, Office of Scientific and Technical Information, P.O. Box 62, Oak Ridge, TN 37831-0062,
phone: (865)576-8401,
fax: (865)576-5728
email: reports@adonis.osti.gov

1173937

WSRC-TR-97-0360, Rev.0

Pilot-Scale Benzene Retention and Release Demonstration (U)

J.C. Marek
W.B. Van Pelt

Publication Date: **December 1997**

Contents

Executive Summary 1

Introduction 4

Mass Transfer 6

Results and Discussion

- Benzene Form 12
- Benzene Generation Rates 20
- Quiescent Releases 22
- Benzene and Water Baseline Test 31
- Agitated Releases 32
- Water Additions 44
- Benzene and KTPB Slurry Baseline Tests 51
- Benzene Balance 52

Quality Assurance 53

Acknowledgments 53

References 54

Appendix A: Experimental A-1

Appendix B: Data B-1

List of Figures

Title		Page Number
Figure 1.	Interphase Mass Transfer	6
Figure 2.	Benzene Globules at Glass Column Wall	14
Figure 3.	Benzene Globule at Aqueous/Slurry Interface	15
Figure 4.	Column Core Sample Showing Benzene Globules	16
Figure 5.	Benzene Globules in Column Sample Port	17
Figure 6.	Benzene Concentrations Required to Float KTPB Slurry	19
Figure 7.	Column 1 Quiescent Release	24
Figure 8.	Column 2 Quiescent Release	25
Figure 9.	Column 3 Quiescent Release	25
Figure 10.	Column 4 Quiescent Release	26
Figure 11.	Column 6 Quiescent Release	26
Figure 12.	Column 7 Quiescent Release	27
Figure 13.	Column 8 Quiescent Release	27
Figure 14.	Column 9 Quiescent Release	28
Figure 15.	Column 10 Quiescent Release	28
Figure 16.	Column 11 Quiescent Release	29
Figure 17.	Column 12 Quiescent Release	29
Figure 18.	Column 13 Quiescent Release	30
Figure 19.	Benzene and Water Baseline Quiescent Release	31
Figure 20.	Column Sample and Pump Port Arrangement	33
Figure 21.	Column 2 Agitated Release	34
Figure 22.	Column 4 Agitated Release	35
Figure 23.	Column 3 Agitated Release	35
Figure 24.	Column 9 Agitated Release	36
Figure 25.	Column 7 Agitated Release	37
Figure 26.	Column 6 Agitated Release	38
Figure 27.	Column 8 Agitated Release	39
Figure 28.	Column 10 Agitated Release	40
Figure 29.	Column 11 Agitated Release	41
Figure 30.	Column 12 Agitated Release	42
Figure 31.	Column 9 Water Addition	44

List of Figures (cont'd)

Title		Page Number
Figure 32.	Column 8 Water Addition	45
Figure 33.	Column 6 Water Addition	46
Figure 34.	Column 10 Water Addition	47
Figure 35.	Column 12 Water Addition	48
Figure 36.	Column 13 Water Addition	50
Figure 37.	Benzene/Slurry Baseline Test	51
Figure 38.	Experimental Apparatus Schematic	A-4
Figure 39.	Column Top Plate Arrangement	A-5
Figure 40.	Water header arrangement	A-5
Figure 41.	Pilot-Scale Benzene Retention Demonstration Columns	A-6
Figure 42.	Pilot-Scale Demonstration Columns 6-10	A-7
Figure 43.	Pilot-Scale Demonstration Columns 11-13	A-8
Figure 44.	Vapor Sampling System	A-9
Figures 45 -56.	Rate of Boron Increase for Columns 1-13	B-13

List of Tables

Title		Page Number
Table 1.	Test Matrix	5
Table 2.	Soluble Potassium in Column 1	20
Table 3.	Quiescent Benzene Loss Summary	23
Table 4.	Benzene Material Balance Summary	52
Table 5.	Target Simulant Compositions	A-12
Table 6.	Column Benzene Analyses	B-1
Table 7.	Boron Data	B-10
Table 8.	Purge Rate Effect Data	B-25
Table. 9	Quiescent Release Data	B-27
Table 10.	Potential Benzene Inventory Calculations	B-31
Table 11.	Actual Benzene Formed Calculations	B-32
Table 12.	Benzene Generation Rate Calculations	B-33

List of Tables (cont'd)

Title		Page Number
Table 13.	Analytical Data	B-36
Table 14.	Temperature Data	B-42
Table 15.	Column Vapor Space Profile Measurement	B-48

Executive Summary

During the initial months of In-Tank Precipitation (ITP) radioactive operation in 1995, the process experienced high rates of tetraphenylborate (NaTPB) decomposition with assumed corresponding high rates of benzene generation. In March 1996, after a two month quiescent period, a water addition to Tank 48H resulted in an unexpected benzene release to the tank vapor phase. This was the first time a "low energy" input resulted in a significant release rate. This led to questions about how benzene, generated in-situ by TPB^- decomposition, was retained in the surrounding potassium tetraphenylborate (KTPB) slurry. It was postulated the retention mechanism may have changed during the quiescent period prior to March so the benzene present became "readily releasable" to the vapor phase with low energy input to the slurry, or that enough benzene accumulated that some of it was in a different, more releasable form. " Readily releasable" is a qualitative term defined as a rapid release of benzene at a rate approaching evaporation of a free benzene layer. It is intended to distinguish between benzene in a form with high liquid phase resistance to mass transfer (diffusion controlled) from benzene in a form with minimal liquid phase resistance to mass transfer (free benzene layer evaporation). If a readily releasable form of benzene was present, the vapor space profile during release tests was anticipated to have an initial benzene vapor space concentration peak followed by a lower vapor concentration, longer duration release.

SRTC initiated a pilot-scale demonstration using glass columns that were six feet tall and eight inches in diameter. The columns were filled to a depth of four feet with various tetraphenylborate slurries. The vapor space above the slurry was continuously purged with nitrogen and the columns were heated (40 - 50 °C) to decompose the NaTPB. Slurry characteristics were monitored through quiescent periods by visual observation and sampling. Benzene releases were measured in quiescent periods and during agitation tests.

The objectives of this experimental program were:

- Determine, if possible, the form and location of benzene formed within the tetraphenylborate slurry as well as if that form and position change with time
- Determine, under both quiescent and agitated conditions, benzene releasability from tetraphenylborate slurry
- Determine the effects, if any, of time, KTPB concentration, and temperature on benzene retention and releasability

The key conclusions from this demonstration are:

Benzene Form and Location

- Dispersed benzene globules coated with TPB and other organics (such as biphenyl) can form in TPB slurries.
- Benzene globules were observed throughout the slurry phase depth and cross-section.
- A larger amount of globules existed at the slurry/salt solution interface for columns with floating slurries, possibly indicating that a large fraction of benzene was formed in the salt solution phase and was then retained by the slurry phase above.
- Benzene globules did not move significantly with time during quiescent periods. Energy input via water addition or recirculation pump was required to move the globules.

Due to an apparent tetraphenylborate decomposition induction period observed in initial test Series 1 (Columns 1-4), chemistry adjustments were made for subsequent test Series 2 (Columns 6-9) and test Series 3 (Columns 10-13) to accelerate the rate of TPB decomposition and hence the rate of benzene formation. Tetraphenylborate decomposition intermediates and boron data were obtained in test Series 2 and 3 verify the induction period observed in test Series 1 was reduced or eliminated. Although not an intended test objective, decomposition kinetic data is presented and discussed to compare benzene generation rates and release rates observed during the quiescent periods for test Series 2 and 3 columns.

- Series 1 columns showed an apparent induction period for TPB decomposition. The factors causing this induction period were not identified.
- Generation rates in Series 2 and 3 columns showed a non-linear relationship with respect to boron consistent with previous studies.

Benzene Releasability

- When dispersed benzene globules were brought to the slurry/vapor interface and subjected to energy input from either water addition or pump discharge flow, benzene was in some cases released at rates that approached the evaporation of a free benzene layer. Therefore, the globules are a readily releasable form of benzene.
- Purge rate variations had significant effect on measured quiescent releases in these tests.
- After correction for the purge rate effect, quiescent release rates did not exceed the calculated peak generation rates.
- Vapor concentrations during agitated releases are directly related to the degree of mixing, particularly the degree of surface motion at the slurry/vapor interface.
- Agitated releases decreased rapidly when mixing or water addition was stopped.

- At the conclusion of the demonstration, Columns 6, 7, 9, and 11 had retained 50 - 60% of the benzene available at the time agitated release tests were initiated on those columns.

Effect of Time, wt % TPB, and Temperature

- There was no apparent time effect on either the quiescent or agitated total integrated releases.
- Since 4 wt % slurries are expected to have higher yield stress than 1 wt % slurries, the 1 wt % slurries were expected to have higher releases.
    - When the slurry floated, 1 wt % slurries had higher quiescent losses than the 4 wt % slurries.
    - 1 wt % slurries were more readily mixed than 4 wt % slurries. As a result, the 1 wt % slurries were more easily depleted of benzene than the 4 wt % slurries during agitated release tests.
- As expected, lower temperatures produced lower quiescent losses.

Other Observations

- Column 12 was the only column with submerged slurry. Observations were:
    - This column had the highest average quiescent releases. This supports the observation that floating slurry acts as a barrier to quiescent releases.
    - As the lower two-thirds of the column were gently recirculated, globules disengaged from the slurry and accumulated at the salt solution/vapor interface without producing significant vapor space benzene concentrations. A subsequent, moderate rate water addition, however, produced a marked increase in vapor space benzene.
    - Tank 48H was mixed in December 1995 then allowed to remain quiescent for two months. A water addition in March 1996 produced an unexpected, although small, benzene release to the vapor space. It is plausible that globules could have formed prior to December and were then available near the liquid surface such that the March water addition produced a release.

- Large dilutions (e.g., specific gravity changes) through subsurface water additions to floating slurry did not produce a significant benzene release.

Introduction

The In-Tank Precipitation (ITP) Process began radioactive operation in August 1995. During the subsequent months of operation, the process experienced high rates of soluble sodium tetraphenylborate (NaTPB) decomposition with corresponding high rates of benzene generation.[1] Energy input from long shaft slurry pumps, used to mix the Tank 48H contents, was typically required to release the generated benzene from the slurry phase to the vapor. However, in March 1996, after a two month quiescent period, a water addition to Tank 48H resulted in an unexpected benzene release to the tank vapor phase. This was the first time a "low" energy input resulted in a significant release rate. Two days after the water addition, the slurry pumps were operated. Benzene release rates into the vapor phase were orders of magnitude higher than previously experienced during pump operations. Even with the higher than expected rates there were no safety concerns because the tank was inerted by a nitrogen purge, and the bulk vapor phase remained at less than 25% of the composite lower flammability limit. Two days later, the pumps were operated again. Release rates were briefly high, but then returned to a range consistent with previous pump operation experience.

The events of March 1996 led to questions about how benzene, generated in-situ by TPB^- decomposition, was retained in the surrounding potassium tetraphenylborate (KTPB) slurry. It was postulated the retention mechanism may have changed during the quiescent period prior to March so the benzene present became "readily releasable" to the vapor phase with low energy input to the slurry, or that enough benzene accumulated that some of it was in a different, more releasable form.

In their August 1996 recommendation to the Secretary of Energy, the Defense Nuclear Facilities Safety Board (DNFSB) requested a more thorough understanding of the benzene retention and release mechanisms in ITP.[2] The Department of Energy (DOE) responded with an implementation plan which committed to study benzene retention and release mechanisms.[3] Commensurate with that implementation plan, High Level Waste Engineering requested the Savannah River Technology Center (SRTC) to determine the effects of significant variables (such as time and KTPB concentration) on benzene retention capacity and release as benzene accumulates beyond the apparent solubility.[4]

SRTC developed a technical task plan which used six -foot tall, eight-inch diameter glass columns filled to a depth of four feet with various tetraphenylborate slurries.[5] This solution depth is approximately the same depth as that in Tank 48H during the initial ITP radioactive operations in September 1995. Therefore the test columns were simulated "core samples" of the ITP slurry. Using nearly isothermal conditions within the bulk slurry, the columns were heated to promote benzene formation from tetraphenylborate (TPB) decomposition. This is not the case in Tank 48H which has a temperature gradient (5-10 °C) within the aqueous phase. The simulant slurry chemistry, while similar to Tank 48H in salt composition, differed from expected compositions in that it contained higher concentrations of potential TPB decomposition catalysts to promote benzene formation. The initial plan included testing with 10 wt % slurry, but this higher wt % slurry could not be made in-situ and no filtration capability existed to concentrate the volumes needed from a lower wt % slurry. Therefore the tests were limited to 1 and 4 wt % slurries. The column vapor spaces were purged with nitrogen. Vapor space sampling was performed using a gas chromatograph to monitor benzene releases during both quiescent and agitated periods. Sample ports using glass stopcocks were provided along the length of each column to obtain aqueous solution and slurry samples. Sample results were used to monitor any changes in slurry chemistry with time. During tests in Columns 1-4, two glass stopcocks failed resulting in suspension of the tests for sample port redesign. The stopcocks were replaced with metal ball valves which provided both increased mechanical stability during sampling and improved chemical resistance. At this time, Columns 1-4 were designated as Series 1 tests with subsequent tests divided into Series 2 (Columns 6-9) and Series 3 (Columns 10-13) tests. These Series are distinguished by the different slurry chemistry in each series. The experimental setup and slurry compositions are given in Appendix A. The test matrix is shown below:

Table 1. Test Matrix

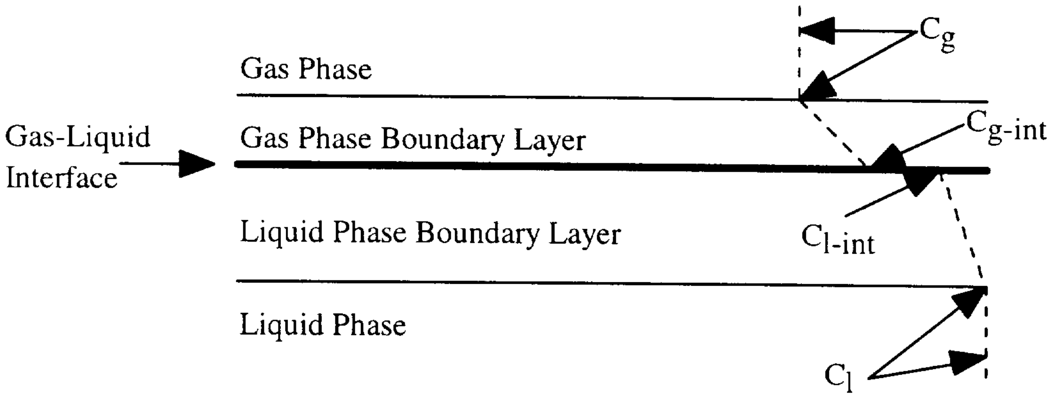
Column #	Sodium molarity (Slurry Basis)	Nominal Temperature, °C	Tetraphenylborate Weight %	Comments
Series 1				
1	4.7	40/50	4	Temp. increased to 50 after 8 days
2	4.7	40/50	4	Temp. increased to 50 after 7 days
3	4.7	40/50	1	Temp. increased to 50 after 7 days
4	4.7	40/50	1	Temp. increased to 50 after 7 days
Series 2				
6	4.7	50	1	Temp. decreased to 40 after 27 days
7	4.7	50	4	Reused Series 1 slurry, temp to 40 after 27 days
8	4.7	50	1	Reused Series 1 slurry, temp to 40 after 27 days
9	4.7/4.3	50	4	Temp. decreased to 40 after 24 days
Series 3				
10	3.7	40	4	Attempted neutrally buoyant slurry
11	3.5	50	4	Attempted neutrally buoyant slurry
12	0.2	50	4	Submerged slurry
13	4.7	50	4	½ full column, add water to sink solids

The results in this report are for the most part more qualitative in nature rather than quantitative. Several conclusions are based on visual observations obtained during testing. While some observed trends may be reasonably extended to ITP operation, great care must be taken when attempting to apply results from these tests directly to Tank 48H. To more definitively explain some conclusions, additional tests specifically designed for that purpose would be needed.

Mass Transfer

Figure 1 describes mass transfer between a liquid with no insoluble solids and a gas. In Figure 1, C_l is the bulk liquid phase benzene concentration, $C_{l\text{-int}}$ is the liquid phase benzene concentration at the interface, $C_{g\text{-int}}$ is the vapor phase benzene concentration at the interface, and C_g is the bulk vapor phase benzene concentration.

Figure 1. Interphase Mass Transfer



Diffusion Controlled Mass Transfer

A boundary layer is a region close to the interface where both phases influence the mass transfer process. Benzene is transported from the bulk liquid phase to the liquid boundary layer. (The edge of the boundary layer is generally defined as the point at which the concentration is within 1% of the bulk phase concentration.) Within the liquid phase boundary layer, mass transfer occurs from the edge of the boundary layer to the vapor-liquid interface by diffusion and the flux is described by equation [1]

$$J = \alpha\ A\ \Delta C/\Delta y \qquad [1]$$

where J is the benzene flux, α is the molecular diffusivity, A is the surface area, C is the benzene concentration, and y is distance. At the vapor-liquid interface, the vapor and liquid are in equilibrium. The benzene is also transported from the vapor-liquid interface to the edge of the vapor boundary layer by diffusion, and then transported from the vapor boundary layer to the bulk vapor.

The flux across either the liquid or vapor phase boundary layer can be changed by changing the molecular diffusivity, the surface area, the concentration difference across the boundary layer (ΔC), or the boundary layer thickness.

The term $\alpha/\Delta y$ can be replaced by a constant, k, which is called the mass transfer coefficient. The mass transfer coefficient can be changed by changing the boundary layer thickness or the molecular diffusivity.

As the benzene is transported from the liquid phase to the vapor phase, the liquid phase boundary layer is replenished from the bulk liquid. If the bulk liquid does not replenish the benzene lost from the liquid phase boundary layer, the liquid phase boundary layer thickness will increase and the mass transfer coefficient will decrease. If the bulk liquid transports benzene to the liquid boundary layer faster than it is removed, the liquid boundary layer thickness decreases and the mass transfer coefficient increases.

Convective Mass Transfer

Convective mass transfer from the liquid phase to the vapor phase can be described with a mass transfer coefficient. The flux is equal to the product of the mass transfer coefficient, the surface area, and the concentration difference (Flux = k A ΔC). The concentration difference can be the concentration difference across the gas phase boundary layer, the concentration difference across the liquid phase boundary layer, the concentration difference between the bulk vapor and the vapor which would be in equilibrium with the bulk liquid, or the concentration difference between the bulk liquid and the liquid which would be in equilibrium with the bulk vapor. If the concentration difference across the gas phase boundary layer is used, the mass transfer coefficient is called a gas phase mass transfer coefficient. If the concentration difference across the liquid phase boundary layer is used, the mass transfer coefficient is called a liquid phase mass transfer coefficient. If the concentration difference between the bulk vapor and the vapor which would be in equilibrium with the bulk liquid is used, the mass transfer coefficient is called an overall mass transfer coefficient. If the concentration difference between the bulk liquid and the liquid which would be in equilibrium with the bulk vapor is used, the mass transfer coefficient is also called an overall mass transfer coefficient.

The vapor phase mass transfer coefficient is described by equation [2] and the liquid phase mass transfer coefficient is described by [3].

$$J = k_g A (C_{g\text{-int}} - C_g) \qquad [2]$$

$$J = k_l A (C_l - C_{l\text{-int}}) \qquad [3]$$

In equations [2] and [3], J is the flux, A is the interfacial area, k_g is the vapor phase mass transfer coefficient, and k_l is the liquid phase mass transfer coefficient.

Since $C_{g\text{-int}}$ and $C_{l\text{-int}}$ are difficult to determine, while C_g and C_l can usually be measured or calculated, the overall mass transfer coefficient can be described by equations [4] and [5]

$$J = K_g A (C_g^* - C_g) = K_g A (H C_l - C_g) \qquad [4]$$

$$J = K_l A (C_l - C_l^*) = K_l A (C_l - C_g/H) \qquad [5]$$

In equations [4] and [5], K_g is the overall mass transfer coefficient based on vapor phase driving force, K_l is the overall mass transfer coefficient based on liquid phase driving force, C_g^* is the concentration of benzene in the vapor phase which is in equilibrium with the bulk liquid, C_l^* is the liquid phase benzene concentration which is in equilibrium with the bulk vapor, and H is the Henry's law constant. The overall mass transfer coefficient based on the vapor concentration (K_g) has been called the overall mass transfer coefficient in previous SRTC work.[6]

Since at steady state, the fluxes calculated with equations [2] - [5] are equal, relationships between the mass transfer coefficients described in the equations are derived in the literature[7] and shown in equations [6] - [8].

$$1/K_g = 1/k_g + H/k_l \qquad [6]$$

$$1/K_l = 1/k_l + 1/H\, k_g \qquad [7]$$

$$K_l = H\, K_g \qquad [8]$$

The overall mass transfer coefficient is a function of the liquid phase and vapor phase mass transfer coefficients; that is, the combined resistance to mass transfer across both the liquid and vapor boundary layers. If the vapor phase mass transfer coefficient (k_g) is much larger than the liquid phase mass transfer coefficient (k_l), the overall mass transfer coefficient (K_l) is approximately equal to the liquid phase mass transfer coefficient: in other words, because the resistance to mass transfer in the vapor phase is low, the liquid phase resistance controls the mass transfer process. Previous research investigating the removal of organic species from water under quiescent and agitated conditions found the vapor phase mass transfer coefficient to be more than 20X the liquid phase mass transfer coefficient. [8, 9]

Therefore, in a system containing a benzene at low concentration, the benzene will be transferred from the liquid phase boundary layer to the vapor phase faster than it can be transferred from the bulk liquid to the liquid phase boundary layer. The liquid phase boundary layer will become depleted in benzene, and increase in thickness. The thicker boundary layer will lead to a smaller mass transfer coefficient and a lower mass transfer rate.

Equations [9] and [10] describe the relationship between overall mass transfer coefficients and individual phase mass transfer coefficients in this work.

$$K_g \cong k_l/H \qquad [9]$$

$$K_l \cong k_l \qquad [10]$$

The reciprocal of the product of the mass transfer coefficient and area is often referred to as the resistance to mass transfer. Even though the liquid phase mass transfer is normally controlling in the system used in this test, equation [4] is typically used to model the mass transfer process. Regardless, the overall resistance to mass transfer through both the liquid and vapor boundary layers is reflected in the overall mass transfer coefficient, either K_g or K_l.

Application to Benzene Release from KTPB Slurries

This mass transfer theory can be applied to following situations expected with TPB slurries:

I. Aqueous solutions without KTPB solids
  A. Quiescent solution with no insoluble solids and benzene at or below the solubility limit
  B. Quiescent solution with no insoluble solids and benzene above the solubility limit
II. Slurries with KTPB solids
  A. Non-isothermal, quiescent, sunk KTPB slurry
  B. Quiescent, floating KTPB slurry
  C. Mixed or agitated KTPB slurry

All of the cases discussed below assume a flowing vapor phase.

Aqueous Solutions Without KTPB Solids

In a vessel containing a quiescent aqueous solution with no insoluble solids and benzene at or below its solubility, the following outcome is expected: The benzene release is liquid phase diffusion controlled. The liquid boundary layer thickness increases until steady-state (constant flux) is reached (the liquid concentration will decrease with time assuming the generation rate is zero or less than the flux established). If temperature is increased, the diffusivity increases as well as the equilibrium vapor concentration. Benzene flux increases until a new steady-state is reached.

If the generation rate is higher than the established flux, the benzene concentration will increase above the solubility limit. The following outcome is expected: A second liquid phase which is almost pure benzene is formed and accumulates at the liquid-vapor interface. No liquid boundary layer exists in the salt solution. Mass transfer is by direct evaporation from the floating benzene phase. Since no liquid phase mass transfer resistance exists, mass transfer is controlled by the vapor phase resistance. Benzene flux and vapor phase benzene concentration are higher than when the liquid benzene level is below the solubility limit. That is, in equation [6] k_l becomes very large and $K_g \cong k_g$.

Slurries with KTPB Solids

With a non-isothermal, quiescent, sunk KTPB slurry, the following outcome is expected: The temperature gradient between the slurry-liquid interface and the liquid-vapor interface creates convective mixing in the liquid layer. The mixing replenishes the liquid boundary layer causing it to become thinner than for the isothermal case. Since the liquid boundary layers are thinner, the mass transfer coefficients increase, flux increases, and the vapor benzene concentration increases. As the temperature gradient within the slurry increases, the convective mixing increases, the liquid boundary layer thickness decreases, and the mass transfer rate increases.

With a quiescent, floating slurry, the following outcome is expected: The slurry is stagnant and consolidated. The slurry contains KTPB and interstitial liquid. Boundary layers exist at slurry-liquid and liquid-vapor interfaces. The benzene is soluble, adsorbed to the KTPB solids, small free benzene droplets, or large globules coated with KTPB, biphenyl, or other organic by-products. The mass transfer of benzene through the liquid takes place in a two step process: (1) soluble benzene evaporates from the salt solution into the vapor, and (2) benzene dissolves from the droplets/globules into unsaturated salt solution. The solids in the slurry reduce the effective mass transfer surface area which lowers the benzene flux and the vapor benzene concentration.

With a mixed or agitated slurry, the following outcome is expected: The slurry becomes homogeneous regardless of whether the slurry was sunk or floating at the start. Two cases are considered, globules and droplets.

Globules are defined as coated benzene droplets which are large enough that if sufficient energy is provided when they reach the surface they break open and form a small benzene pool. The mixing brings the globules to the surface or allows them to reach the surface (due to buoyancy forces). If the globules are present, the release rate is similar to free benzene. The approach of the release rate to free benzene evaporation is determined by the total effective surface area of the open globules.

Droplets are defined as free benzene droplets which are small enough that the mixing energy is insufficient to overcome surface tension and they do not break open. Soluble benzene is transferred to vapor phase by the two step process described previously. The mixing reduces the liquid boundary layer thickness which increases the benzene flux and the benzene vapor concentration as compared to a quiescent, floating slurry. The droplets replenish the soluble benzene which is transferred to the vapor. When mixing stops, the liquid boundary layer thickness increases, and the flux and vapor benzene concentration decrease.

Temperature, viscosity, mixing effectiveness, KTPB concentration, and ventilation rate will affect the boundary layer thickness and mass transfer process. For an aqueous salt solution with no insoluble solids temperature affects the mass transfer process in the following manner: Increasing the temperature increases the molecular diffusivity, decreases solution viscosity, and increases the equilibrium vapor pressure. Increasing the molecular diffusivity increases benzene flux as seen in equation [1]. Since the boundary layer thickness decreases with decreasing viscosity, increasing temperature will decrease the boundary layer thickness and increase mass transfer. Increasing the equilibrium vapor pressure will increase the concentration gradient ($C_g^* - C_g$) which will increase mass transfer.

For an aqueous salt solution with no insoluble solids, mixing effectiveness has the following effect on the mass transfer process: Vigorous mixing will cause the liquid phase boundary layer to become smaller, it will increase the mass transfer coefficient.

When the salt solution contains KTPB precipitate, the KTPB concentration has the following effect on mass transfer. Increasing the KTPB concentration increases the slurry yield stress and consistency. The increase in yield stress and consistency increases the boundary layer thickness, which decreases the flux and the vapor benzene concentration.

Ventilation rate changes will affect the vapor phase boundary layer and mass transfer process in the following manner: If the ventilation rate increases, the vapor phase is diluted, the concentration difference across the vapor phase boundary layer increases, the vapor phase boundary layer thickness decreases, and the vapor phase mass transfer coefficient increases. Since in this test and ITP the vapor phase mass transfer coefficient is typically much larger than the liquid phase mass transfer coefficient, increasing the ventilation rate will have a small effect on the overall mass transfer coefficient. If the ventilation rate is decreased, the flux will decrease while the bulk vapor concentration increases, approaching the equilibrium vapor pressure as the ventilation rate approaches zero. The vapor phase mass transfer coefficient will decrease and eventually approach the magnitude of the liquid phase mass transfer coefficient and will have a significant effect on the overall mass transfer coefficient.

Results and Discussion

Benzene Form

In Series 1 columns, a separate and distinct dispersed organic phase was not observed. However, benzene was clearly associated with the TPB slurry as evidenced by slurry phase sample analysis (see Appendix B). The exact nature (i.e., adsorption, interstitial droplets, or emulsion) of this association was not determined. This finding is consistent with bench-scale observations by Dworjanyn using Sudan Red 7B dye to color benzene in simulant slurries with both externally added and in-situ generated benzene.[10] There was no indication of any benzene gradient within the TPB slurry phase, only a clear distinction between the concentration of benzene found in the floating slurry phase and that found in the clear aqueous phase below the slurry. Aqueous phase benzene concentrations were consistent with previous results for typical ITP salt solutions saturated with benzene.

In all the Series 2 and Series 3 columns, a separate, dispersed organic phase was noted to varying degrees. The identification of this dispersed phase is one of the key findings of this experimental program. The dispersed phase was in the shape of globules and ranged from slightly opaque to white in appearance. The globules had a variety of sizes estimated by visual observation to be from 1/8 inch to ¾ inch in diameter. Globules were observed throughout the slurry depth (Figure 2), and particularly at the slurry/liquid interface just below the slurry (Figure 3). To determine if the phase was present throughout the cross section of the slurry, core samples were taken of several columns. One core sample's photograph in Figure 4, shows globules were present in the bulk slurry as well as at the glass wall. Globules were also noted in some of the pump and sample ports, including some in which potassium tetraphenylborate solids were apparently absent (Figure 5). No movement of the globules was discernible during column quiescent periods. Equation 11 defines the diameter of a buoyant sphere required to rise through a Bingham Plastic[11, 12]:

$$D = T_y / ([\Delta\rho \times g] \times 0.047667) \qquad [11]$$

where:

T_y = yield stress, dynes/cm^2

$\Delta\rho \times g$ = buoyant force per unit volume, dynes/cm^3

Solving equation 1 for the following values [13]:

T_y =50 dynes/cm^2 for 4 wt % slurry

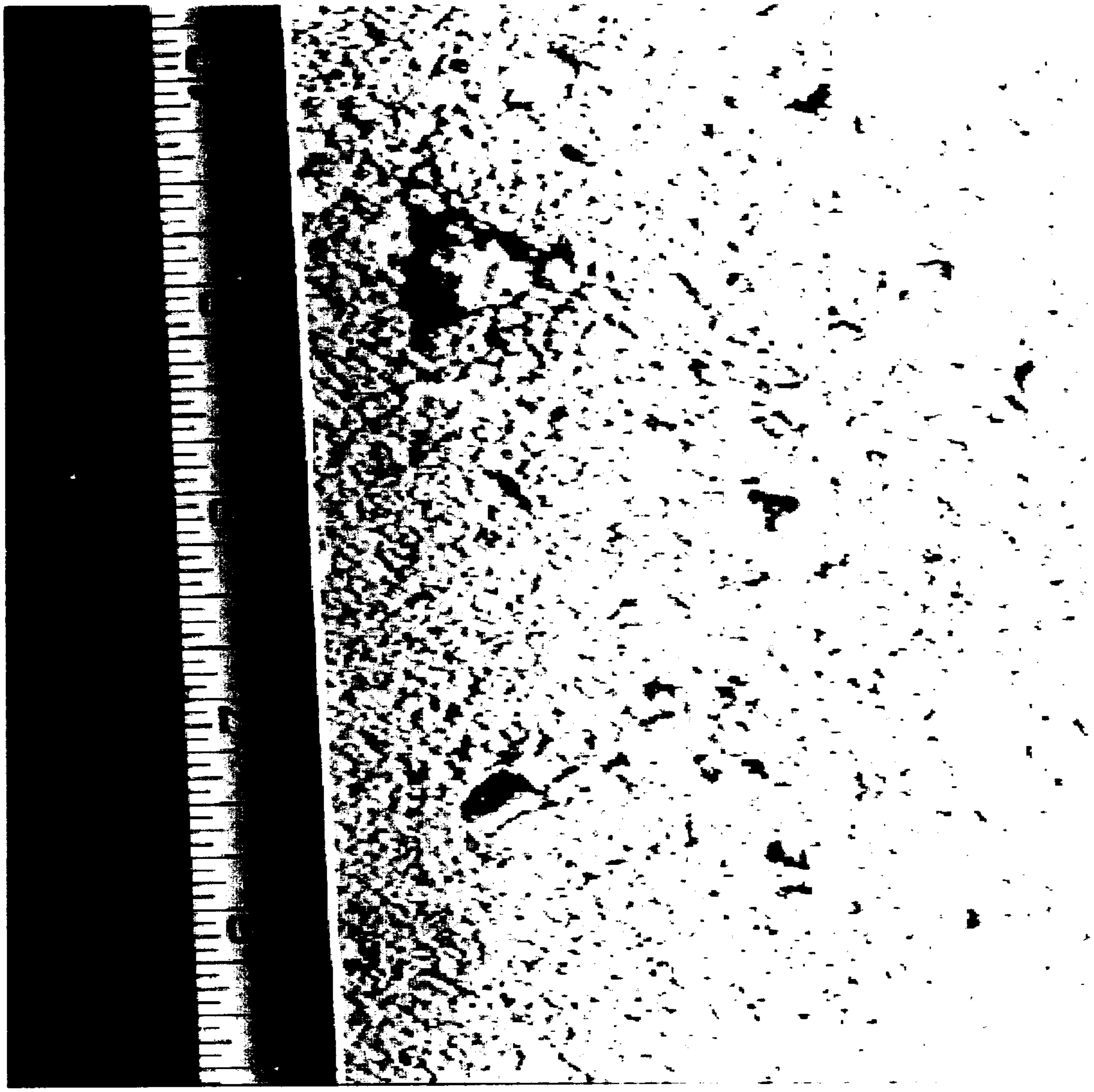
ρ slurry = 1.18 g/ cm^3

ρ benzene = 0.86 g/ cm^3

g = 980 dynes/g

gives a diameter of 3.15 cm or 1.24 inches. Since this diamater is well above that observed for the globules, it is reasonable to conclude that the yield stress of the slurry was higher than the buoyant force of the globules. Globules recovered from the columns were analyzed for chemical content. The interior liquid analyzed about 85 wt % benzene[14], and the outer coating contained 10 - 50 tetraphenylborate and 10 - 30 wt % biphenyl.[15]

Figure 2. Benzene Globules at Glass Column Wall



97-1300-16

Figure 3. Benzene Globules at Aqueous/Slurry Interface

97-1329-1

Figure 4. Column Core Sample Showing Benzene Globules

Figure 5. Benzene Globule in Column Sample Port

97-1264-1

The globules came to the surface readily when the columns were mixed or when water additions were performed in a manner that created a pathway through the slurry to the slurry/vapor interface. In many cases, they were noted to rise in regions of the slurry where KTPB solids were flowing downwards, i.e. they were capable of moving in the opposite direction of the bulk flow when the slurry was circulated and dispersed. During column mixing, globules were observed to rise and accumulate at the slurry/liquid interface, particularly along the interior column circumference or in any small zone not affected by the surface motion or recirculation flow. The globules were stable and required direct energy input from recirculation flow or water addition to break them open. As the globules became directly involved in the surface motion and mixing at the slurry/vapor interface, a thin white film and foam up to a 1 inch thick often formed. The film and foam which formed during agitation tests, did not appear to act as a barrier to benzene release as they were coincident with peak benzene releases. The film and foam are presumed to be the sodium tetraphenylborate and biphenyl analyzed as the outer coating of the globules that remain behind as the benzene evaporates.

Benzene was rapidly released to the vapor with minimal agitation indicating the coated benzene globules were a readily releasable form of benzene. Release tests where concentrations of globules were noted at the slurry/vapor interface produced vapor benzene concentrations in the 25,000 - 30,000 ppm range. This is consistent with baseline tests with free benzene layers at the liquid/vapor interface (30,000 - 45,000 ppm range). Bench-scale results by Dworjanyn show similar releases from slurries with dispersed, externally added benzene droplets.[16] After the globules were no longer visible, the release rates decreased to lower levels sustained for longer time periods. This may indicate another, less releasable form of benzene associated with the KTPB solids

Since the formation of coated globules had not been anticipated, the time period in which globules initially formed in the Series 2 columns was not measured. However, in subsequent Series 3 columns, globules were noted within two weeks of loading the column with slurry. These tests were not designed to determine directly the factors influencing the formation and growth rate of the globules. The accelerated rate of TPB decomposition in the Series 2 and 3 columns intentionally caused by the slurry chemistry (i.e., amount of potential catalysts, excess [NaTPB], TPB decomposition intermediates) and test temperatures of 50 °C created an environment where both the total amount and rate of benzene formation were greater than in Series 1.

Figure 6 shows how benzene concentrations promotes flotation of slurry. All benzene was conservatively assumed to be associated with the solids. Salt solution densities were estimated from previous work by Walker.[17] At a solid KTPB density of 1.18 g/mL, slurry below a nominal level of 4.1M requires benzene to float. Benzene concentration for flotation is proportional to weight percent as shown. Therefore, those columns with 4.7M salt solution (Columns 1-4, 6-9, and 13) had floating slurries as expected.

Column 10 slurry, with 3.7M salt solution, had sufficient benzene at 4,000 - 6,000 mg/L (see Table 6. in Appendix B, page B-6) to float the slurry. The same is true for Column 11 slurry with salt solution at 3.5M and benzene levels of 2000 - 3000 mg/L within the first two weeks of its operation. Benzene is considered a key contributor for slurry flotation in the <4.1M cases, but not the sole contributor. Gas entrainment during slurry makeup and charging of slurry to the column is also a likely contributor as is simple density differences between the salt solution and the slurry. Column 13, which began in 4.7M salt solution, was diluted to cause the slurry to sink. The slurry did not sink, but floated as the dilution continued. It is less clear for Column 13 that benzene was the cause for the flotation due to the scatter in the benzene data (see Table 6 in Appendix B, page B-9). However, 6 of 18 data points show benzene in the range required to float the slurry.

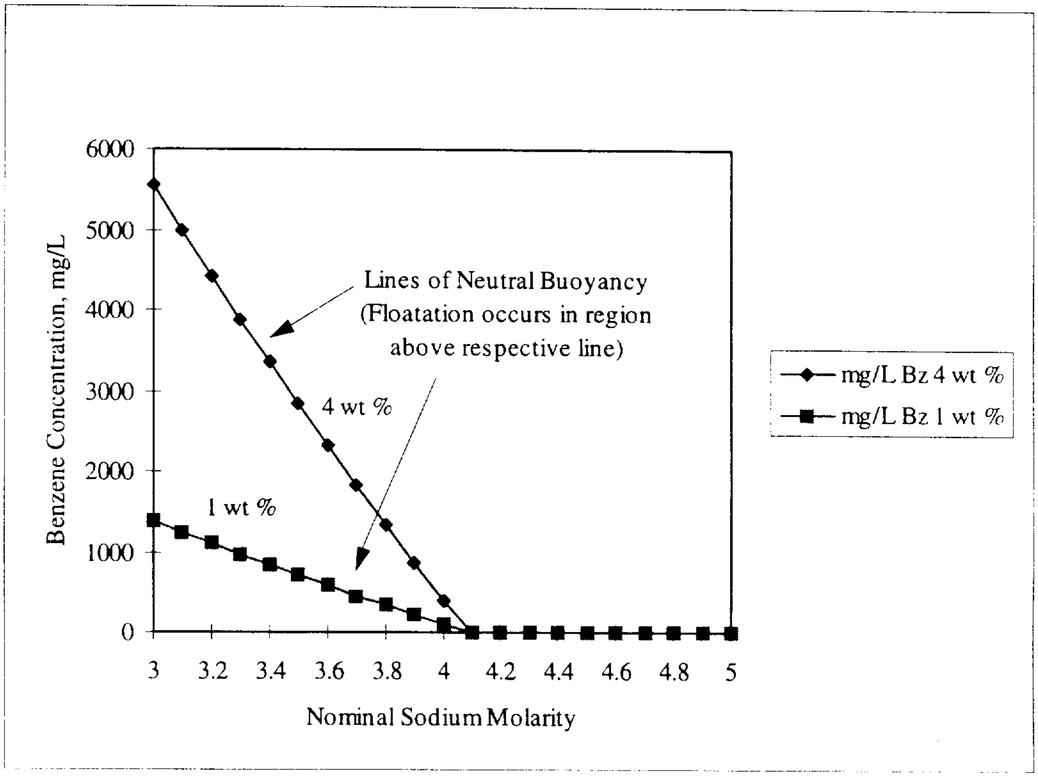


Figure 6. Benzene Concentrations Required to Float KTPB Slurry

In summary, the key findings regarding benzene form are:

- Benzene readily associates with and is retained by stagnant TPB solids
- A dispersed benzene phase in the form of globules coated with TPB and other organics such as biphenyl can form in TPB slurries.
- This dispersed organic phase can be present throughout the depth of a TPB solid phase, but particularly at the solid/liquid interface in the case of floating slurries.
- The globules are a readily releasable form of benzene.

- Accumulated benzene can contribute sufficient buoyancy to TPB slurries to keep TPB solids floating even in dilute solutions.

Benzene Generation

As stated previously, determining the kinetics of benzene generation within the slurries was not one of the original test objectives. Hence, the experimental design and equipment design were not structured specifically for these measurements. However, the low apparent benzene generation rates in the first few weeks of operation in Columns 1 through 4 suggested an induction period retarding the generation of benzene. These results warranted monitoring of species indicative of the benzene generation kinetics for all the columns to ensure sufficient benzene was generated in-situ to meet the test objectives.

The high sodium concentrations (> 5.0 M on a supernate basis) present in most of the slurries caused the majority of the excess sodium tetraphenylborate to be present as an insoluble solid. As tetraphenylborate decomposed, boron increased in the aqueous phase. Benzene generation within the slurries was thus inferred from the change in soluble boron concentration for all the columns except Column 12. For Column 12, the sodium concentration was sufficiently low (0.2 M) that all of the excess tetraphenylborate ion remained soluble. Hence, the benzene generation in Column 12 was inferred from the change in sodium tetraphenylborate concentration with time.

The data obtained in the Series 1 columns suggested that an induction period occurred (i.e., the rate of increase in the soluble boron concentration increased dramatically between 800 and 1000 hours of operation for Column 1 and between 500 and 600 hours of operation for Columns 2-4). Recall that Column 1 was the only column where the TPB precipitation reaction was carried out in the column. Mixing was poor resulting in subsequent slurries being pre-mixed before column loading. Table 2 below shows that soluble potassium was present during initial sampling which confirms that the precipitation was not complete. Therefore it is not suprising that Column 1 reacted more slowly than the other Series 1 columns.

Table 2. Soluble Potassium in Column 1

Cumulative Run Time, h	Soluble Potassium, wt %
0	0.21
0	0.19
192	0.14
192	0.13
360	0.12

Column sampling was performed with the column contents stagnant rather than well-mixed. Samples were collected from several ports on each column. Therefore the sample results contain a high degree of variability which limits the data analysis to general trends rather than precise quantitative conclusions. The equations discussed below are empirical and are used only to describe the demonstration test data. As seen in the graphs of the boron data for Series 1 columns (Appendix B, Pages B-13 through B-24), an exponential equation generally fits the data. The equation [12] is of the form:

$$\text{Boron concentration} = p1 \bullet e^{p2 \bullet \text{time}} \qquad [12]$$

where p1 and p2 are adjustable parameters

An exponential equation is consistent with an initial induction period followed by a more rapid boron increase as the TPB decomposition accelerates.

The data for the Series 2 and Series 3 columns showed no induction period. The data was loosely fitted with an equation [13] of the form:

$$\text{Boron concentration} = p1 + p2 \bullet (\text{time})^{0.5} \qquad [13]$$

The non-linear nature of the TPB decomposition is consistent with previous work by Crawford.[18]

Column 9 was diluted with water additions prior to agitated release tests. Because the column was not stirred immediately after the water additions, the dilution could not effectively be determined on a sample by sample basis. Note the high degree of scatter in the Column 9 boron data graph (Appendix B, page B-10). Therefore, a benzene generation rate was not calculated for Column 9.

Column 12 had a sodium concentration of 0.2M so that all of the TPB was soluble. The benzene generation rate for this column was determined from the rate of soluble [TPB] decrease with time. The data was fitted to an equation [14] of the form:

$$\text{NaTPB Concentration} = p1 \bullet e^{(-\text{time} / p2)} \qquad [14]$$

Calculations of the generation rates are in Table 12, Appendix B, Page B-33.

The key observations regarding benzene generation in these tests are:

- Series 1 columns showed an apparent induction period for TPB decomposition. The factors causing this induction period were not identified.

- Generation rates in Series 2 and 3 columns showed a first order relationship consistent with previous studies.

Quiescent Releases

The vapor space of Series 1 columns were purged with nitrogen at a rate of 50 cc/min during idle periods. The nitrogen purge was increased to about 150 cc/min for vapor benzene concentration measurements. This increased purge rate provided a faster sample transport time to the Gas Chromatograph (GC) and therefore a faster response time. This particular rate was chosen because the GC sample pump injected samples to the unit at 150 cc/min. In the Series 2 and Series 3 columns, the nitrogen purge rate was reduced to about 20 cc/min during the idle periods to minimize benzene losses and evaporation of water. The nitrogen purge rate during vapor measurements was set to about 200 cc/min in the Series 2 and Series 3 columns. The benzene concentrations in the vapor were measured weekly or twice a week. Using the measured concentrations and the nitrogen flows, the rate of benzene loss (mass/time) and the benzene release flux (mass/time/area) were estimated under quiescent conditions. The plot of quiescent losses as a function of the run time was then integrated to estimate the quantity of benzene lost from the columns. Initially, the benzene flux at 200 cc/min nitrogen purge was assumed to be equal to the benzene flux at 20 cc/min nitrogen purge. This assumption would be valid if the benzene release flux was only a function of the resistance to mass transfer within the liquid/slurry phase, since changing the vapor velocity would affect the vapor phase resistance to mass transfer and the system was shown to be well-mixed at 150 cc/min through vapor profile measurements (see Table 15, Appendix B, page B-48)

This effect of purge rate on flux was checked with varied purge rate tests on Column 11 under quiescent conditions at 40 °C and then again at 50 °C (See Table 8, Appendix B, page B-25). At 40 °C, the average flux at 19 cc/min was 0.072 g/m^2 min. At 200 cc/min nitrogen purge, the average flux was 0.123 g/m^2 min. The ratio of the fluxes at the two purge rates was 0.59 versus an expected value of 1. Similarly, at 50 °C the average flux at 22 cc/min was 0.108 g/m^2 min and 0.182 g/m^2 min at 210 cc/min nitrogen purge. The ratio of the fluxes at the two purge rates was again 0.59 versus an expected value of 1. As discussed in the mass transfer section (see pages 11 and 12), increasing the purge rate would reduce the vapor phase boundary layer. However, since the vapor phase resistance is small compared to the liquid phase resistance, the overall effect of increasing the purge on mass transfer , flux, and vapor concentration would be expected to be small. Since a more substantial effect was observed, the liquid phase resistance must have been impacted by the increased purge rate. Higher purge rates could cause cooler vapor phase temperatures resulting in a steeper temperature gradient at the slurry/vapor interface. This would have reduced the liquid phase boundary layer thickness through convective mixing and thus increased mass transfer (i.e., flux). The observed effect was judged substantial enough to apply an empirical correction factor of 0.60 to the measurements of quiescent releases obtained with purge rates of 150 cc/min or greater.

The integrated estimate of quiescent benzene losses, obtained from the vapor measurements, are shown in Table 3 below (Raw data given in Table 9, Appendix B, Page B-27):

Table 3. Quiescent Benzene Loss Summary

Column #	wt % KTPB	Nominal Temperature,°C	Sodium, M (Slurry basis)	Benzene Generation Rate Range, g/h	Peak Measured Quiescent Rate, g/h	Average Quiescent Rate, g/h	Total Quiescent Losses,g
1	4	40/50	4.7	0.05 - 0.34	0.0055	0.002	2
2	4	40/50	4.7	0.09 - 0.71	0.0065	0.003	3
3	1	40/50	4.7	0.04 - 0.93	0.0085	0.004	3
4	1	40/50	4.7	0.09 - 0.39	0.0068	0.003	2
6	1	50	4.7	0.11 - 0.55	0.27	0.08	220
7	4	50	4.7	0.07 - 0.36	0.27	0.06	174
8	1	50	4.7	0.12 - 0.91	0.54	0.11	320
9	4	50	4.7	N/A	0.18	0.06	171
10	4	40	3.7	0.03 - 0.15	0.06	0.02	49
11	4	50	3.5	0.09 - 0.31	0.48	0.06	136
12	4	50	0.2	0.04 - 0.51	0.27	0.15	299
13	4	50	4.7	0.18 - 0.50	0.07	0.03	33

Estimated losses for Series 1 columns were negligible. As seen in the benzene balance section (Table 4, page 44), the Series 1 columns only produced about 20% of the potential benzene inventory. Therefore, the driving force for quiescent release from Columns 1-4 was much less than for Series 2 and Series 3 columns.

For the floating slurries (all but Column 12), the 1 wt % potassium tetraphenylborate slurries (Columns 6 and 8) had higher quiescent release rates than the comparable Columns (7, 9, 11) containing 4 wt % potassium tetraphenylborate slurries. This is expected since the 1 wt % slurries have less yield stress than the 4 wt % slurries and therefore provided less resistance to benzene release from the slurry to the vapor phase.

Column 10, maintained at 40 °C for all but the initial 72 hours of run time, exhibited the least amount of quiescent benzene release in Series 2 and 3. This was expected since at 40 °C the equilibrium vapor pressure for benzene is 50% lower than at 50 °C and there was less convective mixing of both vapor and slurry.

The sunk slurry in Column 12 had the highest average quiescent releases in Series 2 and 3. This suggests that the floating insoluble solids in the other columns were a barrier to the benzene release, particularly those columns with 4 wt % slurry. The observation that the dispersed organic globules in columns with floating slurries were particularly prevalent at the slurry/liquid interface may be the result of the floating slurry barrier effect.

Plots of quiescent losses versus time are given below in Figures 7-18.

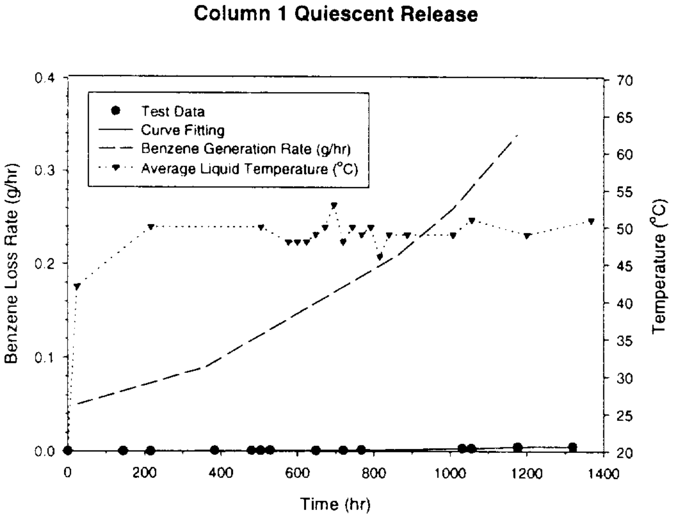


Figure 7. Column 1 Quiescent Release

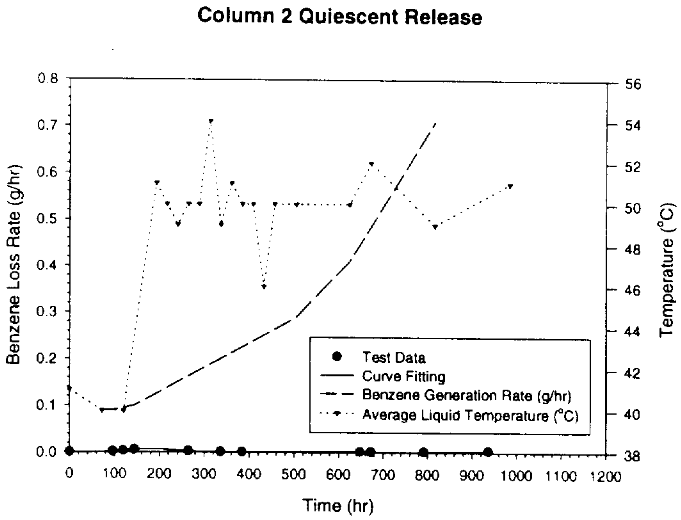


Figure 8. Column 2 Quiescent Release

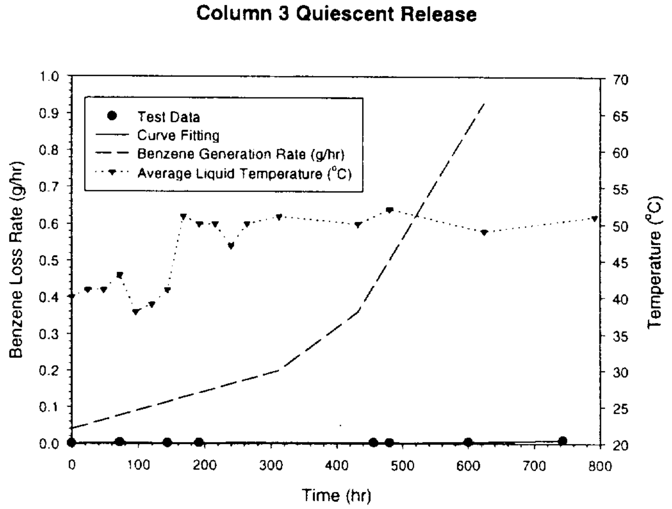


Figure 9. Column 3 Quiescent Release

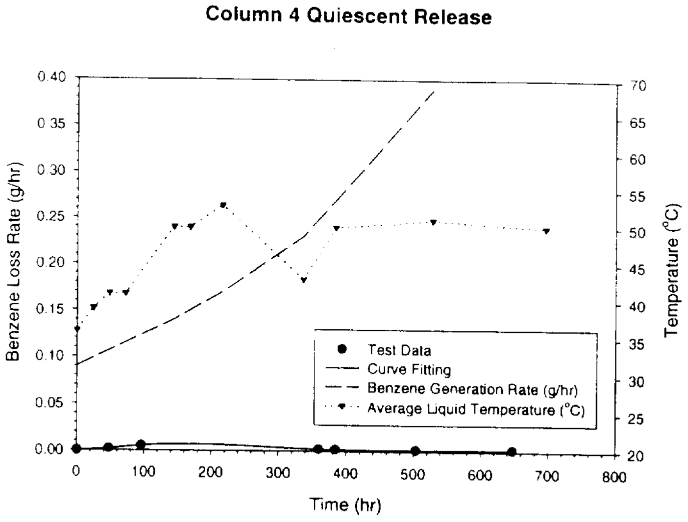


Figure 10. Column 4 Quiescent Release

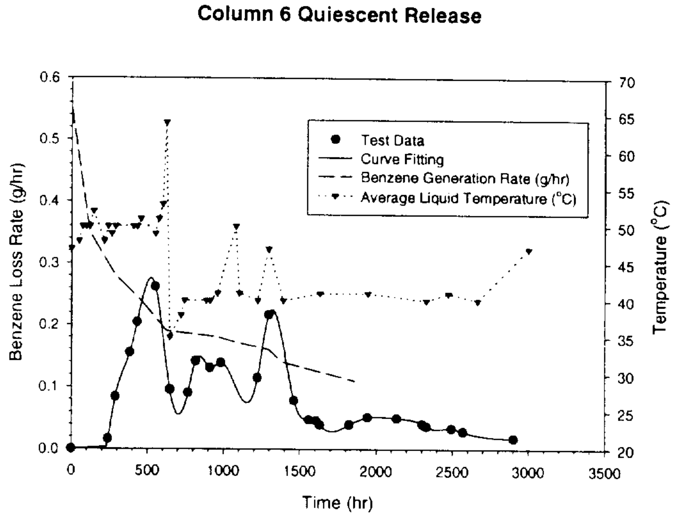


Figure 11. Column 6 Quiescent Release

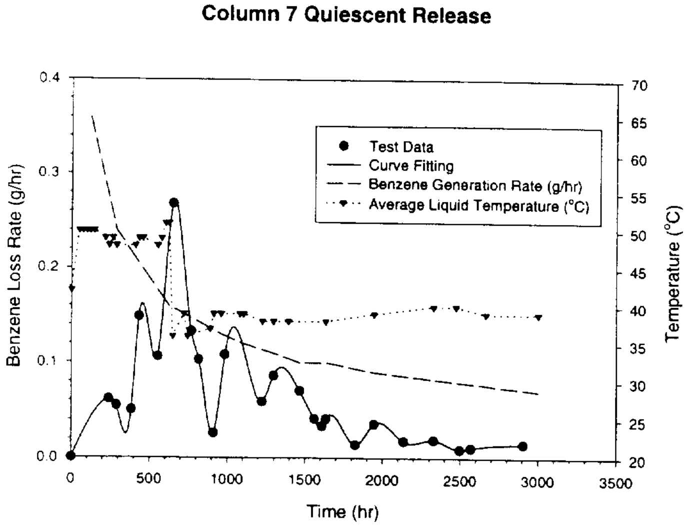


Figure 12. Column 7 Quiescent Release

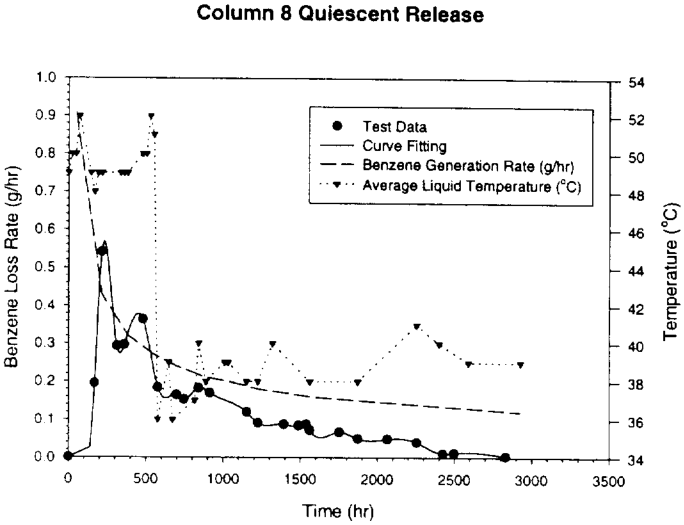


Figure 13. Column 8 Quiescent Release

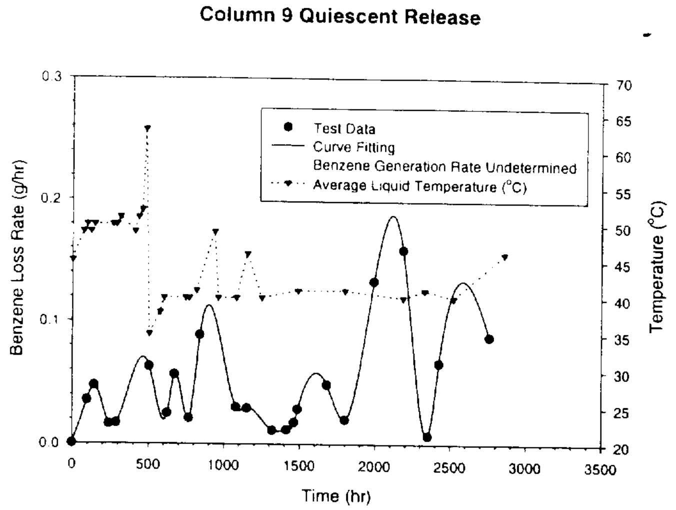


Figure 14. Column 9 Quiescent Release

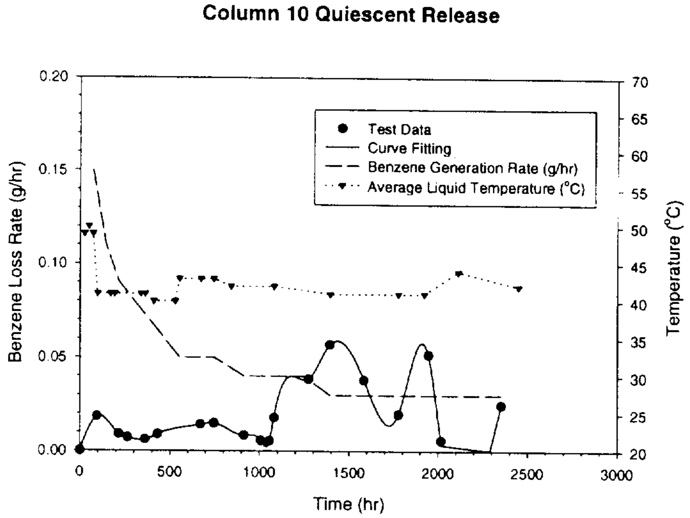


Figure 15. Column 10 Quiescent Release

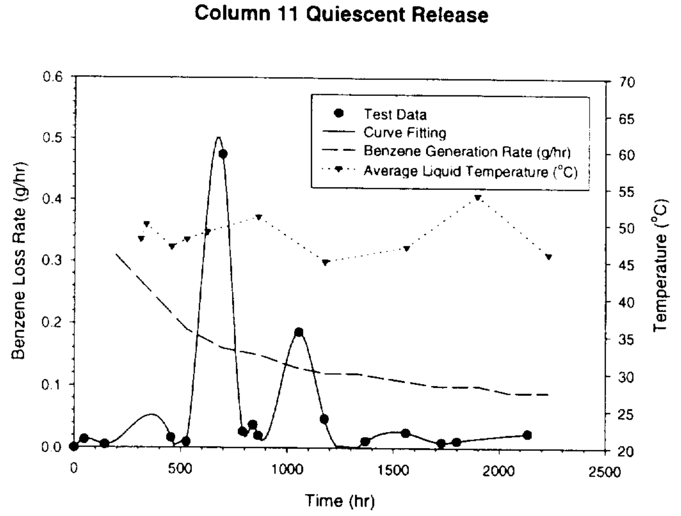


Figure 16. Column 11 Quiescent Release

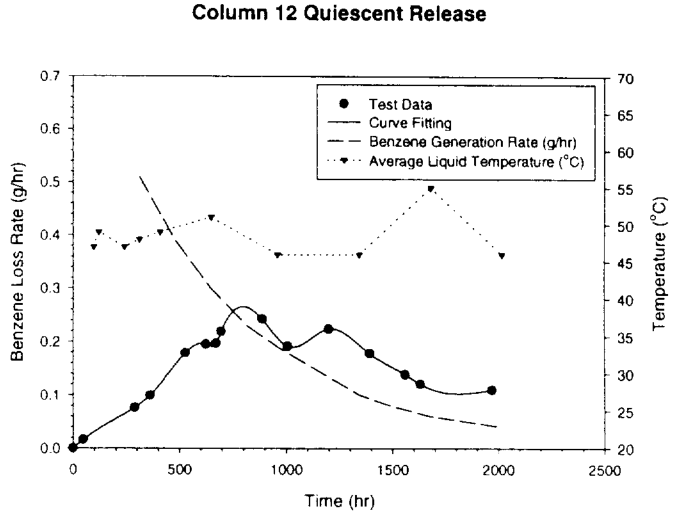


Figure 17. Column 12 Quiescent Release

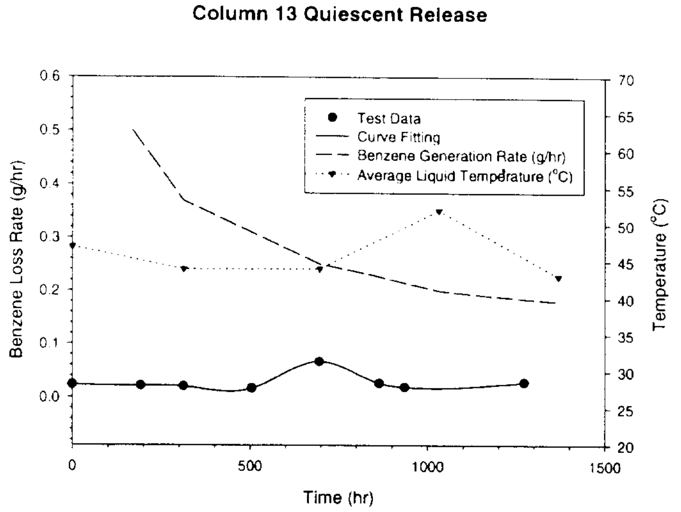


Figure 18. Column 13 Quiescent Release

The calculation of generation rate assumes four moles of benzene immediately form from each mole of boron. This simplifying assumption causes the generation rate to be overpredicted early in the test period and underpredicted later in the period. Stated differently, the generation rate lines on the quiescent plots in reality would have less curvature than shown. Most of the quiescent plots show an increasing release during the initial portions of the tests consistent with accumulating benzene in the slurry. Slurry benzene analyses (Table 6, Appendix B, Page B-1) also show accumulating benzene during initial parts of each test. As benzene was accumulating, the generation rate simultaneously dropped as the available TPB was decomposed. Quiescent rates briefly exceeded the instantaneous generation rate. However, as the accumulated benzene is released and the generation rate continues to fall, the quiescent releases fall below the instantaneous generation rate. The quiescent releases did not exceed the peak generation rate, however, with the exception of a single measurement for Column 11. Although there is no clear explanation for this single point for Column 11, one point does not provide enough data to conclude that the release rate exceeded the generation rate for any significant time period.

Overall, the quiescent release conclusions are:

- Purge rate variations had an effect on measured quiescent releases in these tests. Both the vapor phase and liquid phase boundary layers were reduced resulting in increased mass transfer.
- After correction for the purge rate effect, peak quiescent release rates approached but did not exceed the calculated peak generation rate.

- As expected, lower temperatures produced lower quiescent losses as evidenced by Column 10. At lower temperatures the molecular diffusivity and solution viscosity decrease which in turn increases the thickness of the liquid phase boundary layer decreasing mass transfer.

- The sunk slurry Column 12 had the highest average quiescent losses supporting the observation that floating slurry acts as a barrier to quiescent benzene releases.

Benzene and Water Baseline Test

On 4/3/97 a baseline test was performed by adding benzene to the surface of a full column of water. Sufficient benzene was added (50 mL) to form a free-floating organic layer on top of the water. The resulting quiescent release is shown in Figure 19. As expected the vapor concentration rose and then remained steady as the benzene evaporated. No system perturbation could be identified that would have caused the brief peak at the end of the test. Possible causes include a sudden breakup of the benzene layer into droplets that would come with a release of interfacial surface tension and increased mass transfer of droplets versus the continuous layer. Note that the peak releases observed in several column agitation tests were in the same approximate range as those in this baseline test. This is consistent with the presence of a dispersed benzene phase coming to the surface and releasing to the vapor space during slurry recirculation.

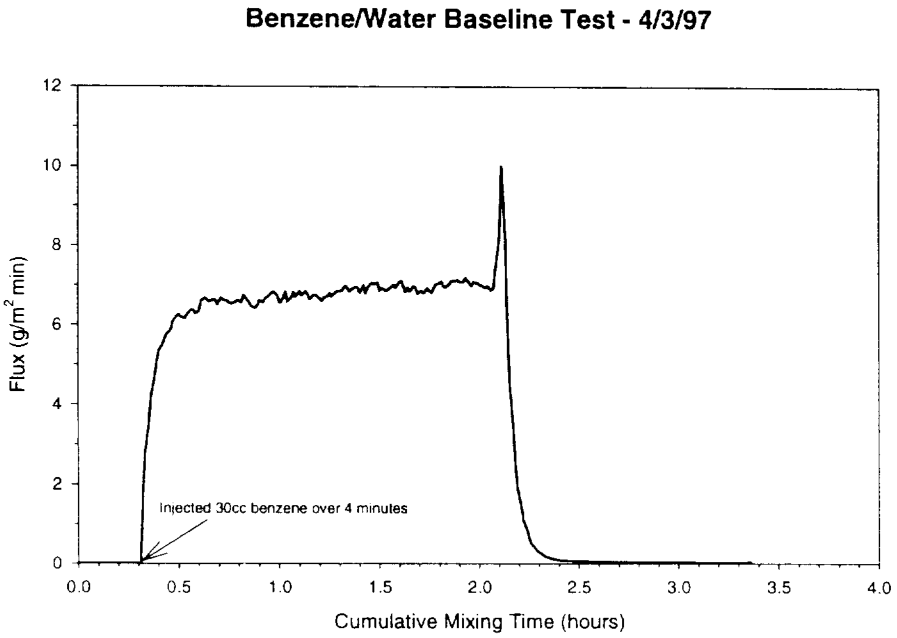


Figure 19. Benzene and Water Baseline Quiescent Release

Agitated Releases

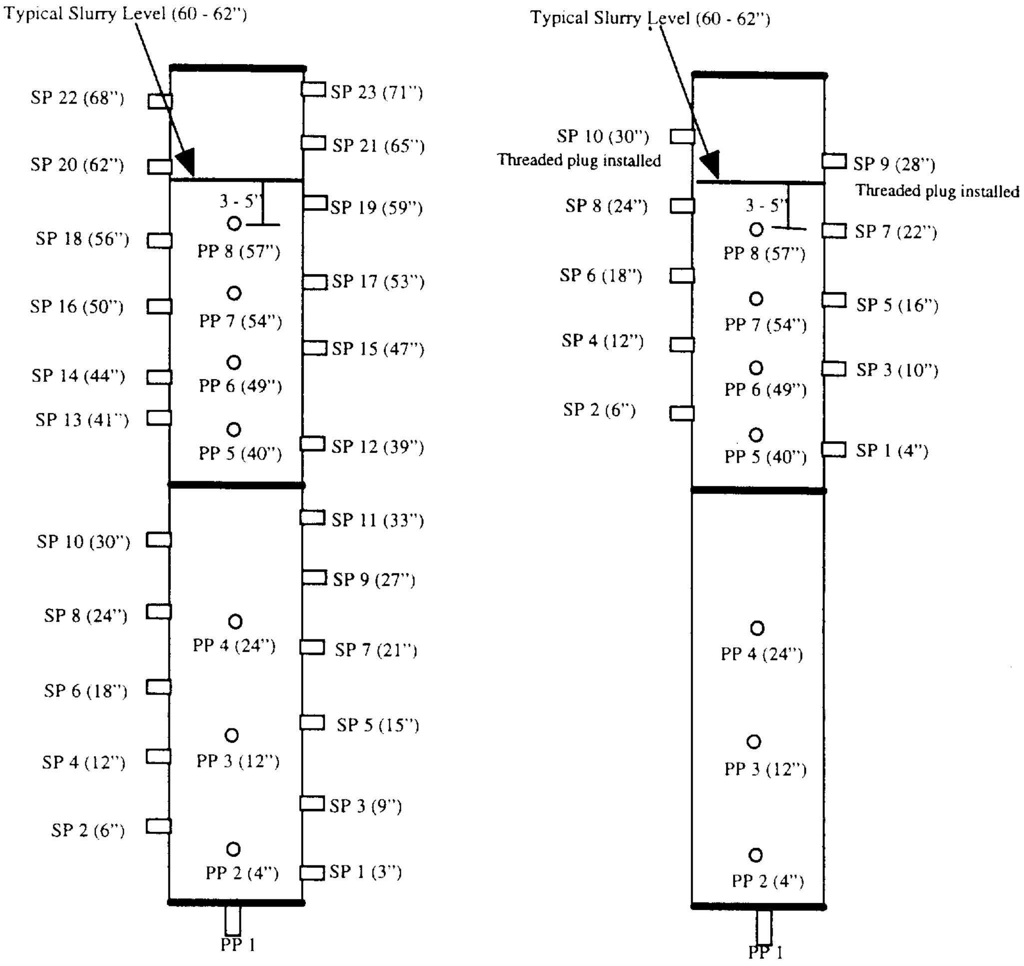
In addition to collecting quiescent release data, each column was agitated to gain insight on the benzene release behavior with energy input to the TPB slurry. Agitation, or more appropriately energy input, took three forms. First, the Series 1 columns (1-4) contained impeller blades to provide mixing. Second, Series 1, Series 2 (columns 6-9), and Series 3 (10-13) all shared a single tubing pump to provide recirculation flow through the columns. Third, water additions were made to Series 2 and Series 3 columns (these are discussed in a subsequent section). More detailed description of the column and supporting equipment is given in Appendix A.

As described in the introduction, the Series 1 column tests were curtailed due to problems with the stopcock sample port design. Agitation tests for these columns were abbreviated to promptly empty the columns. The impeller blades proved to be an ineffective method for mixing the slurry at the slurry/liquid interface. Since the blades were evenly spaced along the column length, several of the blades were beneath the bulk of the slurry which had segregated to the upper portion of the column. Therefore, recirculation became the standard mixing method for the columns. Impeller blades were removed from the column design for Series 2 and Series 3.

Eight pump ports were spaced along the column length as shown in Figure 20. Tubing connected the pump ports with the main trunk connecting PP1 to PP8 with branches to each of the other ports. The basic flow path was suction from one of the bottom two pump ports (PP1 or PP2) with discharge from one of the uppermost pump ports (PP7 or PP8). Intermediate pump ports were briefly used on a case-by-case basis to dislodge accumulated solids or to observe the effect on surface motion of a "lower" discharge. The recirculation pump was capable of adjustable rates (0-8 liters/min) and of reversing flow direction (i.e., PP1 to PP8 or PP8 to PP1) using a toggle switch.

The test sequence consisted of initiating a low recirculation flow (~ 2-4 liters/min) and then ramping the flow up until the surface motion was deemed sufficient. The desired degree of surface motion was a "rolling" surface involving as much of the surface cross-section as possible without spraying or splattering slurry into the vapor space. A judgment of "sufficient" motion was based on the visual observation by the authors. Both flowrate and discharge location were often varied frequently in the early portions of a test to involve as much of the TPB slurry in the recirculation as possible. The best surface motion characteristics were obtained when the slurry/vapor interface was 2-4 inches above the discharge pump port. Once the desired recirculation was established, vapor space data was collected approximately once per minute. Slurry samples were obtained periodically during an agitated release test to analyze for slurry composition changes.

The agitated release tests are presented by column and in the chronological order in which the tests were performed.



Series 1 Column Arrangement

Series 2 and 3 Column Arrangement

Figure 20. Column Sample and Pump Port Arrangement

Columns 2-4

The release test on Column 2 (Figure 21) showed that the slurry depth above the pump discharge port was a critical factor in the amount of surface motion achieved during recirculation. The degree of surface motion, in turn, was a critical factor in the benzene releases during mixing. The release test on Column 4 (Figure 22) was performed using a gentler agitation that moved only a portion of the slurry surface. The benzene release fluxes were low, on the order of 0.6 g/m^2 min.

The release of benzene from the slurry in Column 3 (Figure 23) was performed attempting to replicate the release from Column 4. However, during the pump operation the fluid level decreased to only 1 inch above the discharge port and the release rate rose considerably above 0.6 g/m^2 min. The release data on the Series 1 columns suggested that the degree of surface mixing and motion was an important factor in controlling the rate of benzene evolution. In the release tests for both column 2 and column 3, some spraying into the vapor space occurred because the pump discharge was even with the slurry/vapor interface and is likely the reason for the fluxes briefly exceeding that obtained from an evaporating benzene layer in the benzene and water baseline test.

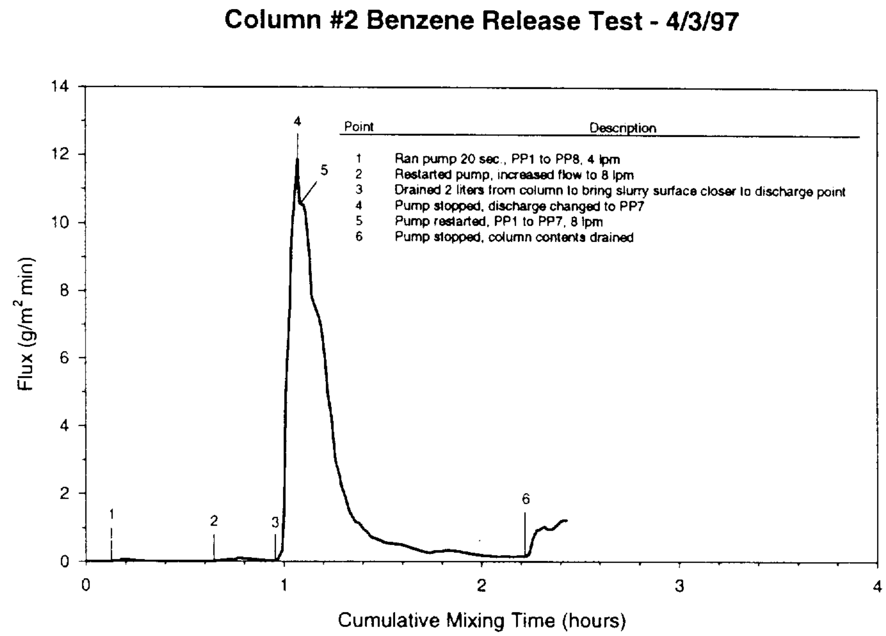


Figure 21. Column 2 Agitated Release

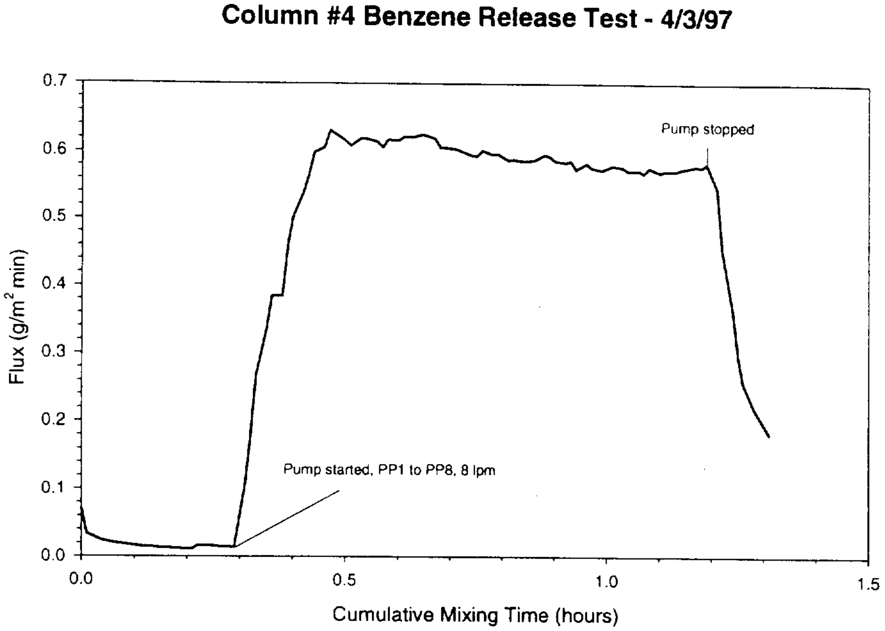
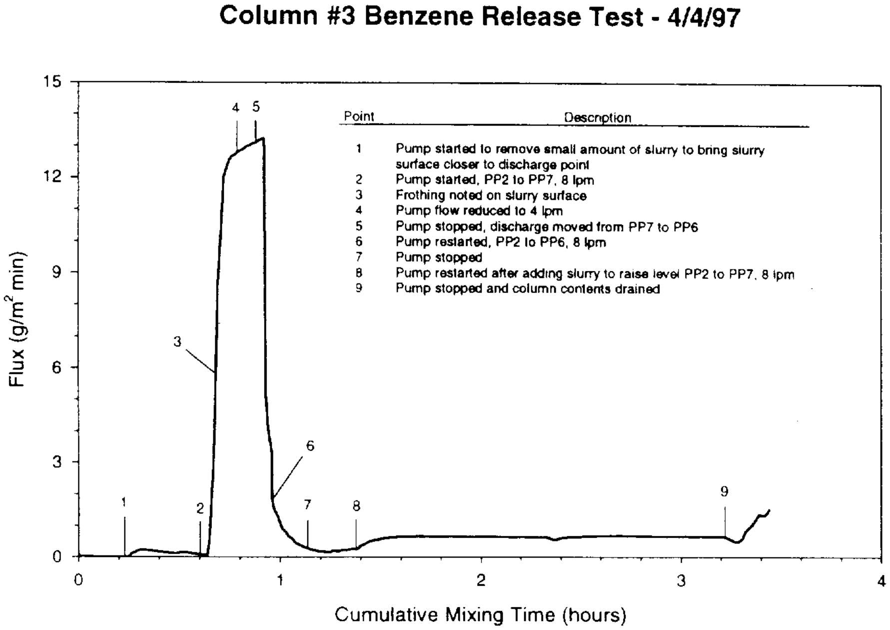


Figure 22. Column 4 Agitated Release



Point	Description
1	Pump started to remove small amount of slurry to bring slurry surface closer to discharge point
2	Pump started, PP2 to PP7, 8 lpm
3	Frothing noted on slurry surface
4	Pump flow reduced to 4 lpm
5	Pump stopped, discharge moved from PP7 to PP6
6	Pump restarted, PP2 to PP6, 8 lpm
7	Pump stopped
8	Pump restarted after adding slurry to raise level PP2 to PP7, 8 lpm
9	Pump stopped and column contents drained

Figure 23. Column 3 Agitated Releases

Column 9

The agitated release tests for Column 9 were designed to provide a structured sequence of steps to better assess the effect of mixing and surface motion on the benzene release rate. The fluid level in Column 9 was 7 to 8 inches above the centerpoint of PP8. During the first day of agitation, both the discharge location and recirculation flowrate were adjusted to obtain the desired surface motion (see points 1-4). No surface motion was observed at the slurry/vapor interface, and the benzene release was negligible.

On the second day, a length of tubing was installed to allow pumping from PP02 through SP09 and SP10. SP09 was located about 2 inches above the slurry/vapor interface. Discharge through both SP09 and SP10 (see point 9) increased the flux to 6.2 g/m^2 min. The benzene globules rose against the recirculating flow and accumulated at the vapor/slurry interface where the surface appeared frothy. Discharge through PP08, SP09, and SP10 at 8 L/min (see point 11) resulted in a peak flux of 13.4 g/m^2 min. Since SP09 and SP10 were above the slurry/vapor interface, some spraying of slurry into the vapor space occurred during recirculation. This resulted in higher flux rates than were obtained in the benzene and water baseline test. During the mixing of Column 9, once surface motion was established, the benzene release rate increased. The floating slurry impeded the benzene release until the floating layer was incorporated in the bulk flowing fluid. The shape of the release curve indicated readily releasable benzene followed by a less-readily releasable form of benzene. The benzene release rate decreased rapidly when the mixing was stopped.

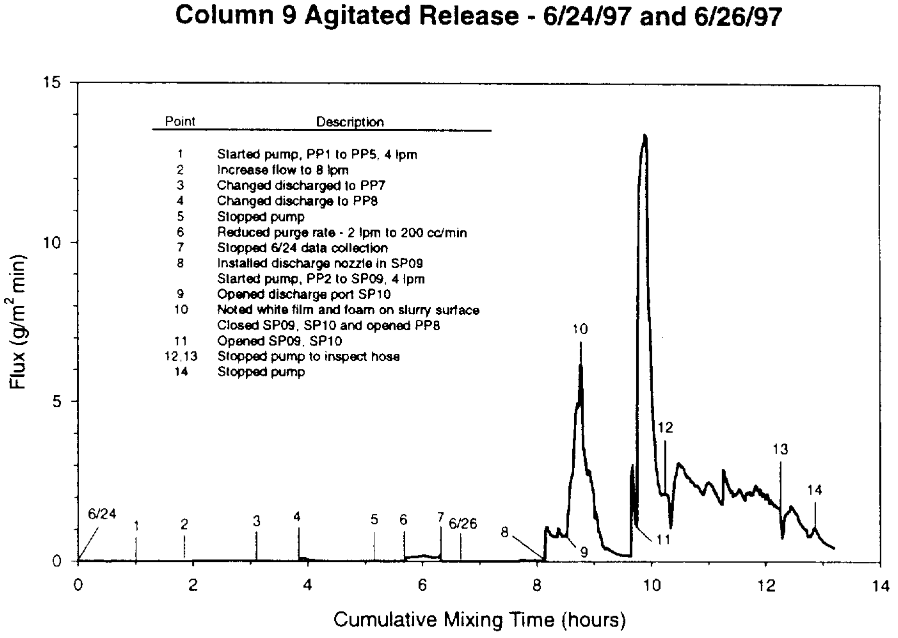


Figure 24. Column 9 Agitated Release

Column 7

The slurry level in Column 7 prior to the release test was about 5 inches above the center of PP08. About 2 liters of slurry were drained out to attempt to adjust the level to 2 inches above the pump port. The benzene release from the 4 wt % KTPB slurry in Column 7 (Figure 25) was accompanied by openings or channels created in the slurry through which white globules were observed to rise. As the release continued, a foam or froth was noted on the inner circumference of the glass column and a large release of benzene (peak at 4 g/m^2 min) was noted (see point 2). After recirculation was run for thirteen minutes the 2 liters of slurry were re-introduced into the column and the pump was re-started at 4 L/min. The release flux was less than 1 g/m^2 min (see points 3 and 4).Various combinations of recirculation schemes and flowrates were tried (see points 5-15) with marginal success. As with column 9, the column 7 tests showed both the importance of mixing and the difficulty in achieving that mixing in 4 wt % TPB slurries. Only when the surface was perturbed did the vapor space benzene increase.

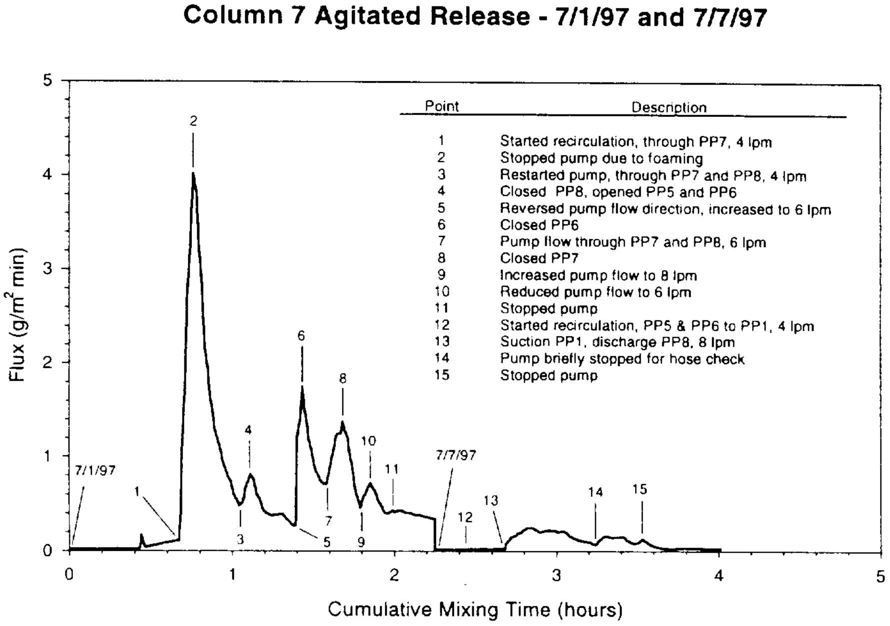
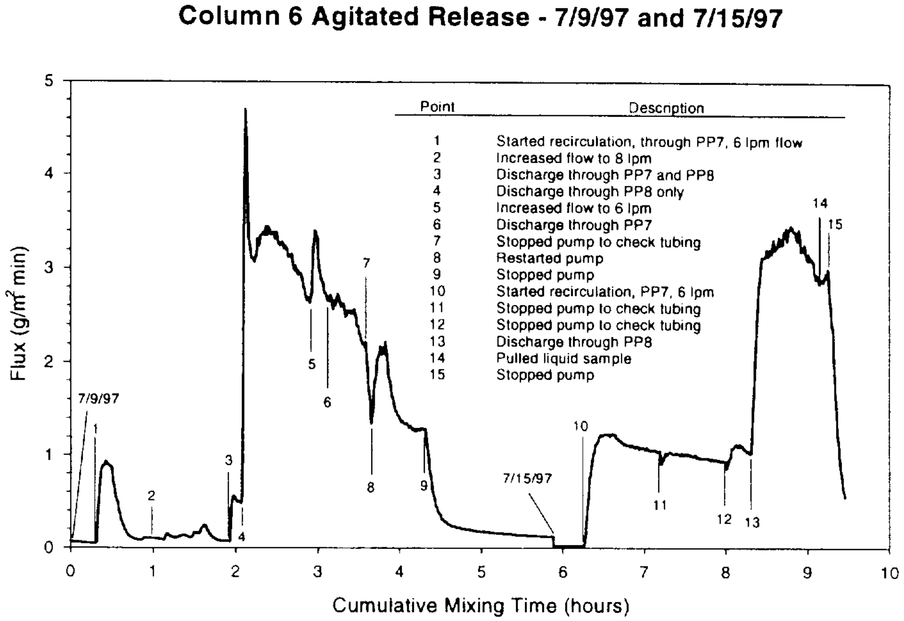


Figure 25. Column 7 Agitated Release

Column 6

Column 6 was the first 1 wt % KTPB slurry to be agitated for benzene release. The slurry recirculation chronology and benzene release flux are shown in Figure 26. Pumping through PP7 at 4 or 6 L/min produced a small increase in the benzene release flux to about 1 g/m^2 min (see point 1). The slurry surface was not flowing under these conditions, although an accumulation white globules were noted near the slurry surface during this portion of the test. Once the discharge port was changed to PP8 and surface motion was established, a large spike in the benzene release occurred to over 4.5 g/m^2 min (see point 4). A frothy surface was observed, and the benzene release fell thereafter to about 3 g/m^2 min. It was possible to affect the release profile by alternating the flow through PP7 and PP8. Although the release decreased when the flow discharge was switched from PP8 to PP7, the release rate was substantially greater than during the first two hours of the test. This may be indicative of the need to break apart floating slurry masses to achieve benzene release. Also, once the pump was stopped the benzene release decreased rapidly.

During a second day of operation, the release rate was fairly steady at about 1 g/m^2 min when the slurry was circulated from PP1 through PP7 at 6 L/min flowrate (see points 10-12). When the discharge port was changed to PP8, the benzene release flux immediately rose to about 3.4 g/m^2 min (see point 13). Finally, the pump was turned off just after the 9 hour point of cumulative mixing. The benzene release rate again dropped sharply to less than 0.5 g/m^2 min.



Point	Description
1	Started recirculation, through PP7, 6 lpm flow
2	Increased flow to 8 lpm
3	Discharge through PP7 and PP8
4	Discharge through PP8 only
5	Increased flow to 6 lpm
6	Discharge through PP7
7	Stopped pump to check tubing
8	Restarted pump
9	Stopped pump
10	Started recirculation, PP7, 6 lpm
11	Stopped pump to check tubing
12	Stopped pump to check tubing
13	Discharge through PP8
14	Pulled liquid sample
15	Stopped pump

Figure 26. Column 6 Agitated Release

Column 8

Column 8 contained a 1 wt % KTPB slurry that was agitated for over 35 hours to deplete the benzene in the slurry to less than 50 mg/L. Following a water addition test, the slurry was recirculated during 7 days of operation (Figure 27). After initial recirculation efforts produced minimal surface motion, the pump was stopped and about 1.2 liters of supernate were drained out before re-starting the pump (see point 2). The surface motion improved and the benzene release flux reached 4.2 g/m^2 min. The balance of the release data on Column 8 was taken with the pump set at 6 L/min with circulation from PP1 to PP8.

Benzene release fluxes obtained in the second day of operation exceeded those of the first day. The pumping performed on the first day undoubtedly helped to break up the floating slurry from a large mass into more dispersible slurry. Each time the pump was stopped, the benzene release rate plummeted. However, in many of the pump re-starts the benzene release rate observed immediately after re-starting the pump exceeded the value just prior to stopping the pump. The slurry segregated rapidly after the pump was stopped, with the insoluble solids rising to the top of the aqueous phase. When the pump was restarted, the discharge flow initiated motion within a slurry layer that had a higher concentration of insoluble solids and organic globules compared to the state just prior to stopping the pump, when the slurry was more homogeneously dispersed throughout the column volume.

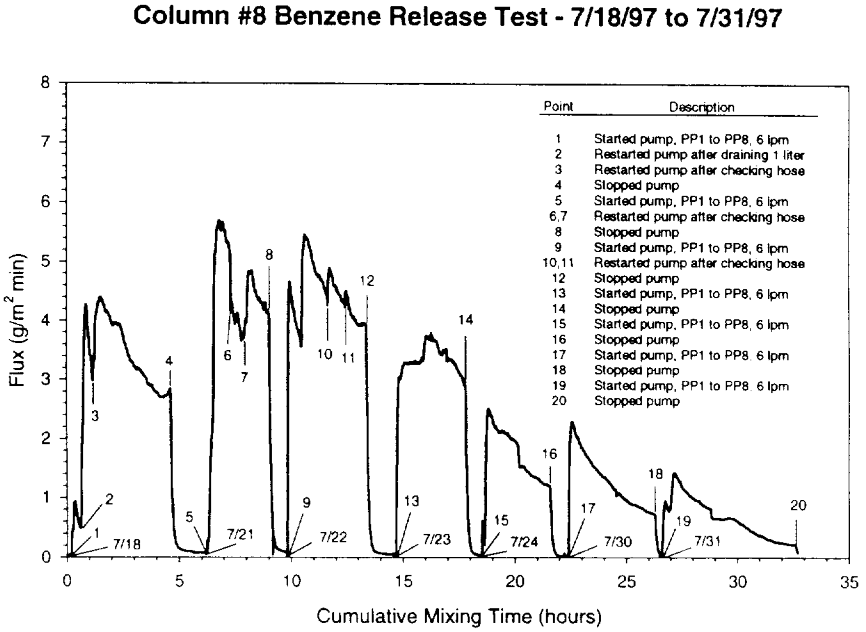


Figure 27. Column 8 Agitated Release

Column 10

Column 10 contained 4 wt % slurry in a 3.7M sodium aqueous solution. The sodium concentration was reduced from the 4.7M (slurry basis) value in previous tests in an attempt to obtain more neutrally buoyant slurry. The floating slurries in Columns 1-9 were atypical of Tank 48H experience to date (based on tank video inspections and operator field observations) although the slurry is expected to float during particular steps in the concentration process. As discussed in the Benzene Form and Location section, there was sufficient benzene in Column 10 to create buoyancy to float the KTPB solids.

The release test on Column 10 is shown in Figure 28. The test began with recirculation discharge approximately 1 inch below the slurry/vapor interface. Additional solution was drained and pump flow increased to create more motion at the slurry surface. The flux increased to approximately 8 g/m^2 min (see point 2). As with columns 2,3, and 9, some foaming and splattering into the vapor space occurred causing the flux to briefly exceed the benzene in water baseline test flux. Each time the pump was stopped, benzene concentrations in the vapor phase dropped sharply. Subsequent tests showed similar trends of release peaks followed by steady declines. It is reasonable to expect that initial benzene releases would be higher as on each day the slurry was accumulated along with its benzene inventory at the top of the column. As slurry was drawn downward throughout the length of the column, vapor benzene concentrations dropped. Also, vapor concentrations were very low until good surface flow and mixing were obtained at the slurry/vapor interface.

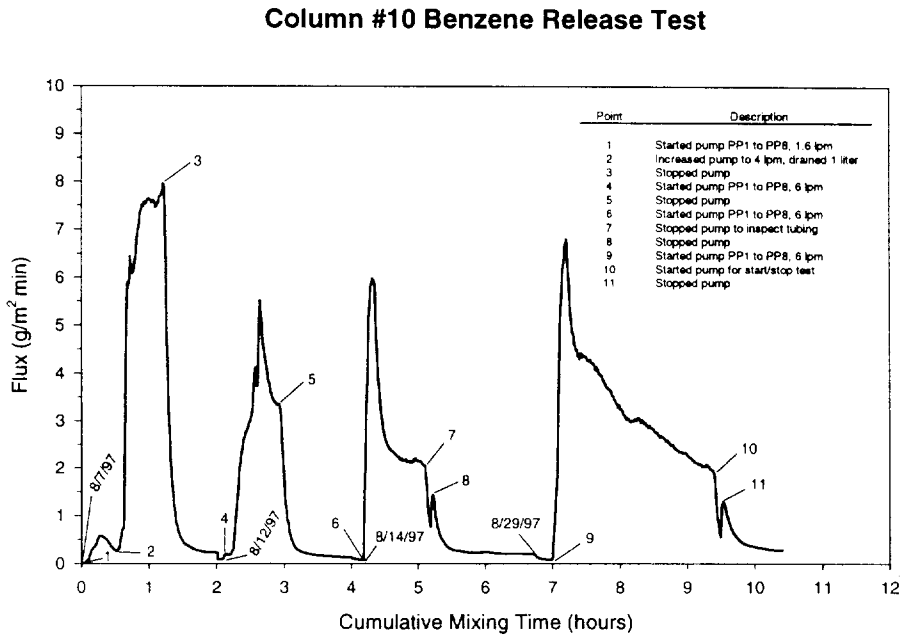
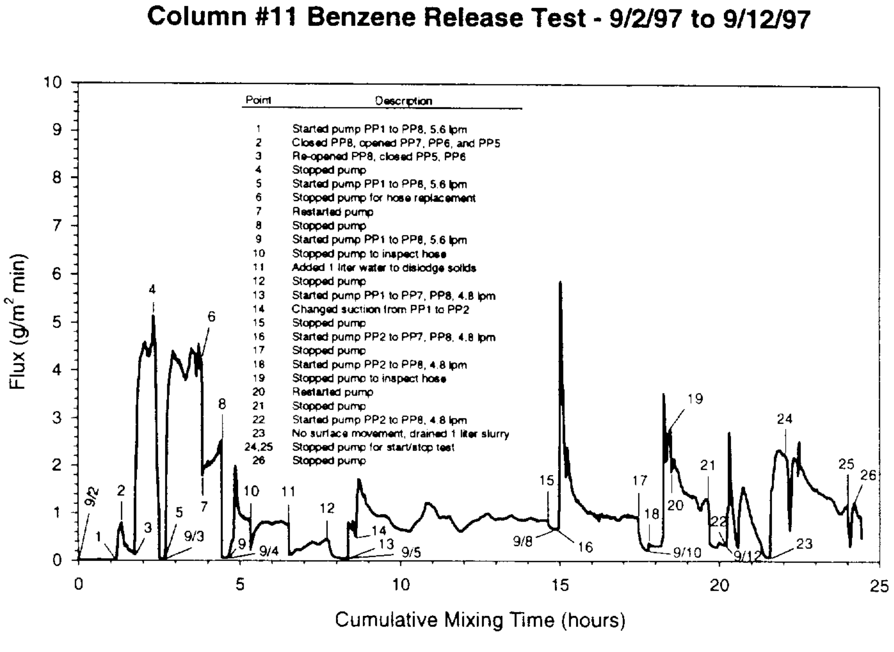


Figure 28. Column 10 Agitated Release

Column 11

Column 11 contained 4 wt % slurry in a 3.5M sodium aqueous solution. The sodium concentration was reduced further from the 3.7M (slurry basis) value in Column 10 in another attempt to obtain more neutrally buoyant slurry. Once again the slurry floated. Figure 29 shows the release test results for Column 11.

Involving a high percentage of the TPB solids in the recirculation flow was more difficult in this test. The Figure 29 annotations show the frequency of pump port and pump speed changes all performed in an attempt to involve more solids and to get adequate surface motion at the slurry vapor interface. The general trend of initial peaks followed by a decreasing release rate was observed again. The sharp drop in vapor concentration when recirculation was stopped was observed again. As expected, the 4 wt % slurry released less of its benzene inventory in approximately 25 hours of mixing than did the 1 wt % slurry in Column 8 over the same time period. Two "stop/start" tests were performed on Column 11 in an attempt to verify the effect noted in Column 8 that in many of the pump re-starts the benzene release rate observed immediately after re-starting the pump exceeded the value just prior to stopping the pump. No clear "stop/start" effect was observed in Column 11.



Point	Description
1	Started pump PP1 to PP8, 5.6 lpm
2	Closed PP8, opened PP7, PP6, and PP5
3	Re-opened PP8, closed PP5, PP6
4	Stopped pump
5	Started pump PP1 to PP8, 5.6 lpm
6	Stopped pump for hose replacement
7	Restarted pump
8	Stopped pump
9	Started pump PP1 to PP8, 5.6 lpm
10	Stopped pump to inspect hose
11	Added 1 liter water to dislodge solids
12	Stopped pump
13	Started pump PP1 to PP7, PP8, 4.8 lpm
14	Changed suction from PP1 to PP2
15	Stopped pump
16	Started pump PP2 to PP7, PP8, 4.8 lpm
17	Stopped pump
18	Started pump PP2 to PP8, 4.8 lpm
19	Stopped pump to inspect hose
20	Restarted pump
21	Stopped pump
22	Started pump PP2 to PP8, 4.8 lpm
23	No surface movement, drained 1 liter slurry
24,25	Stopped pump for start/stop test
26	Stopped pump

Figure 29. Column 11 Agitated Release

Column 12

Column 12 was agitated to disperse the sunken insoluble solids sufficiently to release any organic globules trapped within the slurry. Unlike agitation tests for the other columns, the Column 12 agitation was intentionally gentle and did not bring slurry to the liquid/vapor interface. The lower two thirds of the column were mixed leaving the upper one third as a disengagement section. It was anticipated that freed globules would rise to the surface of the clear liquid standing above the sunken slurry. Various combinations of suction and discharge ports were tested to achieve the dispersion of the solids with minimal success. Better flow of the solids was eventually achieved by squeezing the portion of tubing between PP1 and the pump. During this operation, white colored globules were observed to rise from within the slurry near the bottom of the column up to the surface. The globules accumulated at the liquid/vapor interface and agglomerated to form a thin layer or film. The rise of the globules to the surface was accompanied by a slight increase in the benzene release rate, from about 0.1 g/m^2 min to a peak of 0.3 g/m^2 min. However, the globules did not produce a release of benzene in excess of 1.0 g/m^2 min without external energy input.

Draining solution from the columns did result in an increase in the rate of benzene release, presumably because additional surface area wetted with slurry was exposed to the vapor phase as the liquid level decreased. However, the increases were significantly less than those observed when mixing created surface motion.

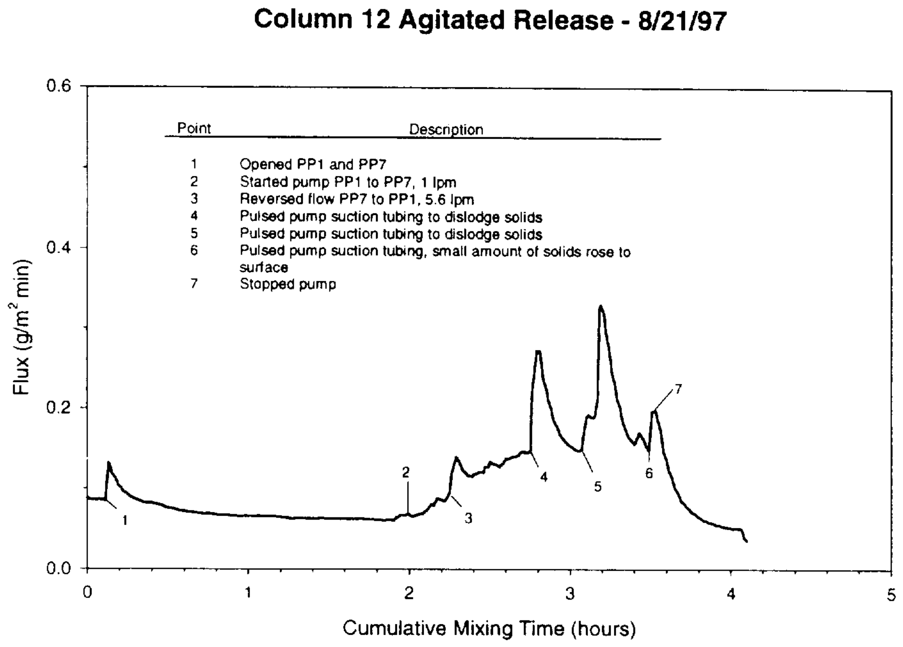

Column 12 Agitated Release - 8/21/97

Figure 30. Column 12 Agitated Release

The overall conclusions from the Agitated Releases are:

- The extent of vapor concentration during agitated releases is directly related to the degree of mixing and particularly the degree of surface motion at the slurry/vapor interface. This is consistent with an increase in mass transfer when the liquid phase boundary layer is reduced.
- During mixing, floating slurry impedes benzene release (by reducing the effective mass transfer surface area) until the floating layer is incorporated into the mixing stream.
- Release decreases rapidly when mixing is stopped. This is consistent with the expectation that when mixing stops the liquid phase boundary layer thickness increases and the flux and vapor concentrations decrease.

Water Additions

Two water spray lines were provided. The first was a ¼" line fitted with a nozzle which produced a conical spray pattern of approximately 8 inches in diameter at a flowrate of 22 mL/second. The second was a simple ¼" line with no spray nozzle which produced a straight stream at a flowrate of 44 mL/second.

Column 9

Addition of 250 mL of water over 125 seconds (0.12 L/min) to the 4 wt% KTPB slurry in Column 9, using the conical spray pattern nozzle, produced only a minor increase in the release of benzene from an otherwise quiescent slurry (Figure 31). The water was noted to impact on the surface of the slurry and collect in a pool. In fact, the benzene release rate several minutes after the water addition was less than the release rate before the water addition.

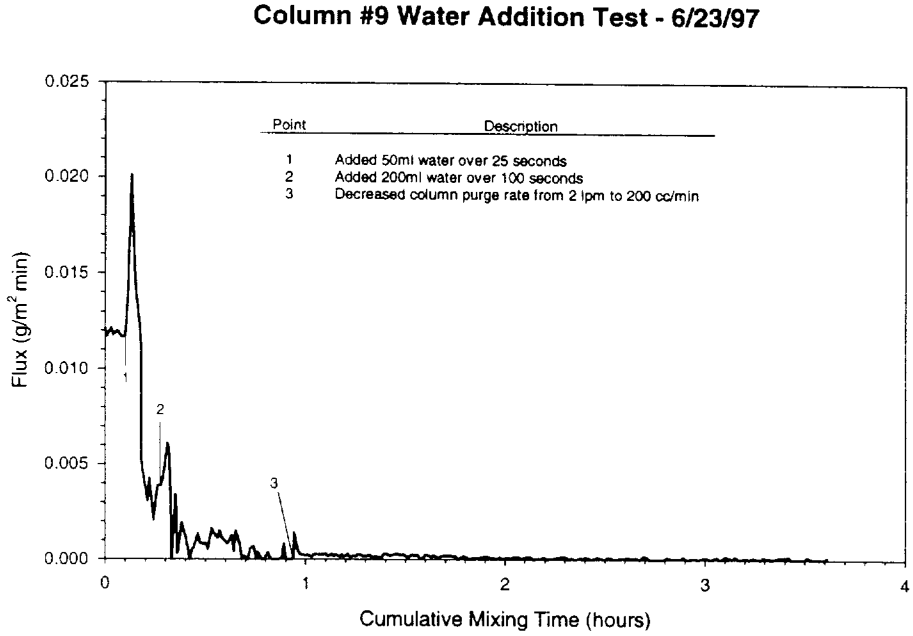


Figure 31. Column 9 Water Addition

Column 8

Two water additions were performed on the 1 wt % KTPB slurry in Column 8 using the ¼ inch conical spray pattern nozzle. The response, as shown in Figure 32, was a slight increase in benzene release, followed by a decrease in the benzene release, as compared to the baseline benzene release rate prior to the test. The water spray nozzle was replaced with the straight ¼ inch tube. A water addition of 0.9 liters over 20 seconds (2.7 L/min) produced a significant release of benzene, with a peak flux of over 7 g/m^2 min. The water jet addition was repeated about 90 minutes later, and again a significant release, although less than the first release, was observed. Visually, it was noted that the water stream from the jet penetrated through the entire floating slurry phase depth and into the salt solution beneath the slurry to a depth of about 2 - 3 inches. The water stream bored a hole through a section of the slurry surface, but did not affect the entire cross-section of the slurry surface. This provides further evidence that floating slurry acts as a physical barrier to benzene release.

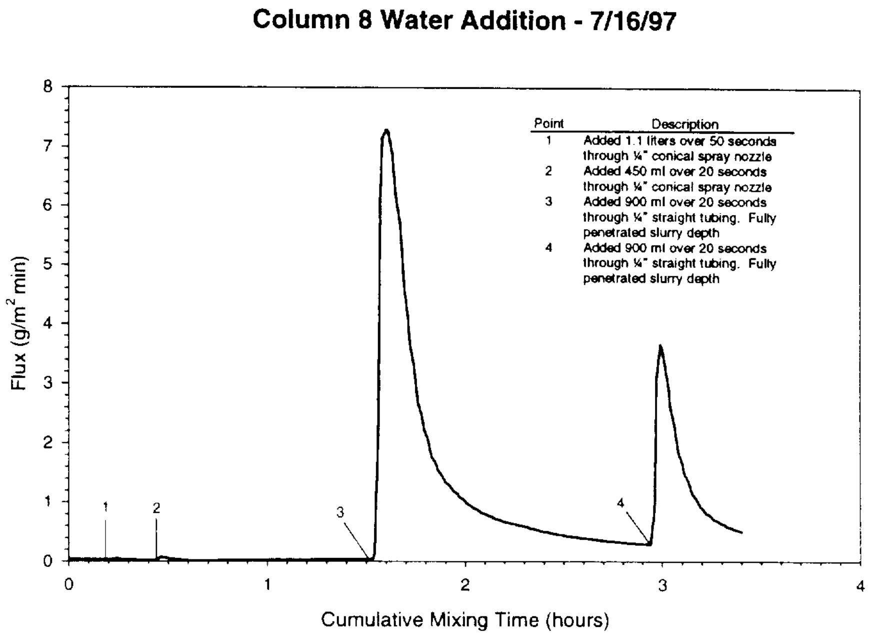


Figure 32. Column 8 Water Addition

Column 6

Water addition was also performed on the Column 6 1 wt % KTPB slurry 24 days after the agitated release tests completed. The behavior of the water jet was similar to that observed in Column 8, the jet penetrated the floating slurry layer and terminated 2-3 inches into the aqueous phase. An increase in the benzene release was noted in the Column 6 test (Figure 33), even though the slurry had previously undergone agitated release tests. This water addition, performed after agitated release tests on Column 6, produced a peak flux of about 1.2 g/m^{2}min which is about 1/3 of the peak fluxes seen during the agitated release test.

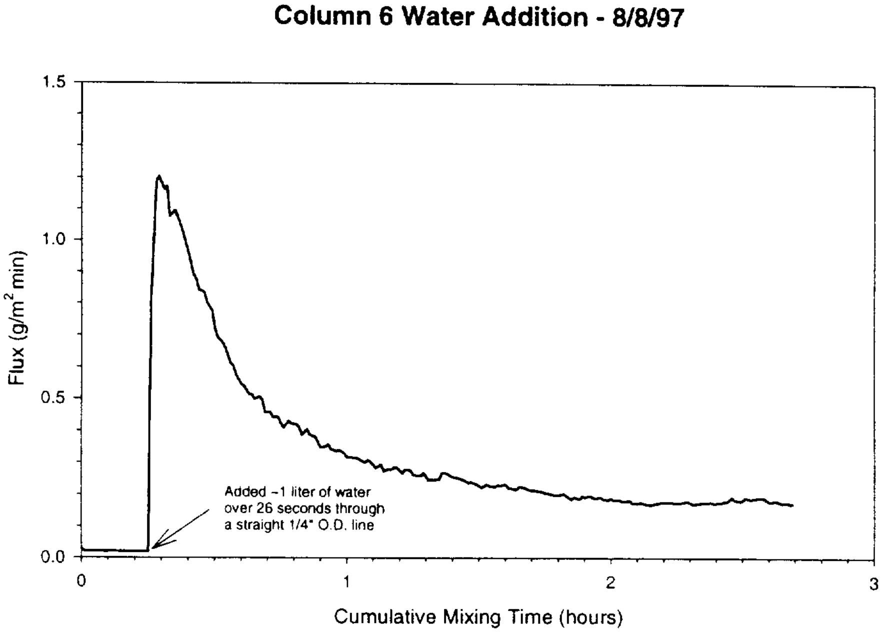


Figure 33. Column 6 Water Addition

Column 12

The Column 12 submerged slurry (4 wt % slurry in 0.2 M sodium salt solution) position of Column 12 was considered more comparable to current tank conditions. The low sodium concentration is not currently the case for Tank 48H, but is expected to occur during the precipitate washing step of the process.

Approximately the bottom two thirds of the column length were recirculated. Based on previous column tests, benzene globules were expected to accumulate at the liquid/vapor interface and remain stable there. The presence of a white, opaque film at the interface was also expected. As the solids were recirculated, the expected film and globule accumulation occurred. This surface appearance was consistent with video inspections of Tank 48H and with operator comments regarding a film or scum observed on the tank liquid surface during initial ITP operations. Initial vapor concentrations of approximately 300 ppm rose to as high as approximately 1200 ppm during the recirculation period. This vapor concentration increase was not considered significant in comparison to previous column tests. The conical spray pattern nozzle was selected for the water addition test to provide the greatest surface area coverage. The spray nozzle was operated to transfer 450 mL of water over 20 seconds (1.35 L/min). This rate is 10 times the rate used for Column 9 and about one half the rate from the straight ¼" tube. The benzene release resulted in a peak benzene flux of about 1.7 g/min/m2 (Figure 35).

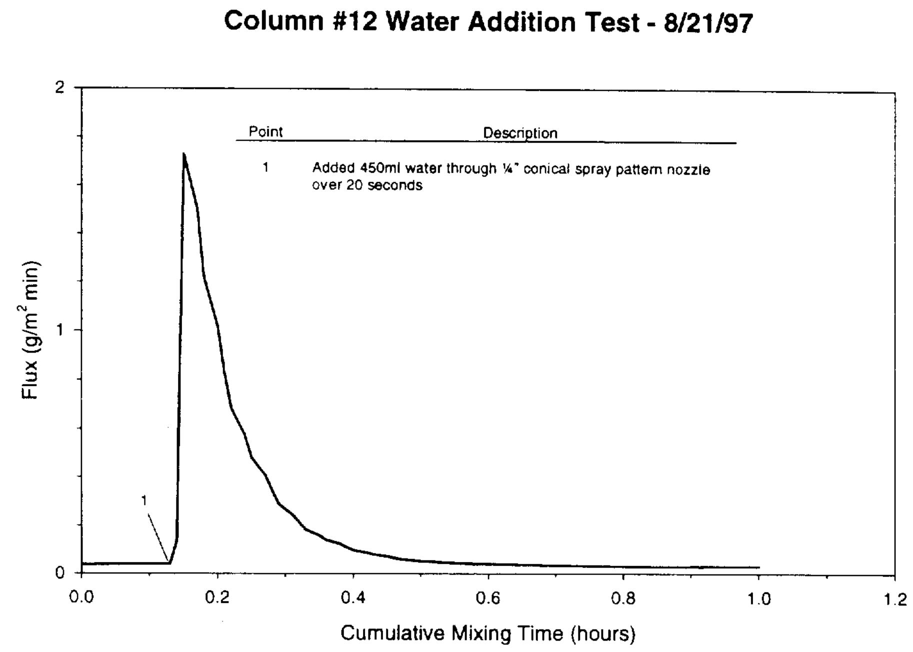


Figure 35. Column 12 Water Addition

Column 10

Column 10 was the first 4 wt % slurry where a water addition was performed (Figure 34). The initial two additions on 8/5/97 using the straight tube did not fully penetrate the deeper 4 wt % slurry layer and therefore did not produce the notable release as was observed in the 1 wt % slurries in Columns 6 and 8. However, a second day of water additions was performed on 8/11/97, 4 days after the initial recirculation test on Column 10. The water additions on 8/11/97 did produce a measurable effect suggesting that the recirculation prior to the water addition either distributed more benzene to the upper portions of the slurry layer or affected the extent of slurry "compaction" such that the water addition penetrated further into the slurry layer.

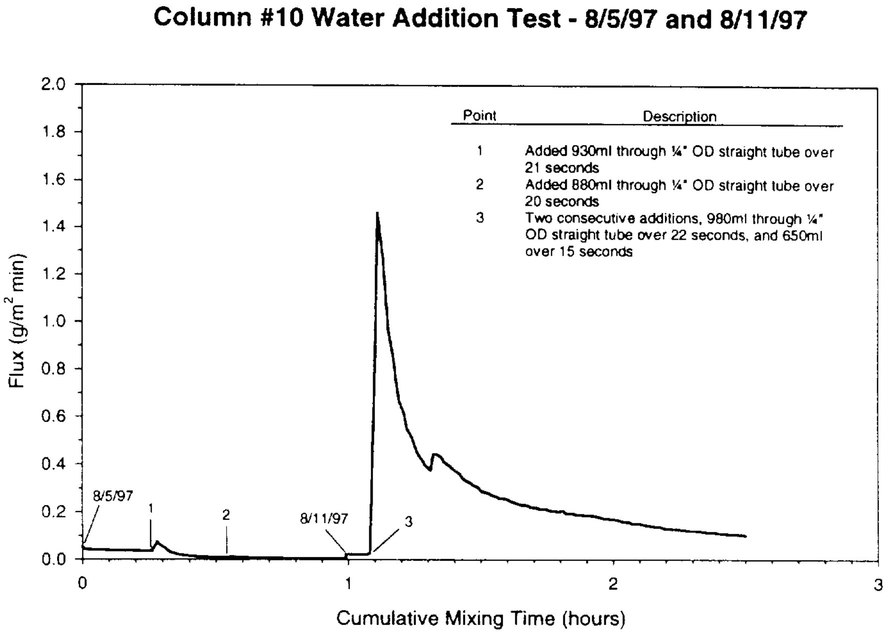


Figure 34. Column 10 Water Addition

The ability to recirculate slurry without significant increase in benzene vapor concentrations, to accumulate a readily releasable form of benzene (i.e., globules) at the liquid surface, and then release that benzene through a water addition represents a plausible scenario for the March 1996 events in Tank 48H. Globules could have formed in the Tank 48H slurry during the initial months of operation. The globules could then have been available for release during the March 1996 water addition and subsequent pump operation in a way similar to that observed in the column 12 test. This observation is very complementary to the primary test objectives and is one of the key findings of the program.

Column 13

Water was added sub-surface to Column 13 in an attempt to dilute the salt solution and cause the floating insoluble solids to sink. A total of 24 liters of water was added through PP2 on the first day of operation on 8/19/97. Initially, some increase in the benzene release rate was observed (see point 3 on Figure 36). During the addition, the entering water stream was visibly rising to the slurry/liquid interface presumably because of buoyant forces created by the difference in specific gravity. However, even though the water addition was sufficient to reduce the sodium concentration on a slurry basis from 4.7 M to 2.4 M, the majority of the solids did not sink. Only about 25% of the solids sank during the first day's operation. When the purge rate was reduced to conserve nitrogen during off-shift vapor space monitoring, a slight and unexplained increase in flux occurred (see point 4). This was the only such occurrence in the column tests and is not considered significant evidence to refute the overall conclusion (supported by every other column) that quiescent fluxes decreased with decreasing purge rate.

Additional water was added on 8/22/97. The additional water was sufficient to reduce the sodium concentration on a slurry basis from 2.4M to 1.7M. Again, only a small portion of the solids sank. During the water additions, white colored globules were observed to rise up to the slurry/vapor interface, but no significant increases in vapor concentration occurred. These test results were significant, because they showed a subsurface water addition did not result in a significant benzene release.

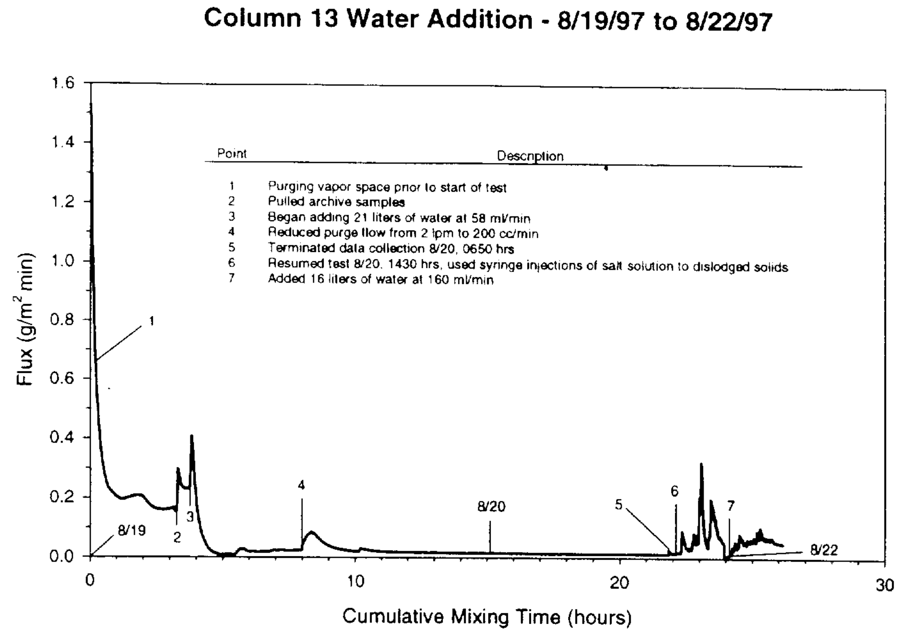


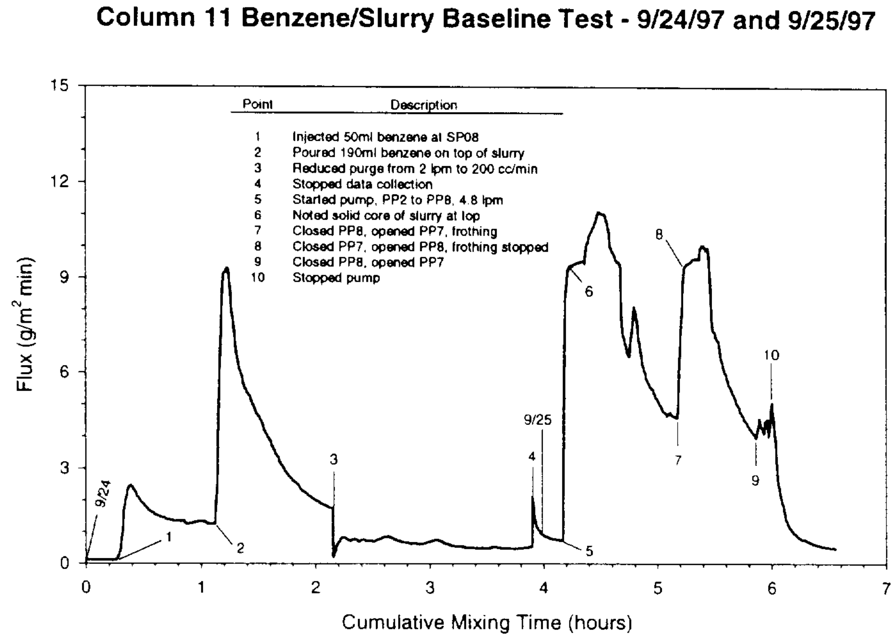
Figure 36. Column 13 Water Addition

The overall conclusions from water additions are:

- Low energy (0.1 L/min) water spray on floating slurry did not result in significant benzene releases.
- A water jet of sufficient energy (2.7 L/min) to fully penetrate the slurry depth produced a notable release in floating slurries.
- Moderate (1.3 L/min) water spray on floating benzene globules produced a significant release.
- Accumulation of globules at liquid surface following recirculation and subsequent release during water addition on Column 12 is a plausible scenario for the March 1996 experience in Tank 48H.
- Large subsurface water additions to floating slurry did not produce a significant benzene release.

Benzene and KTPB Slurry Baseline Tests

On 9/24/97, additional baseline tests were performed where benzene was added to 4 wt % slurry in Column 11. Initially 50 mL of benzene was injected into the upper 2 inches of the slurry depth. As shown in Figure 37, the flux showed a brief response but then quickly began to decay. An additional 190 mL of benzene was then added to the slurry surface in an attempt to form a free benzene layer as was present in the benzene and water test (see point 2). Again there was an initial response, followed by a slower decay. The slurry surface was porous with numerous cracks and pockets. The benzene filled these areas, effectively "soaking" into the slurry layer. This reduced the benzene surface area in contact with the vapor space and possibly caused the decay observed after the 190 mL benzene addition. The column purge rate was reduced to conserve nitrogen and the vapor concentration was monitored overnight. The next day, 9/25/97, a recirculation test was performed. The upper 6 inches of slurry layer had formed a firm mass consistent with observations by Dworjanyn when adding benzene at 50,000 ppm to TPB slurry.[6] The mass appeared mud-like in consistency. The pump discharge cut through the mass, but left the bulk of the mass in place. A large amount of globules had formed and were present throughout the upper portions of the slurry layer. Recirculation dislodged portions of the upper mass and brought the globules to the surface resulting in high vapor phase concentrations.



Column 11 Benzene/Slurry Baseline Test - 9/24/97 and 9/25/97

Figure 3. Benzene and KTPB slurry Quiescent and Agitated Releases

Benzene Balance

Table 4 below shows the benzene material balance for the column tests. The "potential benzene inventory" values were determined from the initial slurry composition and assuming that all available phenylborate compounds completely decomposed (see Table 10, Appendix B, page B-31). The "actual benzene formed" values were determined by taking the difference between the initial and final concentrations of soluble boron, TPB^-, 3PB, 2PB, and 1PB for each column (see Table 11, Appendix B, page B-32). "Quiescent", "Agitated", and "Water Addition" release values are rounded to the nearest gram and were determined from integration of the data presented in the previous sections of this report. "Residual" values were determined from the final slurry benzene analysis for each column (see Table 6, Appendix B, page B-1). The sum of the released and the residual benzene was compared to the actual formed as a measure of the material balance.

Table 4. Benzene Material Balance Summary

Column #	Potential Benzene,g	Actual Benzene Formed, g	Quiescent Release, g	Agitated Release,g	Water Addition Release,g	Residual Benzene,g	Total Released + Residual, g	Ratio of Released + Residual/ Actual, g
1	702	151	2	NA	NA	143	145	0.96
2	702	165	3	5	0	56	64	0.38
3	700	106	3	4	0	132	139	1.32
4	701	70	2	1	0	53	56	0.80
6	701	326	220	20	1	58	299	0.92
7	927	508	174	3	0	164	341	0.67
8	926	663	320	114	5	3	442	0.67
9	702	448	171	20	0	177	368	0.82
10	702	231	49	33	1	470	553	2.39
11	702	505	136	48	0	33	217	0.43
12	602	719	299	0	1	68	368	0.51
13	349	285	33	6	3	26	68	0.24

The calculated values for "actual benzene formed" and "residual benzene" have uncertainty because of the uncertainties associated with sample representativeness and analyses. Note for example that for Columns 3 and 10 that the residual benzene value exceeds the actual formed value which is not possible. Considered as a whole, however, the values in Table 4 show some interesting trends:

- The Series 1 columns formed the lowest amounts of benzene and had the lowest quiescent releases.
- For Series 2 and 3 columns, quiescent releases were 20-70% of the actual benzene formed.
- For Series 2 columns, the 1 wt % slurries (columns 6 and 8) had higher quiescent losses than the 4 wt % slurries (columns 7 and 9). This is consistent with the 1 wt % slurries having lower yield stress than the 4 wt % slurries.
- At the conclusion of the demonstration, Columns 6, 7, 9, and 11 had retained 50 - 60% of the benzene available at the time agitated release tests were initiated on those columns.

Quality Assurance

All work was conducted in accordance with the Technical Task Plan approved for this task.[5] This experimental program was conducted at the request of High Level Waste Engineering. Data collected during this program are contained in the author's notebooks and files.

Acknowledgments

The authors express their sincere gratitude to all the individuals who have contributed to this program. We would specifically like to thank H.L. Bolton, N.E. Gregory, B.A. Gunter, and M.S. Blume for providing outstanding experimental support. This program would not have been possible without the design, fabrication, instrumentation, and operational support skills of SRTC Experimental Thermal Fluids Group members: Z.H. Qureshi, J.W. Corbett, M.L. Armstrong, T.R. Head, J.S. Bollinger, M.D. Fowley, and T.J. Steeper. The challenge to fabricate the glass columns used in this experiment was met by the SRTC Glassblowing Shop personnel: J.G. Dobos and W.C. Sexton. Excellent analytical support was provided by T.L. White of SRTC Analytical Development and K.E. Cheeks of Waste Management Laboratories as well as their respective support personnel. Outstanding support in preparing this report was provided by T. Hang of SRTC-WPT and Janet Johnson of Publications and Media Services.

References

1. D.D. Walker, M.J. Barnes, C.L. Crawford. R.F. Swingle, R.A. Peterson, M.S. Hay, and S.D. Fink, "Decomposition of Tetraphenylborate in Tank 48-H (U)," WSRC-TR-96-0113, May 10, 1996.

2. Defense Nuclear Facilities Safety Board Recommendation 96-1 To The Secretary of Energy pursuant to 42 U.S.C. [[section]] 2286a(a)(5) Atomic Energy Act of 1954, as amended. August 14, 1996.

3. Department of Energy Implementation Plan for Defense Nuclear Facilities Safety Board Recommendation 96-1 To The Secretary of Energy pursuant to 42 U.S.C. DNFSB Recommendation 96-1, Implementation Plan, A. Lee Watkins, October 21, 1996.

4. J.E. Marra, J.T. Carter, "Benzene Retention and Release from KTPB Slurries Beyond Apparent Benzene Solubility (U)," Technical Task Request HLE-TTR-97018, 11/15/96.

5. J.E. Marek to W.L. Tamosaitis, "Task Technical Plan for Pilot-Scale Benzene Retention and Release Demonstration (U)," WSRC-RP-97-7, Revision 0, January 13, 1997.

6. R.A. Peterson, C.L. Crawford, and R.L. Swingle, "Initial Estimates of Mass Transfer Coefficients in Tank 48H and Tank 50H," WSRC-TR-96-256, Rev.0, August 16, 1996.

7. C.O. Bennett and J.E. Myers, Momentum, Heat, and Mass Transfer, 3rd Ed., New York: McGraw-Hill, 1982.

8. Donald Mackay and Paul J. Leinonen, "Rate of Evaporation of Low Solubility Contaminants from Water Bodies to the Atmosphere," Env. Sci. Tech., Vol. 9, No. 13, 1975, pp. 1178-1180.

9. Paul V. Roberts and Paul G. Dandliker , "Mass Transfer of Volatile Organic Contaminants from Aqueous Solution to the Atmosphere during Surface Aeration," Env. Sci. Tech., Vol. 17, No. 8, 1983, pp. 484-489.

10. L.O. Dworjanyn , "Benzene Retention in TPB Slurry - Status Report (U)," WSRC-RP-97-217, Revision 0, March 26, 1997.

11. A.N. Beris, J.A. Tsamopoulos, R.C. Armstrong, and R.A. Brown , "Creeping Motion of a Sphere through a Bingham Plastic," J. Fluid Mech., Vol. 158, 1985, pp. 219-244.

12. Ralph W. Ansley and Terence N. Smith , "Motion of Spherical Particles in a Bingham Plastic," A.I. Ch.E. Journal., Vol. 13, No. 6, Nov. 1967, pp. 1193-1196.

13. M.A. McLain and D. Goren, "Rheology of Non-Radioactive Simulant of Concentrated Tetraphenylborate Precipitate," DPST-84-401, March 30, 1984.

14. J.E. Young to W.B. Van Pelt, "Re: Analysis of Benzene Globules (U)," SRT-ADS-97-0467, September 19, 1997.

15. R.J. Ray to W.B. Van Pelt, "HPLC Results," WSRC-NB-97-60-76, 9/19/97.

16. L.O. Dworjanyn, "Benzene Release Interim Status Report (U)," WSRC-RP-97-903, September 30, 1997.

17. D.D. Walker, "Densities and Weight % Solids of Simulated Salt Solutions (U) ", WSRC-TR-91-00176, April 17, 1991.

References (cont'd)

18. C.L. Crawford, "Decomposition Studies of Tetrphenylborate Slurries (U)," WSRC-TR-97-0046, Rev.0, May 6, 1997

19. W.L. Daugherty to W.B. Van Pelt, "Benzene Demonstration Column Stopcock Failure (U)," SRT-MTS-974007, April 11, 1997.

20. Z.H. Qureshi to D. Muhlbaier, "System Description, Pilot-Scale Benzene Retention and Release Demonstration Tests (U)," SRT-ETF-970012, January 31, 1997.

21. J.S. Bollinger, to D.R. Muhlbaier, "System Description and Operating Instructions, Pilot-Scale Benzene Retention and Release Demonstration Test - Vapor Sampling System, Revision 2 (U)," SRT-ETF-970028, rev.2, June 12,1997.

Appendix A - Experimental

Apparatus

A layout schematic of the experimental equipment and services located in building 786-A is given in Figure 38. The vessels used in this experiment were borosilicate glass columns that were 72 ± 0.5 inches tall with an outside diameter of 7.87 inches (200 mm) and an inside diameter of 7.48 inches (190 mm). Over the course of the experiment, 12 such columns were used. The columns were constructed using two, 36 inch sections flanged together with Viton o-rings. Top and bottom plates of 316 stainless steel were clamped to the glass flanges with Viton o-rings. The upper plate contained several access ports for services, instrumentation, and sampling as shown in Figure 39. The bottom plate contained one access port for column recirculation and draining.

Sample Ports

The initial design used 23 sample ports located 3 inches apart along the length of the column. The sample ports consisted of stopcocks with 4 mm orifice and septa. A total of 11 ports were placed on the lower half of the column while 12 were positioned in the upper section. During the first test Series (Columns 1-4), two stopcock sample ports failed structurally. Subsequent analysis[19]determined that the failures were due to a combination of applied stress and caustic attack of the glass. Glass samples of the stopcock body had deposits of dried slurry and under microscopic inspection showed a network of crack-like features consistent with caustic attack. Repetitive tightening of the nut on the bottom of the Teflon® stopcock body as well as thermal expansion mismatch between the glass and the Teflon® created stresses within the stopcock. These two effects combined to create the failures. The risk of continuing the tests with the stopcock design was considered too great and the Series 1 (Columns 1-4) tests were terminated to redesign the sampling system. The column contents were drained and the columns were dismantled for redesign and retrofit. The stopcocks were replaced with #15 threaded glass fittings with Teflon® bushings with Viton® o-rings installed. Metal ball valves (1/4" Whitey) with septa were threaded into the Teflon® bushings. The valves provided much greater stability during sampling and offered superior chemical resistance compared to the glass stopcocks. All the sample ports in the upper sections of Columns 6-13 used the metal valve design. Based on the observations in Series 1 tests that the slurry filled the upper half of the columns, no sample ports were installed in the lower sections of Series 2 (Columns 6-9) or Series 3 (Columns 10-13) columns. The one exception was Column 12 which had 2 pump ports converted into sample ports in the lower column section since that slurry was designed to remain submerged due to the low salt content of the aqueous phase.

Pump Ports

Ports were also installed to accommodate recirculation pump suction and discharge points. The ports consisted of #15 threaded glass fittings fused to the glass column. Each port was fitted with threaded Teflon® bushing and a barbed Kynar® fitting, which was in turn connected to flexible Viton® tubing. All of the pump ports on a given column were interconnected with Viton® tubing (Masterflex series # 6412-size 73, 3.8" ID) to allow variable pump suction and discharge combinations. A total of 8 pump ports were installed (1 in bottom plate, 3 in lower glass section, 4 in upper glass section) at locations 0, 3, 12, 24, 39, 48, 54 and 57 inches respectively from the bottom of the column. The entire column assembly was bolted to a ¼" stainless steel plate supported on a framework resting in an open-topped (~670 liter capacity) polyethylene secondary containment box. The top of each column was anchored to overhead platforms that are an existing part of the building 786-A super structure.

Water addition method

Two water spray lines were provided. The first was a ¼" line fitted with a nozzle which produced a conical spray pattern of approximately 8 inches in diameter at a flowrate of 22 mL/second. The second was a simple ¼" line with no spray nozzle which produced a straight stream at a flowrate of 44 mL/second.

Column Heating

Each column was fitted with a 3/8" outside diameter serpentine heating coil (316 stainless steel, approximately 72 feet long). Three support rings were installed on each column along the length of the column interior. The coil was connected through the top column plate to hot water supply and return headers as shown in Figure 40. Four independent hot water zones were supplied, consisting of a 2.5 gallon water heater (1350 watts), a centrifugal pump (1/2 hp Teel, 115 volt, polypropylene), rotameter (0-11 gpm), supply header, return header and stand pipe. Each zone was equipped with drain, water fill, air vent, and pump discharge isolation valves. A type J thermocouple was installed in the supply header inlet and in the return header outlet. Each header has 10 ports to allow flexibility to achieve four different temperatures among the test columns. Each hot water heater has a thermostat with safety shutoff (190°F or 88°C). The design flowrate for heating water through the column coils was 1.5 gpm. At this flowrate, the temperature drop through the column heating coil is less than 1°C. Since the heating coil is positioned against the interior glass wall where heat loss occurs, it acts as a guard heater maintaining the interior bulk slurry nearly isothermal thereby minimizing convective thermal gradients within the bulk fluid.

Column Mixing

A slurry recirculation system was provided that consisted of a variable speed (50-650 rpm) Masterflex tubing pump (Cole Parmer catalog # H-07549-50) with Easy-Load head (Cole Parmer catalog # H-07529-80). Only one pump was used and moved as needed to the column being tested. The pump took suction from either the lowest side-mounted pump port or the port off the bottom of the column. Discharge was directed through any one port or combination of ports from the remaining six locations along the column length. Pump ports were isolated by tubing clamps. The flowrates achieved through the size 73, 3/8 inch tubing ranged from 0.2 to 8 liters/minute depending on the pump speed. During the course of testing, the section of Viton® tubing installed in the Easy-Load pump head experienced wear resulting in frequent hose replacement. For the section installed in the pump, the tubing material was changed to Norprene® (Masterflex series # 6404-size 73, 3.8" ID) to provide better durability. Although the tubing life was improved by the material change, the replacement frequency was still unacceptably high. The Easy-Load pump head was replaced with a standard-load head (Cole Parmer catalog # E-07019-41) with much improved overall system performance.

The initial equipment installation included an agitation system for each column consisting of a variable speed mixer drive, a 6 foot long, 3/8 inch diameter shaft, and three impellers (two axial flow Lightnin A-310, ¾ inch diameter impellers and one radial flow Lightnin R-100, 3 inch diameter impeller). The drive was a 115 V, ¼ hp Lightnin motor capable of turning the shaft in the 20-350 rpm range. The shaft speed is directly proportional to the potentiometer setting since the unit is a high torque gear drive. The drive was connected to the shaft via a universal coupling. One drive unit was shared among the columns. After observing the poor degree of mixing achieved in Column 1, the design was changed to add more axial impellers and to increase their diameter. For Columns 2 -4, each column had 5 axial impellers (Midwest Mixing, investment casting , 5x5) of 5 inches in diameter and one radial impeller at the bottom of the shaft. Columns 2-4 operation showed that the impeller blades, while providing better mixing, provided significant surface area to catch or trap TPB slurry rather than allowing the slurry to segregate into clear solids and salt solution layers. This resulted in regions of solids along the column length interspersed with large gaps filled with salt solution. Since this slurry configuration was considered atypical of expectations for Tank 48H, the agitator shaft and impellers were removed from Columns 6-13 (column position 5 was never used). Mixing in Columns 6-13 was then provided solely by the recirculation pump. The two uppermost pump ports in Columns 6-13 were fitted with ¼ inch tubes to provide additional flow velocity and increased mixing in the area where the slurry to vapor interface was most likely to occur.

Because benzene is present in the experimental system, the vapor spaces of the test columns were purged with nitrogen to preclude flammability concerns. Purging the test columns is also consistent with the fact that Tank 48H is nitrogen-purged. The purge system provides capability for two purge rate ranges. The first is for the stagnant or quiescent period of the test where the column purge rate can range from 20 - 500 cc/min. During the agitated or release periods of the test the purge is increased to approximately 2 liters/min. through a separate purge line with a maximum capacity of 11 liters/min. The nitrogen is supplied through two cylinders connected in parallel through pressure regulators. One bottle was on-line at any given time with the second bottle serving as a standby which automatically began to supply nitrogen if the first bottle could not maintain the desired pressure. This arrangement provided an uninterrupted nitrogen supply. The lower purge range header was connected to a manifold of 10 rotameters that allowed individual control for the purge to each column. During testing, a water-filled standpipe was added to the system downstream of the control rotameters to better humidify the nitrogen purge gas. The humidification was added to lessen the degree of slurry surface drying observed in the initial Column 1-4 tests. For the high purge header, argon was introduced as a tracer gas to more accurately measure the nitrogen purge rate. The purges exhausted through a exhaust header into the building H&V system. The exhaust system was capable of pulling 150 cfm at 5 inches of water column vacuum. An atmospheric air bleed line was attached to the exhaust vent to control column vacuum. The column pressure was maintained at approximately 2 inches of water column vacuum as measured by a manometer mounted to the outside of each column. Purge gases were exhausted to the atmosphere at a height of about 42 feet above ground level. [20]

Figure 38. Experimental Apparatus Schematic

Figure 39. Column Top Plate Arrangement

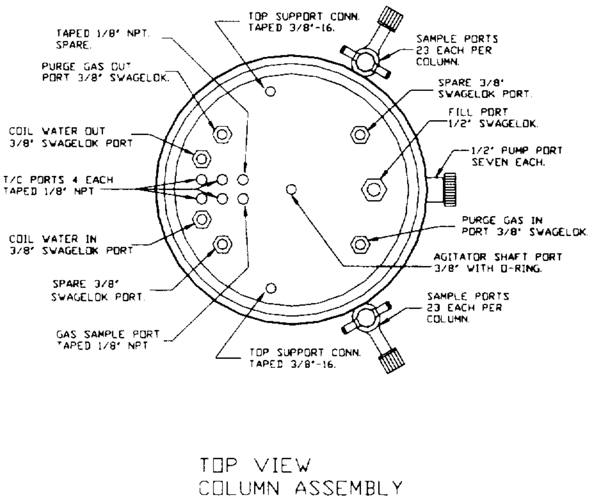


Figure 40. Water Header Arrangement

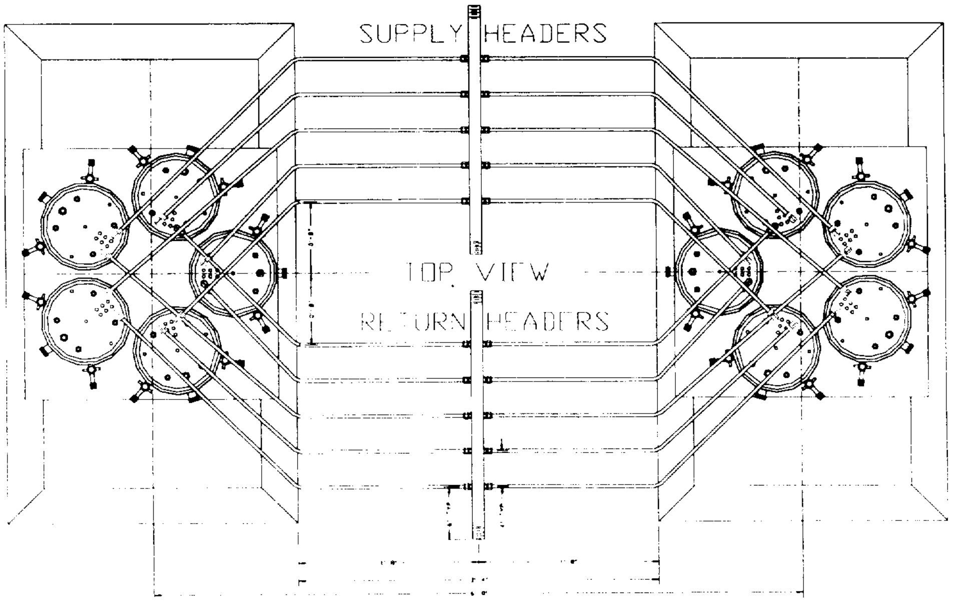


Figure 41. Pilot-Scale Benzene Retention Demonstration Columns

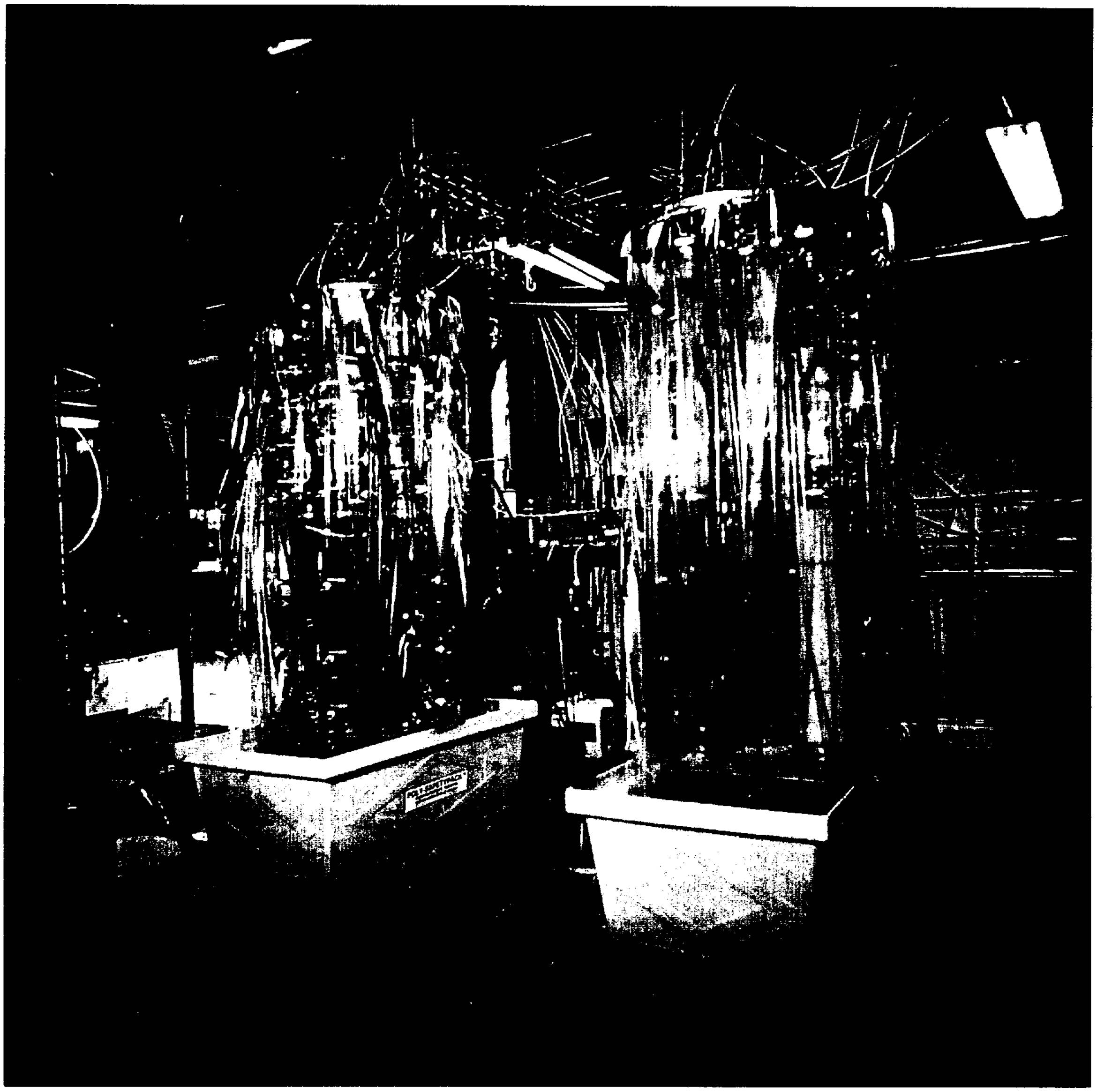


Figure 42. Pilot-Scale Demonstration Columns 6-10

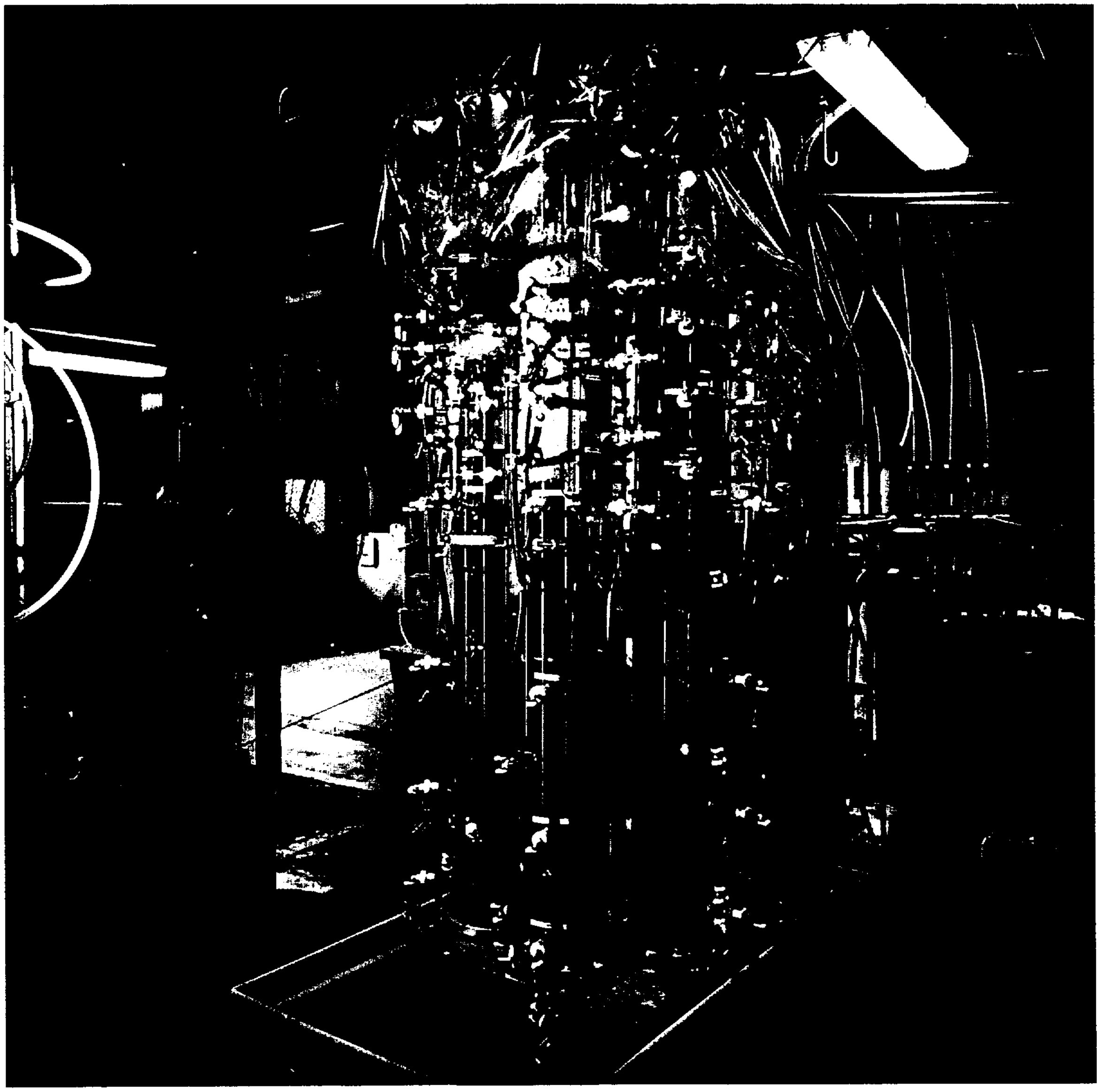
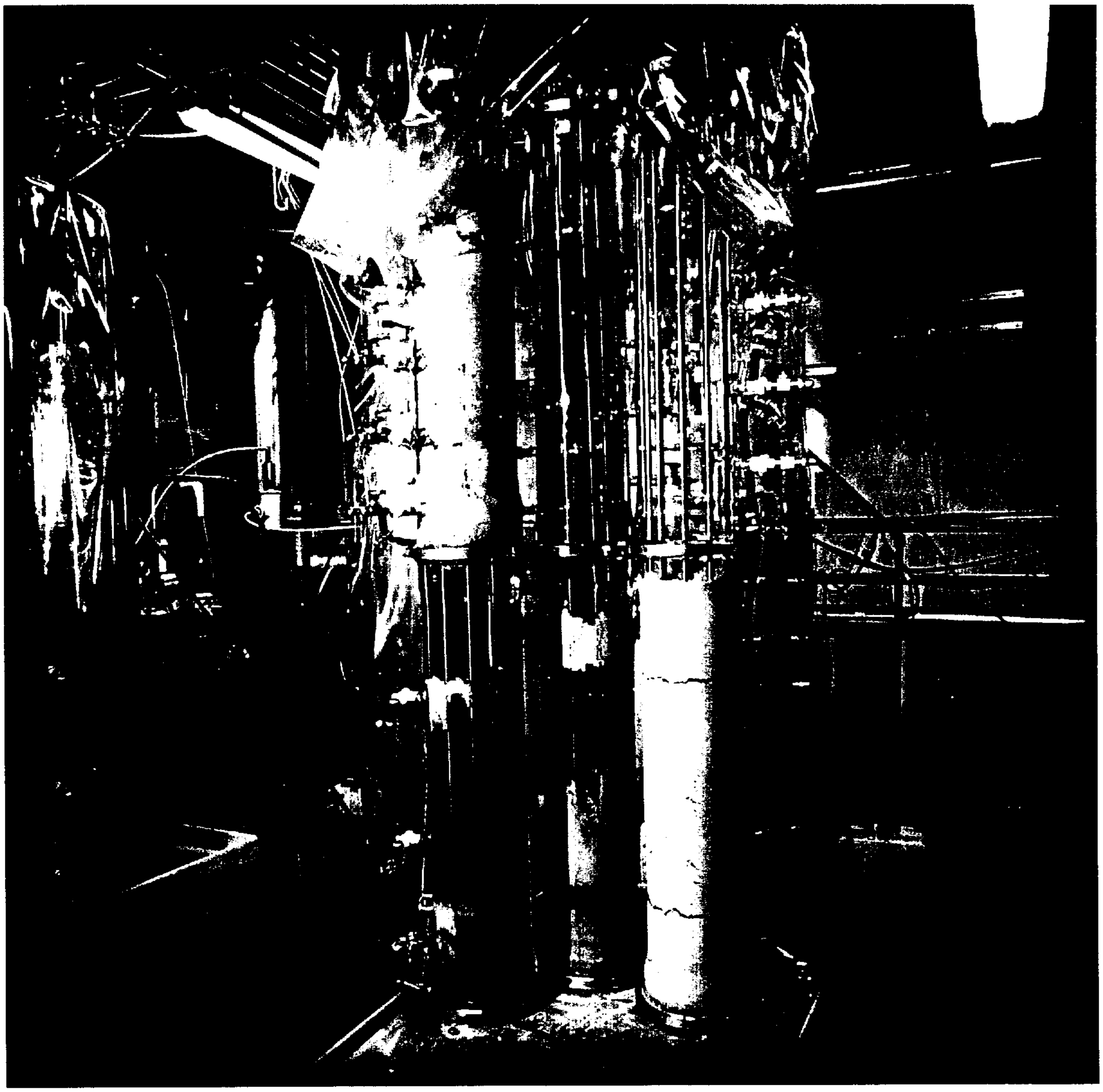


Figure 43. Pilot-Scale Demonstration Columns 11-13



Vapor Sampling System

Figure 44 gives a layout schematic of the vapor sampling system. Vapor samples from the columns are drawn into individual 1/8 inch diameter tubing attached to three-way valves. The three-way valves allow selection of individual columns for sampling. The sample line from the three-way valves to the Nafion dryer is heat traced to preclude any condensate formation. Vapor phase moisture is then removed from the sample by a Nafion dryer. A Nafion dryer is much like a shell and tube heat exchanger with dry air on the shell side and sample gas in the tube side. The shell side dry gas is provided by an O'Keefe dryer. Dry sample gas leaving the Nafion dryer enters the gas chromatograph via a small diaphragm pump.

The gas chromatograph is a MicroSensor Technology Inc. (MTI) unit consisting of two detector columns, an internal vacuum pump, sample injector, and a thermal conductivity detector for each column. Column A is an MS-5A Column used to detect oxygen and uses nitrogen as a carrier gas. Since the test columns are purged with nitrogen, oxygen detected in vapor space samples would indicate a leak in the system. Column B is an OV-73 Column used to detect benzene and uses helium as a carrier gas.

EZChrom 200 Version 4.4 gas chromatagraphy software is being used in this test. The gas chromatograph RS-232 port is connected to a pentium notebook computer which served as the operator interface with the GC.[21]

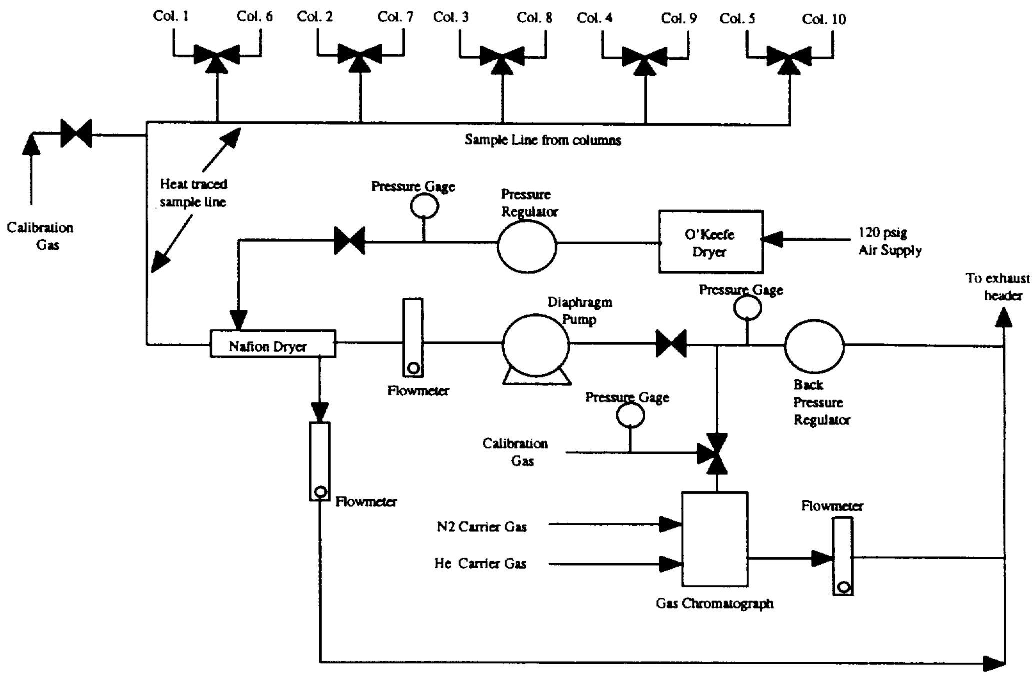


Figure 44. Vapor Sampling System

Slurry Preparation

Precipitate slurry for Column 1 was generated in-situ by reaction of a potassium salt solution with a 0.58 M sodium tetraphenylborate solution provided by ITP (Holly Oak Lot # D1558). The precipitation was performed by pumping the sodium tetraphenylborate solution into the salt solution over a period of 5 +/- 0.5 hours. The recipe for the Column 1 and subsequent slurries incorporated organic intermediates, including benzene, triphenylboron, diphenylborinic acid, phenylboric acid, phenol, biphenyl, methanol, and ethanol. During the preparation of the slurry in Column 1, mixing was observed to be questionable, and this was attributed to the large aspect ratio (L/D = 9) of the column. The precipitate slurries for Columns 2 - 13 were hence prepared in carboys at TNX and transported to the 786-A columns for loading. It was subsequently determined that the precipitation reaction in Column 1 did not initially achieve complete precipitation of all the soluble potassium and hence the soluble tetraphenylborate concentration was extremely low. Hence, the source of the benzene was initially severely diminished in Column 1. In subsequent slurry preparations, a step was included to qualitatively check for the presence of soluble potassium and for soluble tetraphenylborate before the majority of the sodium salts were added to the slurry. This test allowed all subsequent columns to be loaded with slurry having the appropriate properties for benzene generation.

The composition of the slurries used in the Series 1 columns is shown in Table 5. These slurries were prepared in air, with vigorous agitation to provide good mixing. In order to accelerate TPB decomposition, the slurry composition was adjusted in the Series 2 columns to boost the noble metals (ruthenium, palladium, and rhodium) to the levels of the ECC catalyst studies (reference) and the copper concentration was increased from 1.4 to 11.4 mg/L. In addition, the Series 2 columns incorporated silver in the recipe, and were prepared under an inert nitrogen purge to reduce the concentration of soluble oxygen. Three of the slurries prepared for Series 3 had reduced levels of sodium (3.9M in Column 10, 3.7M in Column 11, 0.2M in Column 12). Gas entrainment was suspected of contributing to the slurry buoyancy observed in the columns. The slurries prepared for Columns #12 and #13 were prepared using gentle agitation and with the sodium tetraphenylborate addition line submerged in the salt solution to minimize any incorporation of gases in the slurry.

Columns 1 - 4 were loaded were maintained at 40 C for the first 7-8 days of operation. Thereafter, the columns were adjusted to 50 C for the balance of their operation until they were de-inventoried on 4/3/97. The Series 2 columns (6-9) were loaded with slurry and adjusted to 50 C. These columns were maintained at 50 C until 5/8/97, when they were cooled to 40 C. Column 10 was loaded with slurry at 50 C but was cooled to 40 C within 72 hours and maintained at 40 C throughout the duration of its operation. Columns 11 - 12 were loaded with slurry at 50 C and were maintained at 50 C until their respective release tests when they were cooled to 40 C.

During the quiescent period, the columns were monitored at least weekly. This monitoring included measurements of the vapor phase benzene concentration and sampling of the liquid and slurry phases. Liquid and slurry samples for benzene analysis were taken by inserting a #10 gauge syringe needle through a septa attached to the sample port. This allowed withdrawal of the sample into a 10 cc syringe. Upon removal from the sample port, the needle was removed and a gas-tight syringe valve was attached to the syringe and closed. Liquid and slurry samples for chemical analysis were acquired by opening the appropriate valve and collecting material in a 125 mL glass bottle.

These samples were then filtered and the filtrate submitted for HPLC analysis and for ICP/ES analysis. The HPLC method (HPLC-DPBA) analyzed for the soluble sodium tetraphenylborate and the breakdown intermediates triphenylboron and diphenylborinic acid. A limited set of data was also obtained on the Series 1 columns for phenol and phenylboric acid. The ICP/ES method analyzed for soluble boron and periodically for other species of interest (soluble copper, sodium, potassium, etc.)

Table 5. Target Simulant Compositions

		Column 1	Column 2	Column 3	Column 4
Target wt %		4	4	1	1
Potassium Salt Solution	Formula Wt	grams	grams	grams	grams
NaOH	40	153.76	153.76	153.76	153.76
KNO_3	101.1	579.17	579.17	579.17	579.17
KNO_2	85.1	0	0	0	0
$CsNO_3$	162.91	1.28	1.28	1.28	1.28
$NaNO_3$	85.01	399.78	399.78	765.03	765.03
$NaNO_2$	69.01	2248.0	2248.0	2248.0	2248.0
NaCl	58.45	35.57	35.57	35.57	35.57
NaF	42.00	14.56	14.56	14.56	14.56
Na_2SO_4	142.05	30.85	30.85	30.85	30.85
$Na_3PO_4 \cdot 12H_2O$	380.16	99.10	99.10	99.10	99.10
Water	18.016	6125.40	6125.40	6125.40	6125.40
NaTPB Solution					
NaTPB	342.23	2684.96	2684.96	1214.56	1214.56
NaOH	40.00	75.75	75.75	34.27	34.27
Water	18.016	11334.09	11334.09	5127.06	5127.06
Sodium Salt Solution					
NaOH	40.00	5453.42	5453.42	5453.42	5453.42
$Na_2CO_3 \cdot H_2O$	124.00	828.78	828.78	969.64	969.64
$Al(NO_3)_3 \cdot 9H_2O$	375.14	2281.31	2281.31	2281.31	2281.31
Water	18.016	14867.74	14867.74	15744.51	15744.51
Sludge/Titanate Solutions					
Sludge	12.42 wt %	839.33	839.33	209.83	209.83
$NaTi_2O_5H$	199.80	329.68	329.68	82.42	82.42
Acid Metal Trim Solution					
$Cu(NO_3)_2 \cdot 2.5H_2O$	232.62	0.223	0.223	0.223	0.223
$SnCl_2 \cdot 2H_2O$	225.65	0.190	0.190	0.190	0.190
$Zn(NO_3)_2 \cdot 6H_2O$	297.49	1.522	1.522	1.522	1.522
$Fe(NO_3)_3 \cdot 9H_2O$	404.02	0.440	0.440	0.440	0.440
HNO_3	63.02	0.126	0.126	0.126	0.126
Water	18.016	20.00	20.00	20.00	20.00
Basic Metal Trim Solution					
Na_2CrO_4	162.00	9.879	9.879	9.879	9.879
K_2MoO_4	238.13	1.294	1.294	1.294	1.294
NaOH	40.00	0.4	0.4	0.4	0.4
Water	18.016	100	100	100	100
Noble Metal Solutions					
$RuCl_3$	0.4174	0.083	0.083	0.083	0.083
$Pd(NO_3)_2$	0.1527	0.114	0.114	0.114	0.114
$Rh(NO_3)_3$	0.0493	0.176	0.176	0.176	0.176
Water	18.016	150	150	150	150
Organic Trim Chemicals					
Benzene	78.11	31.3	31.3	31.3	31.3
Phenol	94.11	5.4	5.4	5.4	5.4
Phenylboric Acid	121.9	5.4	5.4	5.4	5.4
Biphenyl	154.2	6.5	6.5	6.5	6.5
Diphenylborinic Acid	182.0	6.7	6.7	6.7	6.7
Triphenylboron	242.1	70.3	70.3	70.3	70.3
Isopropyl Alcohol	60.09	2.2	2.2	2.2	2.2
Methanol	32.04	0.22	0.22	0.22	0.22

Table 5. Target Simulant Compositions

		Column 6	Column 7*	Column 8**	Column 9
Target wt %		4	4	1	1
Potassium Salt Solution	Formula Wt	grams	grams	grams	grams
NaOH	40	153.76			153.76
KNO_3	101.1	144.79			579.17
KNO_2	85.1	0.00			0.00
$CsNO_3$	162.91	1.28			1.28
$NaNO_3$	85.01	765.03			399.78
$NaNO_2$	69.01	2248.00			2248.00
NaCl	58.45	35.57			35.57
NaF	42.00	14.56			14.56
Na_2SO_4	142.05	30.85			30.85
$Na_3PO_4 \cdot 12H_2O$	380.16	99.10			99.10
Water	18.016	6125.40			6125.40
NaTPB Solution					
NaTPB	342.23	1214.56	1040.00	1040.00	2684.96
NaOH	40.00	34.27			75.75
Water	18.016	5127.06			11334.09
Sodium Salt Solution					
NaOH	40.00	5453.42			5453.42
$Na_2CO_3 \cdot H_2O$	124.00	969.64			969.64
$Al(NO_3)_3 \cdot 9H_2O$	375.14	2281.31			2281.31
Water	18.016	15744.51			14867.74
Sludge/Titanate Solutions					
Sludge	12.42 wt %	209.83			839.33
$NaTi_2O_5H$	199.80	82.42			329.68
$AgNO_3$	169.87		0.0342	0.0342	0.034
Acid Metal Trim Solution					
$Cu(NO_3)_2 \cdot 2.5H_2O$	232.62	1.813	1.5924	1.5924	1.813
$SnCl_2 \cdot 2H_2O$	225.65	0.190			0.190
$Zn(NO_3)_2 \cdot 6H_2O$	297.49	1.522			1.522
$Fe(NO_3)_3 \cdot 9H_2O$	404.02	0.440			0.440
HNO_3	63.02	0.1260			0.1260
Water	18.016	20.00			20.00
Basic Metal Trim Solution					
Na_2CrO_4	162.00	9.879			9.879
K_2MoO_4	238.13	1.294			1.294
NaOH	40.00	0.4			0.4
Water	18.016	100			100
Noble Metal Solutions					
$RuCl_3$	0.4174	0.083	0.479	0.479	0.562
$Pd(NO_3)_2$	0.1527	0.114	0.626	0.626	0.740
$Rh(NO_3)_3$	0.0493	0.176	1.057	1.057	1.233
Water	18.016	150	60	60	150
Organic Trim Chemicals					
Benzene	78.11	31.3	31.3	31.3	31.3
Phenol	94.11	5.4			5.4
Phenylboric Acid	121.9	5.4	5.4	5.4	5.4
Biphenyl	154.2	6.5			6.5
Diphenylborinic Acid	182.0	6.7	6.7	6.7	6.7
Triphenylboron	242.1	70.3	70.3	70.3	70.3
Isopropyl Alcohol	60.09	2.2			2.2
Methanol	32.04	0.22			0.22

Table 5. Target Simulant Compositions

		Column 10	Column 11	Column 12*	Column 13
Target wt %		4	4	1	1
Potassium Salt Solution	Formula Wt	grams	grams	grams	grams
NaOH	40	130.97	119.20	193.39	76.88
KNO_3	101.1	579.17	579.17	71.31	289.58
KNO_2	85.1	0.00	0.00	68.17	0.00
$CsNO_3$	162.91	1.28	1.28	1.11	0.64
$NaNO_3$	85.01	268.35	200.43	0.00	199.89
$NaNO_2$	69.01	1914.82	1742.63	0.00	1124.000
NaCl	58.45	30.30	27.57	0.87	17.79
NaF	42.00	12.40	11.28	0.36	7.28
Na_2SO_4	142.05	26.28	23.92	0.76	15.43
$Na_3PO_4 \cdot 12H_2O$	380.16	84.42	76.83	2.44	49.55
Water	18.016	5125.40	5125.40	12430.43	3062.70
NaTPB Solution					
NaTPB	342.23	2684.96	2684.96	2311.79	1342.48
NaOH	40.00	75.75	75.75	65.22	37.87
Water	18.016	11334.09	11334.09	9758.84	5667.04
Sodium Salt Solution					
NaOH	40.00	4645.16	4227.46		2726.71
$Na_2CO_3 \cdot H_2O$	124.00	825.93	751.66		484.82
$Al(NO_3)_3 \cdot 9H_2O$	375.14	1943.20	1768.46	56.10	1140.66
Water	18.016	17722.04	17722.04		5799.40
Sludge/Titanate Solutions					
Sludge	12.42 wt %	983.45	983.45	983.45	419.67
$NaTi_2O_5H$	199.80	329.68	329.68	329.68	164.84
$AgNO_3$	169.87	0.034	0.034	0.034	0.17
Acid Metal Trim Solution					
$Cu(NO_3)_2 \cdot 2.5H_2O$	232.62	1.813	1.813	1.813	0.906
$SnCl_2 \cdot 2H_2O$	225.65	0.190	0.190	0.190	0.095
$Zn(NO_3)_2 \cdot 6H_2O$	297.49	1.522	1.522	1.522	0.761
$Fe(NO_3)_3 \cdot 9H_2O$	404.02	0.440	0.440	0.440	0.220
HNO_3	63.02	0.1260	0.1260	0.1260	0.126
Water	18.016	20.00	20.00	20.00	20.00
Basic Metal Trim Solution					
Na_2CrO_4	162.00	9.879	9.879	9.879	4.940
K_2MoO_4	238.13	1.294	1.294	1.294	0.647
NaOH	40.00	0.4	0.4	0.4	0.4
Water	18.016	100	100	100	100
Noble Metal Solutions					
$RuCl_3$	0.4174	0.562	0.562	0.562	0.2810
$Pd(NO_3)_2$	0.1527	0.740	0.740	0.740	0.370
$Rh(NO_3)_3$	0.0493	1.233	1.233	1.233	0.616
Water	18.016	150	150	150	150
Organic Trim Chemicals					
Benzene	78.11	31.3	31.3	31.3	15.6
Phenol	94.11	5.4	5.4	5.4	2.7
Phenylboric Acid	121.9	5.4	5.4	5.4	2.7
Biphenyl	154.2	6.5	6.5	6.5	3.3
Diphenylborinic Acid	182.0	6.7	6.7	6.7	3.4
Triphenylboron	242.1	70.3	70.3	70.3	35.1
Isopropyl Alcohol	60.09	2.2	2.2	2.2	1.1
Methanol	32.04	0.22	0.22	0.22	0.11

Appendix B - Data

Table 6. Column Benzene Analyses

Column #	Sample Port	Date	ITP Sample #	Dilution Factor	Raw Bz Analysis mg/L	Benzene Corrected for Dilution, mg/L
1	SP13	2/5/97	70092	1	184	184
1	SP13	2/6/97	70093	1	244	244
1	SP13	2/6/97	70094	1	171	171
1	SP18	2/6/97	70095	1	165	165
1	SP03	2/11/97	70096	1	386	386
1	SP13	2/11/97	70097	1	160	160
1	SP18	2/11/97	70098	1	168	168
1	SP19	2/13/97	70100	1	332	332
1	SP18	2/20/97	70099	1	272	272
1	SP03	2/27/97	70126	1	217	217
1	SP13	2/27/97	70127	1	90	90
1	SP18	2/27/97	70128	1	451	451
1	SP03	3/6/97	70173	1	301	301
1	SP13	3/6/97	70178	1	78	78
1	SP18	3/6/97	70177	1	1454	1454
1	SP03	3/13/97	70188	1	198	198
1	SP13	3/13/97	70189	1	73	73
1	SP18	3/13/97	70190	1	1644	1644
1	SP18	3/13/97	70200	1	925	925
1	SP11	3/20/97	70216	1	141	141
1	SP17	3/20/97	70217	1	310	310
1	SP18	3/20/97	70218	1	2442	2442
1	SP19	3/20/97	70219	1	2732	2732
1	SP18	3/26/97	70237	1	2731	2731
1	S-02	4/2/97	70250	1	179	179
1	SP01	4/2/97	70249	1	83	83
1	SP03	4/2/97	70251	1	866	866
1	SP04	4/2/97	70252	1	73	73
1	SP05	4/2/97	70253	1	112	112
1	SP06	4/2/97	70254	1	86	86
1	SP07	4/2/97	70255	1	674	674
1	SP08	4/2/97	70256	1	1390	1390
1	SP09	4/2/97	70257	1	1365	1365
1	SP10	4/2/97	70258	1	117	117
1	SP11	4/2/97	70259	1	93	93
1	SP12	4/2/97	70260	1	130	130
1	SP13	4/2/97	70261	1	72	72
1	SP14	4/2/97	70262	1	69	69
1	SP15	4/2/97	70263	1	66	66
1	SP16	4/2/97	70264	1	465	465
1	SP17	4/2/97	70265	1	2166	2166
1	SP18	4/2/97	70266	1	3256	3256
2	SP03	2/19/97	70101	1	104	104
2	SP12	2/21/97	70102	1	394	394
2	SP18	2/21/97	70103	1	589	589
2	SP03	2/22/97	70104	1	140	140
2	SP12	2/22/97	70105	1	365	365
2	SP18	2/22/97	70106	1	542	542
2	SP12	2/23/97	70107	1	694	694
2	SP18	2/23/97	70108	1	742	742
2	SP03	2/24/97	70109	1	129	129
2	SP18	2/24/97	70110	1	659	659
2	SP03	2/25/97	70111	1	175	175

Table 6. Column Benzene Analyses

Column #	Sample Port	Date	ITP Sample #	Dilution Factor	Raw Bz Analysis mg/L	Benzene Corrected for Dilution, mg/L
2	SP18	2/25/97	70113	1	793	793
2	SP03	2/26/97	70114	1	163	163
2	SP12	2/26/97	70115	1	854	854
2	SP18	2/26/97	70116	1	856	856
2	SP03	2/27/97	70124	1	94	94
2	SP12	2/27/97	70123	1	629	629
2	SP18	2/27/97	70125	1	563	563
2	SP03	3/6/97	70168	1	81	81
2	SP12	3/6/97	70172	1	326	326
2	SP18	3/6/97	70169	1	131	131
2	SP03	3/13/97	70191	1	130	130
2	SP13	3/13/97	70192	1	349	349
2	SP18	3/13/97	70193	1	166	166
2	SP18	3/13/97	70201	1	400	400
2	SP16	3/20/97	70220	1	1390	1390
2	SP17	3/20/97	70221	1	1591	1591
2	SP18	3/20/97	70222	1	975	975
2	SP19	3/20/97	70223	1	2300	2300
2	SP18	3/26/97	70238	1	522	522
2	SP19	3/26/97	70239	1	1270	1270
2	SP12	2/25/	70112	1	733	733
3	SP03	2/28/97	70129	1	300	300
3	SP12	2/28/97	70130	1	309	309
3	SP18	2/28/97	70131	1	276	276
3	SP03	3/1/97	70132	1	25	25
3	SP12	3/1/97	70133	1	122	122
3	SP18	3/1/97	70134	1	560	560
3	SP03	3/2/97	70135	1	79	79
3	SP12	3/2/97	70136	1	257	257
3	SP18	3/2/97	70137	1	616	616
3	SP03	3/3/97	70138	1	81	81
3	SP12	3/3/97	70139	1	233	233
3	SP18	3/3/97	70140	1	724	724
3	SP03	3/4/97	70141	1	91	91
3	SP13	3/4/97	70142	1	107	107
3	SP18	3/4/97	70143	1	714	714
3	SP03	3/5/97	70147	1	101	101
3	SP13	3/5/97	70148	1	121	121
3	SP18	3/5/97	70149	1	182	182
3	SP03	3/6/97	70170	1	112	112
3	SP13	3/6/97	70167	1	110	110
3	SP18	3/6/97	70171	1	440	440
3	SP03	3/13/97	70194	1	132	132
3	SP13	3/13/97	70195	1	144	144
3	SP18	3/13/97	70196	1	1123	1123
3	SP15	3/20/97	70224	1	233	233
3	SP16	3/20/97	70225	1	1156	1156
3	SP17	3/20/97	70226	1	1466	1466
3	SP18	3/20/97	70227	1	3067	3067
3	SP18	3/26/97	70240	1	3541	3541
4	SP03	3/4/97	70144	1	412	412
4	SP13	3/4/97	70145	1	415	415
4	SP18	3/4/97	70146	1	480	480

Table 6. Column Benzene Analyses

Column #	Sample Port	Date	ITP Sample #	Dilution Factor	Raw Bz Analysis mg/L	Benzene Corrected for Dilution, mg/L
4	SP03	3/5/97	70150	1	400	400
4	SP13	3/5/97	70151	1	424	424
4	SP18	3/5/97	70152	1	397	397
4	SP03	3/6/97	70174	1	391	391
4	SP13	3/6/97	70175	1	387	387
4	SP18	3/6/97	70176	1	389	389
4	SP03	3/7/97	70165	1	393	393
4	SP13	3/7/97	70166	1	384	384
4	SP18	3/7/97	70163	1	366	366
4	SP03	3/8/97	70164	1	339	339
4	SP13	3/8/97	70162	1	309	309
4	SP18	3/8/97	70156	1	407	407
4	SP03	3/9/97	70159	1	276	276
4	SP13	3/9/97	70161	1	425	425
4	SP18	3/9/97	70158	1	409	409
4	SP03	3/10/97	70155	1	370	370
4	SP13	3/10/97	70157	1	426	426
4	SP18	3/10/97	70160	1	337	337
4	SP03	3/11/97	70185	1	64	64
4	SP13	3/11/97	70186	1	176	176
4	SP18	3/11/97	70187	1	72	72
4	SP03	3/13/97	70197	1	409	409
4	SP13	3/13/97	70198	1	696	696
4	SP18	3/13/97	70199	1	206	206
4	SP16	3/20/97	70228	1	424	424
4	SP17	3/20/97	70229	1	358	358
4	SP18	3/20/97	70230	1	102	102
4	SP19	3/20/97	70231	1	986	986
4	SP18	3/26/97	70241	1	56	56
4	SP19	3/26/97	70242	1	1201	1201
6	SP01	4/11/97	70280	1	268	268
6	SP01	4/12/97	70281	1	724	724
6	SP01	4/17/97	70304	1	84	84
6	SP04	4/17/97	70313	1	2217	2217
6	SP07	4/17/97	70323	1	2950	2950
6	SP04	4/24/97	70305	1	98	98
6	SP07	4/24/97	70317	1	4473	4473
6	SP08	4/24/97	70319	1	thick	
6	SP05	5/1/97	70353	1	134	134
6	SP06	5/1/97	70354	1	84	84
6	SP07	5/1/97	70355	1	4538	4538
6	SP04	5/8/97	70371	1	88	88
6	SP06	5/8/97	70374	1	384	384
6	SP07	5/8/97	70373	1	730	730
6	SP05	5/14/97	70396	1	1693	1693
6	SP07	5/22/97	70421	1	688	688
6	SP06	5/28/97	70474	1	2524	2524
6	SP07	5/28/97	70475	1	2541	2541
6	SP03	6/5/97	70509	1	98	98
6	SP07	6/5/97	70510	1	3129	3129
6	SP03	6/12/97	70565	1	61	61
6	SP07	6/12/97	70566	1	2553	2553
6	SP07	6/19/97	70601	1	918	918
6	SP01	7/9/97	70720	1	762	762

Table 6. Column Benzene Analyses

Column #	Sample Port	Date	ITP Sample #	Dilution Factor	Raw Bz Analysis mg/L	Benzene Corrected for Dilution, mg/L
6	SP01	7/9/97	70721	1	669	669
6	SP02	7/9/97	70722	1	484	484
6	SP02	7/9/97	70723	1	1906	1906
6	SP06	7/9/97	70718	1	708	708
6	SP07	7/9/97	70719	1	2833	2833
6	SP01	7/15/97	70768	1	1034	1034
6	SP01	7/15/97	70769	1	915	915
6	SP01	7/15/97	70770	1	694	694
6	SP01	7/15/97	70771	1	1045	1045
6	SP01	7/15/97	70772	1	651	651
6	SP07	7/15/97	70773	1	2036	2036
6	SP07	7/15/97	70774	1	1484	1484
7	SP01	4/12/97	70278	1	2503	2503
7	SP01	4/12/97	70279	1	2360	2360
7	SP04	4/17/97	70307	1	thick	
7	SP07	4/17/97	70320	1	thick	
7	SP01	4/24/97	70308	1	213	213
7	SP03	4/24/97	70315	1	1528	1528
7	SP04	4/24/97	70325	1	3503	3503
7	SP07	4/24/97	70318	1	thick	
7	SP04	5/1/97	70356	1.14	3141	3595
7	SP05	5/1/97	70357	3.09	3581	11060
7	SP06	5/1/97	70358	2.07	2710	5613
7	SP03	5/8/97	70375	1	1440	1440
7	SP04	5/8/97	70376	1	1383	1383
7	SP05	5/8/97	70377	1	1999	1999
7	SP06	5/8/97	70378	1	1756	1756
7	SP05	5/14/97	70397	1	2663	2663
7	SP06	5/14/97	70398	1	2291	2291
7	SP03	5/22/97	70427	2.36	2627	6190
7	SP04	5/22/97	70426	1	1431	1431
7	SP05	5/22/97	70438	1	801	801
7	SP06	5/22/97	70436	1	539	539
7	SP05	5/28/97	70476	1	907	907
7	SP06	5/28/97	70477	1	1777	1777
7	SP08	5/28/97	70478	1	651	651
7	SP04	6/5/97	70511	1	4062	4062
7	SP05	6/5/97	70512	1	405	405
7	SP04	6/12/97	70567	1	2687	2687
7	SP05	6/12/97	70568	1	575	575
7	SP07	6/12/97	70569	8.22	358	2942
7	SP03	6/19/97	70602	2.01	2854	5725
7	SP01	7/1/97	70680	1	3718	3718
7	SP03	7/2/97	70685	1	554	554
8	SP01	4/15/97	70282	1	1140	1140
8	SP01	4/17/97	70312	1	140	140
8	SP04	4/17/97	70314	1	3755	3755
8	SP04	4/17/97	70306	1	253	253
8	SP07	4/17/97	70316	1	3510	3510
8	SP06	4/24/97	70310	1	857	857
8	SP07	4/24/97	70309	1	4870	4870
8	SP04	5/1/97	70359	1	65	65
8	SP05	5/1/97	70360	1	138	138

Table 6. Column Benzene Analyses

Column #	Sample Port	Date	ITP Sample #	Dilution Factor	Raw Bz Analysis mg/L	Benzene Corrected for Dilution, mg/L
8	SP06	5/1/97	70361	1	261	261
8	SP07	5/1/97	70362	1.56	5145	8004
8	SP01	5/8/97	70379	1	298	298
8	SP04	5/8/97	70380	1	297	297
8	SP05	5/8/97	70381	1	51	51
8	SP06	5/8/97	70382	1	556	556
8	SP06	5/14/97	70399	1	553	553
8	SP07	5/14/97	70400	1	4323	4323
8	SP06	5/22/97	70434	1	934	934
8	SP07	5/22/97	70423	1	558	558
8	SP05	5/28/97	70479	1	600	600
8	SP06	5/28/97	70480	1	1592	1592
8	SP06	6/5/97	70513	1	656	656
8	SP03	6/12/97	70570	1	100	100
8	SP04	6/12/97	70571	1	141	141
8	SP05	6/12/97	70572	1	136	136
8	SP06	6/12/97	70573	1	3186	3186
8	SP05	6/19/97	70603	1	91	91
8	SP03	7/18/97	70775	1	2358	2358
8	SP03	7/18/97	70776	1	1506	1506
8	SP03	7/18/97	70777	1	1577	1577
8	SP03	7/18/97	70778	1	276	276
8	SP03	7/21/97	70831	1	719	719
8	SP03	7/21/97	70832	1	420	420
8	SP03	7/21/97	70833	1	654	654
8	SP03	7/22/97	70834	1	765	765
8	SP03	7/22/97	70835	1	1121	1121
8	SP03	7/22/97	70836	1	637	637
8	SP03	7/22/97	70837	1	503	503
8	SP03	7/23/97	70838	1	436	436
8	SP03	7/23/97	70839	1	389	389
8	SP03	7/23/97	70840	1	315	315
8	SP03	7/24/97	70841	1	182	182
8	SP03	7/24/97	70842	1	151	151
8	SP03	7/30/97	70858	1	79	79
8	SP03	7/30/97	70859	1	64	64
8	SP03	7/30/97	70860	1	87	87
8	SP03	7/30/97	70861	1	31	31
9	SP01	4/18/97	70311	1	776	776
9	SP03	4/24/97	70322	1	3814	3814
9	SP04	4/24/97	70324	1	4302	4302
9	SP07	4/24/97	70321	1	3686	3686
9	SP03	5/1/97	70363	1	3303	3303
9	SP04	5/1/97	70364	1	4285	4285
9	SP05	5/1/97	70365	1	3861	3861
9	SP07	5/1/97	70366	3.85	3574	13751
9	SP04	5/8/97	70383	1	3462	3462
9	SP05	5/8/97	70384	1	3340	3340
9	SP06	5/8/97	70385	1	3241	3241
9	SP07	5/8/97	70386	1	1721	1721
9	SP03	5/14/97	70401	1	444	444
9	SP04	5/14/97	70402	1	426	426
9	SP06	5/14/97	70403	1	3071	3071
9	SP07	5/14/97	70404	1	355	355

Table 6. Column Benzene Analyses

Column #	Sample Port	Date	ITP Sample #	Dilution Factor	Raw Bz Analysis mg/L	Benzene Corrected for Dilution, mg/L
9	SP08	5/14/97	70405	2.01	2641	5319
9	SP05	5/22/97	70435	1	1514	1514
9	SP06	5/22/97	70431	1	1971	1971
9	SP07	5/22/97	70428	1	336	336
9	SP08	5/22/97	70432	1	642	642
9	SP05	5/28/97	70481	1	1059	1059
9	SP06	5/28/97	70482	1	2895	2895
9	SP07	5/28/97	70483	1	1888	1888
9	SP08	5/28/97	70484	1	1124	1124
9	SP05	6/5/97	70514	1	832	832
9	SP06	6/5/97	70515	1	2516	2516
9	SP07	6/5/97	70516	1	545	545
9	SP08	6/5/97	70517	1	3134	3134
9	SP05	6/12/97	70574	1	392	392
9	SP06	6/12/97	70575	1	502	502
9	SP07	6/12/97	70576	1	271	271
9	SP08	6/12/97	70577	1	3020	3020
9	SP06	6/19/97	70604	1	1788	1788
9	SP01	6/24/97	70593	1	84	84
9	SP01	6/24/97	70594	1	125	125
9	SP01	6/24/97	70595	1	87	87
9	SP01	6/24/97	70596	1	92	92
9	SP01	6/24/97	70597	1	63	63
9	SP01	6/24/97	70598	1	307	307
9	SP01	6/24/97	70599	1	2555	2555
9	SP01	6/24/97	70600	1	726	726
9	SP02	6/24/97	70586	1	66	66
9	SP03	6/24/97	70587	1	1204	1204
9	SP04	6/24/97	70588	1	705	705
9	SP05	6/24/97	70589	1	2586	2586
9	SP07	6/24/97	70591	1	395	395
9	SP08	6/24/97	70592	1	2618	2618
9	SP01	6/26/97	70694	2.00	4511	9021
9	SP01	6/26/97	70695	1	2896	2896
9	SP01	6/26/97	70688	1	1410	1410
9	SP02	6/26/97	70703	1	2741	2741
10	SP01	5/5/97	70367	1	983	983
10	SP04	5/8/97	70387	1	181	181
10	SP05	5/8/97	70388	1	1876	1876
10	SP06	5/8/97	70389	2.45	2062	5047
10	SP07	5/8/97	70390	1.75	2488	4359
10	SP04	5/14/97	70406	1	1031	1031
10	SP05	5/14/97	70407	1	3028	3028
10	SP06	5/14/97	70408	2.1	2859	5989
10	SP07	5/14/97	70409	2.01	2943	5901
10	SP08	5/14/97	70410	2.00	3078	6158
10	SP01	5/22/97	70437	2.10	2302	4828
10	SP06	5/22/97	70422	2.05	3025	6189
10	SP07	5/22/97	70430	2.10	2698	5655
10	SP08	5/22/97	70425	2.03	2896	5870
10	SP02	5/28/97	70485	2.34	2960	6919
10	SP07	5/28/97	70486	2.20	3111	6853
10	SP08	5/28/97	70487	2.11	2844	5994
10	SP05	6/5/97	70518	2.00	2720	5435

Table 6. Column Benzene Analyses

Column #	Sample Port	Date	ITP Sample #	Dilution Factor	Raw Bz Analysis mg/L	Benzene Corrected for Dilution, mg/L
10	SP06	6/5/97	70519	2.00	3020	6046
10	SP07	6/5/97	70520	1.99	3102	6186
10	SP08	6/5/97	70521	2.01	1122	2256
10	SP05	6/12/97	70578	1	2977	2977
10	SP06	6/12/97	70579	1	2465	2465
10	SP07	6/12/97	70580	1	3118	3118
10	SP08	6/12/97	70581	1	3052	3052
10	SP06	6/19/97	70605	1	2364	2364
10	SP07	6/19/97	70606	2.06	2491	5128
10	SP05	6/26/97	70689	2.13	1839	3923
10	SP06	6/26/97	70693	1	2381	2381
10	SP07	6/26/97	70700	2.14	3739	7990
10	SP08	6/26/97	70699	3.37	3175	10697
10	SP05	7/2/97	70683	2.00	3533	7067
10	SP07	7/2/97	70684	1	3659	3659
10	SP08	7/2/97	70686	1.71	2153	3676
10	SP05	7/18/97	70779	1.90	3600	6844
10	SP06	7/18/97	70780	1.79	2674	4799
10	SP07	7/18/97	70781	2.57	1409	3621
10	SP05	7/24/97	70843	1	3869	3869
10	SP06	7/24/97	70844	1	3071	3071
10	SP07	7/24/97	70845	1	2456	2456
10	SP05	8/1/97	70862	1	2067	2067
10	SP06	8/1/97	70863	1	1476	1476
10	SP07	8/1/97	70864	2.43	2920	7090
10	SP05	8/7/97	70889	1	3501	3501
10	SP06	8/7/97	70890	2.15	3906	8384
10	SP07	8/7/97	70891	2.15	3436	7371
10	SP05	8/12/97	70927	1	3492	3492
10	SP05	8/15/97	70928	2.44	4453	10866
10	SP06	8/15/97	70929	2.32	4497	10435
10	SP07	8/15/97	70930	2.52	4265	10758
11	SP01	5/14/97	70411	1	178	178
11	SP01	5/22/97	70424	2.06	517	1067
11	SP02	5/22/97	70433	2.01	731	1472
11	SP07	5/22/97	70420	2.01	559	1121
11	SP08	5/22/97	70429	2.04	404	825
11	SP01	5/28/97	70488	2.07	2715	5624
11	SP02	5/28/97	70489	2.17	2410	5228
11	SP07	5/28/97	70490	2.03	2813	5707
11	SP08	5/28/97	70491	2.02	2773	5591
11	SP01	6/5/97	70522	2.05	5694	11680
11	SP02	6/5/97	70523	2.01	5001	10043
11	SP07	6/5/97	70524	2.01	6199	12438
11	SP08	6/5/97	70525	2.00	6076	12151
11	SP01	6/12/97	70582	1	243	243
11	SP02	6/12/97	70583	1	88	88
11	SP06	6/12/97	70584	1	2504	2504
11	SP07	6/12/97	70585	1	2278	2278
11	SP04	6/19/97	70607	1	460	460
11	SP05	6/19/97	70608	1	638	638
11	SP06	6/19/97	70609	1	450	450
11	SP07	6/19/97	70610	1	708	708
11	SP01	6/26/97	70704	1	255	255

Table 6. Column Benzene Analyses

Column #	Sample Port	Date	ITP Sample #	Dilution Factor	Raw Bz Analysis mg/L	Benzene Corrected for Dilution, mg/L
11	SP02	6/26/97	70701	1	293	293
11	SP06	6/26/97	70691	1	876	876
11	SP07	6/26/97	70690	1	292	292
11	SP02	7/2/97	70678	1	181	181
11	SP05	7/2/97	70681	1	1979	1979
11	SP07	7/2/97	70679	1	541	541
11	SP02	7/18/97	70782	1	108	108
11	SP05	7/18/97	70783	1	2750	2750
11	SP07	7/18/97	70784	1	1530	1530
11	SP02	7/24/97	70846	1	193	193
11	SP05	7/24/97	70847	1	1663	1663
11	SP07	7/24/97	70848	1	888	888
11	SP02	8/1/97	70865	1	153	153
11	SP05	8/1/97	70866	1	577	577
11	SP07	8/1/97	70867	1	716	716
11	SP02	8/7/97	70892	1	190	190
11	SP05	8/7/97	70893	1	583	583
11	SP07	8/7/97	70894	1	1383	1383
11	SP02	8/15/97	70931	1	178	178
11	SP05	8/15/97	70932	1	519	519
11	SP07	8/15/97	70933	1	1587	1587
12	SP03	5/23/97	70527	1	2489	2489
12	SP01	5/28/97	70493	2.12	10	21
12	SP03	5/28/97	70492	2.05	38	78
12	SP01	6/5/97	70526	2.00	303	606
12	PP03	6/19/97	70613	1	2325	2325
12	PP04	6/19/97	70611	1	489	489
12	PP05	6/19/97	70612	1	605	605
12	PP03	6/26/97	70696	1	3562	3562
12	SP01	6/26/97	70698	1	596	596
12	PP03	7/2/97	70675	1	2712	2712
12	PP04	7/2/97	70682	1	606	606
12	PP03	7/18/97	70785	1	399	399
12	PP04	7/18/97	70786	1	441	441
12	PP03	7/24/97	70849	1	1746	1746
12	PP04	7/24/97	70850	1	381	381
12	PP03	8/1/97	70868	1	1390	1390
12	PP04	8/1/97	70869	1	249	249
12	PP03	8/7/97	70895	1	1221	1221
12	PP04	8/7/97	70896	1	385	385
12	PP03	8/15/97	70934	1	1555	1555
12	PP04	8/15/97	70935	1	178	178
13	PP03	6/19/97	70614	1.91	1981	3792
13	PP05	6/19/97	70615	1.89	20	38
13	PP03	6/26/97	70697	1	103	103
13	PP04	6/26/97	70687	1.93	4194	8091
13	PP06	6/26/97	70702	6.25	20	125
13	SP05	6/26/97	70692	2.57	654	1684
13	PP03	7/2/97	70677	1	144	144
13	SP05	7/2/97	70676	1.98	8768	17324
13	PP03	7/18/97	70787	1	132	132
13	PP05	7/18/97	70788	1	4766	4766
13	PP03	7/24/97	70851	1	1143	1143

Table 6. Column Benzene Analyses

Column #	Sample Port	Date	ITP Sample #	Dilution Factor	Raw Bz Analysis mg/L	Benzene Corrected for Dilution, mg/L
13	PP05	7/24/97	70852	1	4898	4898
13	PP03	8/1/97	70870	1	82	82
13	PP05	8/1/97	70871	1	346	346
13	PP03	8/7/97	70897	1	20	20
13	PP04	8/7/97	70898	1	3311	3311
13	PP03	8/15/97	70936	1	79	79
13	PP04	8/15/97	70937	1	582	582

Table 7. Boron Analytical Data

Column 1

Cumulative Column Time, Hrs.	Boron, mg/L
24	40.13
24	36.38
192	40.98
192	39.90
360	51.90
864	58.07
864	61.76
864	77.28
1008	170.90
1008	142.70
1176	145.60
1176	156.20
1176	190.10

Column 2

Cumulative Column Time, Hrs.	Boron, mg/L
0	34.68
0	35.53
72	39.03
96	38.98
144	53.53
504	57.57
504	63.81
504	79.12
624	97.89
624	101.50
624	122.00
816	149.80
816	180.60
816	277.10

Column 3

Cumulative Column Time, Hrs.	Boron, mg/L
0	33.77
0	35.19
0	34.05
312	38.81
312	40.57
432	49.51
432	41.31
432	33.22
624	116.70
624	118.10
624	217.80

Column 4

Cumulative Column Time, Hrs.	Boron, mg/L
0	30.61
0	32.04
0	33.87
144	41.20
168	36.43
216	38.34
216	38.48
216	39.09
336	85.63
528	102.35
528	114.40
528	118.20

Table 7. Boron Analytical Data (Cont'd)

Column 6

Cumulative Column Time, Hrs.	Boron, mg/L
0	29.04
120	61.40
288	140.97
456	175.60
624	338.70
960	447.30
1104	411.50
1296	443.50
1380	418.5
1860	461.2
2916	387

Column 7

Cumulative Column Time, Hrs.	Boron, mg/L
0	228.80
120	294.88
288	274.22
624	587.30
960	872.30
1104	728.00
1296	476.7
1464	487.6
1632	435.1
1944	539.2
3000	514.7

Column 8

Cumulative Column Time, Hrs.	Boron, mg/L
0	108.00
48	165.01
216	288.39
384	334.70
552	423.40
696	495.30
888	318.7
1032	506.80
1224	539.70
1392	578.20
1560	538.6
1872	611
2928	562.5

Column 9

Cumulative Column Time, Hrs.	Boron, mg/L
0	78.09
144	110.80
312	637.00
480	748.30

Table 7. Boron Analytical Data (Cont'd)

Column 10

Cumulative Column Time, Hrs.	Boron, mg/L
72	195.30
144	270.30
216	385.10
552	274.20
744	365.70
912	374.60
1080	397.90
1248	509.30
1392	403.40
1704	336
1920	325
2040	303.3
2184	339.7
2376	357.7
2376	332.7

Column 11

Cumulative Column Time, Hrs.	Boron, mg/L
0	93.70
192	109.60
528	310.70
696	390.30
864	283.50
864	292.30
1032	389.90
1176	355.20
1320	378.30
1704	360.6
1896	344.4
2040	389.1
2232	407.3

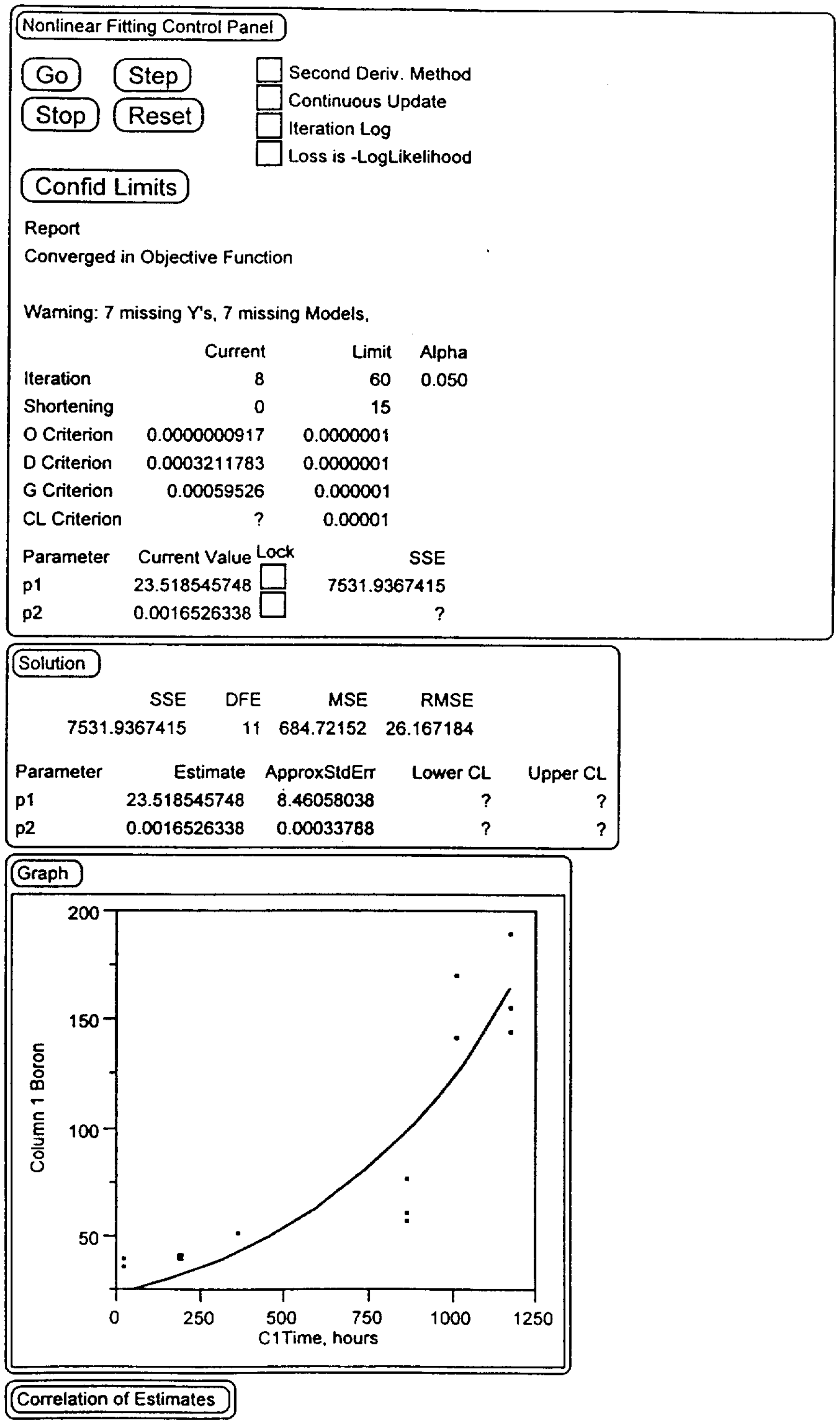
Column 12

Cumulative Column Time, Hrs.	TPB, mg/L*
0	13,349
312	3,646
480	12,982
648	518
648	498
816	3,512
816	3,434
960	5,968
960	5,520
1344	357
1344	2,833
1488	566
1488	1,995
1680	233
1680	1,118
2016	9
2016	91

* At 0.2M [Na], all excess TPB was soluble. Hence, the benzene generation was inferred from the change in [TPB].

Column 13

Cumulative Column Time, Hrs.	Boron, mg/L
0	62.06
168	188.10
312	341.30
696	322.60
840	313.30
1032	322.00
1176	441.60
1368	525.00



☐ Second Deriv. Method
☐ Continuous Update
☐ Iteration Log
☐ Loss is -LogLikelihood

Report
Converged in Objective Function

Warning: 7 missing Y's, 7 missing Models,

	Current	Limit	Alpha
Iteration	8	60	0.050
Shortening	0	15	
O Criterion	0.0000000917	0.0000001	
D Criterion	0.0003211783	0.0000001	
G Criterion	0.00059526	0.000001	
CL Criterion	?	0.00001	

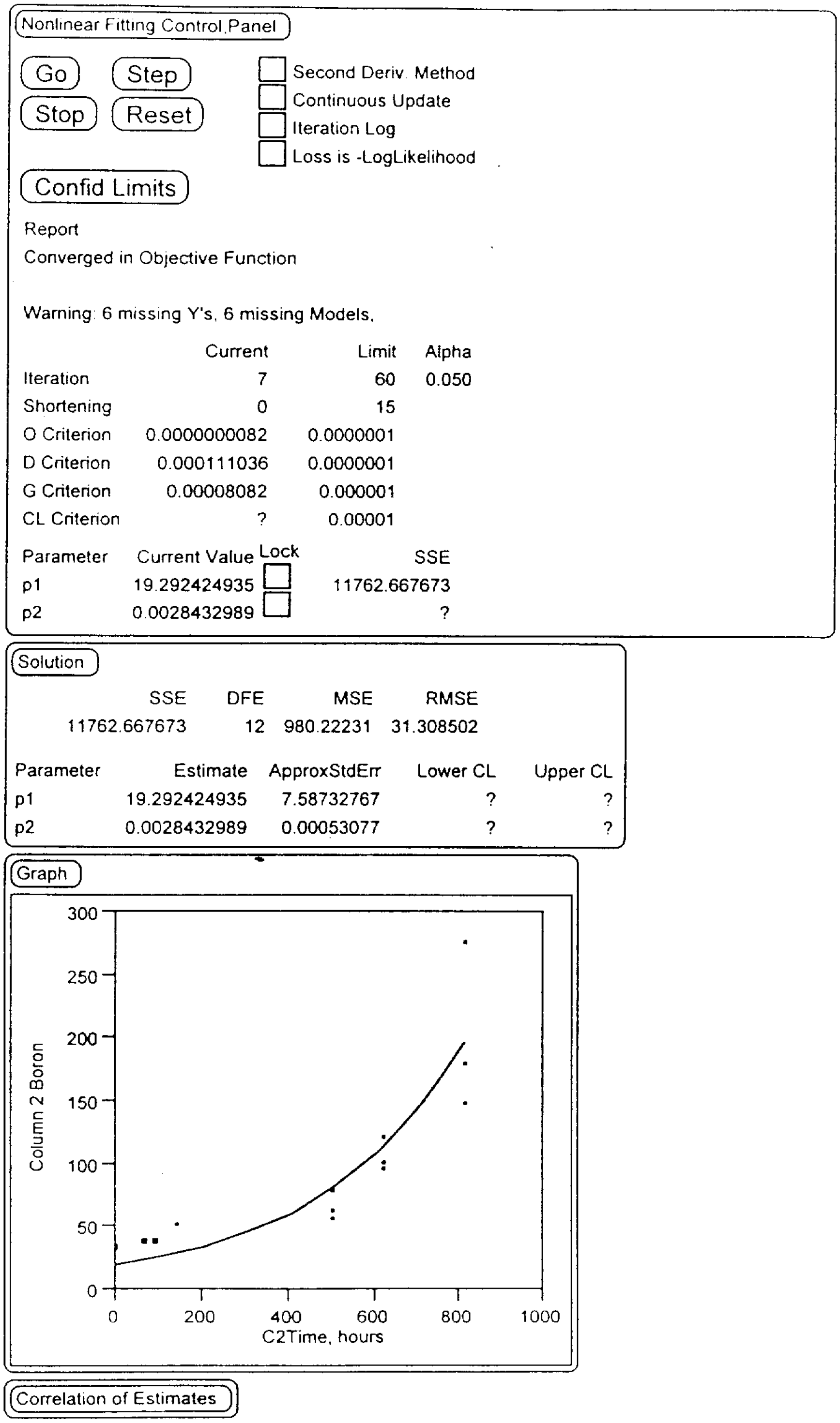
Parameter	Current Value	Lock	SSE
p1	23.518545748	☐	7531.9367415
p2	0.0016526338	☐	?

Solution

SSE	DFE	MSE	RMSE
7531.9367415	11	684.72152	26.167184

Parameter	Estimate	ApproxStdErr	Lower CL	Upper CL
p1	23.518545748	8.46058038	?	?
p2	0.0016526338	0.00033788	?	?

Figure 45. Rate of Boron Increase for Column 1



☐ Second Deriv. Method
☐ Continuous Update
☐ Iteration Log
☐ Loss is -LogLikelihood

Report
Converged in Objective Function

Warning: 6 missing Y's, 6 missing Models,

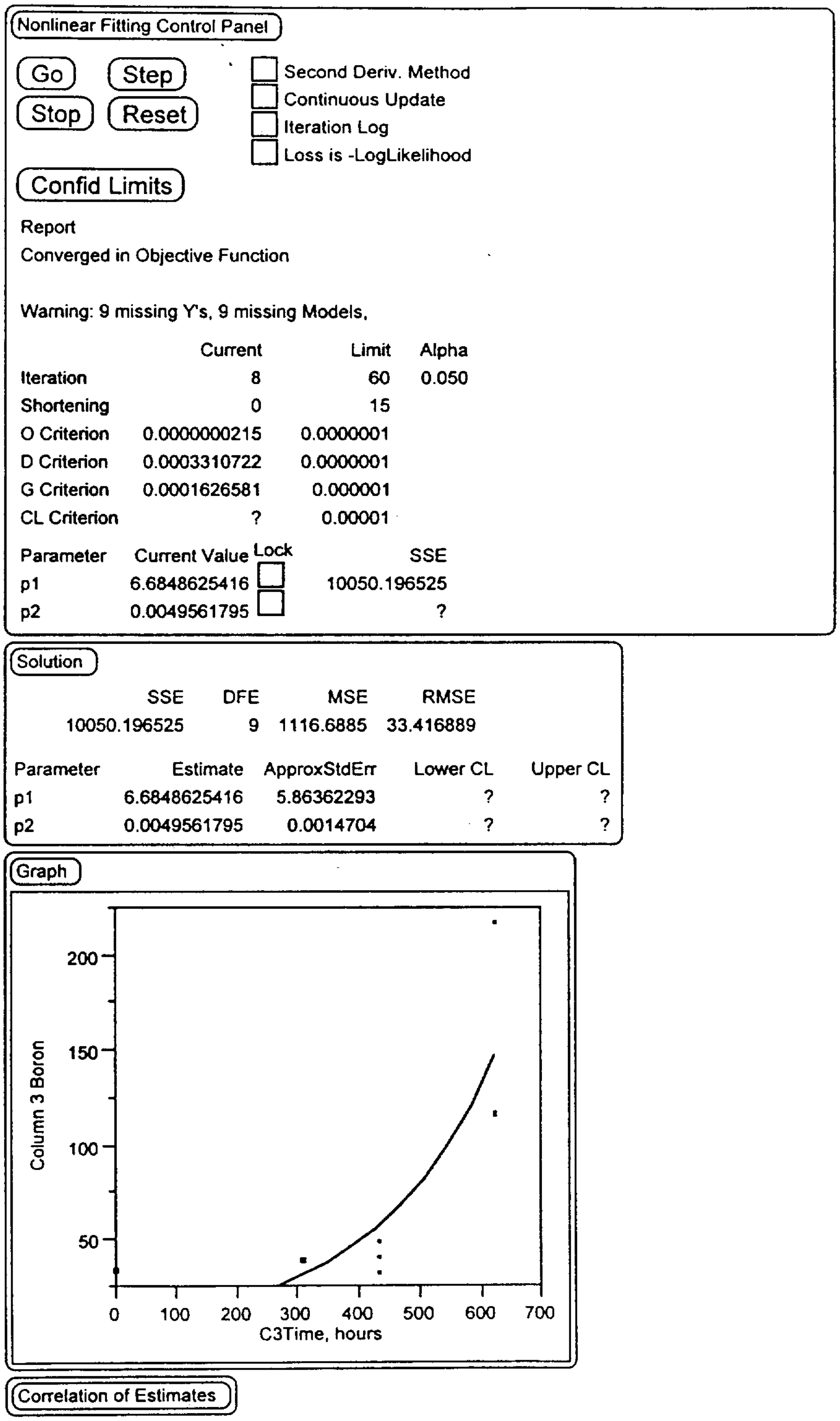
	Current	Limit	Alpha
Iteration	7	60	0.050
Shortening	0	15	
O Criterion	0.0000000082	0.0000001	
D Criterion	0.000111036	0.0000001	
G Criterion	0.00008082	0.000001	
CL Criterion	?	0.00001	

Parameter	Current Value	Lock	SSE
p1	19.292424935	☐	11762.667673
p2	0.0028432989	☐	?

SSE	DFE	MSE	RMSE
11762.667673	12	980.22231	31.308502

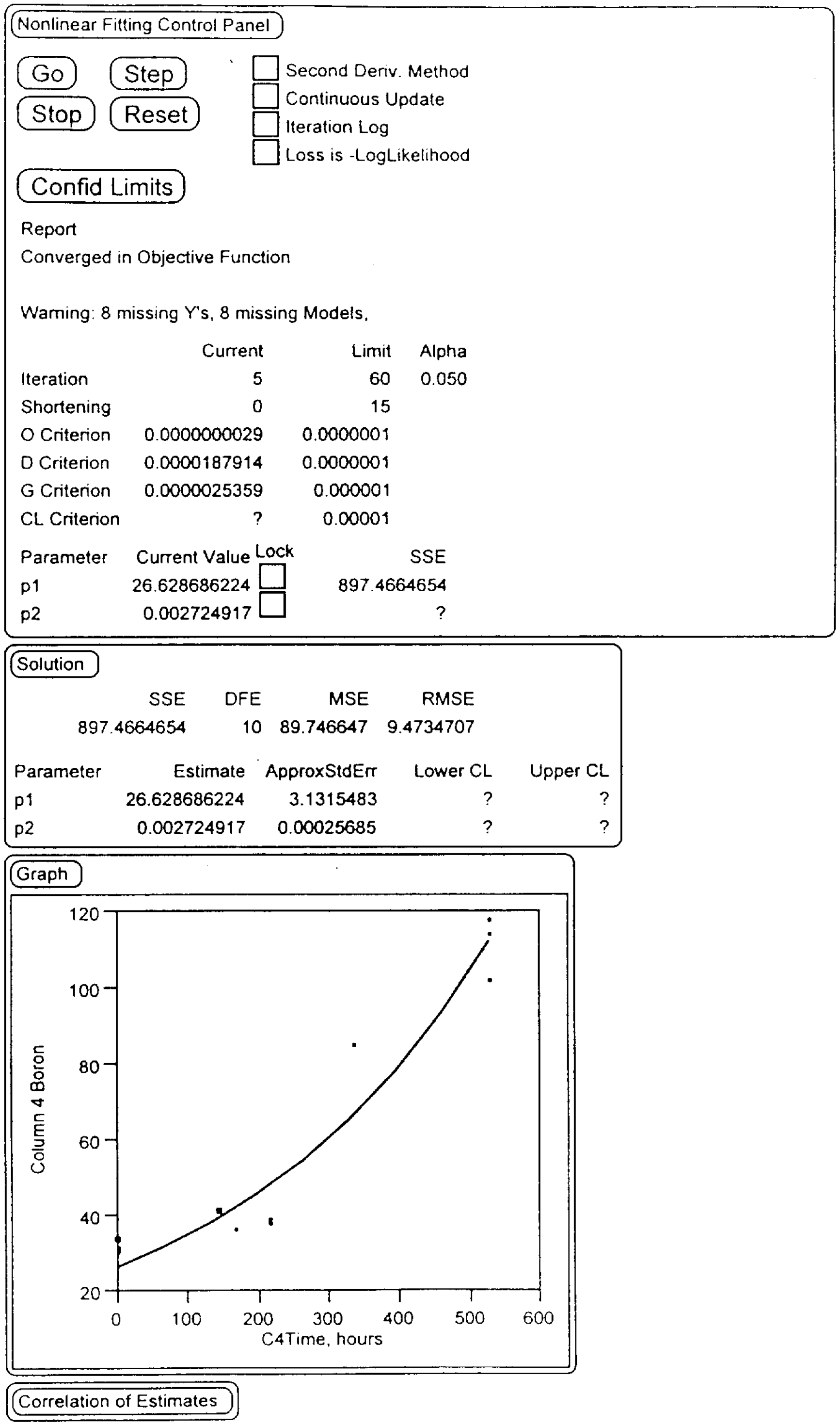
Parameter	Estimate	ApproxStdErr	Lower CL	Upper CL
p1	19.292424935	7.58732767	?	?
p2	0.0028432989	0.00053077	?	?

Figure 46. Rate of Boron Increase for Column 2



```
☐ Second Deriv. Method
☐ Continuous Update
☐ Iteration Log
☐ Loss is -LogLikelihood

Report
Converged in Objective Function

Warning: 9 missing Y's, 9 missing Models.
                     Current       Limit   Alpha
Iteration                  8          60   0.050
Shortening                 0          15
O Criterion     0.0000000215   0.0000001
D Criterion     0.0003310722   0.0000001
G Criterion     0.0001626581    0.000001
CL Criterion               ?     0.00001

Parameter   Current Value  Lock          SSE
p1          6.6848625416    ☐    10050.196525
p2          0.0049561795    ☐               ?

Solution
            SSE     DFE        MSE        RMSE
   10050.196525       9  1116.6885   33.416889

Parameter       Estimate  ApproxStdErr   Lower CL   Upper CL
p1          6.6848625416    5.86362293          ?          ?
p2          0.0049561795     0.0014704          ?          ?
```

Figure 47. Rate of Boron Increase for Column 3



☐ Second Deriv. Method
☐ Continuous Update
☐ Iteration Log
☐ Loss is -LogLikelihood

Report
Converged in Objective Function

Warning: 8 missing Y's, 8 missing Models,

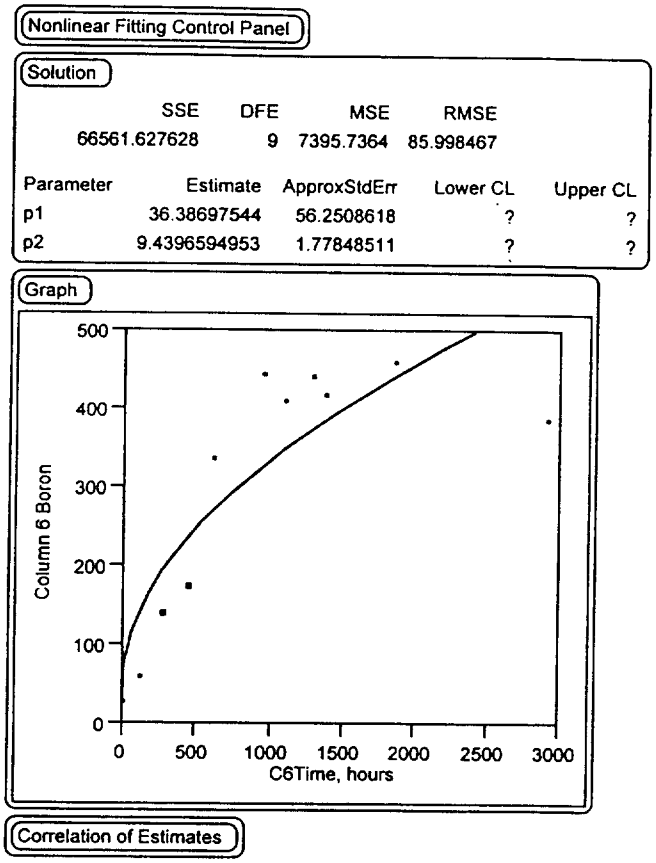
	Current	Limit	Alpha
Iteration	5	60	0.050
Shortening	0	15	
O Criterion	0.0000000029	0.0000001	
D Criterion	0.0000187914	0.0000001	
G Criterion	0.0000025359	0.000001	
CL Criterion	?	0.00001	

Parameter	Current Value	Lock	SSE
p1	26.628686224	☐	897.4664654
p2	0.002724917	☐	?

SSE	DFE	MSE	RMSE
897.4664654	10	89.746647	9.4734707

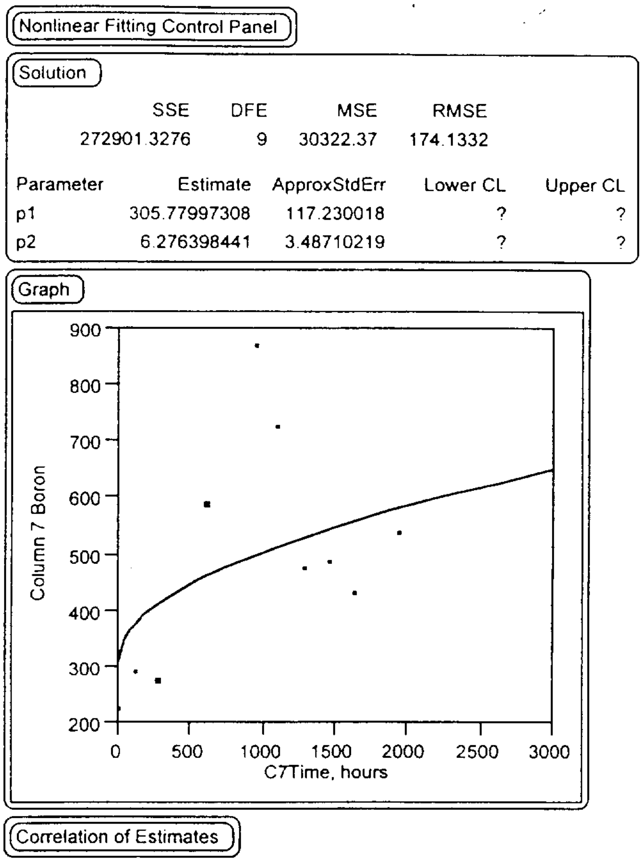
Parameter	Estimate	ApproxStdErr	Lower CL	Upper CL
p1	26.628686224	3.1315483	?	?
p2	0.002724917	0.00025685	?	?

Figure 48. Rate of Boron Increase for Column 4



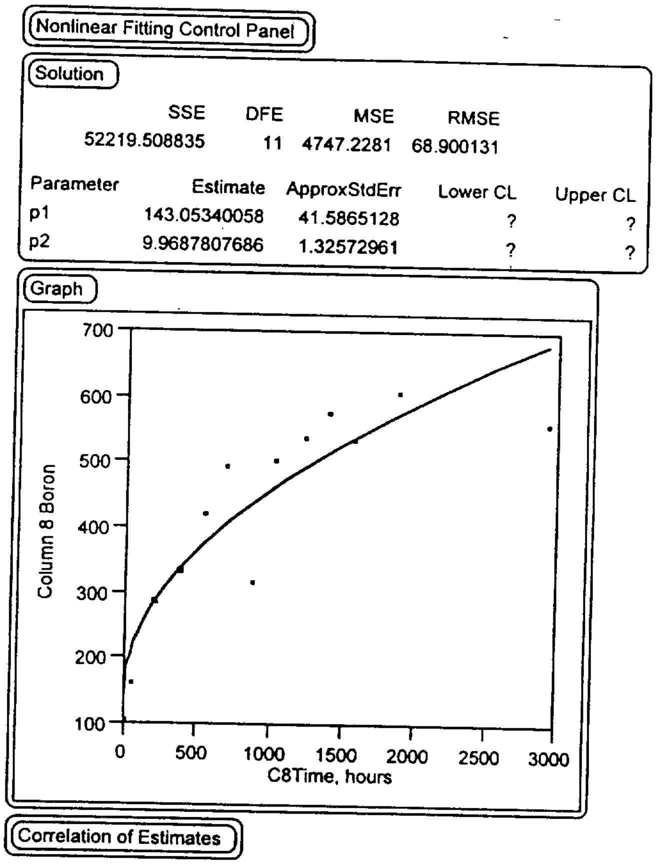
```
            SSE    DFE        MSE       RMSE
   66561.627628      9  7395.7364  85.998467

Parameter        Estimate  ApproxStdErr   Lower CL   Upper CL
p1            36.38697544    56.2508618          ?          ?
p2           9.4396594953    1.77848511          ?          ?
```

Figure 49. Rate of Boron Increase for Column 6



```
            SSE     DFE        MSE       RMSE
    272901.3276       9   30322.37   174.1332

Parameter        Estimate   ApproxStdErr   Lower CL   Upper CL
p1           305.77997308     117.230018          ?          ?
p2            6.276398441     3.48710219          ?          ?
```

Figure 50. Rate of Boron Increase for Column 7



SSE	DFE	MSE	RMSE
52219.508835	11	4747.2281	68.900131

Parameter	Estimate	ApproxStdErr	Lower CL	Upper CL
p1	143.05340058	41.5865128	?	?
p2	9.9687807686	1.32572961	?	?

Figure 51. Rate of Boron Increase for Column 8

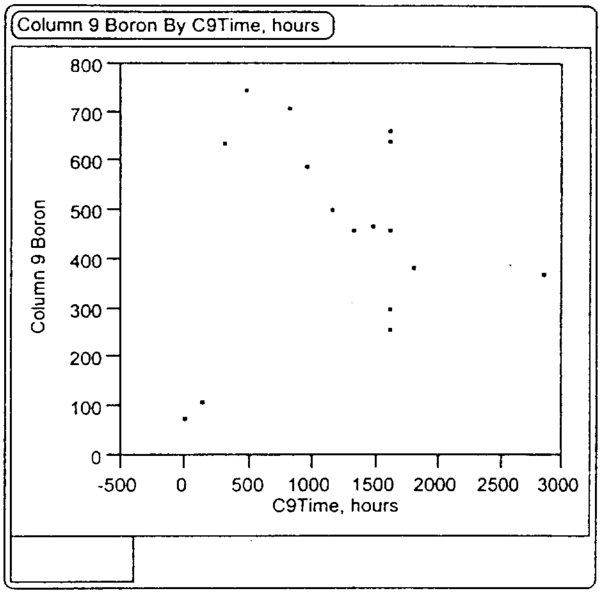
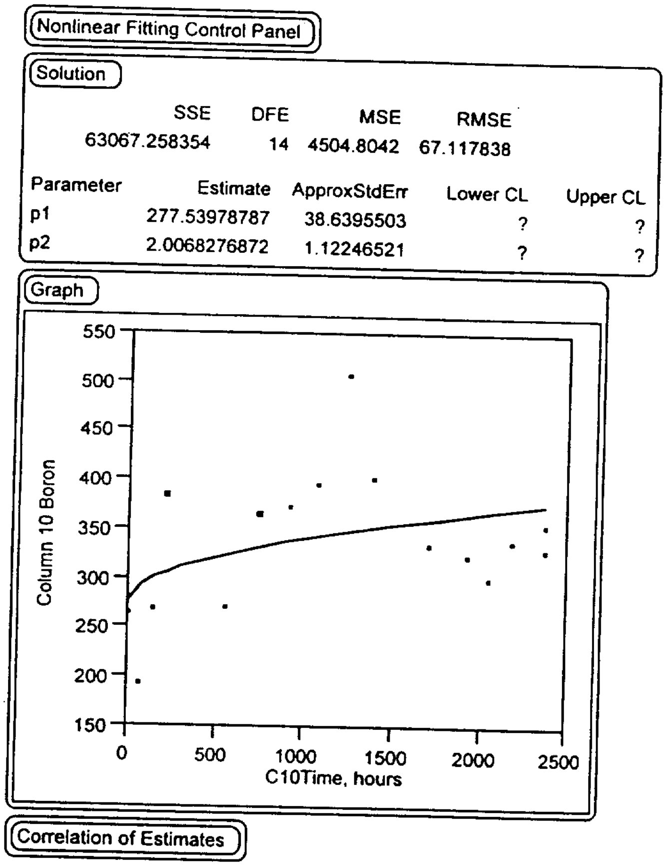


Figure 52. Rate of Boron Increase for Column 9



Nonlinear Fitting Control Panel

Solution

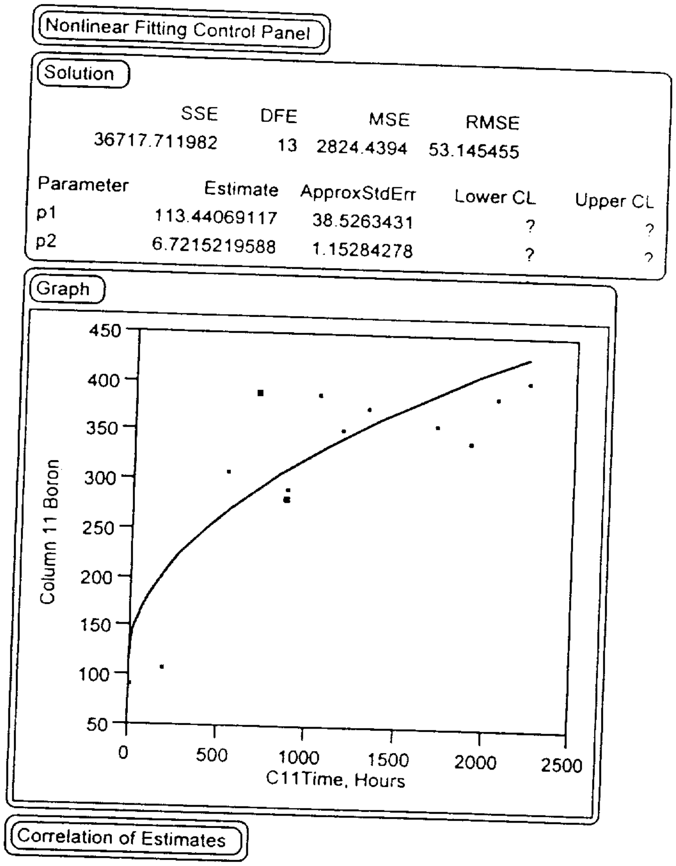
SSE	DFE	MSE	RMSE
63067.258354	14	4504.8042	67.117838

Parameter	Estimate	ApproxStdErr	Lower CL	Upper CL
p1	277.53978787	38.6395503	?	?
p2	2.0068276872	1.12246521	?	?

Graph

Correlation of Estimates

Figure 53. Rate of Boron Increase for Column 10



Nonlinear Fitting Control Panel

Solution

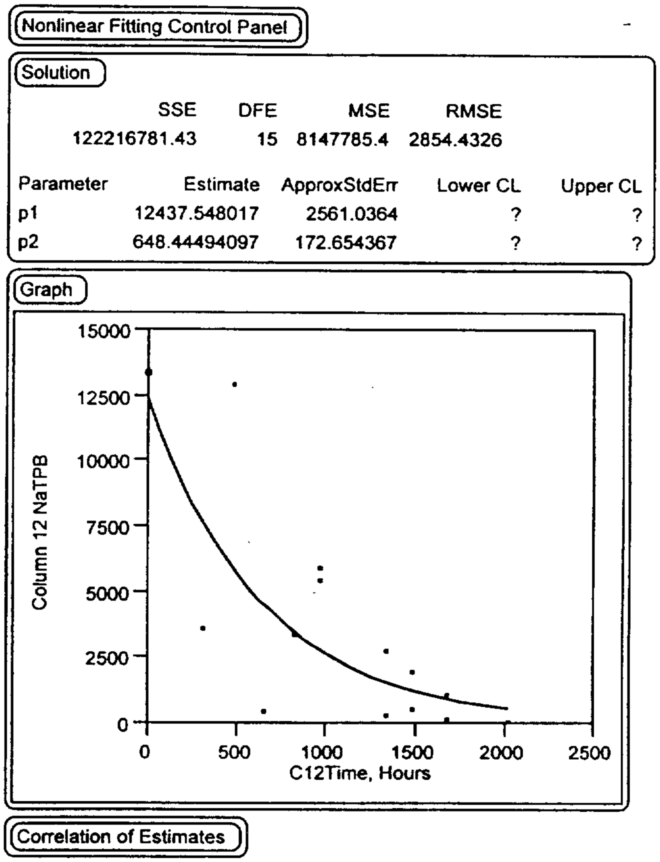
SSE	DFE	MSE	RMSE
36717.711982	13	2824.4394	53.145455

Parameter	Estimate	ApproxStdErr	Lower CL	Upper CL
p1	113.44069117	38.5263431	?	?
p2	6.7215219588	1.15284278	?	?

Graph

Correlation of Estimates

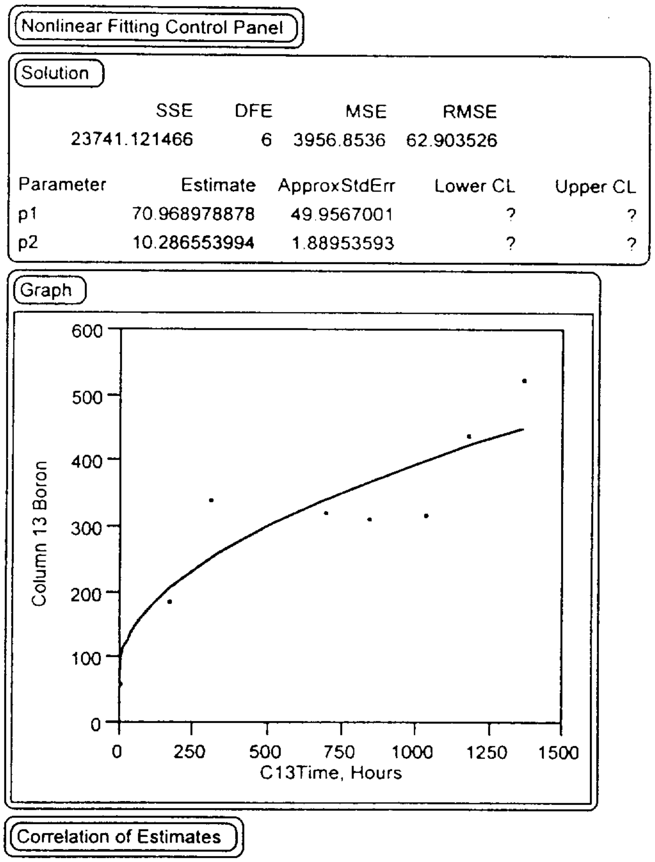
Figure 54. Rate of Boron Increase for Column 11



SSE	DFE	MSE	RMSE
122216781.43	15	8147785.4	2854.4326

Parameter	Estimate	ApproxStdErr	Lower CL	Upper CL
p1	12437.548017	2561.0364	?	?
p2	648.44494097	172.654367	?	?

Figure 55. Rate of TPB$^-$ Decrease for Column 12



```
         SSE     DFE        MSE       RMSE
 23741.121466      6  3956.8536  62.903526

Parameter       Estimate  ApproxStdErr  Lower CL  Upper CL
p1          70.968978878    49.9567001         ?         ?
p2          10.286553994    1.88953593         ?         ?
```

Figure 56. Rate of Boron Increase for Column 13

Table 8. Purge Rate Effect Data

Column	Date	N2 Purge cc/min	Benzene Conc., ppm	RATE g/min	RELEASE g/min/m^2	Benzene Loss Rate, g/day	Hours from Column Start	Benzene Loss Rate, g/hr
Test for Correction Factor of 9/23/97 (50 C):								
C11		22	41770	0.0030835	0.10876	4.411		0.18380
		22	41900	0.0030935	0.10912	4.426		0.18440
		22	41854	0.0030899	0.10899	4.420		0.18418
		22	41606	0.0030708	0.10832	4.393		0.18305
		22	42054	0.0031054	0.10954	4.442		0.18510
		22	41852	0.0030898	0.10899	4.420		0.18418
		22	41741	0.0030812	0.10869	4.408		0.18367
		22	41362	0.0030521	0.10766	4.366		0.18193
		22	41064	0.0030291	0.10685	4.333		0.18056
		22	40684	0.0029999	0.10582	4.292		0.17882
		22	41363	0.0030521	0.10766	4.366		0.18193
		22	41396	0.0030547	0.10775	4.370		0.18208
		22	40790	0.0030081	0.10610	4.303		0.17930
		22	40750	0.0030050	0.10600	4.299		0.17912
		22	40613	0.0029944	0.10562	4.284		0.17849
		22	40845	0.0030123	0.10625	4.309		0.17956
		22	41324	0.0030491	0.10755	4.362		0.18175
Avg @ 22cc/min:		22	41351	0.0031	0.1076	4.365		0.182
		210	7476	0.0050859	0.17940	7.276		0.30316
		210	7495	0.0050990	0.17986	7.294		0.30394
		210	7504	0.0051051	0.18007	7.303		0.30431
		210	7524	0.0051188	0.18056	7.323		0.30512
		210	7532	0.0051243	0.18075	7.331		0.30545
		210	7531	0.0051236	0.18073	7.330		0.30541
		210	7576	0.0051545	0.18182	7.374		0.30725
		210	7593	0.0051661	0.18223	7.391		0.30794
		210	7615	0.0051812	0.18276	7.412		0.30884
		210	7629	0.0051908	0.18310	7.426		0.30941
		210	7646	0.0052025	0.18351	7.443		0.31011
		210	7666	0.0052162	0.18399	7.462		0.31093
		210	7692	0.0052340	0.18462	7.488		0.31199
		210	7704	0.0052422	0.18491	7.499		0.31248
		210	7733	0.0052621	0.18561	7.528		0.31366
Avg @ 210cc/min:		210	7594.4	0.005	0.182	7.39		0.30800
Factor to correct benzene losses/fluxes at 200 cc/min to 20 cc/min:						0.591		

Table 8. Purge Rate Effect Data

Column	Date	N2 Purge cc/min	Benzene Conc., ppm	RATE g/min	RELEASE g/min/m^2	Benzene Loss Rate, g/day	Hours from Column Start	Benzene Loss Rate, g/hr
Test for Correction Factor of 9/19/97 (40 C):								
C11		19	32858	0.0020755	0.07321	2.969		0.12372
		19	33095	0.0020910	0.07376	2.991		0.12464
		19	32802	0.0020719	0.07308	2.964		0.12350
		19	32664	0.0020629	0.07276	2.951		0.12296
		19	32047	0.0020226	0.07134	2.894		0.12056
		19	31724	0.0020015	0.07060	2.863		0.11931
		19	31658	0.0019972	0.07045	2.857		0.11905
		19	31603	0.0019937	0.07032	2.852		0.11884
Avg @ 19cc/min:		19	31269	0.002040	0.071941	2.91774		0.11754
		200	5456	0.0035278	0.12444	5.047		0.21028
		200	5434	0.0035135	0.12393	5.026		0.20943
		200	5426	0.0035083	0.12375	5.019		0.20912
		200	5444	0.0035200	0.12416	5.036		0.20982
		200	5407	0.0034959	0.12331	5.001		0.20839
		200	5392	0.0034862	0.12297	4.987		0.20780
		200	5383	0.0034803	0.12276	4.979		0.20746
		200	5387	0.0034829	0.12285	4.983		0.20761
		200	5371	0.0034725	0.12249	4.968		0.20699
		200	5361	0.0034660	0.12226	4.958		0.20660
		200	5344	0.0034550	0.12187	4.943		0.20594
Avg @ 200cc/min:		200	5400	0.0035	0.123	4.995		0.20813

Factor to correct benzene losses/fluxes at 200 cc/min to 20 cc/min: 0.584

Table 9. Quiescent Release Data

Column	Date	N2 Purge cc/min	Benzene Conc., ppm	RATE g/min	RELEASE g/min/m^2	Benzene Loss Rate, g/day	Hours from Column Start	Benzene Loss Rate, g/hr	Corrected Bz Loss Rate, g/hr	Hours from Column Start	Bz Generation Rate, g/hr
1	2/10/97	150	11	0.0000053	0.00019	0.008	144	0.00032	0.00019	24.00	0.05
1	2/13/97	150	12	0.0000058	0.00020	0.008	216	0.00034	0.00021	192.00	0.07
1	2/20/97	150	42	0.0000203	0.00071	0.029	384	0.00121	0.00072	360.00	0.09
1	2/24/97	150	43	0.0000207	0.00073	0.030	480	0.00124	0.00074	864.00	0.21
1	2/25/97	150	47	0.0000227	0.00080	0.032	504	0.00135	0.00081	1,008.00	0.26
1	2/26/97	150	57	0.0000275	0.00097	0.039	528	0.00164	0.00098	1,176.00	0.34
1	3/3/97	150	53	0.0000256	0.00090	0.037	648	0.00152	0.00091		
1	3/6/97	150	58	0.0000280	0.00099	0.040	720	0.00167	0.00100		
1	3/8/97	150	87	0.0000420	0.00148	0.060	768	0.00250	0.00150		
1	3/19/97	150	191	0.0000921	0.00325	0.132	1032	0.00549	0.00330		
1	3/20/97	150	188	0.0000907	0.00320	0.130	1056	0.00541	0.00324		
1	3/25/97	150	287	0.0001385	0.00488	0.198	1176	0.00825	0.00495		
1	3/25/97	150	271	0.0001307	0.00461	0.187	1176	0.00779	0.00468		
1	3/31/97	150	308	0.0001486	0.00524	0.213	1320	0.00886	0.00531		
2	2/24/97	150	103	0.0000497	0.00175	0.071	96	0.00296	0.00178	72.00	0.09
2	2/25/97	150	195	0.0000941	0.00332	0.135	120	0.00561	0.00336	96.00	0.09
2	2/26/97	150	334	0.0001611	0.00568	0.231	144	0.00961	0.00576	144.00	0.10
2	3/3/97	150	167	0.0000806	0.00284	0.115	264	0.00480	0.00288	504.00	0.29
2	3/6/97	150	100	0.0000482	0.00170	0.069	336	0.00288	0.00173	624.00	0.41
2	3/8/97	150	96	0.0000463	0.00163	0.066	384	0.00276	0.00166	816.00	0.71
2	3/19/97	150	159	0.0000767	0.00271	0.110	648	0.00457	0.00274		
2	3/20/97	150	144	0.0000695	0.00245	0.099	672	0.00414	0.00248		
2	3/25/97	150	148	0.0000714	0.00252	0.102	792	0.00426	0.00255		
2	3/25/97	150	194	0.0000936	0.00330	0.134	792	0.00558	0.00335		
2	3/31/97	150	231	0.0001114	0.00393	0.159	936	0.00664	0.00399		
3	3/3/97	150	179	0.0000863	0.00305	0.124	72	0.00515	0.00309	0.00	0.04
3	3/6/97	150	59	0.0000285	0.00100	0.041	144	0.00170	0.00102	312.00	0.20
3	3/8/97	150	102	0.0000492	0.00174	0.070	192	0.00293	0.00176	432.00	0.36
3	3/19/97	150	178	0.0000859	0.00303	0.123	456	0.00512	0.00307	624.00	0.93
3	3/20/97	150	182	0.0000878	0.00310	0.126	480	0.00523	0.00314		
3	3/25/97	150	249	0.0001201	0.00424	0.172	600	0.00716	0.00430		
3	3/25/97	150	246	0.0001187	0.00419	0.170	600	0.00707	0.00424		
3	3/31/97	150	491	0.0002369	0.00836	0.339	744	0.01412	0.00847		
4	3/6/97	150	110	0.0000531	0.00187	0.076	48	0.00316	0.00190	0.00	0.09
4	3/8/97	150	287	0.0001385	0.00488	0.198	96	0.00825	0.00495	144.00	0.14
4	3/19/97	150	122	0.0000588	0.00208	0.084	360	0.00351	0.00210	168.00	0.15
4	3/20/97	150	103	0.0000497	0.00175	0.071	384	0.00296	0.00178	216.00	0.17
4	3/25/97	150	94	0.0000453	0.00160	0.065	504	0.00270	0.00162	336.00	0.23
4	3/25/97	150	96	0.0000463	0.00163	0.066	504	0.00276	0.00166	528.00	0.39
4	3/31/97	150	115	0.0000555	0.00196	0.079	648	0.00331	0.00198		
6	4/22/97	150	931	0.000449	0.016	0.643	240	0.02679	0.01607	0.00	0.55
6	4/24/97	160	4539	0.002346	0.083	3.356	288	0.13982	0.08389	120.00	0.35
6	4/28/97	130	10237	0.004323	0.152	6.185	384	0.25770	0.15462	288.00	0.28
6	4/30/97	130	13467	0.005706	0.201	8.163	432	0.34012	0.20407	456.00	0.24
6	5/5/97	160	14035	0.007323	0.258	10.476	552	0.43651	0.26191	624.00	0.19
6	5/9/97	200	4145	0.002677	0.094	3.829	648	0.15955	0.09573	960.00	0.18
6	5/14/97	145	5392	0.002527	0.089	3.616	768	0.15066	0.09039	1,104.00	0.17
6	5/16/97	200	6109	0.003953	0.139	5.655	816	0.23561	0.14136	1,296.00	0.16
6	5/20/97	180	6271	0.003652	0.129	5.225	912	0.21770	0.13062	1,380.00	0.14
6	5/23/97	210	5716	0.003882	0.137	5.553	984	0.23138	0.13883	1,860.00	0.11
6	6/2/97	215	4619	0.003208	0.113	4.589	1224	0.19122	0.11473		
6	6/5/97	180	10332	0.006042	0.213	8.644	1296	0.36016	0.21609		
6	6/12/97	200	3373	0.002176	0.077	3.114	1464	0.12973	0.07784		
6	6/16/97	215	1901	0.001317	0.046	1.884	1560	0.07848	0.04709		
6	6/18/97	205	1936	0.001279	0.045	1.829	1608	0.07621	0.04573		
6	6/19/97	200	1687	0.001087	0.038	1.555	1632	0.06477	0.03886		
6	6/27/97	210	1642	0.001111	0.039	1.589	1824	0.06620	0.03972		
6	7/2/97	220	2021	0.001432	0.051	2.049	1944	0.08539	0.05123		
6	7/10/97	200	2158	0.001391	0.049	1.990	2136	0.08290	0.04974		

Table 9. Quiescent Release Data

Column	Date	N2 Purge cc/min	Benzene Conc., ppm	RATE g/min	RELEASE g/min/m^2	Benzene Loss Rate, g/day	Hours from Column Start	Benzene Loss Rate, g/hr	Corrected Bz Loss Rate, g/hr	Hours from Column Start	Bz Generation Rate, g/hr
6	7/17/97	190	1835	0.001123	0.040	1.607	2304	0.06694	0.04017		
6	7/18/97	250	1264	0.001017	0.036	1.455	2328	0.06064	0.03638		
6	7/25/97	200	1439	0.000927	0.033	1.326	2496	0.05524	0.03314		
6	7/28/97	220	1117	0.000791	0.028	1.132	2568	0.04715	0.02829		
6	8/11/97	290	522	0.000487	0.017	0.697	2904	0.02903	0.01742		
7	4/22/97	150	3547	0.001717	0.061	2.456	240	0.10233	0.06140	120.00	0.36
7	4/24/97	150	3143	0.001521	0.054	2.175	288	0.09064	0.05439	288.00	0.24
7	4/28/97	125	3482	0.001404	0.050	2.009	384	0.08371	0.05023	624.00	0.16
7	4/30/97	140	9153	0.004158	0.147	5.949	432	0.24786	0.14872	960.00	0.13
7	5/5/97	180	5095	0.002964	0.105	4.240	552	0.17667	0.10600	1,104.00	0.12
7	5/9/97	190	12128	0.007500	0.265	10.729	648	0.44706	0.26824	1,296.00	0.11
7	5/14/97	190	6054	0.003721	0.131	5.323	768	0.22180	0.13308	1,464.00	0.10
7	5/16/97	180	4952	0.002880	0.102	4.120	816	0.17169	0.10301	1,632.00	0.10
7	5/20/97	175	1279	0.000721	0.025	1.031	912	0.04295	0.02577	1,944.00	0.09
7	5/23/97	230	4083	0.003032	0.107	4.337	984	0.18072	0.10843	3,000.00	0.07
7	6/2/97	220	2321	0.001646	0.058	2.354	1224	0.09809	0.05886		
7	6/5/97	185	4040	0.002413	0.085	3.452	1296	0.14383	0.08630		
7	6/12/97	200	3043	0.001963	0.069	2.808	1464	0.11700	0.07020		
7	6/16/97	220	1612	0.001142	0.040	1.634	1560	0.06808	0.04085		
7	6/18/97	220	1320	0.000935	0.033	1.338	1608	0.05573	0.03344		
7	6/19/97	200	1765	0.001137	0.040	1.627	1632	0.06777	0.04066		
7	6/27/97	230	522	0.000386	0.014	0.553	1824	0.02302	0.01381		
7	7/2/97	200	1539	0.000991	0.035	1.418	1944	0.05908	0.03545		
7	7/10/97	200	757	0.000487	0.017	0.697	2136	0.02904	0.01742		
7	7/18/97	250	662	0.000532	0.019	0.762	2328	0.03174	0.01904		
7	7/25/97	190	418	0.000255	0.009	0.365	2496	0.01523	0.00914		
7	7/28/97	220	421	0.000298	0.011	0.426	2568	0.01776	0.01066		
7	8/11/97	230	558	0.000413	0.015	0.591	2904	0.02461	0.01477		
8	4/22/97	150	11170	0.005448	0.192	7.794	168	0.32475	0.19485	48.00	0.91
8	4/24/97	150	30409	0.015126	0.534	21.639	216	0.90164	0.54098	216.00	0.43
8	4/28/97	130	19202	0.008183	0.289	11.707	312	0.48779	0.29268	384.00	0.32
8	4/30/97	120	20989	0.008272	0.292	11.834	360	0.49307	0.29584	552.00	0.27
8	5/5/97	140	22108	0.010177	0.359	14.559	480	0.60661	0.36397	696.00	0.24
8	5/9/97	190	8357	0.005148	0.182	7.365	576	0.30688	0.18413	888.00	0.21
8	5/14/97	140	10128	0.004606	0.162	6.589	696	0.27454	0.16472	1,032.00	0.20
8	5/16/97	180	7323	0.004269	0.151	6.108	744	0.25450	0.15270	1,224.00	0.18
8	5/20/97	180	8741	0.005104	0.180	7.301	840	0.30421	0.18253	1,392.00	0.17
8	5/23/97	190	7713	0.004749	0.167	6.793	912	0.28305	0.16983	1,560.00	0.16
8	6/2/97	200	5142	0.003324	0.117	4.755	1152	0.19812	0.11887	1,872.00	0.15
8	6/5/97	195	4022	0.002532	0.089	3.622	1224	0.15092	0.09055	2,928.00	0.12
8	6/12/97	205	3709	0.002454	0.087	3.510	1392	0.14627	0.08776		
8	6/16/97	210	3463	0.002346	0.083	3.357	1488	0.13986	0.08392		
8	6/18/97	220	3434	0.002437	0.086	3.487	1536	0.14529	0.08718		
8	6/19/97	195	3224	0.002028	0.072	2.901	1560	0.12088	0.07253		
8	6/27/97	220	2648	0.001878	0.066	2.687	1752	0.11195	0.06717		
8	7/2/97	210	2049	0.001386	0.049	1.983	1872	0.08264	0.04958		
8	7/10/97	220	1923	0.001363	0.048	1.950	2064	0.08124	0.04874		
8	7/18/97	200	1744	0.001123	0.040	1.607	2256	0.06697	0.04018		
8	7/25/97	200	442	0.000284	0.010	0.407	2424	0.01695	0.01017		
8	7/28/97	195	539	0.000338	0.012	0.484	2496	0.02016	0.01209		
8	8/11/97	225	141	0.000102	0.004	0.146	2832	0.00608	0.00365		
9	4/22/97	150	2045	0.000988	0.035	1.414	96	0.05891	0.03535		
9	4/24/97	150	2732	0.001321	0.047	1.890	144	0.07876	0.04725		
9	4/28/97	140	1002	0.000451	0.016	0.646	240	0.02691	0.01615		
9	4/30/97	130	1136	0.000475	0.017	0.680	288	0.02834	0.01700		
9	5/9/97	200	2729	0.001760	0.062	2.517	504	0.10489	0.06294		
9	5/14/97	190	1146	0.000701	0.025	1.003	624	0.04178	0.02507		
9	5/16/97	200	2453	0.001581	0.056	2.262	672	0.09426	0.05656		
9	5/20/97	180	1010	0.000585	0.021	0.837	768	0.03488	0.02093		
9	5/23/97	200	3850	0.002485	0.088	3.556	840	0.14815	0.08889		

Table 9. Quiescent Release Data

Column	Date	N2 Purge cc/min	Benzene Conc., ppm	RATE g/min	RELEASE g/min/m^2	Benzene Loss Rate, g/day	Hours from Column Start	Benzene Loss Rate, g/hr	Corrected Bz Loss Rate, g/hr	Hours from Column Start	Bz Generation Rate, g/hr
13	6/19/97	220	917	0.000649	0.023	0.929	0	0.03870	0.02322	168.00	0.50
13	6/27/97	240	775	0.000599	0.021	0.856	192	0.03568	0.02141	312.00	0.37
13	7/2/97	220	793	0.000561	0.020	0.803	312	0.03346	0.02008	696.00	0.25
13	7/10/97	220	655	0.000464	0.016	0.663	504	0.02764	0.01658	840.00	0.23
13	7/18/97	270	2145	0.001866	0.066	2.670	696	0.11124	0.06674	1,032.00	0.20
13	7/25/97	215	1066	0.000738	0.026	1.055	864	0.04397	0.02638	1,176.00	0.19
13	7/28/97	220	750	0.000531	0.019	0.760	936	0.03165	0.01899	1,368.00	0.18
13	8/11/97	240	1015	0.000784	0.028	1.122	1272	0.04674	0.02804		

Table 10. Potential Benzene Inventory Calculations

Column Number	1	2	3	4	6	7 (adj #2)	8 (adj #4)	9	10	11	12	13
NaTPB Added, moles	7.85	7.85	3.55	3.55	3.55	7.85	3.55	7.85	7.85	7.85	6.755	3.92
K added, moles	5.740	5.740	1.443	1.443	1.443	5.740	1.443	5.740	5.740	5.740	4.965	2.870
Cs added, moles	0.00784	0.00788	0.00787	0.00786	0.00787	0.00788	0.00786	0.00786	0.00786	0.00785	0.00684	0.00394
Excess NaTPB, moles	2.102	2.102	2.099	2.099	2.099	2.102	2.099	2.102	2.103	2.102	1.783	1.046
Moles Benzene Equivalent	8.409	8.409	8.396	8.396	8.396	8.409	8.396	8.409	8.410	8.409	7.133	4.185
Benzene Added, g	31.319	31.307	31.298	31.3022	31.322	31.307	31.3022	31.307	31.3	31.3	31.2978	15.6
Moles Benzene Equivalent	0.4010	0.4008	0.4007	0.4007	0.4010	0.4008	0.4007	0.4008	0.4007	0.4007	0.4007	0.1997
1-PB added, g.	5.4255	5.408	5.4053	5.4258	5.4231	10.8112	10.8285	5.4053	5.3983	5.4025	5.3994	2.7
Moles Benzene Equivalent	0.0445	0.0444	0.0443	0.0445	0.0445	0.0887	0.0888	0.0443	0.0443	0.0443	0.0443	0.0221
2-PB sol'n added, g.	6.7198	6.7028	6.7117	6.7198	6.7216	13.4027	13.4224	6.703	6.7	6.7	6.7	3.4
Moles Benzene Equivalent	0.0597	0.0596	0.0596	0.0597	0.0597	0.1191	0.1193	0.0596	0.0595	0.0595	0.0595	0.0302
3-PB sol'n added, g.	70.301	70.3085	70.3027	70.3226	70.3159	140.609	140.628	70.305	70.2998	70.3026	70.2959	35.1
Moles Benzene Equivalent	0.0673	0.0673	0.0673	0.0673	0.0673	0.1346	0.1346	0.0673	0.0673	0.0673	0.0673	0.0336
Additional Benzene Added	0	0	0	0	0	31.3	31.3055	0	0	0	0	0
Moles Benzene Equivalent	0	0	0	0	0	0.40072	0.40079	0	0	0	0	0
Additional NaTPB sol'n Added, g.	0	0	0	0	0	1040	1040	0	0	0	0	0
Moles Benzene Equivalent	0	0	0	0	0	2.32	2.32	0	0	0	0	0
Net Moles Benzene Equivalent	8.982	8.981	8.968	8.968	8.969	11.873	11.860	8.981	8.982	8.981	7.705	4.470
Net Mass Benzene Potential, g.	702	702	700	701	701	927	926	702	702	702	602	349

Table 11. Actual Benzene Formed Calculations

		Units
Nominal Column volume	44.00	liters
Boron	10810.00	mg/gmole
4PB	34223.00	mg/gmole
3PB	243190.00	mg/gmole
2PB	171090.00	mg/gmole
1PB	121930.00	mg/gmole
C6H6	78.11	g/gmole

	Col. 1	Col.2	Col.3	Col.4	Col.6	Col.7	Col.8	Col.9	Col.10	Col.11	Col. 12	Col.13
Initial concentrations												
Date	2/5/97	2/20/97	2/28/97	2/13/97	4/11/97	4/12/97	4/15/97	4/18/97	5/8/97	5/22/97	5/23/97	6/19/97
Soluble Boron, mg/L	38.25	35.10	34.35	38.48	158.50	228.80	108.00	78.09	231.00	93.70	527.70	62.06
4PB, mg/L	10.00	10.00	116.00	123.00	68.00	91.00	168.00	164.00	48.00	1120.00	13349.00	118.00
3PB, mg/L	10.00	10.00	221.00	346.00	219.00	1774.00	1124.00	194.00	937.00	336.00	458.00	429.00
2PB, mg/L	72.00	65.00	100.00	42.00	75.00	589.00	225.00	78.00	870.00	68.00	130.00	69.00
1PB, mg/L	111.00	114.00	103.00	96.00	106.00	481.00	233.00					
Moles boron	0.16	0.14	0.14	0.16	0.65	0.93	0.44	0.32	0.94	0.38	2.15	0.25
Moles 4PB	0.01	0.01	0.15	0.16	0.09	0.12	0.22	0.21	0.06	1.44	17.16	0.08
Moles 3PB	0.00	0.00	0.04	0.06	0.04	0.32	0.20	0.04	0.17	0.06	0.08	0.04
Moles 2PB	0.02	0.02	0.03	0.01	0.02	0.15	0.06	0.02	0.22	0.02	0.03	0.01
Moles 1PB	0.04	0.04	0.04	0.03	0.04	0.17	0.08	0.00	0.00	0.00	0.00	0.00
Equivalent moles benzene												
from Boron (Boric acid)	0.33	0.28	0*	0*	1.84	0.67	0*	0.21	1.94	0*	0*	0.52
from 3PB	0.00	0.00	0.04	0.06	0.04	0.32	0.20	0.04	0.17	0.06	0.08	0.04
from 2PB	0.04	0.03	0.05	0.02	0.04	0.30	0.12	0.04	0.45	0.03	0.07	0.02
from 1PB	0.12	0.12	0.11	0.10	0.11	0.52	0.25	0.00	0.00	0.00	0.00	0.00
Total	0.49	0.44	0.20	0.19	2.04	1.82	0.57	0.28	2.56	0.10	0.15	0.57
Final concentrations												
Date	3/26/97	3/26/97	3/26/97	3/26/97	8/15/97	8/15/97	8/15/97	8/15/97	8/15/97	8/15/97	8/15/97	8/15/97
Soluble Boron, mg/L	164.00	202.50	151.00	112.00	387.00	514.70	562.50	373.60	345.20	407.30	599.10	525.00
4PB, mg/L	10.00	25.00	58.00	65.00	10.00	10.00	13.00	10.00	10.00	10.00	50.00	10.00
3PB, mg/L	196.00	812.00	779.00	637.00	60.00	10.00	45.00	10.00	52.00	18.00	139.00	20.00
2PB, mg/L	88.00	200.00	243.00	62.00	21.00	13.00	13.00	13.00	43.50	26.00	138.00	64.00
1PB, mg/L	134.00	185.00	154.00	93.00								
Moles boron	0.67	0.82	0.61	0.46	1.58	2.09	2.29	1.52	1.41	1.66	2.44	1.07
Moles 4PB	0.01	0.03	0.07	0.08	0.01	0.01	0.02	0.01	0.01	0.01	0.06	0.01
Moles 3PB	0.04	0.15	0.14	0.12	0.01	0.00	0.01	0.00	0.01	0.00	0.03	0.00
Moles 2PB	0.02	0.05	0.06	0.02	0.01	0.00	0.00	0.00	0.01	0.01	0.04	0.01
Moles 1PB	0.05	0.07	0.06	0.03	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Equivalent moles benzene												
from Boron (Boric acid)	2.19	2.11	1.12	0.83	6.18	8.31	9.05	6.01	5.49	6.54	9.25	4.21
from 3PB	0.04	0.15	0.14	0.12	0.01	0.00	0.01	0.00	0.01	0.00	0.03	0.00
from 2PB	0.05	0.10	0.12	0.03	0.01	0.01	0.01	0.01	0.02	0.01	0.07	0.02
from 1PB	0.15	0.20	0.17	0.10	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Total	2.42	2.56	1.56	1.08	6.21	8.32	9.06	6.02	5.52	6.56	9.35	4.23
Net moles benzene (Final - Initial)	1.93	2.12	1.35	0.89	4.17	6.50	8.49	5.74	2.96	6.46	9.20	3.65
Net mass benzene , g	151	165	106	70	326	508	663	148	231	505	719	285
Projected mass had all PB decomp	702	702	700	701	701	927	926	702	702	702	602	349
Quiescent releases,g	2.00	3.00	3.00	2.00	220.00	174.00	320.00	171.00	49.00	136.00	299.00	33.00
Agitated + Water addition releases,g	0.00	4.66	4.15	0.93	21.36	2.78	118.95	19.54	33.66	48.03	1.00	9.00
Total Releases	2.00	7.66	7.15	2.93	241.36	176.78	438.95	190.54	82.66	184.03	300.00	42.00
Total residual benzene in columns	143.00	56.00	132.00	53.00	58.00	164.00	3.00	177.00	470.00	33.00	68.00	26.00
Residual + Releases	145	64	139	56	299	341	442	368	553	217	368	68
Total available	151	165	106	70	326	508	663	448	231	505	719	285
Difference	0.96	0.38	1.32	0.80	0.92	0.67	0.67	0.82	2.39	0.43	0.51	0.24

Table 12. Benzene Generation Rate Calculations

	Col. 1	2	3	4	6	7	8.00	10	11	12	13
P1	23.52	19.29	6.68	26.63	36.39	305.78	143.05	277.54	113.44	12437.55	70.97
P2	0.00165	0.00284	0.00496	0.00272	9.44000	6.28000	9.97	2.01000	6.72000	648.44000	10.29000

Col.#	Cummulative Hrs	Measured Boron, mg/L	Calculated Boron, mg/L	Rate of boron Change, mg/L/Hr	Rate of benzene Change, mg/L/Hr	Benzene Generation, g/hr	
1	24	40.13	24.47	0.04	1.17	0.05	
	24	36.38	24.47	0.04	1.17	0.05	
	192	40.98	32.29	0.05	1.54	0.07	
	192	39.90	32.29	0.05	1.54	0.07	
	360	51.90	42.60	0.07	2.03	0.09	
	864	58.07	97.85	0.16	4.67	0.21	
	864	61.76	97.85	0.16	4.67	0.21	
	864	77.28	97.85	0.16	4.67	0.21	
	1008	170.90	124.10	0.20	5.92	0.26	
	1008	142.70	124.10	0.20	5.92	0.26	
	1176	145.60	163.74	0.27	7.81	0.34	
	1176	156.20	163.74	0.27	7.81	0.34	
	1176	190.10	163.74	0.27	7.81	0.34	
2	0	34.68	19.29	0.05	1.58	0.07	
	0	35.53	19.29	0.05	1.58	0.07	
	72	39.03	23.67	0.07	1.94	0.09	
	96	38.98	25.34	0.07	2.08	0.09	
	144	53.53	29.04	0.08	2.38	0.10	
	504	57.57	80.72	0.23	6.62	0.29	
	504	63.81	80.72	0.23	6.62	0.29	
	504	79.12	80.72	0.23	6.62	0.29	
	624	97.89	113.49	0.32	9.32	0.41	
	624	101.5	113.49	0.32	9.32	0.41	
	624	122	113.49	0.32	9.32	0.41	
	816	149.8	195.79	0.56	16.07	0.71	
	816	180.6	195.79	0.56	16.07	0.71	
	816	277.1	195.79	0.56	16.07	0.71	
3	0	33.77	6.68	0.03	0.96	0.04	
	0	35.19	6.68	0.03	0.96	0.04	
	0	34.05	6.68	0.03	0.96	0.04	
	312	38.81	31.39	0.16	4.50	0.20	
	312	40.57	31.39	0.16	4.50	0.20	
	432	49.51	56.93	0.28	8.16	0.36	
	432	41.31	56.93	0.28	8.16	0.36	
	432	33.22	56.93	0.28	8.16	0.36	
	624	116.7	147.55	0.73	21.15	0.93	
	624	118.1	147.55	0.73	21.15	0.93	
	624	217.8	147.55	0.73	21.15	0.93	
4	0	30.61	26.63	0.07	2.09	0.09	
	0	32.04	26.63	0.07	2.09	0.09	
	0	33.87	26.63	0.07	2.09	0.09	
	144	41.2	39.40	0.11	3.10	0.14	
	168	36.43	42.06	0.11	3.31	0.15	
	216	38.34	47.92	0.13	3.77	0.17	
	216	38.48	47.92	0.13	3.77	0.17	
	216	39.09	47.92	0.13	3.77	0.17	
	336	85.63	66.42	0.18	5.22	0.23	
	528	102.35	111.97	0.30	8.80	0.39	
	528	114.4	111.97	0.30	8.80	0.39	
	528	118.2	111.97	0.30	8.80	0.39	
6	0	29.04	36.39	#DIV/0!	#DIV/0!	#DIV/0!	
	120	61.4	139.80	0.43	12.45	0.55	0.55
	288	140.97	196.59	0.28	8.04	0.35	0.35
	456	175.6	237.97	0.22	6.39	0.28	0.28
	624	338.7	272.20	0.19	5.46	0.24	0.24
	960	447.3	328.88	0.15	4.40	0.19	0.19
	1104	411.5	350.05	0.14	4.11	0.18	0.18
	1296	443.5	376.23	0.13	3.79	0.17	0.17
	1380	418.5	387.07	0.13	3.67	0.16	0.16
	1860	461.2	443.52	0.11	3.16	0.14	0.14
	2916	387	546.15	0.09	2.53	0.11	0.11

Table 12. Benzene Generation Rate Calculations

Col.#	Cummulative Hrs	Measured Boron, mg/L	Calculated Boron, mg/L	Rate of boron Change, mg/L/Hr	Rate of benzene Change, mg/L/Hr	Benzene Generation, g/hr	
7	0	228.8	305.78	#DIV/0!	#DIV/0!	#DIV/0!	
	120	294.88	374.57	0.29	8.28	0.36	0.36
	288	274.22	412.36	0.19	5.35	0.24	0.24
	624	587.3	462.65	0.13	3.63	0.16	0.16
	960	872.3	500.36	0.10	2.93	0.13	0.13
	1104	728	514.44	0.09	2.73	0.12	0.12
	1296	476.7	531.86	0.09	2.52	0.11	0.11
	1464	487.6	546.07	0.08	2.37	0.10	0.10
	1632	435.1	559.48	0.08	2.25	0.10	0.10
	1944	539.2	582.67	0.07	2.06	0.09	0.09
	3000	514.7	649.75	0.06	1.66	0.07	0.07
8	48	165.01	212.12	0.72	20.79	0.91	0.91
	216	288.39	289.58	0.34	9.80	0.43	0.43
	384	334.7	338.42	0.25	7.35	0.32	0.32
	552	423.4	377.29	0.21	6.13	0.27	0.27
	696	495.3	406.08	0.19	5.46	0.24	0.24
	888	318.7	440.15	0.17	4.83	0.21	0.21
	1032	506.8	463.33	0.16	4.48	0.20	0.20
	1224	539.7	491.86	0.14	4.12	0.18	0.18
	1392	578.2	515.03	0.13	3.86	0.17	0.17
	1560	538.6	536.83	0.13	3.65	0.16	0.16
	1872	611	574.42	0.12	3.33	0.15	0.15
	2928	562.5	682.54	0.09	2.66	0.12	0.12
10	72	195.3	294.60	0.12	3.42	0.15	0.15
	144	270.3	301.66	0.08	2.42	0.11	0.11
	216	385.1	307.08	0.07	1.98	0.09	0.09
	552	274.2	324.76	0.04	1.24	0.05	0.05
	744	365.7	332.37	0.04	1.06	0.05	0.05
	912	374.6	338.24	0.03	0.96	0.04	0.04
	1080	397.9	343.60	0.03	0.88	0.04	0.04
	1248	509.3	348.55	0.03	0.82	0.04	0.04
	1392	403.4	352.53	0.03	0.78	0.03	0.03
	1704	336	360.51	0.02	0.70	0.03	0.03
	1920	325	365.61	0.02	0.66	0.03	0.03
	2040	303.3	368.32	0.02	0.64	0.03	0.03
	2184	339.7	371.47	0.02	0.62	0.03	0.03
	2376	357.7	375.52	0.02	0.60	0.03	0.03
	2376	332.7	375.52	0.02	0.60	0.03	
11	192	109.6	206.56	0.24	7.01	0.31	0.31
	528	310.7	267.85	0.15	4.23	0.19	0.19
	696	390.3	290.73	0.13	3.68	0.16	0.16
	864	283.5	310.97	0.11	3.30	0.15	0.15
	864	292.3	310.97	0.11	3.30	0.15	0.15
	1032	389.9	329.32	0.10	3.02	0.13	0.13
	1032	389.9	329.32	0.10	3.02	0.13	0.13
	1176	355.2	343.89	0.10	2.83	0.12	0.12
	1176	355.2	343.89	0.10	2.83	0.12	0.12
	1320	378.3	357.59	0.09	2.67	0.12	0.12
	1704	360.6	390.84	0.08	2.35	0.10	0.10
	1896	344.4	406.05	0.08	2.23	0.10	0.10
	2040	389.1	416.96	0.07	2.15	0.09	0.09
	2232	407.3	430.92	0.07	2.06	0.09	0.09
12	312	3646	7687.27	11.86	11.61	0.51	0.51
	480	12982	5932.72	9.15	8.96	0.39	0.39
	648	518	4578.62	7.06	6.91	0.30	0.30
	648	498	4578.62	7.06	6.91	0.30	0.30
	816	3512	3533.59	5.45	5.33	0.23	0.23
	816	3434	3533.59	5.45	5.33	0.23	0.23
	960	5968	2829.91	4.36	4.27	0.19	0.19
	960	5520	2829.91	4.36	4.27	0.19	0.19
	1344	357	1565.26	2.41	2.36	0.10	0.10
	1344	2833	1565.26	2.41	2.36	0.10	0.10
	1488	566	1253.55	1.93	1.89	0.08	0.08
	1488	1995	1253.55	1.93	1.89	0.08	0.08
	1680	233	932.29	1.44	1.41	0.06	0.06

Table 12. Benzene Generation Rate Calculations

Col.#	Cummulative Hrs	Measured Boron, mg/L	Calculated Boron, mg/L	Rate of boron Change, mg/L/Hr	Rate of benzene Change, mg/L/Hr	Benzene Generation, g/hr	
	1680	1118	932.29	1.44	1.41	0.06	0.06
	2016	9	555.28	0.86	0.84	0.04	0.04
	2016	91	555.28	0.86	0.84	0.04	0.04
13	168	188.1	204.34	0.40	11.47	0.50	0.50
	312	341.3	252.73	0.29	8.42	0.37	0.37
	696	322.6	342.44	0.20	5.64	0.25	0.25
	840	313.3	369.20	0.18	5.13	0.23	0.23
	1032	322	401.53	0.16	4.63	0.20	0.20
	1176	441.6	423.84	0.15	4.34	0.19	0.19
	1368	525	451.56	0.14	4.02	0.18	0.18

Table 13. Analytical Data From Benzene Retention Columns

Col. No.	Sample Port	Date	ADS No.	4-PB	3-PB	2-PB	1-PB	Phenol	Boron mg/L	Cu mg/L	K (sol) wt%	Na (sol) wt%	Hg (tot) ppm	K (sol) mg/L
1	SP13	2/5/97	3-88278						40.13	0.345	0.2116	11.64		
1	SP13	2/5/97	3-88288	<10	<10	76	114	584						
1	SP13	2/5/97	3-88279						36.38	0.365	0.1863	10.13		
1	SP13	2/5/97	3-88289	<10	<10	68	107	551						
1	SP09	2/13/97	3-88285						40.98	1.025	0.1391	9.97		
1	SP09	2/13/97	3-88295	<10	52	19	99	615						
1	SP12	2/13/97	3-88286						39.90	0.79	0.1328	9.66		
1	SP12	2/13/97	3-88296	<10	52	21	98	616						
1	SP13	2/20/97	3-88283						51.90	0.813	0.1167	10.92		
1	SP13	2/20/97	3-88293	<10	88	<10	50	677						
1	SP03	3/13/97	3-88823						58.07	0.51				
1	SP03	3/13/97	3-88802	<10	72	10	22	664						
1	SP13	3/13/97	3-88824						61.76	0.62				
1	SP13	3/13/97	3-88803	<10	95	12	24	675						
1	SP18	3/13/97	3-88825						77.28	0.65				
1	SP18	3/13/97	3-88804	<10	361	104	95	729						
1	SP03	3/18/97	3-89069						303.3					
1	SP03	3/18/97	3-89077						103.6					
1	SP03	3/18/97	3-89057	<10	78	14	27	720						
1	SP13	3/18/97	3-89070						170.9					
1	SP13	3/18/97	3-89058	<10	117	21	40	715						
1	SP18	3/18/97	3-89071						142.7					
1	SP18	3/18/97	3-89059	<10	385	176	135	737						
1	SP03	3/26/97	3-89391						145.63					
1	SP03	3/26/97	3-89379	<10	89	16	53	718						
1	SP13	3/26/97	3-89392						156.21					
1	SP13	3/26/97	3-89380	<10	180	62	124	755						
1	SP18	3/26/97	3-89393						190.08					
1	SP18	3/26/97	3-89381	<10	319	186	226	787						
2	SP12	2/20/97	3-88280						34.68	0.288	0.0455	9.88		
2	SP12	2/20/97	3-88290	<10	<10	67	113	563						
2	SP12	2/20/97	3-88281						35.53	0.295	0.0412	10.1		
2	SP12	2/20/97	3-88291	<10	<10	63	114	538						
2	SP03	2/23/97	3-88284						39.03	0.605	0.0513	10.57		
2	SP03	2/23/97	3-88294		<10	75	114	588						
2	SP12	2/24/97	3-88287						38.98	0.453	<0.0001	7.19		
2	SP12	2/24/97	3-88297	80	320	78	123	577						
2	SP12	2/26/97	3-88282						53.53	0.335	<0.0001	9.94		
2	SP12	2/26/97	3-88292	70	363	62	76	626						
2	SP03	3/13/97	3-88826						57.57	0.6				
2	SP03	3/13/97	3-88805	46	324	18	35	666						
2	SP13	3/13/97	3-88827						63.81	0.54				
2	SP13	3/13/97	3-88806	51	447	34	50	671						
2	SP18	3/13/97	3-88828						79.12	0.53				
2	SP18	3/13/97	3-88807	100	854	78	89	720						
2	SP03	3/18/97	3-89072						97.89					
2	SP03	3/18/97	3-89060	31	366	29	41	710						
2	SP13	3/18/97	3-89073						101.5					
2	SP13	3/18/97	3-89061	40	523	56	65	720						
2	SP18	3/18/97	3-89074						122					
2	SP18	3/18/97	3-89078						33.22					
2	SP18	3/18/97	3-89062	84	1093	164	134	813						
2	SP03	3/26/97	3-89394						149.85					
2	SP03	3/26/97	3-89382	25	502	49	74	733						
2	SP13	3/26/97	3-89395						180.59					
2	SP13	3/26/97	3-89383	23	711	124	142	738						
2	SP18	3/26/97	3-89396						277.08					
2	SP18	3/26/97	3-89384	27	1223	425	340	780						

Table 13. Analytical Data From Benzene Retention Columns

Col. No.	Sample Port	Date	ADS No.	4-PB	3-PB	2-PB	1-PB	Phenol	Boron mg/L	Cu mg/L	K (sol) wt%	Na (sol) wt%	Hg (tot) ppm	K (sol) mg/L
3	SP03	2/28/97	3-88815						33.77	0.59				
3	SP03	2/28/97	3-88793	106	222	104	104	326						
3	SP12	2/28/97	3-88816						35.19	0.43				
3	SP12	2/28/97	3-88795	122	224	101	103	348						
3	SP18	2/28/97	3-88817						34.09	0.26				
3	SP18	2/28/97	3-88796	119	217	95	101	336						
3	SP03	3/13/97	3-88829						38.81	0.51				
3	SP03	3/13/97	3-88808	125	307	25	75	356						
3	SP13	3/13/97	3-88830						40.57	0.52				
3	SP13	3/13/97	3-88809	128	310	37	72	354						
3	SP18	3/13/97	3-88831						40.93	0.5				
3	SP18	3/13/97	3-88810	128	385	42	83	356						
3	SP03	3/18/97	3-89075						49.51					
3	SP03	3/18/97	3-89063	102	356	31	64	373						
3	SP13	3/18/97	3-89076						41.31					
3	SP13	3/18/97	3-89064	81	359	33	61	372						
3	SP18	3/18/97	3-89065	111	1206	267	183	466						
3	SP03	3/26/97	3-89397						116.69					
3	SP03	3/26/97	3-89385	59	484	63	81	383						
3	SP13	3/26/97	3-89398						118.10					
3	SP13	3/26/97	3-89386	50	489	68	82	385						
3	SP18	3/26/97	3-89399						217.8					
3	SP18	3/26/97	3-89387	64	1365	598	300	428						
4	SP13	2/13/97	3-88833						38.48	0.43				
4	SP13	2/13/97	3-88812	123	346	42	96	351						
4	SP03	3/4/97	3-88818						30.61	0.26				
4	SP03	3/4/97	3-88797	142	216	98	99	330						
4	SP13	3/4/97	3-88819						32.04	<0.21				
4	SP13	3/4/97	3-88798	131	224	101	101	339						
4	SP18	3/4/97	3-88820						33.87	<0.23				
4	SP18	3/4/97	3-88799	125	216	98	99	311						
4	SP13	3/10/97	3-88821						41.2	0.27				
4	SP13	3/10/97	3-88800	134	281	52	100	336						
4	SP13	3/11/97	3-88822						36.43	0.35				
4	SP13	3/11/97	3-88801	133	291	48	99	336						
4	SP03	3/13/97	3-88832						38.34	0.62				
4	SP03	3/13/97	3-88811	129	327	39	92	342						
4	SP18	3/13/97	3-88834						39.09	0.8				
4	SP18	3/13/97	3-88813	116	333	41	93	349						
4	SP03	3/18/97	3-89066	80	391	42	90	381						
4	SP13	3/18/97	3-89079						190.3					
4	SP13	3/18/97	3-89067	86	407	43	90	386						
4	SP18	3/18/97	3-89080						85.63					
4	SP18	3/18/97	3-89068	86	441	48	93	392						
4	SP03	3/26/97	3-89400						102.35					
4	SP03	3/26/97	3-89388	53	529	43	80	382						
4	SP13	3/26/97	3-89401						114.41					
4	SP13	3/26/97	3-89389	66	660	67	95	397						
4	SP18	3/26/97	3-89402						118.24					
4	SP18	3/26/97	3-89390	77	722	76	104	399						
6	SP01	4/11/97	3-90102						158.5					
6	SP01	4/11/97	3-90097	68	219	75	106	369						
6	SP01	4/12/97	3-90103						29.04					
6	SP01	4/12/97	3-90098	61	227	68	108	381						
6	SP02	4/12/97	3-94111										0.019	
6	SP01	4/17/97	3-90224						61.41					

Table 13. Analytical Data From Benzene Retention Columns

Col. No.	Sample Port	Date	ADS No.	4-PB	3-PB	2-PB	1-PB	Phenol	Boron mg/L	Cu mg/L	K (sol) wt%	Na (sol) wt%	Hg (tot) ppm	K (sol) mg/L
6	SP01	4/17/97	3-90228	42	705	112								
6	SP01	4/24/97	3-90589						140.97					
6	SP01	4/24/97	3-90585	12	850	269								
6	SP06	5/1/97	3-90880						175.6					
6	SP06	5/1/97	3-90876	53	563	178								
6	SP06	5/1/97	3-90872	120	253	70								
6	SP06	5/8/97	3-91163	<10	196	99								
6	SP06	5/8/97	3-91175						338.7					
6	SP06	5/8/97	3-91169	<10	98	45								
6	SP06	5/22/97	3-91873	<10	83	84								
6	SP06	5/22/97	3-91879						447.3					
6	SP06	5/22/97	3-91872											
6	SP06	5/28/97	3-92323	<10	56	60								
6	SP06	5/28/97	3-92328						411.5	6.47				
6	SP06	6/5/97	3-92596	<10	41	38								
6	SP06	6/5/97	3-92588						443.6	12.25				
6	SP06	6/12/97	3-92859	<10	30	27								
6	SP06	6/12/97	3-92866						418.5	0.005				
6	SP02	7/2/97	3-93700	<10	38	11								
6	SP02	7/2/97	3-93709						461.2	4.01				
6	SP02	7/2/97	3-94124								0.0012			
6	SP06	8/15/97	3-95996	<10	60	21								
6	SP06	8/15/97	3-96006						387	3.272	<0.0006			
7	SP01	4/12/97	3-90104						228.8					
7	SP01	4/12/97	3-90099	91	1774	589	481	785						
7	SP02	4/12/97	3-94117										0.075	
7	SP01	4/17/97	3-90225						294.88					
7	SP01	4/17/97	3-90229	24	1228	1033								
7	SP01	4/24/97	3-90590						274.22					
7	SP01	4/24/97	3-90586	<10	342	655								
7	SP04	5/1/97	3-90877	18	2898	1424								
7	SP04	5/1/97	3-90873	215	764	344								
7	SP03	5/8/97	3-91164	<10	24	108								
7	SP03	5/8/97	3-91176						587.3					
7	SP03	5/8/97	3-91170	10	13	51								
7	SP05	5/22/97	3-91874	<10	20	62								
7	SP05	5/22/97	3-91880						872.3					
7	SP04	5/28/97	3-92324	<10	15	64								
7	SP04	5/28/97	3-92329						728	5.95				
7	SP03	6/5/97	3-92597	<10	12	44								
7	SP03	6/5/97	3-92589						476.7	7.99				
7	SP03	6/12/97	3-92860	<10	<10	37								
7	SP03	6/12/97	3-92867						487.6	0.005				
7	SP02	6/19/97	3-93396	<10	121	29								
7	SP02	6/19/97	3-93409						435.1	3.07				
7	SP02	7/2/97	3-93701	<10	18	7								
7	SP02	7/2/97	3-93710						539.2	1.61				
7	SP02	7/2/97	3-94125								0.0016			
7	SP02	8/15/97	3-95997	<10	<10	13								
7	SP02	8/15/97	3-96007						514.7	2.417	0.0013			
8	SP01	4/15/97	3-90105						108					
8	SP01	4/15/97	3-90100	168	1124	225	233	437						
8	SP02	4/15/97	3-94118										0.039	
8	SP01	4/17/97	3-90226						165.01					
8	SP01	4/17/97	3-90230	106	1977	356								
8	SP01	4/24/97	3-90591						288.39					
8	SP01	4/24/97	3-90587	7	1341	682								

Table 13. Analytical Data From Benzene Retention Columns

Col. No.	Sample Port	Date	ADS No.	4-PB	3-PB	2-PB	1-PB	Phenol	Boron mg/L	Cu mg/L	K (sol) wt%	Na (sol) wt%	Hg (tot) ppm	K (sol) mg/L
8	SP06	5/1/97	3-90884						334.7					
8	SP06	5/1/97	3-90878	34	730	460								
8	SP06	5/1/97	3-90874	112	365	194								
8	SP06	5/8/97	3-91165	<10	163	227								
8	SP06	5/8/97	3-91177						423.4					
8	SP06	5/8/97	3-91171	10	82	106								
8	SP06	5/14/97	3-91448	<10	113	190								
8	SP06	5/14/97	3-91452						495.3					
8	SP06	5/22/97	3-91875	<10	68	134								
8	SP06	5/22/97	3-91881						318.7					
8	SP06	5/28/97	3-92325	<10	47	93								
8	SP06	5/28/97	3-92330						506.8	6.49				
8	SP06	6/5/97	3-92598	<10	34	63								
8	SP06	6/5/97	3-92590						539.7	10.7				
8	SP06	6/12/97	3-92861	<10	25	39								
8	SP06	6/12/97	3-92868						578.2	4.69				
8	SP05	6/19/97	3-93397	<10	<10	25								
8	SP05	6/19/97	3-93410						538.6	4.18				
8	SP02	7/2/97	3-93702	<10	20	15								
8	SP02	7/2/97	3-93711						611	3.83				
8	SP02	7/2/97	3-94126								0.0006			
8	SP04	8/15/97	3-95998	13	45	13								
8	SP04	8/15/97	3-96008						562.5	3.997	<0.0006			
9	SP01	4/18/97	3-90227						78.09					
9	SP01	4/18/97	3-90231	164	194	78								
9	SP01	4/24/97	3-90592						110.79					
9	SP01	4/24/97	3-90588	32	491	288								
9	SP06	5/1/97	3-90885						637					
9	SP06	5/1/97	3-90879	<10	110	1383								
9	SP06	5/1/97	3-90875	35	18	167								
9	SP06	5/8/97	3-91166	<10	20	203								
9	SP06	5/8/97	3-91178						748.3					
9	SP06	5/8/97	3-91172	4	10	114								
9	SP03	5/14/97	3-91449	<10	20	23								
9	SP03	5/14/97	3-91453						264					
9	SP05	5/22/97	3-91876	<10	<10	84								
9	SP05	5/22/97	3-91882						711					
9	SP04	5/28/97	3-92326	<10	<10	50								
9	SP04	5/28/97	3-92331						589.7	3.88				
9	SP04	6/5/97	3-92599	<10	<10	31								
9	SP04	6/5/97	3-92591						503.1	7.6				
9	SP04	6/12/97	3-92862	<10	<10	21								
9	SP04	6/12/97	3-92869						459.7	3.29				
9	SP04	6/19/97	3-93398	<10	<10	25								
9	SP04	6/19/97	3-93411						467.9	2.89				
9	SP01	6/24/97	3-93403	<10	<10	<10								
9	SP01	6/24/97	3-93416						259.1	4.32				
9	SP03	6/24/97	3-93404	<10	<10	11								
9	SP03	6/24/97	3-93417						301.1	3.66				
9	SP04	6/24/97	3-93405	<10	<10	25								
9	SP04	6/24/97	3-93418						461.6	3.11				
9	SP05	6/24/97	3-93406	<10	<10	46								
9	SP05	6/24/97	3-93419						661.3	2.52				
9	SP07	6/24/97	3-93407	<10	9	83								
9	SP07	6/24/97	3-93420						642.3	2.31				
9	SP01	7/2/97	3-93703	<10	9	22								
9	SP01	7/2/97	3-93712						384.7	1.85				
9	SP01	7/2/97	3-94127								0.0057			

Table 13. Analytical Data From Benzene Retention Columns

Col. No.	Sample Port	Date	ADS No.	4-PB	3-PB	2-PB	1-PB	Phenol	Boron mg/L	Cu mg/L	K (sol) wt%	Na (sol) wt%	Hg (tot) ppm	K (sol) mg/L
9	SP04	8/15/97	3-95999	<10	<10	13								
9	SP04	8/15/97	3-96009						373.6	2.75	0.0072			
10	SP06	5/8/97	3-91167	10	1220	1244								
10	SP06	5/8/97	3-91179						266.1					
10	SP06	5/8/97	3-91173	84	651	610								
10	SP06	5/8/97	3-91168	50	941	757								
10	SP06	5/8/97	3-91180						195.3					
10	SP06	5/8/97	3-91174	147	484	361								
10	SP04	5/14/97	3-91450	<10	552	1204								
10	SP04	5/14/97	3-91454						270.3					
10	SP06	5/22/97	3-91877	<10	250	1658								
10	SP06	5/22/97	3-91883						385.1					
10	SP01	5/28/97	3-92327	7	288	693								
10	SP01	5/28/97	3-92332						274.2	2.442				
10	SP05	6/5/97	3-92600	<10	88	309								
10	SP05	6/5/97	3-92592						365.7	7.2				
10	SP05	6/12/97	3-92863	<10	30	139								
10	SP05	6/12/97	3-92870						374.6	3.68				
10	SP06	6/19/97	3-93399	<10	14	76								
10	SP06	6/19/97	3-93412						397.9	2.38				
10	SP05	6/26/97	3-93630	<10	8	49								
10	SP05	6/26/97	3-93626						509.3	2.22				
10	SP02	7/2/97	3-93704	<10	46	60								
10	SP02	7/2/97	3-93713						403.4	1.64				
10	SP02	7/2/97	3-94128								0.0064			
10	SP02	7/18/97	3-94516	<10	13	12								
10	SP02	7/18/97	3-94521						336	6.16				
10	SP02	7/24/97	3-94873	<10	13	<10								
10	SP02	7/27/97	3-94868						325	2.517				
10	SP02	8/1/97	3-95029	<10	12	<10								
10	SP02	8/1/97	3-95034						303.3	2.464				
10	SP02	8/7/97	3-95354											
10	SP02	8/7/97	3-95359						339.7	3.883				
10	SP03	8/15/97	3-96000	<10	50	39								
10	SP03	8/15/97	3-96010						357.7	1.748	<0.0006			
10	SP05	8/15/97	3-96001	<10	55	48								
10	SP05	8/15/97	3-96011						332.7	1.739	<0.0006			
11	SP01	5/14/97	3-91451	1120	366	68								
11	SP01	5/14/97	3-91455						93.7					
11	SP06	5/22/97	3-91878	488	553	89								
11	SP06	5/22/97	3-91884						109.6					
11	SP07	6/5/97	3-92601	17	452	706								
11	SP07	6/5/97	3-92593						310.7	8.84				
11	SP05	6/12/97	3-92864	<10	155	421								
11	SP07	6/12/97	3-92871						390.3	2.88				
11	SP03	6/19/97	3-93400	<10	57	116								
11	SP03	6/19/97	3-93413						283.5	2.44				
11	SP04	6/19/97	3-93395	<10	132	71								
11	SP04	6/19/97	3-93408						292.3	4.29				
11	SP05	6/26/97	3-93631	<10	31	110								
11	SP05	6/26/97	3-93627						389.9	3.43				
11	SP01	7/2/97	3-93714						355.2	3.15				
11	SP01	7/2/97	3-94129								0.0034			
11	SP01	7/18/97	3-94517	<10	16	9								
11	SP01	7/18/97	3-94522						378.3	6.93				
11	SP01	7/24/97	3-94874	9	16	<10								
11	SP01	7/24/97	3-94869						360.6	3.139				

Table 13. Analytical Data From Benzene Retention Columns

Col. No.	Sample Port	Date	ADS No.	4-PB	3-PB	2-PB	1-PB	Phenol	Boron mg/L	Cu mg/L	K (sol) wt%	Na (sol) wt%	Hg (tot) ppm	K (sol) mg/L
11	SP01	8/1/97	3-95030	12	15	<10								
11	SP01	8/1/97	3-95035						344.4	3.073				
11	SP01	8/7/97	3-95355											
11	SP01	8/7/97	3-95360						389.1	2.12				
11	SP06	8/15/97	3-96002	<10	18	26								
11	SP06	8/15/97	3-96012						407.3	2.512	0.006			
11	SP01	7/2*97	3-93705	<10	59	28								
12	SP01	5/23/97	3-92603	13349	458	130								
12	SP01	5/23/97	3-92595						527.7	6.67				
12	SP03	6/5/97	3-92602	3646	5580	3804								
12	SP03	6/5/97	3-92594						662.8	6.75				
12	SP03	6/12/97	3-92865	12982	155	<10								
12	SP03	6/12/97	3-92872						583.5	0.005				
12	PP03	6/19/97	3-93401	518	2083	1414								
12	PP03	6/19/97	3-93414						629.3	0.005				
12	PP03	6/26/97	3-93632	3434	2027	510								
12	PP03	6/26/97	3-93628						689.4	0.624				
12	PP03	7/2/97	3-93706	5968	509	129								
12	PP03	7/2/97	3-93715						725.8	0.48				
12	PP04	7/2/97	3-93707	5520	673	175								
12	PP04	7/2/97	3-93716						612.4	0.354				
12	PP03	7/18/97	3-94518	357	553	319								
12	PP03	7/18/97	3-94523						652.8	4.998				
12	PP04	7/18/97	3-94519	2833	413	146								
12	PP04	7/18/97	3-94524						637.4	5.136				
12	PP03	7/24/97	3-94875	566	434	297								
12	PP03	7/24/97	3-94870						621.3	<0.0054				
12	PP04	7/24/97	3-94876	1995	379	145								
12	PP04	7/24/97	3-94871						611.9	0.2956				
12	PP03	8/1/97	3-95031	233	385	216								
12	PP03	8/1/97	3-95036						654.1	0.401				
12	PP04	8/1/97	3-95032	1118	239	120								
12	PP04	8/1/97	3-95037						655.5	0.912				
12	PP03	8/7/97	3-95356											
12	PP03	8/7/97	3-95361						678.1	0.936				
12	PP03	8/15/97	3-96003	9	153	167								
12	PP04	8/15/97	3-96004	91	124	108								
12	PP04	8/15/97	3-96014						599.1	0.2978	<0.0007			
12	PP03	8/18/97	3-96013						617.7	0.1731	0.0012			
13	PP03	6/19/97	3-93402	118	429	69								
13	PP03	6/19/97	3-93415						62.06	0.005				
13	PP04	6/26/97	3-93633	154	1098	323								
13	PP04	6/26/97	3-93629						188.1	1.098				
13	PP04	7/2/97	3-93708	15	1759	1779								
13	PP04	7/2/97	3-93717						341.3	0.81				
13	PP04	7/18/97	3-94520	<10	161	262								
13	PP04	7/18/97	3-94525						322.6	4.925				
13	PP04	7/24/97	3-94877	<10	94	140								
13	PP04	7/24/97	3-94872						313.3	2.076				
13	PP04	8/1/97	3-95033	<10	41	67								
13	PP04	8/1/97	3-95038						322	2.614				
13	PP04	8/7/97	3-95357											
13	PP04	8/7/97	3-95362						441.6	3.27				
13	PP04	8/15/97	3-96005	<10	20	64								
13	PP04	8/15/97	3-96015						525	2.995	<0.0006			

Table 14. Temperature Data From Benzene Retention Columns

Column	Date	Cummulative Run Time, Hours	Average Liquid Temp, C	Upper Col. Temp, C	Mid Col Temp, C	Lower Col Temp, C	Col Vapor Temp, C
1	2/4/97	0	20	20	20	20	20
	2/5/97	24	42	42	42	41	38
	2/13/97	216	50	50	51	49	45
	2/25/97	504	50	51	51	49	46
	2/28/97	576	48	49	49	47	44
	3/1/97	600	48	49	49	47	46
	3/2/97	624	48	48	48	47	45
	3/3/97	648	49	50	50	48	46
	3/4/97	672	50	50	50	49	45
	3/5/97	696	53	54	54	52	48
	3/6/97	720	48	49	49	47	45
	3/7/97	744	50	50	51	49	46
	3/8/97	768	49	50	50	48	45
	3/9/97	792	50	50	50	49	45
	3/10/97	816	46	46	46	45	43
	3/11/97	840	49	51	49	48	46
	3/13/97	888	49	49	50	49	47
	3/18/97	1008	49	49	50	48	45
	3/20/97	1056	51	51	52	51	47
	3/26/97	1200	49	49	50	48	45
	4/2/97	1368	51	51	51	50	48
2	2/20/97	0	41	41	41	41	37
	2/23/97	72	40	40	40	40	38
	2/25/97	120	40	40	40	39	38
	2/28/97	192	51	50	51	51	48
	3/1/97	216	50	49	50	50	49
	3/2/97	240	49	49	49	49	48
	3/3/97	264	50	49	50	50	48
	3/4/97	288	50	50	50	50	47
	3/5/97	312	54	54	54	53	50
	3/6/97	336	49	49	50	49	48
	3/7/97	360	51	51	51	51	49
	3/8/97	384	50	50	50	49	47
	3/9/97	408	50	50	50	50	48
	3/10/97	432	46	46	47	46	45
	3/11/97	456	50	50	50	49	48
	3/13/97	504	50	50	51	50	50
	3/18/97	624	50	50	50	50	48
	3/20/97	672	52	52	53	52	49
	3/26/97	816	49	50	49	49	48
	4/2/97	984	51	51	51	51	51

Table 14. Temperature Data From Benzene Retention Columns

Column	Date	Cummulative Run Time, Hours	Average Liquid Temp, C	Upper Col. Temp, C	Mid Col Temp, C	Lower Col Temp, C	Col Vapor Temp, C
3	2/28/97	0	40	40	40	41	39
	3/1/97	24	41	41	41	41	39
	3/2/97	48	41	40	41	41	39
	3/3/97	72	43	42	43	43	40
	3/4/97	96	38	39	38	38	37
	3/5/97	120	39	39	39	39	38
	3/6/97	144	41	41	41	41	39
	3/7/97	168	51	51	52	51	48
	3/8/97	192	50	50	50	50	46
	3/9/97	216	50	50	51	50	47
	3/10/97	240	47	46	47	47	44
	3/11/97	264	50	51	50	49	48
	3/13/97	312	51	52	51	50	49
	3/18/97	432	50	49	51	50	47
	3/20/97	480	52	51	53	53	48
	3/26/97	624	49	49	50	49	47
	4/2/97	792	51	51	52	51	49
4	3/4/97	0	36	36	36	36	37
	3/5/97	24	39	39	39	39	37
	3/6/97	48	41	41	41	41	38
	3/7/97	72	41	41	41	41	38
	3/10/97	144	50	40	40	40	37
	3/11/97	168	50	51	50	50	46
	3/13/97	216	53	52	53	53	48
	3/18/97	336	43	43	43	42	40
	3/20/97	384	50	49	50	50	44
	3/26/97	528	51	50	51	51	46
	4/2/97	696	50	50	50	50	48
6	4/12/97	0	47	47	46	49	44
	4/14/97	48	48	48	48	48	44
	4/15/97	72	50	50	51	50	46
	4/16/97	96	50	50	51	50	46
	4/17/97	120	50	50	50	49	45
	4/18/97	144	52	52	52	51	46
	4/21/97	216	48	49	48	48	45
	4/22/97	240	50	50	50	49	45
	4/23/97	264	49	49	49	48	45
	4/24/97	288	50	50	50	49	46
	4/29/97	408	50	50	50	49	45
	4/30/97	432	50	51	50	50	46
	5/1/97	456	51	51	51	50	45
	5/5/97	552	49	49	49	49	44
	5/6/97	576	51	52	51	50	47
	5/7/97	600	53	54	53	52	49

Table 14. Temperature Data From Benzene Retention Columns

Column	Date	Cummulative Run Time, Hours	Average Liquid Temp, C	Upper Col. Temp, C	Mid Col Temp, C	Lower Col Temp, C	Col Vapor Temp, C
6	5/8/97	624	64	64	64	63	56
	5/9/97	648	35	35	35	35	33
	5/12/97	720	38	39	38	38	36
	5/13/97	744	40	41	40	40	38
	5/19/97	888	40	41	40	40	37
	5/20/97	912	40	40	40	40	37
	5/22/97	960	41	42	41	41	38
	5/27/97	1080	50	51	50	49	46
	5/28/97	1104	41	41	41	40	39
	6/2/97	1224	40	41	40	40	38
	6/5/97	1296	47	48	47	46	44
	6/9/97	1392	40	40	40	39	37
	6/19/97	1632	41	42	41	40	39
	7/2/97	1944	41	43	40	41	38
	7/18/97	2328	40	41	40	40	38
	7/24/97	2472	41	42	41	40	39
	8/1/97	2664	40	40	40	40	38
	8/15/97	3000	47	48	46	46	42
7	4/12/97	0	42	42	42	41	43
	4/14/97	48	50	48	52	50	45
	4/15/97	72	50	48	51	50	44
	4/16/97	96	50	49	51	50	45
	4/17/97	120	50	49	51	50	45
	4/18/97	144	50	50	51	50	44
	4/21/97	216	49	49	49	49	45
	4/22/97	240	48	48	49	48	43
	4/23/97	264	49	49	50	49	44
	4/24/97	288	48	48	49	48	45
	4/29/97	408	48	48	49	48	45
	4/30/97	432	49	49	49	49	44
	5/1/97	456	49	49	49	48	43
	5/5/97	552	48	48	49	48	45
	5/6/97	576	49	49	50	49	45
	5/7/97	600	51	51	52	51	46
	5/8/97	624	51	51	51	51	45
	5/9/97	648	36	36	36	36	34
	5/12/97	720	39	40	39	39	36
	5/13/97	744	36	36	36	36	34
	5/19/97	888	37	37	37	37	34
	5/20/97	912	39	39	40	39	37
	5/22/97	960	39	39	39	39	36
	5/27/97	1080	39	39	39	39	37
	5/28/97	1104	39	39	39	39	37
	6/2/97	1224	38	38	38	38	36
	6/5/97	1296	38	38	38	38	36

Table 14. Temperature Data From Benzene Retention Columns

Column	Date	Cummulative Run Time, Hours	Average Liquid Temp, C	Upper Col. Temp, C	Mid Col Temp, C	Lower Col Temp, C	Col Vapor Temp, C
7	6/9/97	1392	38	38	38	37	36
	6/19/97	1632	38	38	38	38	36
	7/2/97	1944	39	39	39	38	36
	7/18/97	2328	40	39	41	41	38
	7/24/97	2472	40	39	40	40	37
	8/1/97	2664	39	39	39	39	38
	8/15/97	3000	39	39	39	38	36
8	4/15/97	0	49	48	48	51	43
	4/16/97	24	50	50	51	50	45
	4/17/97	48	50	50	51	50	45
	4/18/97	72	52	51	51	53	44
	4/21/97	144	49	50	49	49	44
	4/22/97	168	48	49	48	48	46
	4/23/97	192	49	50	49	49	44
	4/24/97	216	49	50	49	48	45
	4/29/97	336	49	50	49	48	44
	4/30/97	360	49	50	49	49	44
	5/1/97	384	49	49	49	48	43
	5/5/97	480	50	50	49	50	45
	5/6/97	504	50	50	50	49	45
	5/7/97	528	52	52	52	51	45
	5/8/97	552	51	52	51	51	45
	5/9/97	576	36	36	36	35	33
	5/12/97	648	39	39	39	39	36
	5/13/97	672	36	36	36	35	34
	5/19/97	816	37	37	37	36	34
	5/20/97	840	40	40	40	39	36
	5/22/97	888	38	39	38	38	35
	5/27/97	1008	39	39	39	39	36
	5/28/97	1032	39	39	39	39	36
	6/2/97	1152	38	38	38	38	36
	6/5/97	1224	38	38	38	38	36
	6/9/97	1320	40	40	40	39	37
	6/19/97	1560	38	39	38	38	35
	7/2/97	1872	38	39	38	38	36
	7/18/97	2256	41	41	41	41	37
	7/24/97	2400	40	40	40	40	38
	8/1/97	2592	39	39	39	39	37
	8/15/97	2928	39	39	39	38	36
9	4/18/97	0	45	45	46	45	44
	4/21/97	72	49	49	49	48	44
	4/22/97	96	50	50	50	49	44
	4/23/97	120	49	49	49	48	43
	4/24/97	144	50	50	50	49	42

Table 14. Temperature Data From Benzene Retention Columns

Column	Date	Cummulative Run Time, Hours	Average Liquid Temp, C	Upper Col. Temp, C	Mid Col Temp, C	Lower Col Temp, C	Col Vapor Temp, C
9	4/29/97	264	50	50	50	49	44
	4/30/97	288	50	51	50	50	44
	5/1/97	312	51	52	51	50	45
	5/5/97	408	49	50	49	48	45
	5/6/97	432	51	51	52	50	48
	5/7/97	456	52	52	53	52	48
	5/8/97	480	63	63	64	63	62
	5/9/97	504	35	35	35	35	33
	5/12/97	576	38	39	38	37	35
	5/13/97	600	40	41	40	39	37
	5/19/97	744	40	41	40	40	37
	5/20/97	768	40	40	40	39	37
	5/22/97	816	41	41	41	40	36
	5/27/97	936	49	47	50	49	44
	5/28/97	960	40	40	40	40	38
	6/2/97	1080	40	41	40	40	38
	6/5/97	1152	46	44	47	46	42
	6/9/97	1248	40	40	40	39	37
	6/19/97	1488	41	41	41	40	38
	7/2/97	1800	41	41	41	40	37
	7/18/97	2184	40	40	41	40	37
	7/24/97	2328	41	41	41	40	38
	8/1/97	2520	40	40	40	39	38
	8/15/97	2856	46	47	46	46	42
10	5/5/97	0	#DIV/0!				
	5/6/97	24	49	49	50	49	44
	5/7/97	48	50	50	50	49	45
	5/8/97	72	49	49	50	49	44
	5/9/97	96	41	41	41	40	37
	5/12/97	168	41	41	41	41	37
	5/13/97	192	41	41	41	40	37
	5/19/97	336	41	41	41	41	37
	5/20/97	360	41	41	41	41	34
	5/22/97	408	40	40	40	40	36
	5/27/97	528	40	40	40	40	37
	5/28/97	552	43	42	42	44	38
	6/2/97	672	43	43	43	42	39
	6/5/97	744	43	43	43	42	39
	6/9/97	840	42	42	42	42	39
	6/19/97	1080	42	42	42	41	38
	7/2/97	1392	41	41	42	41	38
	7/18/97	1776	41	40	41	41	37
	7/24/97	1920	41	41	41	40	37
	8/1/97	2112	44	44	45	44	41
	8/15/97	2448	42	42	42	42	39

Table 14. Temperature Data From Benzene Retention Columns

Column	Date	Cummulative Run Time, Hours	Average Liquid Temp, C	Upper Col. Temp, C	Mid Col Temp, C	Lower Col Temp, C	Col Vapor Temp, C
11	5/14/97	0	#DIV/0!				
	5/27/97	312	48	48	48	47	42
	5/28/97	336	50	51	50	49	45
	6/2/97	456	47	48	47	46	42
	6/5/97	528	48	49	48	47	44
	6/9/97	624	49	50	49	48	44
	6/19/97	864	51	49	52	51	44
	7/2/97	1176	45	45	46	45	41
	7/18/97	1560	47	47	47	46	41
	8/1/97	1896	54	52	55	54	48
	8/15/97	2232	46	46	46	45	41
12	5/23/97	0	#DIV/0!				
	5/27/97	96	47	48	47	47	45
	5/28/97	120	49	49	49		
	6/2/97	240	47	47	46	47	44
	6/5/97	312	48	48	48	48	44
	6/9/97	408	49	49	49	49	46
	6/19/97	648	51	50	52	51	35
	7/2/97	960	46	46	46	47	45
	7/18/97	1344	46	45	46	48	45
	8/1/97	1680	55	55	55	54	53
	8/15/97	2016	46	46	45	46	43
13	6/19/97	0	47	47	45	49	49
	7/2/97	312	44	43	42	46	44
	7/18/97	696	44	43	43	46	44
	8/1/97	1032	52	51	50	55	52
	8/15/97	1368	43	42	42	45	43

Table 15. Column Vapor Space Profile Measurement

Column 2 Vapor Space Profile
Purge rate 150cc/min

Reading #	Sample tube distance from slurry/vapor interface	Vapor Benzene ppm
1	1 inch	128
2	1 inch	129
3	1 inch	128
4	1 inch	131
5	1 inch	124
1	10.5 inches	143
2	10.5 inches	142
3	10.5 inches	141
4	10.5 inches	143
5	10.5 inches	140
1	6 inches	134
2	6 inches	135
3	6 inches	133
4	6 inches	139
5	6 inches	131
1	vent line	132
2	vent line	133
3	vent line	131
4	vent line	125
5	vent line	131