

Evaluating Defoaming Agents for the Stripping Columns at the In-Tank Precipitation Facility

by

J. F. McGlynn

Westinghouse Savannah River Company

Savannah River Site

Aiken, South Carolina 29808

RECEIVED

SEP 16 1998

OSTI

DOE Contract No. DE-AC09-89SR18035

This paper was prepared in connection with work done under the above contract number with the U. S. Department of Energy. By acceptance of this paper, the publisher and/or recipient acknowledges the U. S. Government's right to retain a nonexclusive, royalty-free license in and to any copyright covering this paper, along with the right to reproduce and to authorize others to reproduce all or part of the copyrighted paper.

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED 

MASTER

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 to DOE and DOE contractors from the Office of Scientific and Technical Information, P.O. Box 62, Oak Ridge, TN 37831; prices available from (615) 576-8401.

Available to the public from the National Technical Information Service, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, VA 22161.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

Westinghouse Savannah River Company
Savannah River Technology Center

WSRC-RP-93-928

Keywords: Foaming,
In-Tank Precipitation,
Sodium Tetraphenylborate,
Benzene Stripper Column
Tributylphosphate

Retention: Lifetime

Page 1 of 23

- CC: G. T. Wright, 5002H
- W. L. Tamosaitis 773-A
- C. J. Baker, 5002H
- J. N. Brooke, 241-119H
- W. B. Van Pelt, 241-152H
- B. L. Lewis, 703-H
- J. E. Marra, 703-H
- L. F. Landon, 704-T
- L. O. Dworjany, 779-2A
- D. D. Walker, 773-A
- M. J. Barnes, 773-A
- M. Baich, 704-T
- R. F. Swingle, 773-A
- G. K. Georgetown, 703-H
- P. L. Thorpe, 241-119H
- T. P. Gaughan, 241-152H
- G. A. Taylor, 241-152H
- SRTC Records (4)

June 30, 1993

To: S. D. Fink, 773-A

From: J. F. McGlynn, 773-A *J. F. McGlynn*

EVALUATING DEFOAMING AGENTS FOR THE STRIPPING COLUMNS AT THE IN-TANK PRECIPITATION FACILITY (U)

SUMMARY

During initial testing of the stripper columns in the In-Tank Precipitation facility, differential pressures in excess of design specifications have been observed. The root cause of the excessive pressure drops has been attributed to foaming of the simulant salt solution used for acceptance testing. An experimental program was completed to confirm foaming as a cause, to identify and evaluate chemical additives to inhibit foaming, and to provide operating guidance for these defoaming agents in

support of facility acceptance testing, vendor pilot scale testing, and normal operations. These tests indicate that:

- Tributylphosphate, at concentrations no greater than 150 ppm in the filtrate, is the defoaming agent recommended for use in the In-Tank Precipitation benzene stripper columns.
- Sparging tests show that tributylphosphate up to a concentration of 300 ppm does not adversely affect benzene removal.
- The tributylphosphate can be cleaned from the temporary feed tank at ITP using deionized water at 5% of the tank volume.
- Benzene can be added to the temporary feed tank at ITP using the benzene injection port within a 4-6 hour period to obtain a saturated solution.

INTRODUCTION

The In-Tank Precipitation (ITP) process will concentrate the Tank 48 contents to approximately 10 wt.% tetraphenylborate solids by filtration. The filtrate produced during the process flows to the ITP stripping columns where the soluble benzene is removed from the solution. It has been observed that a large pressure differential occurs across the column packing when the filtrate is processed in the column. One potential explanation for the pressure differential is that the filtrate is foaming in the column. Small scale stripping tests have verified that the salt solution foams.^{1,8}

Waste Management requested assistance from SRTC in solving the foaming problem through technical task requests HLE-TTR-93013A/B (Benzene Stripper Performance Evaluation) and HLE-TTR-93044 (Kinetics of Benzene and Dissolution). Various tests were completed to determine an effective defoaming agent for use in the stripping columns. This document discusses the tests and the conclusions.

The conclusions from this testing were used as information in developing a test plan at the vendor's (Koch Engineering Company) facility. Koch Engineering performed several tests on an eight inch diameter column at their testing facility. These tests verified that tributylphosphate inhibits foaming of the salt solution.

EXPERIMENTAL

The study consisted of several phases:

- (1) scouting tests to determine the impact various defoaming reagents have on the foaming behavior of simulated ITP salt solutions,
- (2) simulation of ITP column performance in the presence of selected reagents,
- (3) estimation of the effect of selected defoaming agents on volatility of benzene from the simulated salt solutions,
- (4) qualitative evaluation of the wetting behavior of selected defoaming agents on stainless steel,
- (5) simulation of the ITP benzene injection system for the ITP stripper column acceptance test, and
- (6) testing the efficiency of tributylphosphate (the optimal defoaming reagent) removal from process equipment.

Salt Solution Formulation

Filtrate was made simulating the actual salt expected in the ITP facility. The filtrate was obtained from two sources: (1) a simulated salt solution was made using a lab procedure (see Table 1), and (2) filtrate supplied by M. Morrissey from the Experimental Laboratory Filter apparatus at TNX (see Table 2).

Defoaming Agent Tests

A standardized test device was used to determine the ability of various defoaming agents to reduce the foam height and shorten the collapse time of the salt solution. These tests identified defoaming agents to test in the small scale stripping column. Several defoaming agents were identified for testing by J. P. Bibler² and C. A. Langton: Gafac[®] RA-600 surfactant; n-tributylphosphate, Pegol[®] L-62, Triton[®] X-100 surfactant, surfynol 104E, Dow Corning[®] antifoam 544, and Triton[®] X-114 surfactant. Surfynol 104E and Dow Corning[®] antifoam 544 have been proposed for use in the Defense Waste Processing Facility.

The experimental unit consisted of a 500 mL graduated cylinder with a sparge tube placed at the center of the cylinder approximately one half inch from the bottom (see Figure 1). The sparge tube is a standard stores stocked item with a coarse frit of pore size 40 - 60 μm . The fritted portion at the bottom of the tube has a cylindrical shape with length of approximately 21 mm and a diameter of 11 mm. Each sparge tube has a varying amount of actual sparging area (i.e., the amount of bubbles produced varies slightly).

The 500 mL graduated cylinder was filled with 200 mL of salt solution. The initial level of the solution with the sparge tube immersed was marked. Nitrogen was supplied to the sparge tube at 0.6 L/min causing the solution to foam (flow rate based on a previous test performed by D.D. Walker⁴). The stable height of the foam was marked. The sparge tube was removed and the collapse time of the foam measured. Defoaming agent was added in small increments. The test was repeated after each addition to determine the effect of defoaming agent concentration on foam height and stability.

Simulating Foaming of the Decontaminated Stripping Column

These tests were performed to establish conditions which cause foaming, and to evaluate the ability of selected foaming agents to inhibit the foaming. The stripping columns at ITP have nitrogen and salt solution flowing countercurrently. The experimental stripper is intended as a small scale version of the Decontaminated Salt Solution benzene stripper column in the ITP process. The experimental column was designed on the following assumptions.

- The gas and liquid flow rates are scaled by the same factor and are based on the cross-sectional area of the column (SCFM of nitrogen/ft² and GPM of filtrate/ft²).
- The height and diameter of the column packing are scaled by the same factor (15:1).
- The experimental column packing is stainless steel sponge material. It is a random packing whereas the ITP column packing is a structured stainless steel packing (Koch 1Y Flexipak®).
- Various packing materials were tested in the column before choosing the stainless steel sponge (i.e., glass beads, Pro-Pak protruded metal distillation packing, and stainless steel sponge).

A 680 mL reservoir was used as both the feed and hold tank, unlike the ITP facility which has separate feed and hold tanks. A centrifugal pump supplied the column with the desired filtrate flow rate. Nitrogen was supplied to the column at the calculated flow rate using the laboratory nitrogen supply. Figure 2 and Table 3 detail the equipment setup and operating parameters.

The system was operated at a condition which produced foaming in the column. In the small scale column, it has been observed that foaming increases when the nitrogen flow rate is increased. Therefore filtrate was supplied at its maximum flow rate, and nitrogen was increased gradually until foaming occurred in the column.

After determining the conditions of foaming in the experimental column, the salt solution was spiked with defoaming agent in 25 ppm increments to determine the concentration at which the defoaming agent controls foaming in the column.

Effect of Defoaming Agents on Benzene Removal

The proposed Late Wash Facility will use sparging to remove the benzene in solution. Previous tests were performed in 1992 to determine if surfynol 104E inhibits benzene removal.⁷ Additional testing was done as part of this study using tributylphosphate. The results from these tests were compared to determine the effects of defoaming agent on the removal of benzene.

The sparging apparatus is a small scale version of the sparge tank in the proposed Late Wash Facility (see Figure 3 for details). The nitrogen sparge rate for the tests was 0.62 SCFM/ft² (this is the proposed design rate for the Late Wash facility).

The sparge tank was filled with 691 mL of salt solution premixed with a specified amount of benzene and defoaming agent. The solution was sampled prior to any sparging. Nitrogen was sparged through the solution at the specified rate. Samples were taken every two minutes for the first ten minutes, followed by two additional samples at ten minute intervals. Several tests were run with different concentrations of the defoaming agent to determine its effect on benzene removal.

Simulation of Benzene Addition to the ITP Test Facility

To effectively test the ITP benzene strippers, it is necessary to achieve a specified concentration of benzene near the solubility limit in the feed to the stripping column. At the ITP facility it is planned to recirculate the salt solution through the feed tank and use an injection port to add the benzene. The benzene addition apparatus of this study is a small scale version of the ITP temporary facility feed tank. The test simulated the addition of benzene through an injection port in the piping system.

The apparatus (see Figure 4 for details) was designed on the following assumptions.

- The reservoir was scaled to approximately 1:5,000 of the existing ITP design.
- The reservoir contained 66.7% liquid by volume and 33.3% vapor by volume.
- The benzene was added at the rate of 0.1 mL every 10 minutes over a four hour period to simulate the injection profile proposed for the facility during benzene acceptance testing.

A 5785 mL reservoir was filled with 3856 mL of salt solution. A total of 2.5 mL of benzene was added to the salt solution through an in-line injection port. The solution was recirculated continuously for six hours at one gallon per minute using a micro pump. A sample of the reservoir contents was taken each hour.

Surface Wetting Tests

Wetting tests were done to determine if the defoaming agents would spread over a stainless steel coupon in a salt free environment and when the coupon is coated with salt solution. A drop of tributylphosphate and a drop of surfynol 104E were added onto stainless steel coupons with and without a coating of salt solution to determine if the defoaming agents would spread across the surface.

Residual Cleaning Tests

Tests were performed to determine an efficient cleaning method of tributylphosphate contaminated equipment. A 500 mL graduated cylinder was filled with 200 mL of salt solution and sparged with nitrogen to obtain an original foam height. Tributylphosphate was added to the solution, and sparged with nitrogen to determine the effect of the defoaming agent on foam height. The graduated cylinder was rinsed with a small volume of deionized water and removed with a pipette. Clean salt solution was added to the cylinder and sparged with nitrogen to determine if the solution foamed. If it did not foam, then the cleaning was not sufficient to remove the tributylphosphate. Various quantities of deionized water were used to rinse the cylinder to determine the appropriate amount needed to clean the glassware.

RESULTS AND OBSERVATIONS

Analytical Support

The benzene concentrations reported in this document were provided by the DWPT analytical laboratory at TNX. Organic and inorganic chemical concentrations were provided by both the DWPT laboratory and ADS laboratory in 773-A.

Defoaming Agent Tests

J. P. Bibler researched a variety of defoaming agents available for testing.² This study eliminated ineffective defoamers, and identified defoamers which inhibit the salt solution foaming. Tributylphosphate, Gafac[®] RA-600, and surfynol 104E were most effective in eliminating foaming (see Table 4 and Figure 5). The remaining agents proved relatively ineffective. It was determined to test surfynol 104E and tributylphosphate.

Simulating Foaming of the Decontaminated Stripping Column

After identifying the defoaming agents which inhibit foaming, an experimental stripping column was installed. Various random packing materials were placed in the column to determine the material which provided reproducible foaming. The following packing materials were tested: glass beads, Pro-Pak protruded metal distillation packing, and stainless steel sponge padding. The tests revealed that the stainless steel sponge padding offered the best foaming reproducibility.

Tributylphosphate and surfynol 104E were tested in the small scale column to determine if they were effective in eliminating foaming in packed columns. Although Gafac[®] RA-600 successfully inhibited foam formation, it was not tested. Gafac[®] RA-600 created a residual foam layer which would not collapse rapidly. The foam layer was approximately 25 mm in height and was a white froth. The effectiveness of tributylphosphate and its expected compatibility with saltstone allowed the elimination of Gafac[®] RA-600 from further testing. Defoaming agent was added to the salt solution in 25 ppm increments. The solution was stirred in a flask for approximately one-half hour. The solution was added to the small scale stripping column and observed visually for foaming.

From these tests tributylphosphate is recommended for use in the process at a concentration equal to or less than 150 ppm. The testing illustrated that 75 ppm was the initial concentration that controlled foaming at the baseline conditions. At a concentration of 150 ppm the system did not foam at the baseline conditions as well as conditions of higher nitrogen flow rates. In several tests solids were observed in solution at a tributylphosphate concentration of approximately 100 ppm.

Surfynol 104E did not control foaming at or above the baseline conditions. The surfynol 104E was added in increments up to a concentration of 550 ppm. At all concentrations this defoaming agent failed to inhibit foaming. Solids began to form at a concentration of approximately 100 ppm.

Effect of Defoaming Agents on Benzene Removal

After demonstrating that tributylphosphate eliminates foaming in the stripping column, its effect on benzene removal from the salt solution was tested. The sparging apparatus was used for this testing. Salt solution spiked with benzene and with tributylphosphate concentrations of 0 ppm, 150 ppm, and 300 ppm was sparged with nitrogen at a flow rate of 0.6 L/min.

From the results (see Table 5 and Figure 6) it was determined that the defoaming agent tributylphosphate did not adversely affect the benzene removal. After 10 minutes of sparging the benzene concentration was less than 1 ppm for all three tributylphosphate concentrations.

Simulation of Benzene Addition to the ITP Test Facility

An experiment simulating benzene addition to the test facility was completed. The purpose of this test was to determine if a benzene concentration near the solubility limit could be achieved in the temporary facility tank. The results (see Table 6 and Figure 7) indicate that the facility should be able to achieve the desired liquid phase concentration of benzene by recirculating the salt solution between four to six hours.

Surface Wetting Tests

Wetting tests were completed to compare if a drop of tributylphosphate and a drop of surfynol 104E would spread across the surface of a stainless steel coupon. Drops of each were placed on a coupon and allowed to spread for five minutes before observing. Tributylphosphate covered the most surface area in a salt free environment (see Figure 8). The coupons were placed in salt solution overnight. A drop of tributylphosphate and a drop of surfynol 104E were placed on the salt covered coupons. The amount of area covered by the surfynol 104E was slightly greater than the area covered by tributylphosphate.

Residual Cleaning Tests

A cleaning test was completed to determine how much deionized water is needed to clean the surface of the tank after the addition of tributylphosphate. Various rinsings were done to determine the volume percent needed to clean the tributylphosphate from the walls of the cylinder. The cleaning test results indicated that deionized water at 5% the tank volume would be sufficient for cleaning (see Table 7).

CONCLUSIONS

An experimental program was conducted to identify an effective defoaming agent for the ITP stripping columns. From this program the following results were determined:

- Tributylphosphate is the defoaming agent recommended for use in the ITP stripping columns at a concentration of no more than 150 ppm.
- Tributylphosphate does not adversely affect benzene removal.
- The tributylphosphate can be cleaned from the temporary feed tank at ITP using deionized water at 5% of the tank volume.
- Benzene can be added to the temporary feed tank at ITP using the benzene injection port within a 4-6 hour period to obtain a saturated solution.

QUALITY ASSURANCE

All laboratory procedures, data, and observations discussed in this report are recorded in laboratory notebook WSRC-NB-92-145. The following documents defined this experimental program:

Technical task requests:

- HLE-TTR-93013A/B "Benzene Stripper Performance Evaluation"
- HLE-TTR-93044 "Kinetics of Benzene Dissolution"

Program plans:

- SRT-LWP-93-035 "Program Plan to Evaluate and Compare how Various Surfactants Effect Foaming of a Simulated Decontaminated Salt Solution which Feeds the In-tank Precipitation Stripping Columns"
- SRT-LWP-93-036 "Program Plan to Evaluate the Characteristics of a Simulated Salt Solution in the Small Scale Version of the In-Tank Precipitation (ITP) Decontaminated Salt Solution (DSS) Stripping Column"
- SRT-LWP-93-037 "Program Plan to Evaluate how Tributylphosphate (TBP) affects Benzene Removal from Salt Solution Using Nitrogen Sparging"
- SRT-LWP-93-041 "Program Plan to Evaluate Benzene's Solubility in a Salt Solution"

REFERENCES

- (1) Barnes, M. J. and McGlynn, J. F., "Lab Scale Defoaming Tests on the In-Tank Precipitation Benzene Stripper Column (U)", SRT-LWP-93-021, March 19, 1993.
- (2) Bibler, J. P., "Other Antifoaming Agents for the ITP Stripper", SRT-LWP-93-032, April 13, 1993.
- (3) Georgeton, G. K., "Development and Application of a Mathematical Model for the Benzene Stripping Columns in the ITP Process (U)", WSRC-RP-89-1442, December 28, 1989.
- (4) Georgeton, G. K., Barnes, M. J., Bibler, J. P., Hobbs, D. T., Walker, D. D., and Langton, C. A., private communication, September, 1992 - May, 1993.
- (5) Georgeton, G. K., and Wilson, B. M., "Analysis of ETC Test Results for Benzene Removal with Structured Packing (U)", WSRC-RP-89-871, September 12, 1989.
- (6) Grant, Roger and Grant, Claire, Grant & Hackh's Chemical Dictionary, 5th Edition, McGraw-Hill, Inc., 1987.
- (7) Landon, L. F., "Benzene Striping Late Wash Facility - Status Report", SRTC-PTD-92-036, July 31, 1992.
- (8) McGlynn, J. F., "Results of the Small Scale Testing on how Late Washing Affects the In Tank Precipitation (ITP) Decontaminated Salt Solution (DSS) Stripping Column (U)", SRT-LWP-92-090, December 8, 1992.
- (9) Perry, Robert H., Green, Don W., Maloney, James O., Perry's Chemical Engineers' Handbook, 6th Edition, McGraw-Hill Book Company, Inc., 1984.
- (10) Treybal, Robert E., Mass Transfer Operations, 3rd Edition, McGraw-Hill Book Company, Inc., 1987.
- (11) Walker, D. D., "Vapor Pressure of Benzene, Methanol, and Isopropanol Over Salt Solutions (U)", DPST-88-661, March 28, 1989.
- (12) Weast, Robert C., Astle, Melvin J., and Beyer, William H., CRC Handbook of Chemistry and Physics, 64th Edition 1983-1984, Chemical Rubber Publishing Company, 1985.

TABLE 1. CONCENTRATION OF A TYPICAL SIMULATED SALT SOLUTION

Chemical	Concentration (Moles/Liter)
NaOH	1.30
NaAl(OH) ₄	0.27
NaNO ₃	1.53
NaNO ₂	0.59
Na ₂ CO ₃	0.14
Na ₂ SO ₄	0.13
Na ₂ SiO ₄	0.00
Na ₂ C ₂ O ₄	0.013
NaB(C ₆ H ₅) ₄	0.0051 (sat.)
Na ⁺	4.83

TABLE 2. CONCENTRATION OF SALT SOLUTION FROM THE EXPERIMENTAL LABORATORY FILTER

Chemical	Concentration (Moles/Liter)	Concentration (ppm)
Hydroxide	1.701	28929
Carbonate	0.017	1020
Aluminate	0.255	24228
Aluminum	> 0.265	> 7148
Sodium	> 3.192	> 73393
Silicon	<0.000	13.694
Phosphorous	<0.000	< 0.9
Phosphate	<0.001	< 100
Oxalate	<0.001	< 100
Sulfate	0.019	1858
Nitrate	0.828	51318
Nitrite	0.211	9705
Chloride	0.009	307
Fluoride	0.001	23
Tetraphenylborate	0.0006	N/A

FIGURE 1. GRADUATED CYLINDER FOR SALT SOLUTION

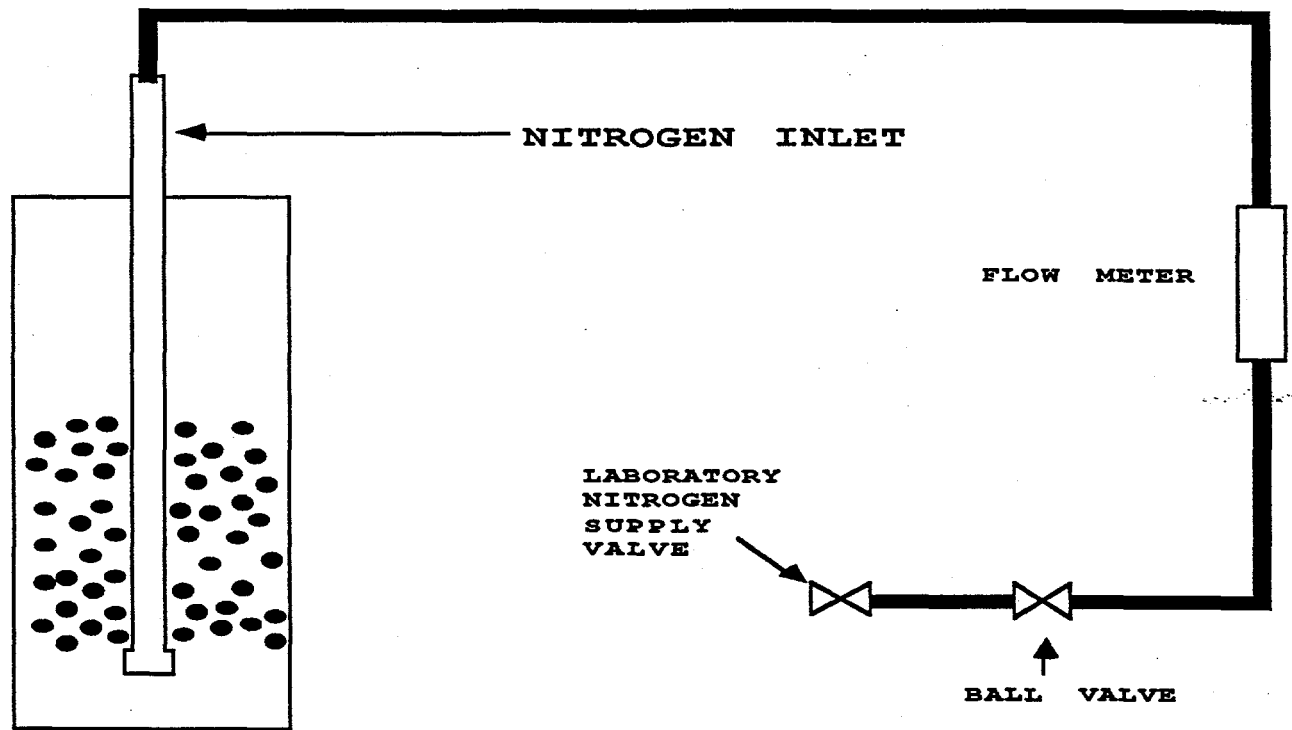
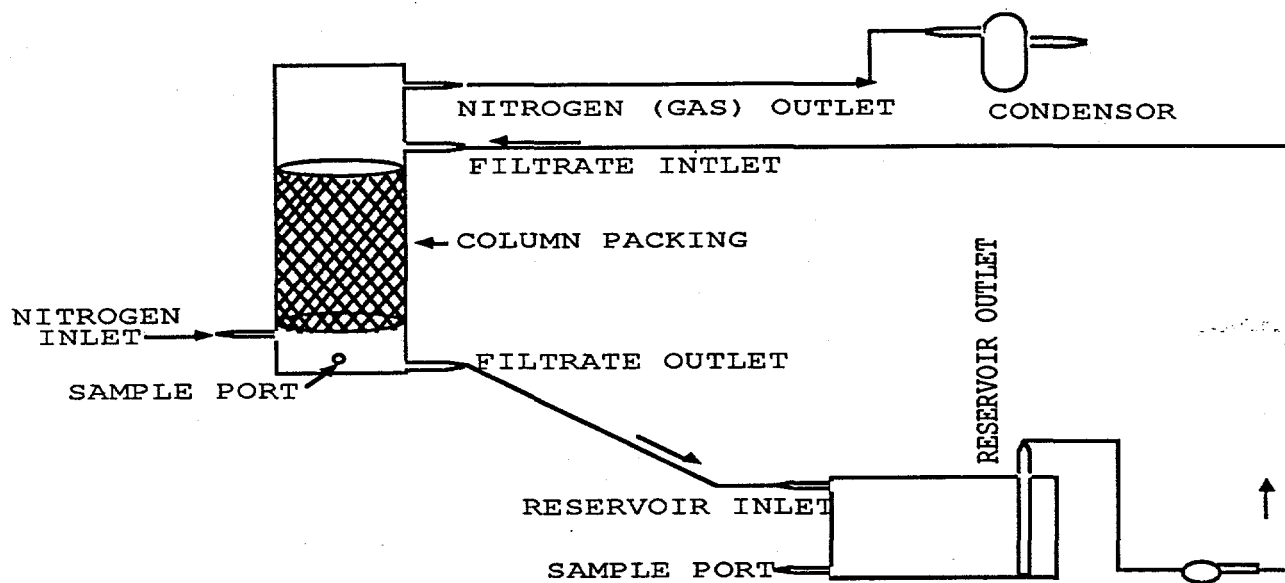


FIGURE 2. SMALL SCALE DECONTAMINATION SALT SOLUTION APPARATUS



Dimensions and Flow Rates

Column packing height:	40.64 cm
Column packing diameter:	5.08 cm
Nitrogen flow rate:	1.93 SCFM = 54.65 L/min*
Filtrate flow rate:	1.89 L/min*
Reservoir volume:	680 mL

* These values correspond to the maximum flow rates of 115 GPM filtrate and 440 SCFM nitrogen in the large scale column.

TABLE 3. NITROGEN AND FILTRATE FLOW RATES AS COMPARED TO THE IN-TANK PRECIPITATION PROCESS

ITP Column	<u>Nitrogen Flow Rate</u>	
	Small Scale Column	% of Maximum
440 SCFM	54.65 L/Min	100%
403 SCFM	50.00 L/Min	91.5%
382 SCFM	47.50 L/Min	86.9%
362 SCFM	45.00 L/Min	82.3%
342 SCFM	42.50 L/Min	77.8%
322 SCFM	40.00 L/Min	73.2%
282 SCFM	35.00 L/Min	64.0%
100 SCFM	12.40 L/Min	22.7%
50 SCFM	6.20 L/Min	11.4%

ITP Column	<u>Filtrate Flow Rate</u>	
	Small Scale Column	% of Maximum
115 GPM	0.5 GPM	100%
92 GPM	0.4 GPM	80%
69 GPM	0.3 GPM	60%
46 GPM	0.2 GPM	40%
23 GPM	0.1 GPM	20%

FIGURE 3. SMALL SCALE SPARGING APPARATUS FOR
BENZENE REMOVAL EFFICIENCY TESTS

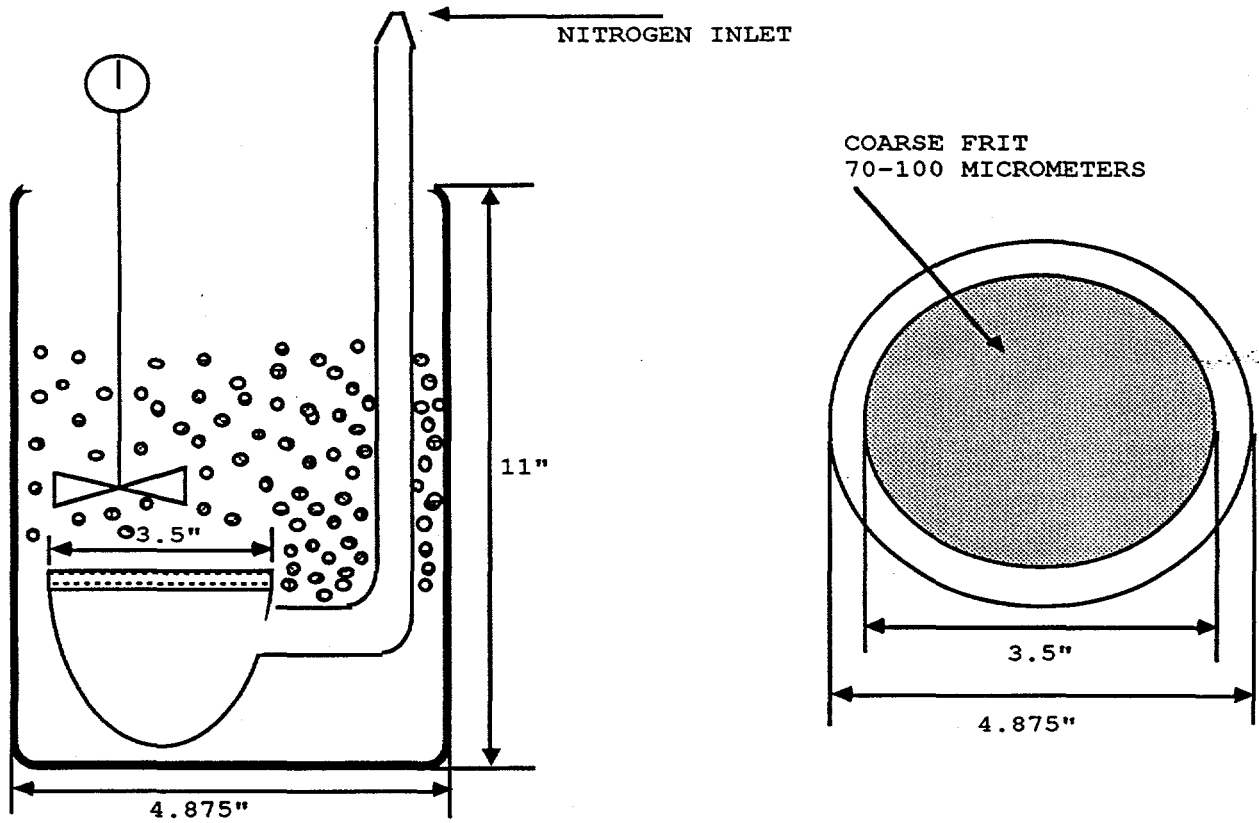
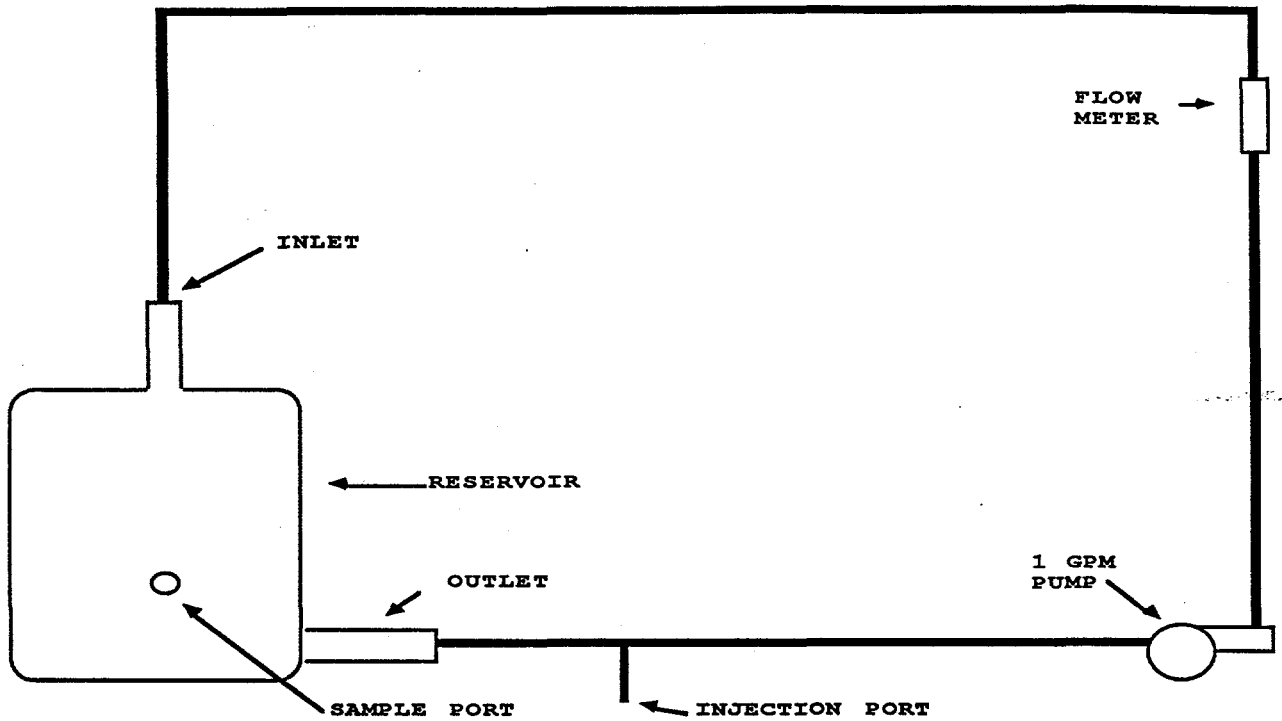


FIGURE 4. SMALL SCALE BENZENE ADDITION APPARATUS



Dimensions and Flow Rates

Tank volume:	5785 mL
Tank Vapor Volume:	1929 mL
Tank Liquid Volume:	3856 mL
Total Benzene Added:	2.5 mL
Benzene Addition Rate:	0.1 mL every 10 min.
Pipe Diameter:	1.27 cm
Pipe Length After Injection Port:	245.11 cm
Flow Rate:	3.79 L/min

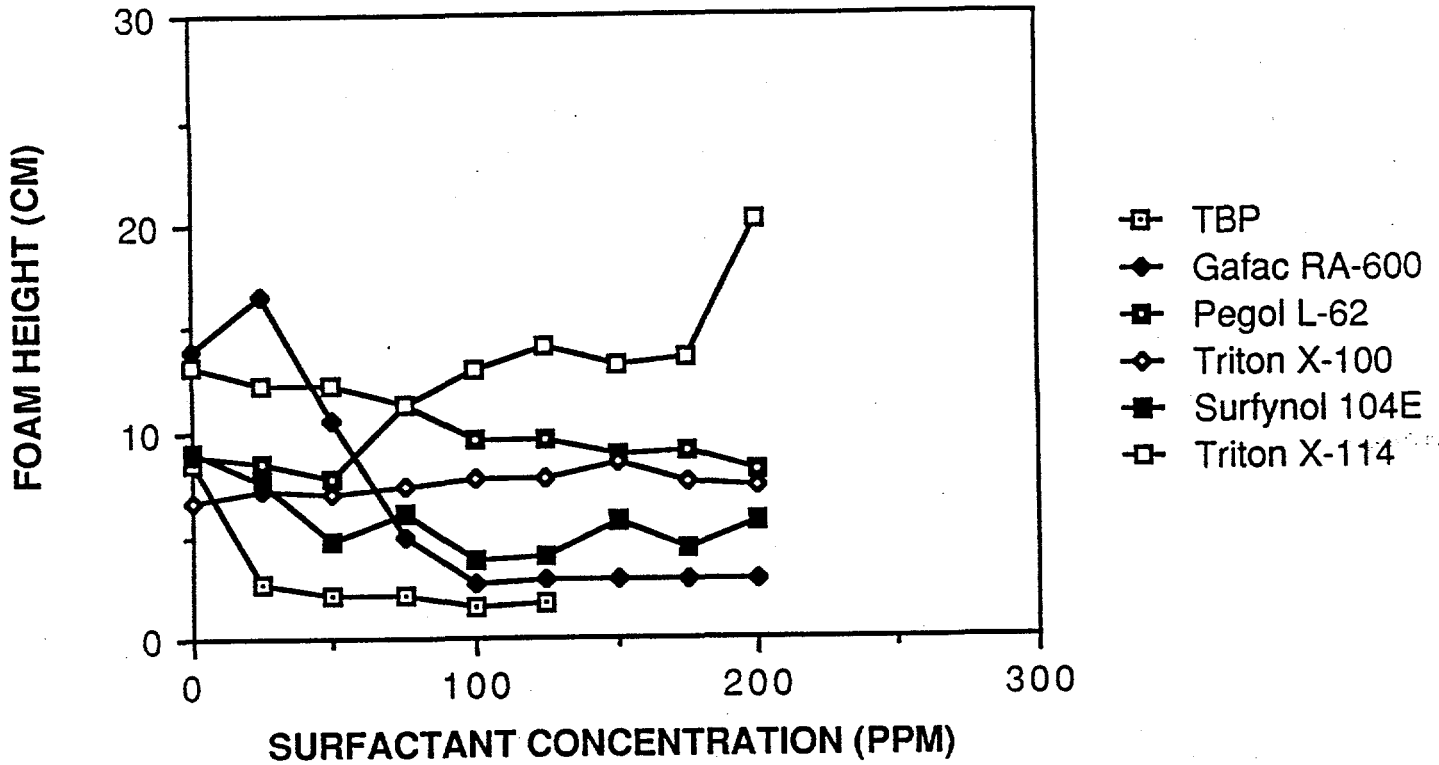
TABLE 4. DEFOAMING AGENTS EFFECT ON FOAM HEIGHT MEASUREMENTS

<u>Defoaming Agent</u> <u>Concentration (ppm)</u>	<u>Foam Height (cm)</u>		
	<u>TBP</u>	<u>Gafac® RA-600</u>	<u>Pegol® L-62</u>
0	8.55	13.9	8.8
25	2.60	16.55	8.5
50	2.10	10.65	7.8
75	2.00	5.0	11.25
100	1.50	2.55	9.6
125	1.65	2.75	9.55
150		2.8	8.9
175		2.8	9.0
200		2.85	8.15

	<u>Triton® X-100</u>	<u>Triton® X-114</u>	<u>Surfynol 104E</u>
0	6.65	13.3	9.0
25	7.25	12.2	7.5
50	6.95	12.2	4.7
75	7.45	11.4	6.0
100	7.7	13.1	3.75
125	7.8	14.1	4.05
150	8.55	13.3	5.75
175	7.6	13.6	4.35
200	7.4	20.1	5.65

	<u>Dow Corning® Antifoam 544</u>
0	8.28
35.5	8.85
60.0	8.75
147.5	8.35
183.5	8.6

FIGURE 5. RESULTS FROM THE GRADUATED CYLINDER TESTING APPARATUS



** The Dow Corning® Antifoam 544 was not included on the graph because it was measured in different concentration increments than the other defoaming agents tested, and this defoaming agent was ineffective in inhibiting the foam formation.

TABLE 5. RESULTS FROM THE SPARGING TEST APPARATUS

	TBP 0 ppm	TBP 150 ppm	TBP 300 ppm
Time (min)	Benzene ppm	Benzene ppm	Benzene ppm
0	97.49	25.29	68.94
2	13.20	2.82	8.27
4	1.14	0.92	0.77
6	0.27	0.10	0.42
8	0.83	0.62	1.39
10	0.92	0.79	0.15
20	0.97	2.09	0.93
30	0.70	1.13	1.52

FIGURE 6. GRAPH OF SPARGE TEST RESULTS

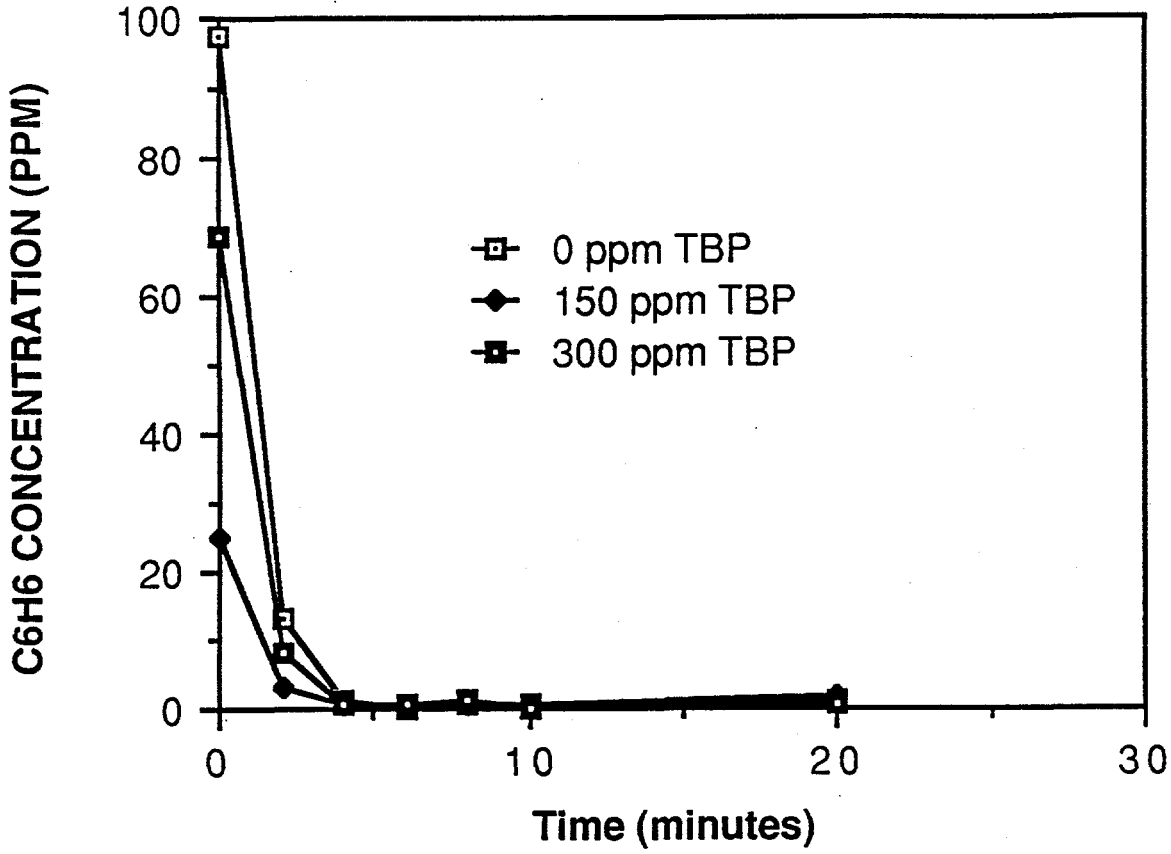


TABLE 6. RESULTS FROM THE BENZENE ADDITION APPARATUS

Time (hours)	Benzene (ppm)
0	0
1	68.7
2	108.5
3	184.7
4	251.8
5	208.2
6	206.3

FIGURE 7.

GRAPH OF BENZENE ADDITION TEST RESULTS

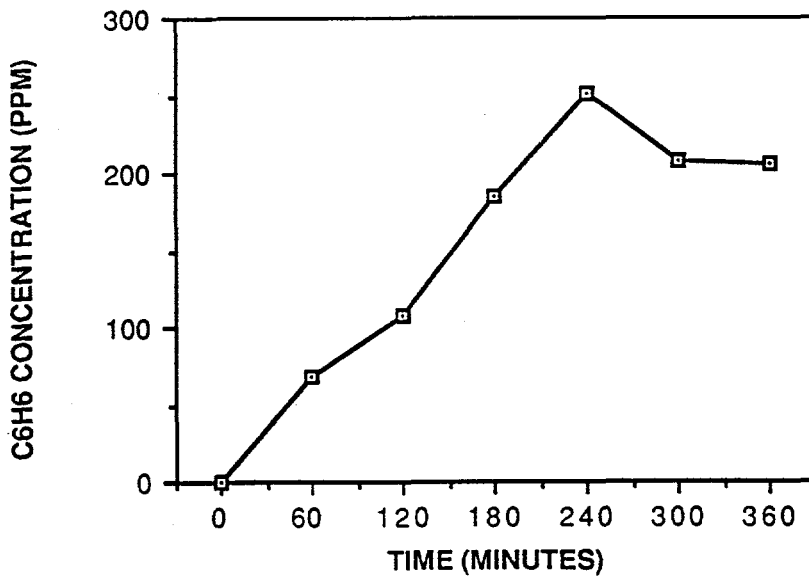


FIGURE 8. RESULTS OF WETTING TEST

OBSERVATIONS:

Dimensions of the coupon

L = 50 mm

W = 19 mm

Coated surface in a salt free environment

Salt solution after 5 minutes

L = 24 mm

W = 6 mm

Surfynol 104E after 5 minutes

L = 16 mm

W = 7 mm

Tributylphosphate after 5 minutes

L = 41 mm

W = 10 mm

Coated surface after the coupon was covered with salt solution

Tributylphosphate after 5 minutes

L = 8 mm

W = 8 mm

Surfynol 104E after 5 minutes

L = 12 mm

W = 7 mm

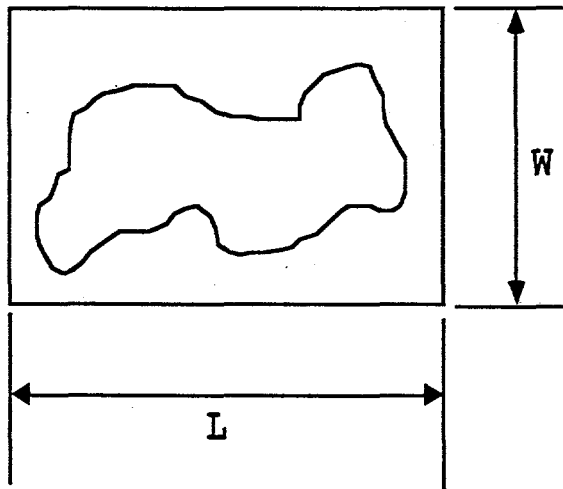


TABLE 7. CLEANING TEST RESULTS

ANTIFOAM	FOAM HEIGHT (mm)	ANTIFOAM CONC. (ppm)	OBSERVATIONS ** & REMARKS
N/A *	97	0	Initial measure
TBP *	28	124.5	
N/A *	39	0	New salt solution added without rinsing the surface
N/A @	114	0	Initial measure
TBP @	17	130	
N/A @	45	0	After rinsing with 5 mL DI H ₂ O
N/A #	124	0	Initial measure
TBP #	82	32.5	
N/A #	76	0	New salt solution added without rinsing surface
N/A ^	112	0	Initial measure
TBP ^	52	126	
N/A ^	90	0	After rinsing with 25 mL DI H ₂ O

* These tests were performed to observe how the salt solution foams after a run with 124.5 ppm tributylphosphate and without rinsing the cylinder walls.

@ These tests were performed to observe how the salt solution foams after a run with 130 ppm tributylphosphate and rinsing the cylinder walls with 5 mL of DI water.

% These tests were performed to observe how the salt solution foams after a run with 32.5 ppm tributylphosphate and without rinsing the cylinder walls.

^ These tests were performed to observe how the salt solution foams after a run with 126 ppm tributylphosphate and rinsing with 25 mL DI water.

** The initial volume of the solutions in these tests was 200 mL.