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Testing of Stripping Columns for the Removal of Benzene from Aqueous Radioactive Salt Solution (U)

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TESTING OF STRIPPING COLUMNS FOR THE REMOVAL OF BENZENE FROM AQUEOUS RADIOACTIVE SALT SOLUTIONS

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DISCLAIMER

INTRODUCTION

Radioactive high level wastes (HLW) generated from production of special nuclear materials at the Savannah River Site (SRS) are held in interim storage in 51 underground, million gallon tanks. Radioactive cesium (^{137}Cs) is segregated by evaporation of aqueous waste solution for interim storage in a salt matrix comprised of Na and K salts or in concentrated salt solution. The saltcake will be dissolved and ^{137}Cs will be separated from the nonradioactive salts in solution in the In-Tank Precipitation (ITP) Process. The cesium will be combined with other radioactive species and glass formers to be melted and poured into stainless steel canisters in the Defense Waste Processing Facility (DWPF). The salt solution remaining after decontamination in the ITP process will be incorporated into grout for disposal at the site's Saltstone facility.

In the ITP facility, sodium tetraphenylborate (STPB) will be added to precipitate the cesium. Potassium in the waste solution also reacts with STPB and precipitates. Due to radiolytic and chemical degradation of the tetraphenylborate (TPB) precipitate, benzene is generated. The benzene dissolves into the decontaminated salt solution (DSS) and into water (WW) used to "wash" the precipitate to lower the soluble salt content of the slurry. Safety and processing requirements for disposal of the DSS and for temporary storage of the WW dictate that the benzene concentration be reduced.

Two columns loaded with structured packing will be used to strip the benzene - one column dedicated to each flow stream. Humidified nitrogen will be used as the stripping medium, and will flow countercurrent to the liquid through the packing. The diameter of the DSS column is 30 inches (0.76 m), and the diameter of the WW column is 16 inches (0.41 m). Both columns contain 20 ft (6.9 m) of Type 1Y Flexipac™ structured packing. The DSS column was designed to process between 20 (1.3) and 115 gpm (7.3 l/sec) of salt solution (5M Na^+) using 440 cfm (0.21 m^3/sec) of nitrogen to reduce the benzene concentration to less than 2.5 mg/l. The WW column was designed for a maximum liquid rate of 33 gpm (2.1 l/sec), a typical flow of 14 gpm (0.88 l/sec), and a nitrogen rate of 220 cfm (0.11 m^3/sec), with an effluent benzene concentration below 5 mg/l. The WW will initially have the same concentration as DSS, but will be gradually diluted until the Na^+ content is below 0.2M.

During run-in testing of the large ITP column with simulated DSS, the differential pressure (DP) limit was reached at liquid and gas flow rates well below the maximum design rates. The limit was set at 40 in (1.02 m) water column (wc), based on the heuristic that flooding begins to occur at a DP of about 2 in wc/ft (0.16 m wc/m) of packing. The maximum liquid flow achieved before the column DP limit was reached was 60 gpm (3.8 l/sec), with the nitrogen flow rate set at 50 scfm ($2.4*10^{-2}$ m^3/sec), well below the design flow rates. The design liquid and gas flows were far from the column's flooding point, and the impact of density and viscosity of the salt solution on the DP across the packing was determined to be relatively insignificant. The cause of the high DP was identified to be foaming in the solution caused by the presence of tetraphenylborate. When soluble TPB is present, as in the ITP solutions, lab and pilot scale experiments have shown that a large and stable volume of foam is generated, even when the gas rate is relatively low.

To determine the impact of foaming on ITP column operation and performance, a test program was implemented at Koch Engineering Company's (the packing vendor) test facility using a pilot scale stripping column. The objective of the testing was to verify that the benzene in DSS and WW could be removed to specified levels at design feed throughputs, in a test program conducted in parallel with the startup testing of the full production facility. A series of tests was identified to determine maximum processing capability in the columns, to evaluate potential methods to reduce and/or eliminate foaming, and to confirm that stripping efficiency requirements will be satisfied.

TESTING PROGRAM

Program Description

An 8 inch (0.2 m) diameter pilot column was configured with two approximately 10 ft (3.05 m) beds of Type 1Y Flexipac™ structured packing (total height of 20.3 ft (6.2 m)). A collector/distributor was installed between the beds to allow sample collection for benzene analysis and to provide proper feed distribution in the bottom bed. For

the final test runs, the intermediate distributor was removed, and the beds combined to form one continuous 20 ft (6.1 m) bed (analogous to the ITP process columns).

Liquid was pumped to the column from either a 150 gal (570 l) feed tank for recycled feed operation or from a tanker truck for single-pass operation. Before entering the column, the feed solution was passed through 1 μm (nominal) cartridge filters to remove suspended solids. The liquid feed was maintained at constant temperature using a heat exchanger. Nitrogen was drawn from the facility's central supply, and was passed through a vessel filled with water to prehumidify the gas before introduction into the column. Nitrogen was maintained at 10 psig ($6.9*10^4$ Pa)and 30°C, and the column pressure was also maintained at 10 psig ($6.9*10^4$ Pa). A schematic diagram of the test facility is shown in Figure 1.

Superficial liquid flow rates were varied between 1 (0.68 l/sec/m^2) and 25 gpm/ft^2 (17 l/sec/m^2). The feed rates covered the range of flow conditions expected in the full scale DSS and WW columns, with 25 gpm/ft^2 (17 l/sec/m^2) corresponding to maximum liquid design rates of 115 gpm (7.3 l/sec) and 33 gpm (2.1 l/sec), respectively. The nominal flow rate for the WW column is expected to be 14 gpm (0.89 l/sec), equivalent to the superficial flow rate of 10 gpm/ft^2 (6.8 l/sec/m^2). The feed solutions (as described below) were nonradioactive simulants of the decontaminated salt solution and of the wash water at the midpoint in the washing operation.

The rate of nitrogen flow was varied over a range of conditions to simulate flow in both ITP columns. The flow was tracked using Koch's normalized vapor flow variable F_S, as defined in Eqn. 1. For the ITP process, F_S is 0.52 for a nitrogen flow rate of 440 cfm (0.21 m^3/sec) at 10 psig ($1.7*10^5$ Pa) and 30°C in the DSS column. For the WW column, F_S is 0.89 for a nitrogen rate of 220 cfm (0.11 m^3/sec) at 10 psig ($1.7*10^5$ Pa) and 30°C. The nitrogen rate was varied over the range $0.2 \leq F_S \leq 1.2$.

$$F_s\ [(ft/sec)\cdot(lb/ft^3)^{0.5}] = v_g * \rho_g^{0.5} \quad (1)$$

v_g = superficial gas velocity (ft/sec)

ρ_g = gas density (lb/ft^3)

Column instrumentation included DP measurement across each bed of packing and across the entire column, and an overall column pressure measurement. Thermocouples were used to measure the temperatures of the column feed streams. Mass flow meters were used to measure the liquid and nitrogen flow rates. Liquid and gas flow rates and differential pressure data were monitored using the test facility's data acquisition system. Once flow rates were adjusted, the column DP was tracked and an average and standard deviation calculated for a 15 minute period. The column was considered to be operating in a stable regime when the DP remained steady for 15 minutes.

Test Methodology

The testing program was designed to evaluate both the hydraulic performance and the stripping efficiency of the column. The goals of the hydraulic evaluation were to reproduce the high DP observed in start up testing, determine maximum flow rates through the column, and evaluate several means of reducing and/or eliminating foaming (the cause of the high DP) to achieve design throughput. For evaluating the stripping efficiency, the benzene concentration at the bounding liquid and gas flow rates would be evaluated for simulated DSS and WW solutions. (The stripping efficiency was not measured through the use of transfer units, but was evaluated based on the bottoms concentration (i.e., proof in the pudding). If the bottoms concentration was perceived to be close to the limits, then more detailed evaluation and testing would have been undertaken.) A series of tests was developed in which the process "variables" of interest were systematically evaluated. A summary of the conditions for each test is listed in Table I.

Test 1

The dry bed pressure drop for the column was determined initially to verify column operation. Nitrogen was passed through the column at various rates, and the differential pressure across the column was measured. Differential pressures per unit height of packing are shown in Figure 2.

Test 2

Following the dry bed runs, a 150 gal (570 l) batch of Baseline feed solution was prepared to the composition shown in Table II. The amount of STPB added was only about 25% of the amount needed to fully precipitate all the potassium and saturate the remaining solution with STPB. As described earlier, the high DPs in the ITP runs were attributed to foaming caused by the presence of soluble TPB. With only a minimal amount of TPB added, the solution provided a baseline of a minimally foaming salt solution.

The Baseline feedstock was processed through the column at liquid rates ranging from 1 (0.68 l/sec/m^2) to 25 gpm/ft^2 (17 l/sec/m^2). The column bottoms were recycled to the feed tank and the liquid temperature was maintained at 30°C. Nitrogen flow was varied to cover the range of F_S between 0.2 and 1.2 for each constant liquid rate. The family of DP curves was generated and plotted along with the dry bed DP data in Figure 2. It should be noted that the slopes of the 20 (14 l/sec/m^2) and 25 gpm/ft^2 (1.7 l/sec/m^2) curves begin to increase at higher N_2 flow rates, indicating the approach to flooding conditions.

Test 3

In this test, the impact of a higher feed temperature was evaluated. An increase in temperature typically reduces the density and viscosity of liquids, and was consequently expected to reduce the foaming tendency of the solution. The feed temperature was raised to 60°C, which is the maximum expected temperature during ITP processing. Liquid and gas rates were again varied over the range of flows to generate the loading curves. The differential pressure at various loadings is shown in Figure 3. Because of difficulty in maintaining the feed solution at the

higher temperature and evaluation of preliminary results, operation at 25 gpm/ft^2 (17 l/sec/m^2) was stopped after data were collected for a nitrogen F_S rate of 0.6.

Test 4

A new batch of salt solution (Standard Solution) was prepared for the fourth test at the composition shown in Table II. Sodium TPB was added in excess to precipitate all of the potassium and to saturate the remaining solution. The objective of this test was to gradually increase the liquid and vapor flow rates to determine the maximum stable operating point that could be expected in the ITP facility, without reduction or elimination of the high DP. However, even at low liquid and nitrogen rates, a stable operating point could not be obtained, and the program was moved forward into Test 5.

Test 5

As the Standard solution was recirculated through the test column, antifoam (AF) was injected into the feed line to reduce the foam and lower the DP across the packing. Tri-n-butyl phosphate was used as the AF, and was injected at a target concentration of 100 ppm. Tri-n-butyl phosphate was recommended based on results of internal laboratory testing at SRS of several antifoams and a screening evaluation of the general classes of commercially available antifoams. The DP across the column immediately dropped to manageable levels and conditions within the column rapidly stabilized. Column operation remained stable over the entire range of liquid and gas rates. Differential pressures for the conditions in Test 5 are shown in Figure 4.

At the conclusion of this test, the column was flushed and cleaned with oxalic acid to remove any solids that may have accumulated in the packing or in the distributors and to flush the system of residual antifoam.

Test 6

In the sixth test, the stripping efficiency of the column when processing DSS was evaluated. Simulated DSS was prepared by a third-party vendor with the concentration shown in Table II and shipped to the test facility in a

tanker trailer. The solution was spiked with benzene and TPB during preparation, but additional benzene and TPB were added by Koch personnel to make up for benzene losses to the tanker vapor space and for reaction of STPB with potassium impurities. The solution was drawn directly from the tanker and was processed through the column in single pass mode (i.e., no recycle). Nitrogen flow was set to correspond to an F_S of 0.5, the DSS column design rate. Liquid feed rate was initially set to 10 gpm/ft^2 (6.8 l/sec/m^2) to simulate WW column feed during initial precipitate washing at ITP. The feed rate was then increased to 25 gpm/ft^2 (17 l/sec/m^2) to simulate DSS column operation at the maximum liquid feed rate. No AF was injected at this point, as the column DP remained relatively stable and below 40 in (1.02 m) wc. After about 1.5 hours of operation, the antifoam injection system was started. Differential pressure across the column dropped to levels comparable to those in Test 5. Samples of the feed, bottoms and mid-column flows were collected throughout Test 6 for benzene analysis. A time trace of the column DP (including highlights of significant events) is shown in Figure 5.

Test 7

The stripping efficiency of the column when processing simulated WW was evaluated in Test 7. Deionized water was added to the feed solution remaining in the tanker to dilute the salt concentration to represent the wash water at the mid-point of the washing cycle (as shown in Table II). Additional TPB and benzene were added, and the solution was mixed overnight in the tanker. The initial feed rate was set to 10 gpm/ft^2 (6.8 l/sec/m^2; nominal WW column processing rate) and processed through the column in single pass mode. The nitrogen rate was set to correspond to an F_S of 0.5. After 2 hours of operation, the feed rate was increased to about 23 gpm/ft^2 (16 l/sec/m^2) . The DP across the column steadily increased over the next 3.5 hours of operation, as is shown in the time trace in Figure 6. At that point, continuous AF injection was started. The DP dropped almost instantly and remained steady until the conclusion of the run. Feed, bottoms and mid-column samples were collected for benzene analysis throughout the experiment.

Test 8

Some supplementary testing was performed as Test 8 to qualitatively examine the effects of changes in some operating variables and conditions. The column was flushed and cleaned with oxalic acid to remove any traces of solids and antifoam that may have collected. In preparation for the final testing phase, the packing was reconfigured to a single, 20 ft (6.1 m) bed of packing. A 150 gal (570 l) batch of Supplemental feedstock solution was prepared to the concentrations shown in Table II. Excess STPB was added to precipitate the potassium (present as impurities in the sodium salts) and saturate the solution. The system was operated in recycle mode. Changes in operating parameters were conducted without interrupting flow. Feed temperature was maintained at 30°C. Feed and bottoms samples were collected periodically.

In Test 8A, the impact of operation at a lower nitrogen rate was evaluated. The liquid rate was set to 25 gpm/ft^2 (17 l/sec/m^2), and the nitrogen flow was set to an F_S of 0.2. No antifoam was used in this experiment. Benzene was injected at a rate such that the resulting concentration in the feed was 100 mg/l. The column DP remained steady and below the 40 inches (1.02 m) wc limit, as shown in the time trace in Figure 7.

The liquid and vapor flow rates and the benzene concentration in the feed were varied in Test 8B to again attempt to establish a stable upper operating point without the use of antifoam. The DP increased well above 60 inches wc (1.52 m); steady conditions (identified by a stable DP) were not achieved. After about 5 hours of column operation without being able to establish steady conditions, antifoam injection was started (approximately 100 ppm) and liquid and gas rates were set to design flows. Benzene was injected to give a target concentration of 200 mg/l. The operating conditions were maintained for about 2.5 hours after the start of antifoam injection.

Finally, in Test 8C, the nitrogen flow was increased to a rate corresponding to an F_S of 0.9 while keeping the liquid feed rate constant, thus representing the design nitrogen flow in the WW column. The antifoam injection rate was increased to counter the increase in DP, resulting in an estimated concentration of about 200 ppm. The AF

injection system continued to run through Tests 8B and 8C, gradually increasing the concentration of AF in the column feed.

DISCUSSION OF TEST RESULTS

Column Hydraulics

The high differential pressure observed in the ITP columns during start up testing was reproduced in the testing at the Koch facility. Four different solutions were used as feedstocks during the test program, and all were observed to foam to differing degrees. Consequently, the magnitude of the pressure drop across the packing varied depending on the solution being processed. For the DSS simulant in Tests 4 and 5, the large amount of foaming precluded establishing a stable operating point. In Test 6, only a pseudo steady state was achieved. Three methods were examined to reduce the differential pressure across the packing and thereby improve throughput - increasing the feed temperature, lowering the N_2 flow rate, and injecting antifoam into the feed stream.

The effect of temperature on reducing the column DP was found to be only marginal. The temperature of the baseline solution was raised to 60°C, simulating operation in ITP at the upper temperature limit. The increased temperature was expected to reduce the "consistency" of the liquid, making it much harder for a stable foam to form. As seen in Figure 8, the DP at 60°C was somewhat less than that seen for the same solution at 30°C, at liquid rates of 10 (6.8 l/sec/m^2) and 25 gpm/ft^2 (17 l/sec/m^2). However, the reduction of DP was not substantial enough to restore the design throughput.

Reduced nitrogen flow was also evaluated as a way of reducing the packing DP to levels that are acceptable, presuming that the bottoms benzene concentration will remain below the specified limit. In Test 8A, the nitrogen rate was reduced to correspond to an F_S of 0.2. The DP across the column was not steady throughout the experiment, and the average DP was calculated to be about 23 inches (0.58 m) wc. As seen in the time trace in Figure 7 (after about 60 minutes into the test), the DP continued to slowly decrease, indicating that a steady flow

pattern had not been established. By comparison, when foaming was not present, the column flows would quickly reach a steady flow pattern, and the DP would stabilize within 5-10 minutes.

The most drastic reduction in the column DP was observed when antifoam was present in the feed solution. The DP values were expected to be roughly the same as those of the baseline solution, but as is evident in Figure 9 for liquid loading of 25 gpm/ft^2 (17 l/sec/m^2), the DP across the column was well below the DP observed when the baseline solution was run through the column. For comparison, calculated DP values for the nitrogen-water system [Koch Engineering Co.] are also shown in Figure 9, indicating that the antifoam reduces the pressure drop to levels approaching an "ideal" limit. At liquid flows comparable to maximum rates in the DSS and WW columns, the observed column DP was 1.9 inches (0.05 m) and 11 inches (0.28 m) wc, respectively.

Thus, the greatest success in consistently and effectively reducing the column DP was (not unexpectedly) concluded to be due to the addition of antifoam. Increased temperature did result in a small but only marginally significant reduction in DP. Operation at low nitrogen rates necessarily reduced the pressure drop, but steady flow conditions (and consequently DP) were not observed consistently. Only the antifoam addition reduced/eliminated the foam, putting the column into a stable operating regime.

Benzene Removal Capability

In Tests 6, 7 and 8, the columns were challenged with DSS and WW simulants saturated with benzene at design feed and gas flow rates (superficial flow rates). These tests were performed to show that the concentration limits for benzene (2.5 mg/l for DSS, 5 mg/l for WW) in the column bottoms could be satisfied. The success of the column operation was simply based on the benzene concentration in the bottoms relative to the concentration in the feed, and whether the limits were achieved.

Samples were collected throughout these three tests and analyzed by Koch personnel for benzene concentration. The benzene analysis was performed using the purge and trap method, and the data are reported in Table IV.

Some of the DSS feed samples reflected benzene concentrations above the solubility limit (approximately 180 - 200 mg/l for solution with 5M Na^+). The column performance was conservatively evaluated, however, using the solubility concentration. Any amount above this concentration would be present as a second phase and would be relatively easy to strip.

Benzene was consistently stripped from the high Na^+ solution (DSS simulant) in Test 6, both before and after initiation of antifoam injection. At a liquid rate of 10 gpm/ft^2 (6.8 l/sec/m^2) (representing WW flow at the start of the wash cycle), a nitrogen rate corresponding to an F_S of 0.5 and with no antifoam addition, the benzene concentration in the feed ranged from about 60 to 260 mg/l, and was reduced in all cases to 1.5 mg/l or less. The feed, mid-column and bottoms concentrations are shown in Figure 10. When the liquid rate was increased to 25 gpm/ft^2 (17 l/sec/m^2) (maximum DSS feed), the benzene concentration was reduced to less than 0.5 mg/l. Feed concentrations ranged from about 170 to 280 mg/l. Finally, when the antifoam injection system was started (still keeping the feed and nitrogen flows constant), the benzene feed concentration of 210 to 250 mg/l was reduced to less than 1.5 mg/l.

For the simulated WW solution (Test 7), the benzene concentration was consistently reduced to 2.1 mg/l or less. Starting at a liquid rate of 10 gpm/ft^2 (6.8 l/sec/m^2) and a nitrogen rate corresponding to an F_S of 0.5 and no antifoam, benzene in the feed (560 to 1050 mg/l) was reduced to a concentration less than 0.9 mg/l. The feed rate was then increased to about 23 gpm/ft^2 (16 l/sec/m^2). For benzene feed concentrations ranging from 250 to 950 mg/l, the bottoms benzene concentration was less than or equal to 1.7 mg/l. During antifoam addition at the same feed and nitrogen rates, the bottoms concentration was analyzed to be 2.1 mg/l or less for feed concentrations from 450 to 970 mg/l. Benzene concentrations in the feed, mid-column and bottoms samples collected during Test 7 are shown in Figure 11.

The dependence of stripping "completeness" on well established flow conditions through the column, as indicated by the DP, was obvious during the supplemental testing. The benzene concentration in the bottoms during the first

part of Test 8B varied between 1 and 30 mg/l for feed concentrations ranging between 210 and 360 mg/l. Comparison with the data from Test 6 (under the same flow conditions) reflects that the bottoms concentration in Test 8B varied by about 30 mg/l for a feed increase of 150 mg/l, whereas in Test 6, there was essentially no variation for a feed concentration range of 110 mg/l. During this time, the DP was observed to go as high as 85 in (2.2 m) wc. Some variation in the bottoms was expected based on variations in the feed concentration; however, the relative difference indicated that good flow distribution was necessary to satisfy process requirements.

The presence of the antifoam appeared to have a slight negative effect on the benzene stripping, and the effect was proportional to the amount of AF added. In Test 8B, the AF was continuously injected at a concentration of 76 ppm into the feed, which was passed through the column and recycled into the 150 gal (570 l) feed tank. At a feed rate of 25 gpm/ft^2 (17 l/sec/m^2), the tank contents were turned over every 20 minutes. Towards the latter part of the supplemental testing, the concentration of AF in the tank was estimated to have reached approximately 800 ppm. The amount of antifoam actually flowing to the column (as dissolved and/or entrained) was not determined, however. The benzene concentration in the bottoms at the end of Test 8B was over 20 mg/l. When the N_2 rate was almost doubled for Test 8C, the bottoms concentration did not decrease as would have been expected, but remained over 20 mg/l.

The test results also indicated that less N_2 than designed could be used to strip the benzene from solution, and still satisfy the concentration requirements for the column bottoms. In Test 8A, with the nitrogen flow corresponding to an F_s of 0.2, the liquid rate set to 25 gpm/ft^2 (17 l/sec/m^2) and no antifoam injection, the bottoms concentration was reduced to below 1.0 mg/l for feed ranging from 110 to 150 mg/l benzene. From an alternative point of view, there is enough nitrogen to counteract any negative effect that the antifoam may have on stripping.

Therefore, when the low DP indicated that flow through the column was well distributed, the benzene concentration in the DSS simulant was reduced to below 1.5 mg/l, and to below 2.1 mg/l in the WW simulant.

Application to ITP Column Operation

The data obtained from the testing program were used to make direct inferences regarding full scale column operation. The projections for the ITP columns are based on the most conservative data collected from all of the experiments, as presented in the discussions above.

Maximum liquid flow rates will only be attainable when an antifoam is used to eliminate foaming and control the DP to values well below 40 inches (1.02 m) wc. Each batch of salt solution will necessarily be different from all others, resulting in varying degrees of foaming and pressure drop across the packing. Consequently, the maximum processing rate through the column (without antifoam) would also be varied to keep the DP below the 40 inch wc (1.02 m) limit. The four solutions used during the test program foamed to varying extents, but the variability was removed by the use of an antifoam. As shown in Figure 12 for Tests 6, 7 and 8, the degree of variability was reflected in the wide changes in column DP. However, once the addition of AF was initiated, the column DPs fell to a level of about 2 inches (0.05 m) wc and remained steady. The solutions were forced to a common condition, such that DP was reproducible for the different solutions. Therefore, based on the testing and the vendor experience with foaming systems, the antifoam must be used to consistently meet design flow rates for the columns.

The differential pressure across the packing in the DSS column is projected to be on the order of 6-8 inches wc (0.15-0.20 m; with ca. 75 ppm antifoam) at a nitrogen rate of 440 cfm (0.21 m^3/sec). For a benzene concentration in the feed up to 200 mg/l, the bottoms concentration is expected to be approximately 1.5 mg/l at the maximum feed rate of 115 gpm (7.3 l/sec). The upper acceptance value for benzene concentration in the decontaminated solution transferred to the grouting facility is 2.5 mg/l. As the feed rate to the stripper decreases due to production upstream of the columns, stripping efficiency will improve, lowering the bottoms concentration further. The amount of nitrogen needed to satisfy the Saltstone limit may be less than 440 cfm (0.21 m^3/sec), depending on the impact that antifoam will have on stripping efficiency in actual operation.

Similarly, for the WW column, the DP across the column during the initial stages of precipitate washing will be on the order of 22 inches (0.56 m) wc at 220 cfm (F_S of 0.9; 0.10 m^3/sec) and maximum feed rate of 33 gpm (2.1 l/sec). At the nominal feed rate of 14 gpm (0.88 l/sec), the DP is projected to be 8-10 inches (0.20-0.25 m) wc. Benzene in the bottoms should be at a concentration well below the 5 mg/l limit for wash water being transferred to the storage tank. The maximum concentration observed during the testing was 2.1 mg/l, at feed concentrations on the order of 900 mg/l and at a nitrogen rate of approximately 110 cfm (half the design rate; 0.052 m^3/sec). Thus, the nitrogen rate can be significantly reduced from the design rate for the WW column.

The use of an antifoam appeared to have a slight negative effect on the stripping efficiency. The quantity of AF used should be minimized to maintain the stripping efficiency as high as possible. However, the nitrogen rate can be increased to accomodate the slight loss in efficiency (or even an exceptionally high benzene concentration) without causing the DP limit to be exceeded.

CONCLUSIONS

The ITP stripping columns will be capable of reducing benzene concentrations to satisfy downstream feed acceptance and wash water storage limits. The salt solutions fed to the ITP process will foam to varying degrees. An antifoam is required to remove the variability and maintain the column differential pressure below the vendor recommendation of 40 inches (1.02 m) wc so that design feed rates can be achieved. The antifoam slightly reduces the stripping efficiency of the columns, but the resulting product streams still satisfy benzene concentration limits. Alternatively, the nitrogen rate can be increased to compensate for the loss of efficiency or for exceptionally high benzene levels in the feed without jeopardizing the DP limit. With AF injection, the benzene concentration in the DSS column bottoms is projected to be approximately 1.5 mg/l, as compared to the downstream processing limit of 2.5 mg/l. For the WW column, the outlet concentration is expected to be less than 2.1 mg/l, as compared to the storage limit of 5 mg/l.

REFERENCE

Koch Engineering Co., Inc., Koch Tower Design™ Tray and Packing Rating Software, calculations performed using software for the nitrogen-water system; nitrogen at 10 psig and 30°C, water at 30°C.

Table I. Summary of Testing Program

Test	Feed Solution	N2 Rate (Fs)	Feed Solutions: Feed Rate (gpm/ft^2)	Feed Solutions: Temperature (C)	Feed Solutions: TPB Added	Feed Solutions: Benzene Conc'n (mg/l)	Feed Solutions: Antifoam Conc'n (ppm)	Feed Arrangement
1	-	0.2 - 1.2	0	-	-	-	-	-
2	Baseline	0.2 - 1.2	1 - 25	30	Minimal	0	0	Recycle
3	Baseline	0.2 - 1.2	1 - 25	60	Minimal	0	0	Recycle
4	Standard	0.2 - 1.2	1 - 25	30	Excess	0	0	Recycle
5	Standard	0.2 - 1.2	1 - 25	30	Excess	0	100	Recycle
6	DSS	0.5	10, 25	30	Excess	150 - 200	0, 100	Single Pass
7	WW	0.5	10, 25	30	Excess	>500	0, 100	Single Pass
8A	Supplemental	0.2	10, 25	30	Excess	150 - 200	0	Recycle
8B	Supplemental	0.5	1 - 25	30	Excess	150 - 200	0, 100	Recycle
8C	Supplemental	0.9	25	30	Excess	150 - 200	200	Recycle

Table II. Feedstock Concentrations for Pilot Scale Testing Program

Component	Amount Charged (lb)	Resulting Solution Concentration (M)
Baseline Solution, 150 gal		
Sodium Hydroxide	128.5	2.57
Sodium Nitrate	144	1.35
Sodium Sulfate	2.70	0.015
Sodium Nitrite	33.5	0.39
Sodium Carbonate	1.32	0.010
Sodium meta Aluminate	46.0	0.45
Potassium Hydroxide	0.32	Not Determined
Sodium TPB	2.26	Not Detected
DI Water	Balance	-
Standard Solution, 150 gal		
Sodium Hydroxide	128.5	2.57
Sodium Nitrate	144.0	1.35
Sodium Sulfate	2.7	0.015
Sodium Nitrite	33.5	0.39
Sodium Carbonate	1.32	0.010
Sodium meta Aluminate	46.0	0.45
Potassium Hydroxide	0.33	Not Detected
Sodium TPB	3.57	0.003
DI Water	Balance	-
DSS Simulant - Trailer from Third Party Vendor		
Sodium Hydroxide		3.07
Sodium Nitrate		1.44
Sodium Sulfate		0.018
Sodium Nitrite		0.44
Sodium Carbonate		0.019
Sodium meta Aluminate		0.49
Potassium Hydroxide	11.0	Not Detected
Sodium TPB	42.4 (+65 lb at Koch)	Excess
Benzene	22.4	Excess
DI Water	Balance	-
WW Simulant - 2100 gal Remaining of DSS Simulant Diluted With DI Water		
Sodium TPB	237.5	Excess
Benzene	23.2	Excess
DI Water	15,000 (1800 gal)	-
Supplemental Feedstock, 150 gal		
Sodium Hydroxide	128.5	2.57
Sodium Nitrate	144	1.35
Sodium Sulfate	2.70	0.015
Sodium Nitrite	33.5	0.39
Sodium Carbonate	1.32	0.010
Sodium meta Aluminate	46.0	0.45
Potassium Hydroxide (from impurities)	0	Not Detected
Sodium TPB	7.00	Excess
DI Water	Balance	-

Table III. Benzene Analysis of Samples

Test	Sample #/Time	Benzene Concentration (mg/l) Feed	Mid-Column	Bottoms
6	6-1 / 55	59.8	4.7	0.5
	6-2 / 85	103	8.7	0.7
	6-3 / 115	258	16.2	1.4
	6-4 / 145	237	13.3	1.5
	6-5 / 175	170	3.7	0.4
	6-6 / 205	262	5.1	0.4
	6-7 / 235	279	4.1	0.4
	6-8 / 325	211	9.7	0.8
	6-9 / 350	247	15.1	1.4
7	7-1 / 30	558	12.7	0.8
	7-2 / 60	744	21.2	0.8
	7-3 / 90	1050	16.1	0.7
	7-4 / 120	1030	17.4	0.7
	7-5 / 150	276	36.4	1.7
	7-6 / 180	247	12.3	0.9
	7-7 / 210	781	6.3	1.5
	7-8 / 240	855	5.5	0.5
	7-9 / 270	949	3.3	0.2
	7-10 / 300	912	2.9	0.4
	7-11 / 345	966	37.9	1.8
	7-12 / 375	453	26.8	2.1
	7-13 / 405	828	18.0	2.1
8A	8-1 / 65	147	-	0.8
	8-2 / 80	129	-	0.5
8B	8-3 / 170	354	-	8.2
	8-4 / 200	300	-	0.9
	8-5 / 245	356	-	5.6
	8-6 / 272	208	-	31.3
	8-7 / 455	185	-	2.5
	8-8 / 485	83.2	-	3.5
	8-9 / 530	219	-	21.2
	8-10 / 560	338	-	22.1
8C	8-11 / 620	116	-	25.4
	8-12 / 650	256	-	20.4

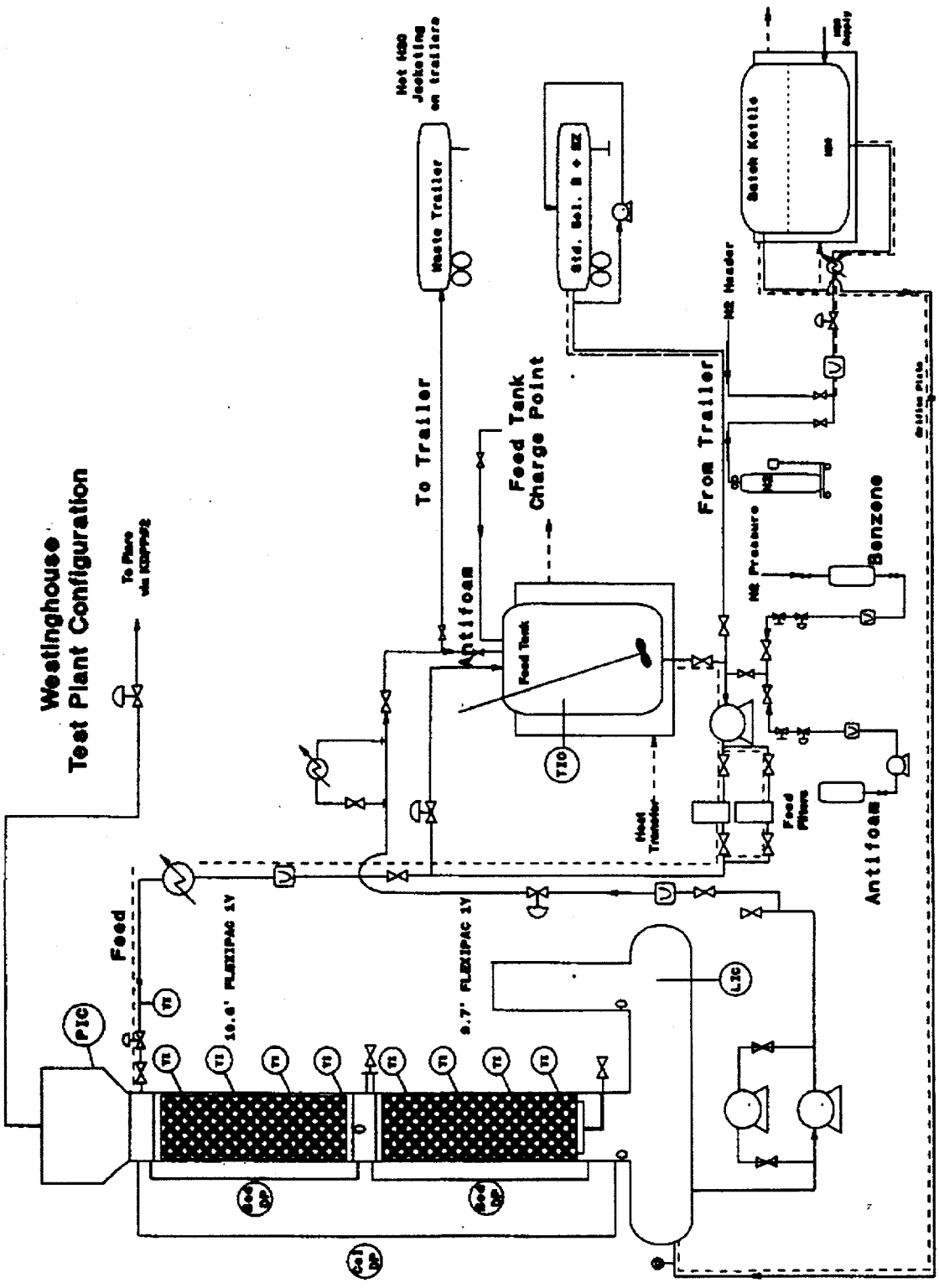

Figure 1. Schematic Diagram of Koch Pilot Scale Test Facility

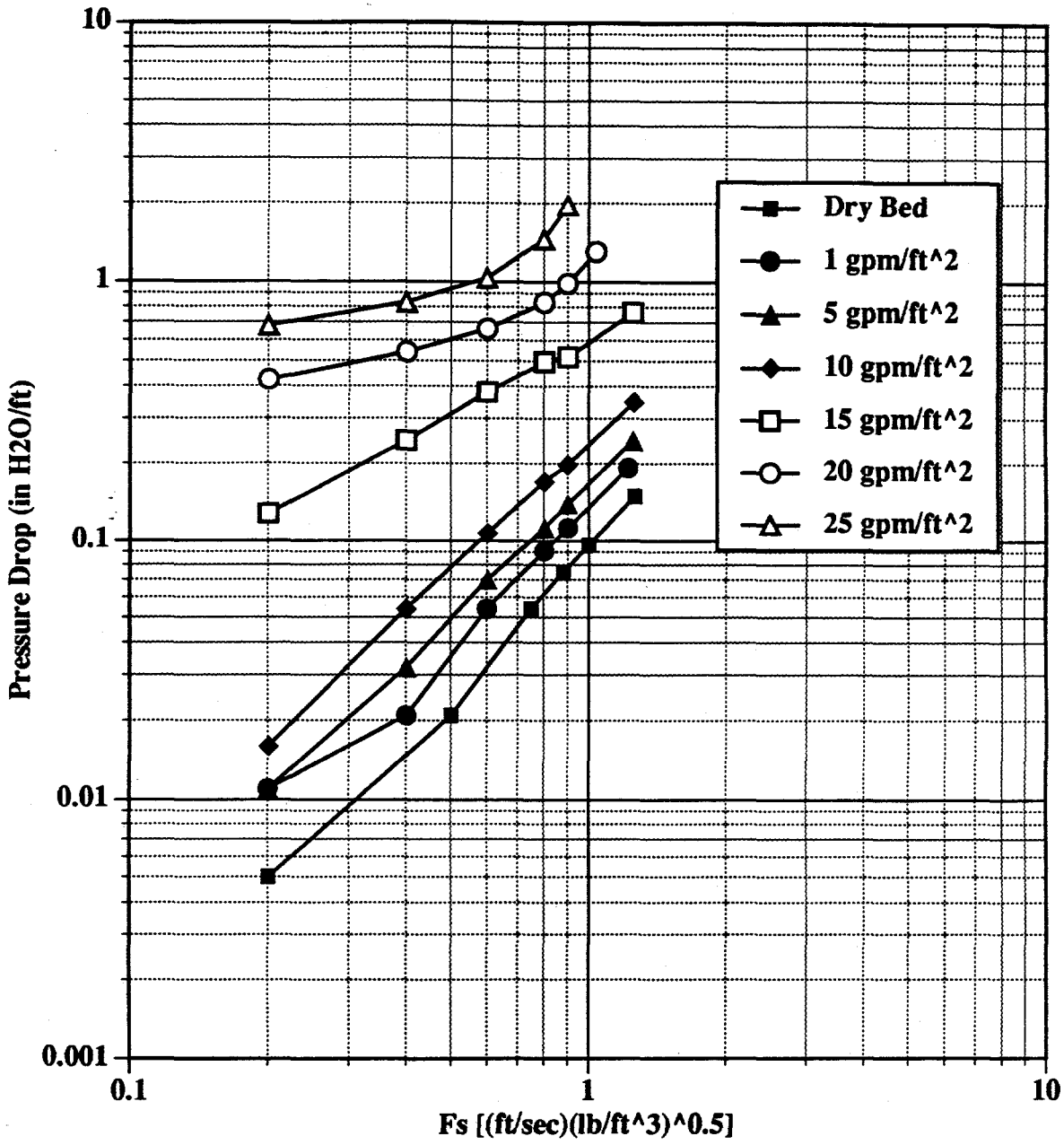

Figure 2. Differential Pressure Data for Baseline Solution

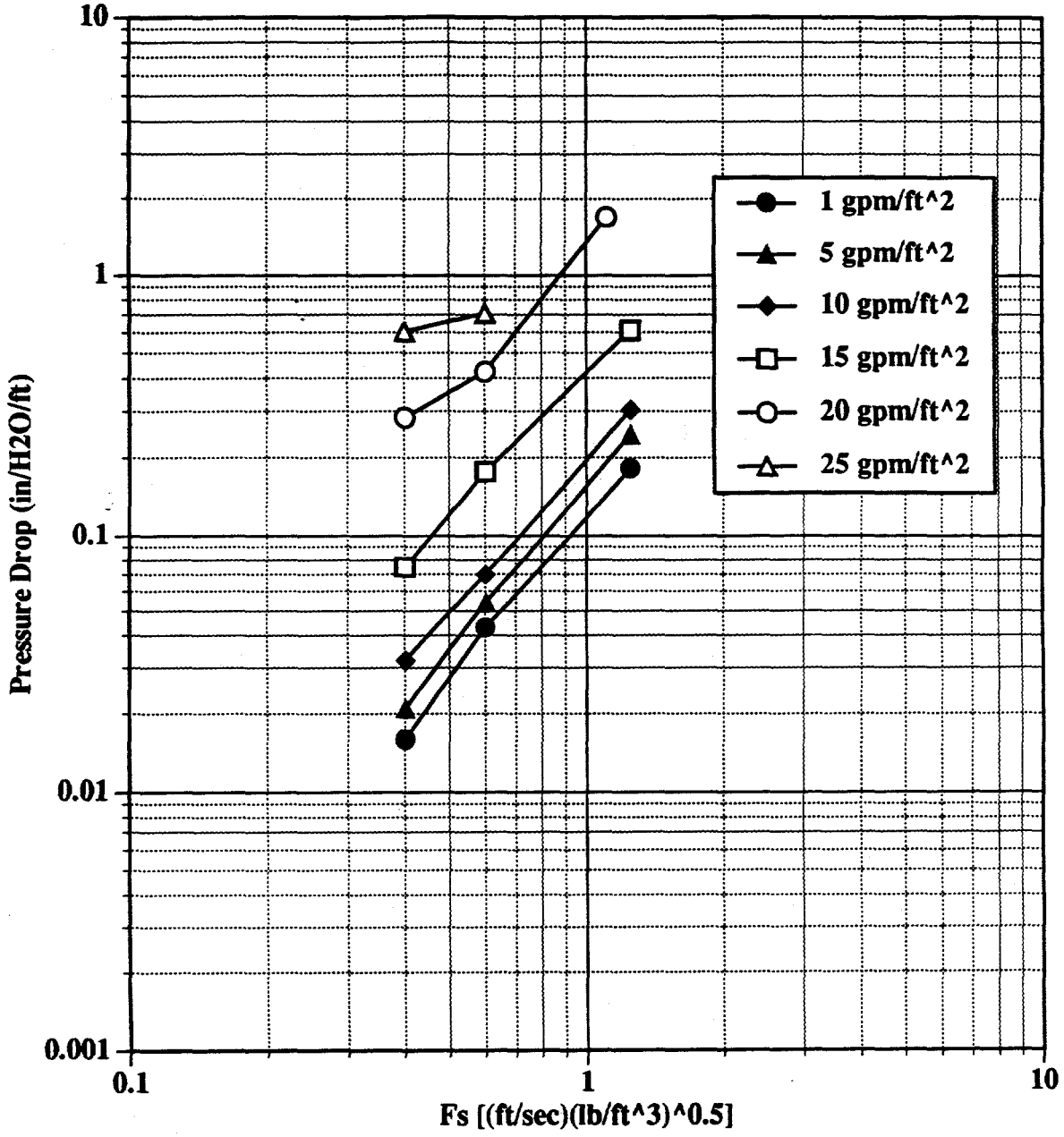

Figure 3. Differential Pressure Data for Baseline Solution at 60°C

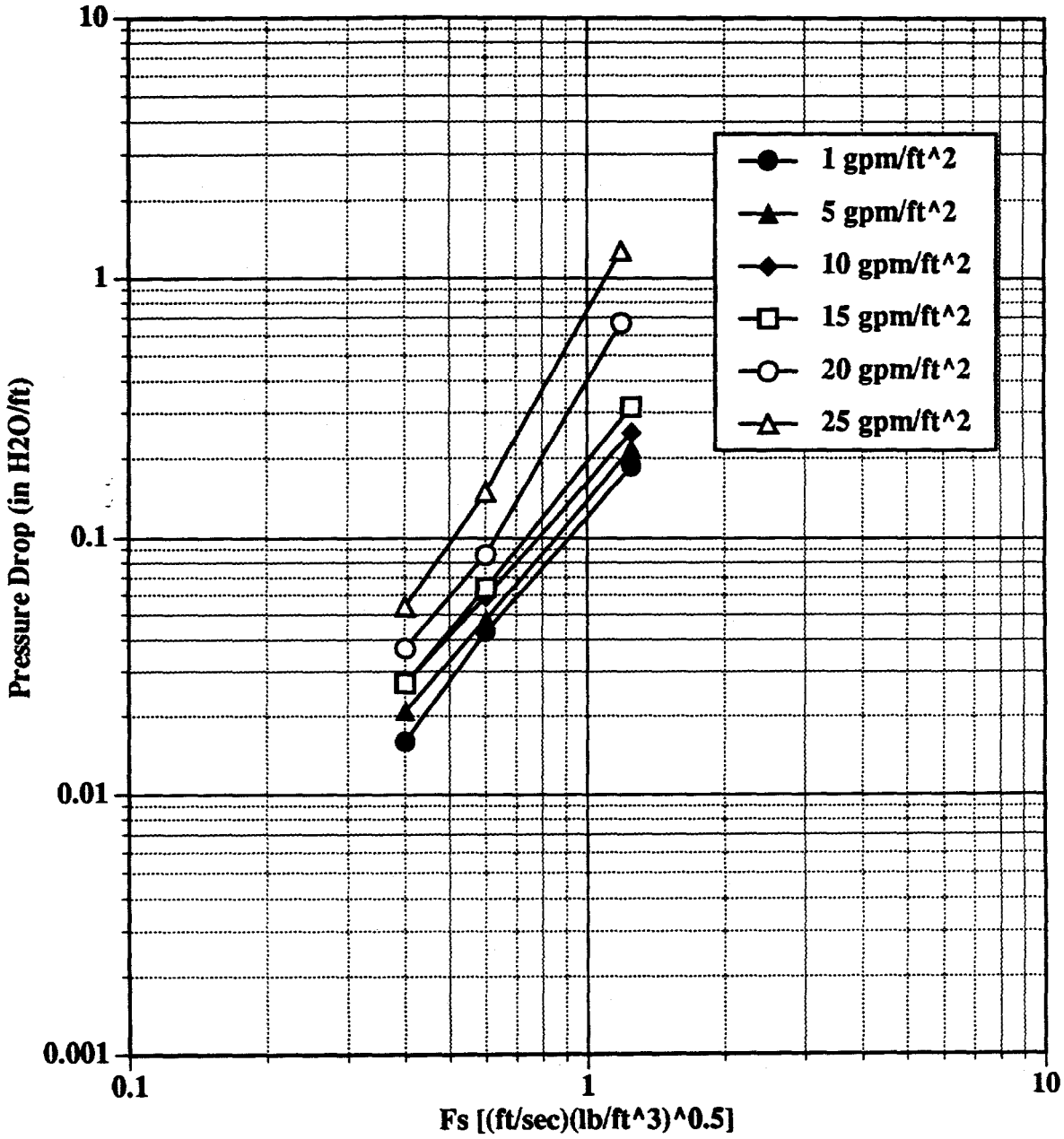

Figure 4. Differential Pressure Of Standard Solution with 100 ppm Antifoam Addition

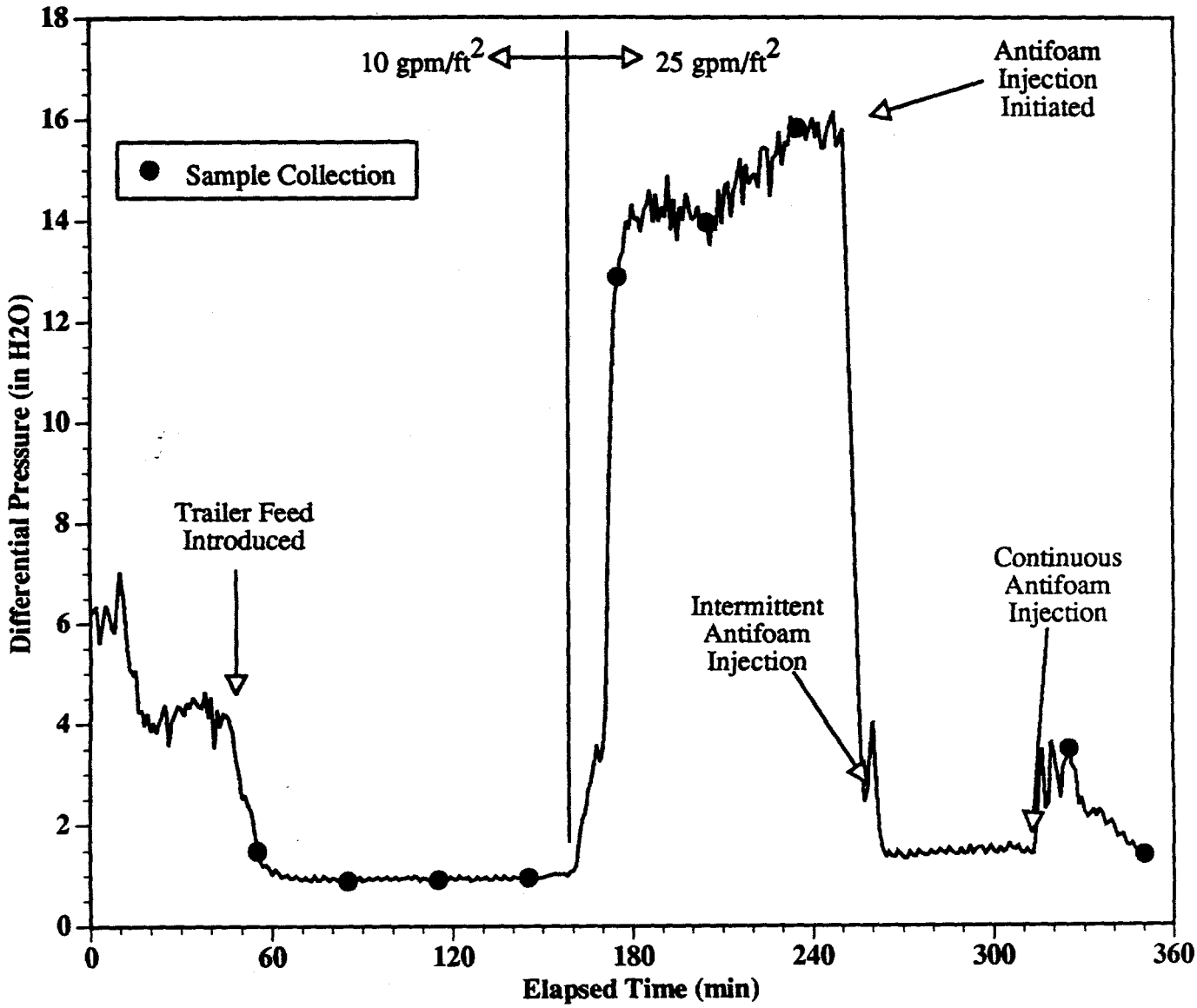

Figure 5. Differential Pressure During DSS Column Simulation

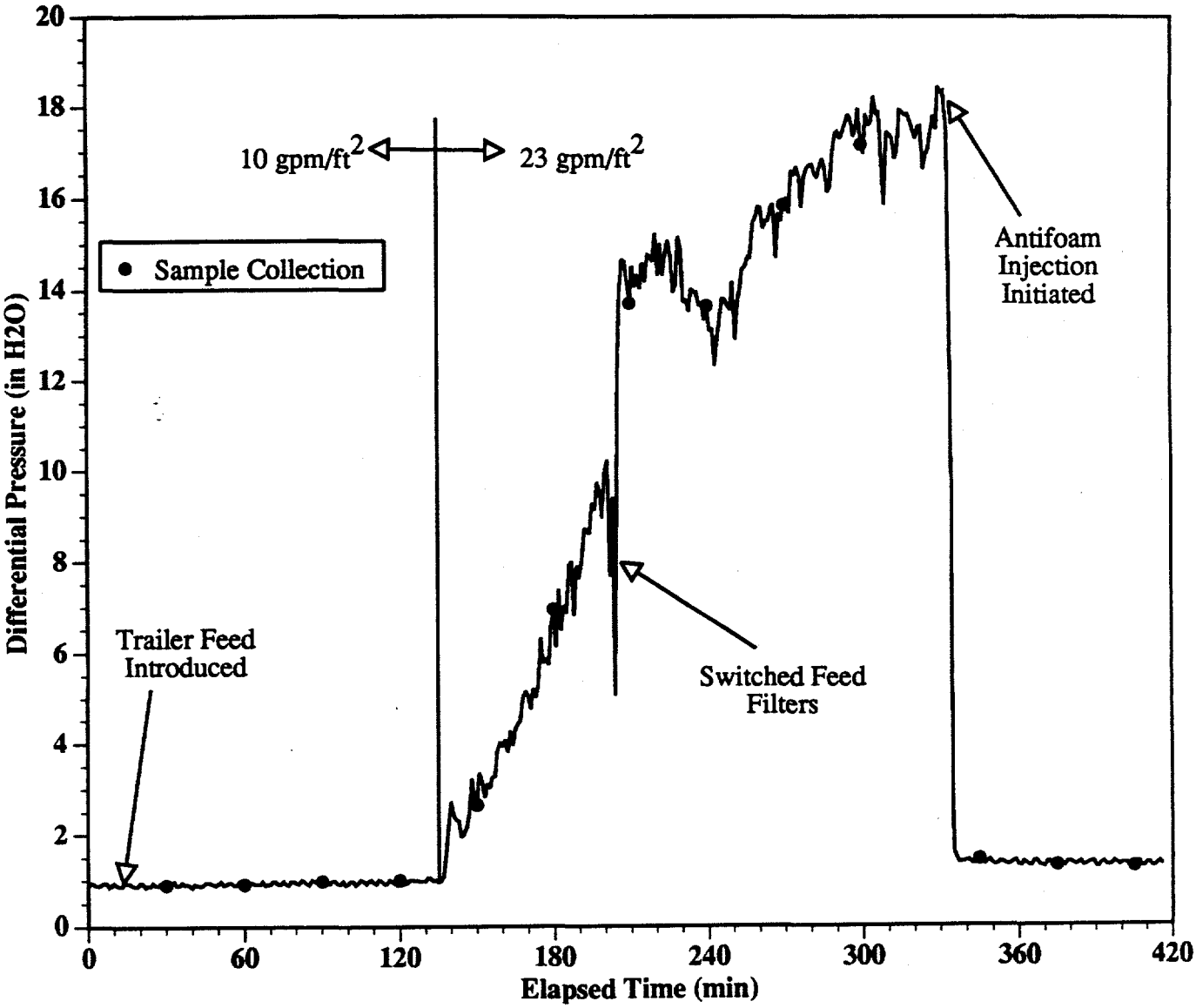

Figure 6. Differential Pressure During WW Column Simulation

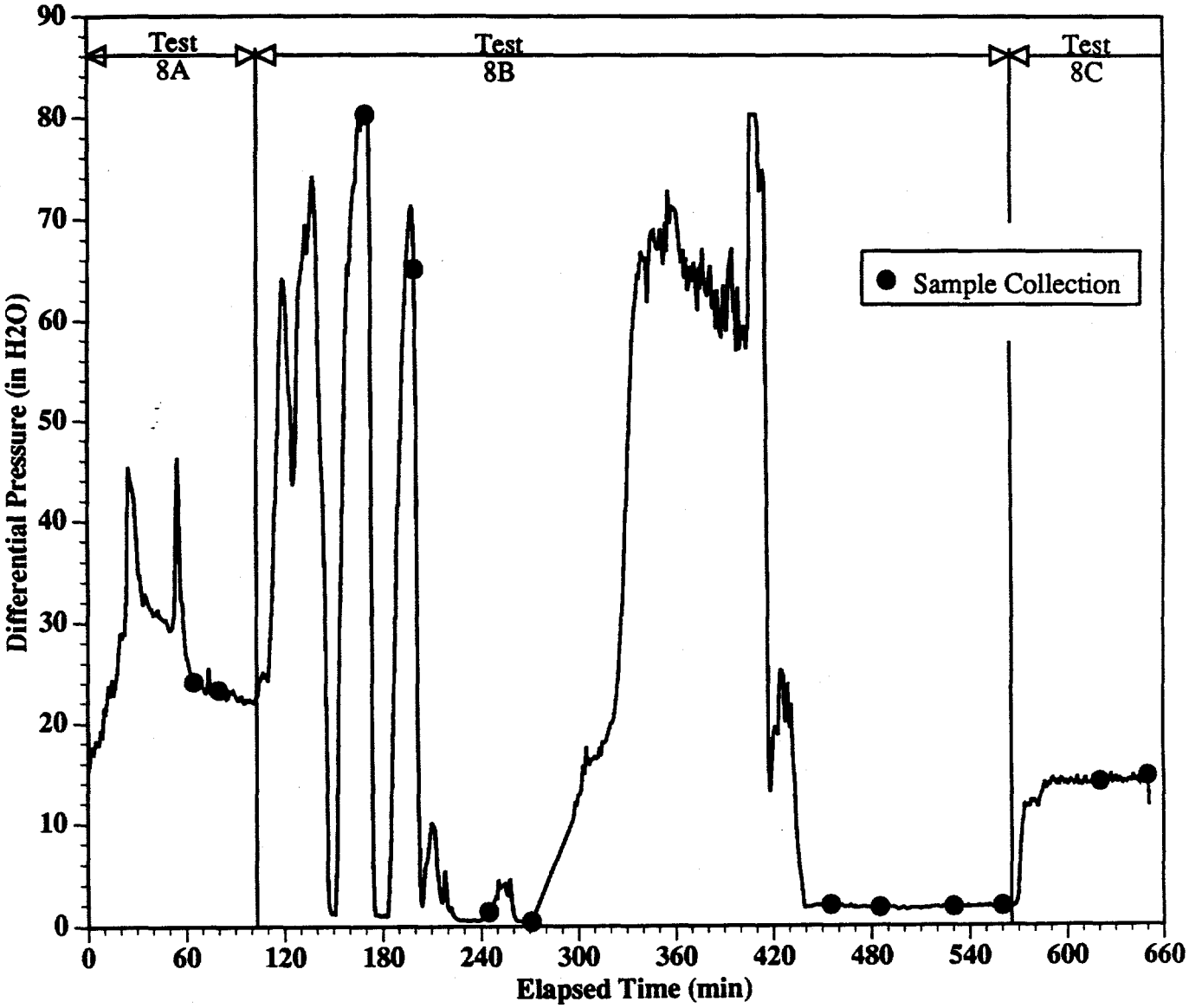

Figure 7. Differential Pressure During Supplemental Testing

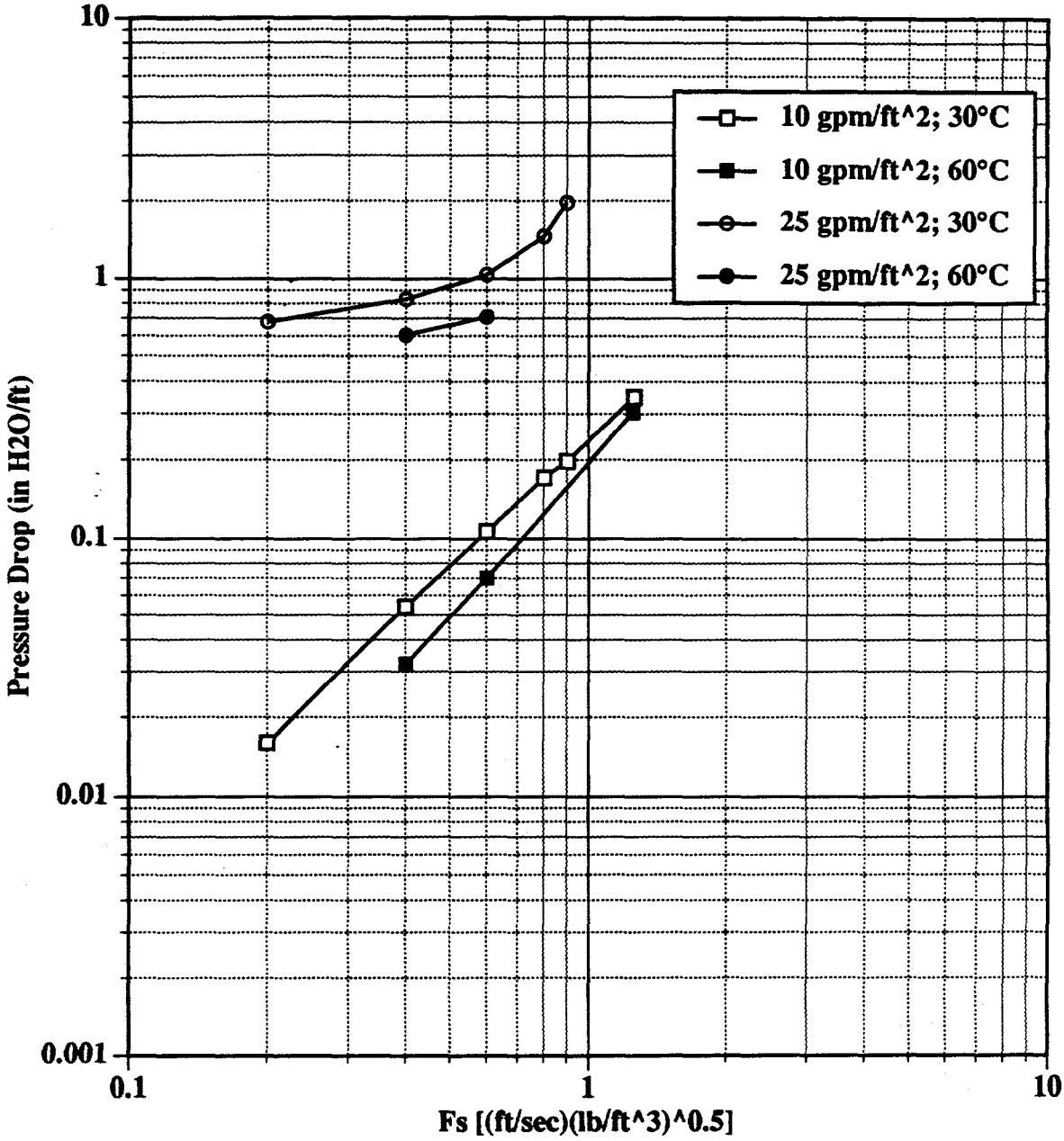

Figure 8. Impact of Temperature on Column Differential Pressure

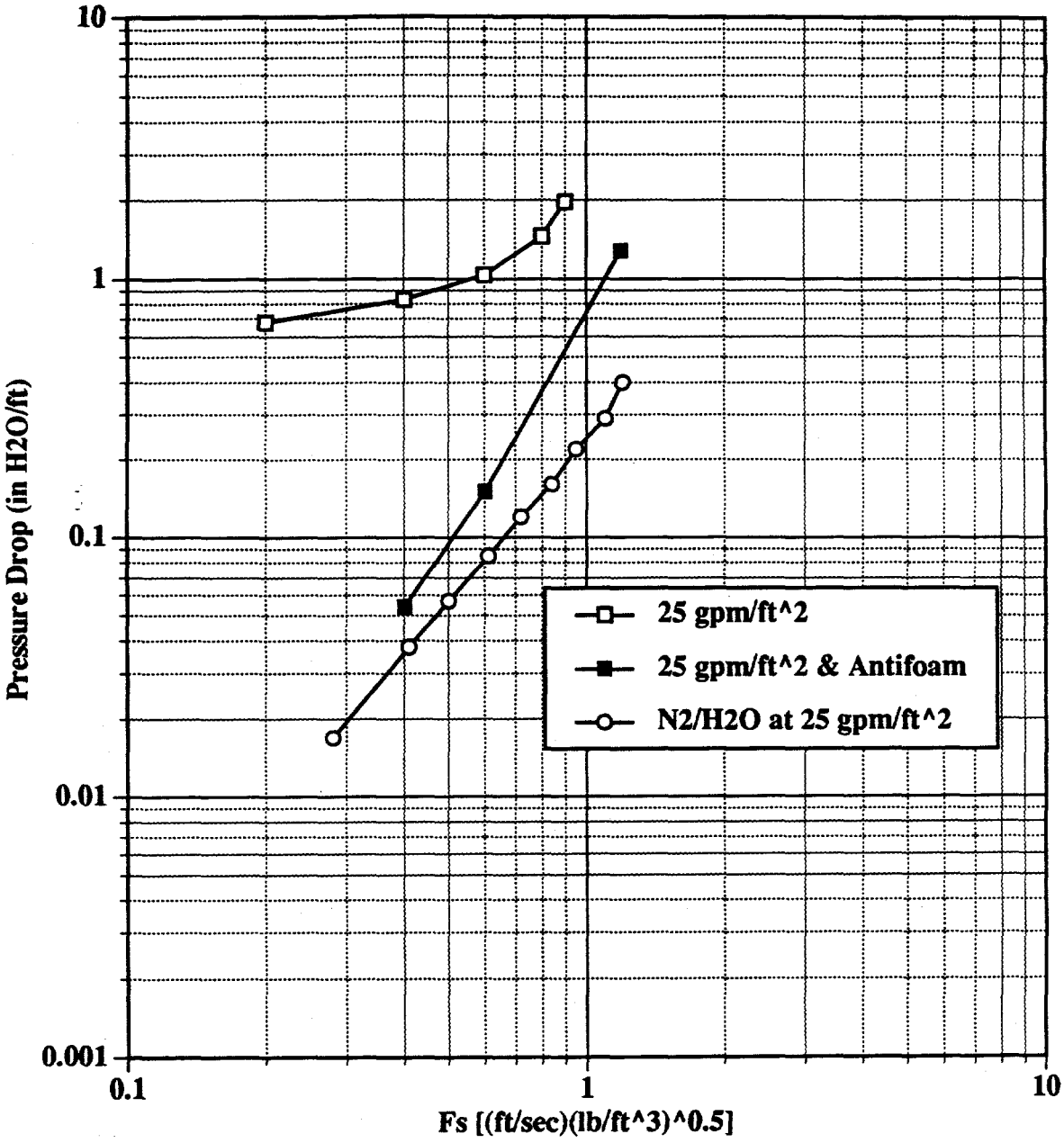

Figure 9. Impact of 100 ppm Antifoam Addition on Column Differential Pressure

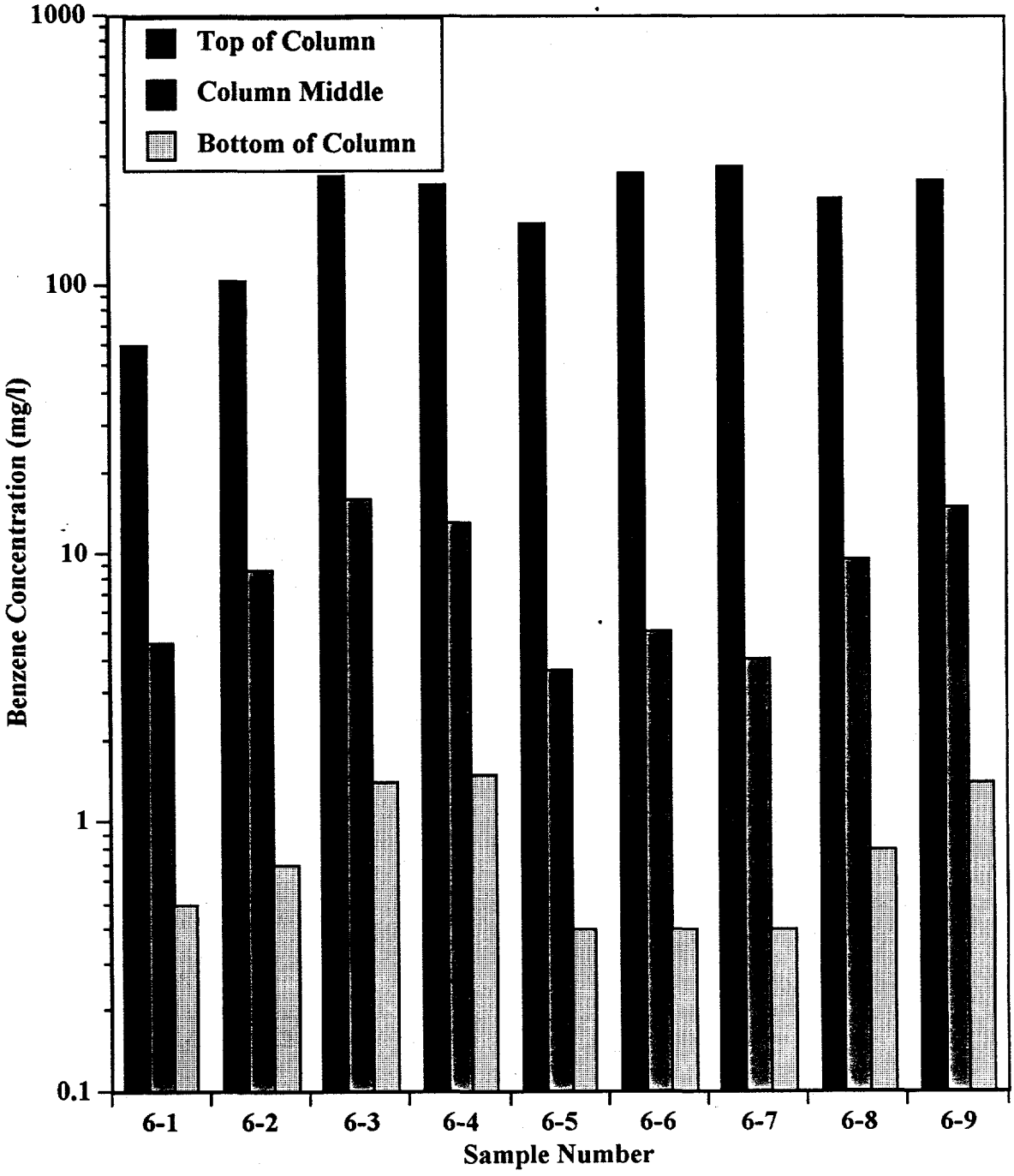

Figure 10. Benzene Removal During DSS Column Simulation

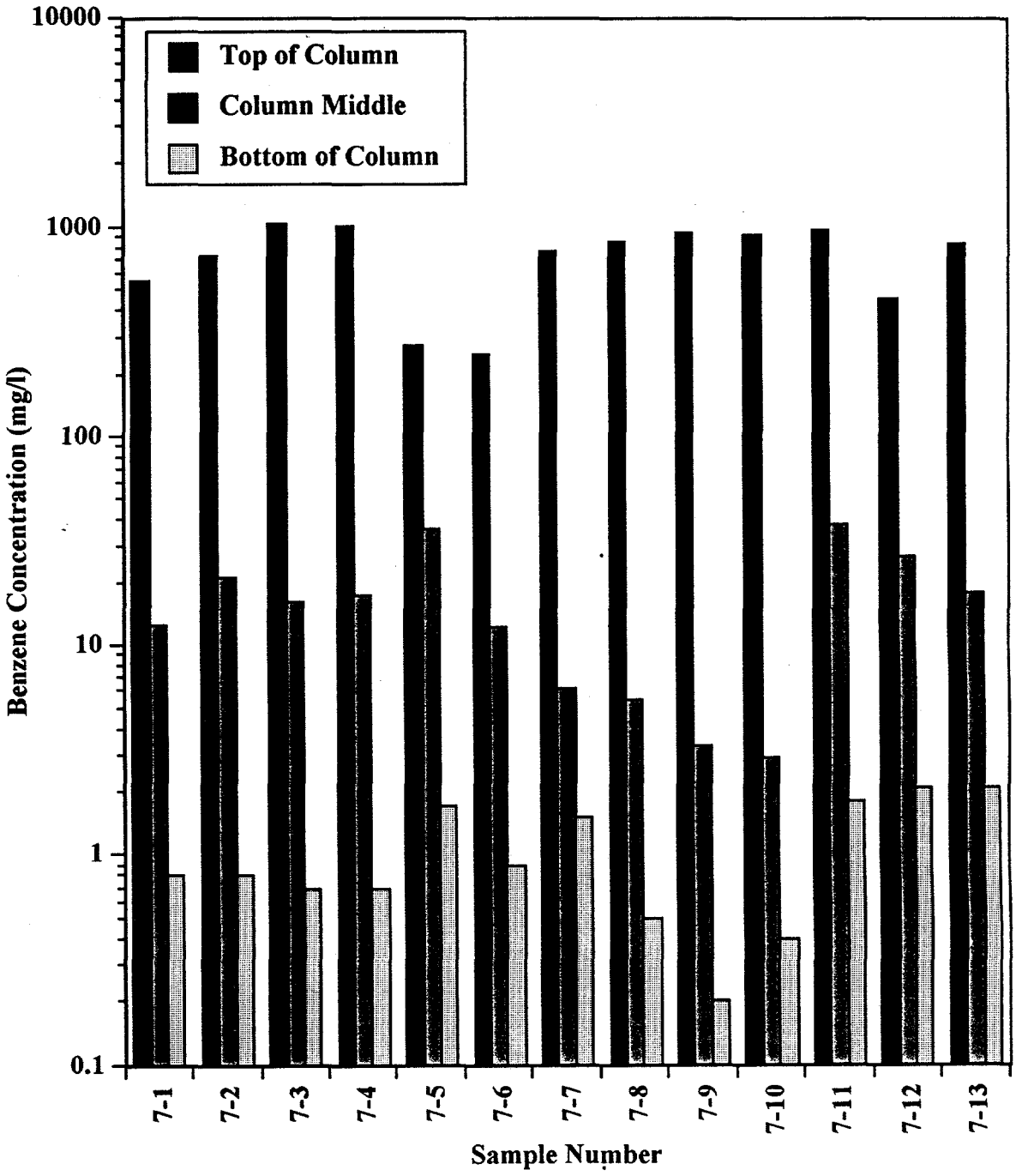

Figure 11. Benzene Removal During WW Column Simulation

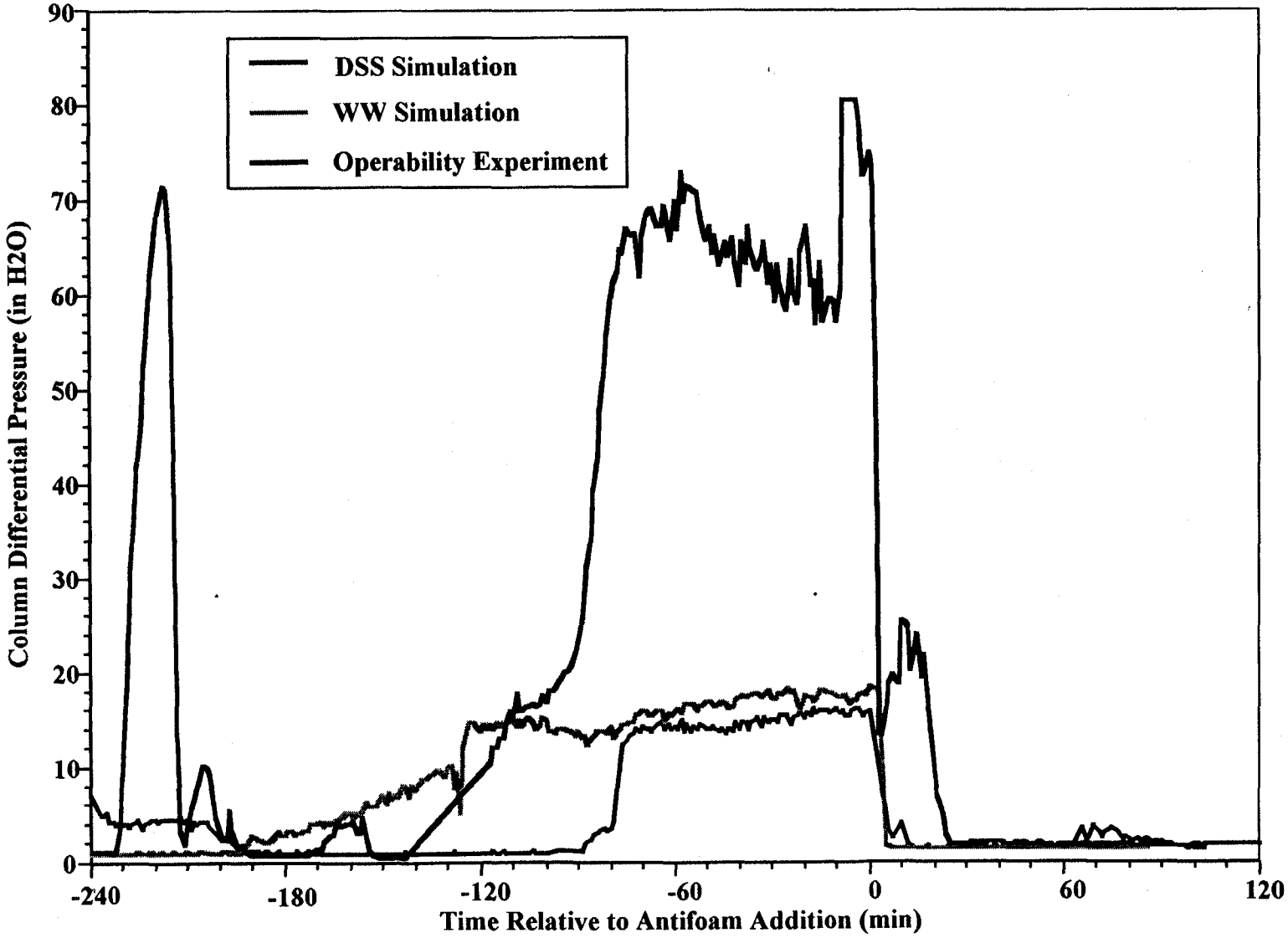

Figure 12. Elimination of Variability in Column Differential Pressure With Antifoam Use