TUNG FLUE GAS DESULFURIZATION PILOT PLANT DEMONSTRATION

PHASE II FINAL REPORT

DRAFT

July 1995

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TUNG FDG TEST FACILITY
PHASE II - PILOT PLANT DEMONSTRATION
FINAL REPORT

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ABSTRACT

The Tung FGD Process is a regenerative process which extracts SO_2 from a scrubbing liquor into an organic medium using mixer-settlers followed by steam-stripping the SO_2 off from the organic medium. For the process to operate satisfactorily, (1) the organic must be stable, (2) phase separation must be relatively fast, (3) crud (i.e. solids in-between two phases) must not form and (4) SO_2 must be able to be stripped off from the organic medium readily. The demonstration confirmed that the first three conditions can be met satisfactorily. Much lower stripping efficiency was attained in the pilot plant demonstration than what was previously attained in a bench-scale demonstration. Engineering analysis showed that the pilot plant stripping column was scaled up from the bench-scale column incorrectly. A new scale-up criterion for stripping a relatively viscous liquid medium is proposed based upon pilot plant data.

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EXECUTIVE SUMMARY

SECTION 1

SECTION 1

EXECUTIVE SUMMARY

The Tung FGD Process utilizes liquid-to-liquid extraction, regeneration of reagents, and yields liquid SO₂ as a salable byproduct. The Tung FGD Process represents a major improvement to state-of-the-art regenerative FGD technology. In a conventional thermal regenerative FGD process, the aqueous absorption spent liquor is steam vaporized directly. In such a system, not only is SO₂ difficult to vaporize but also the solvent water is more easily vaporized than the SO₂. In the Tung FGD Process, the co-vaporization of water is avoided and hence, the energy consumption in regeneration is drastically reduced. Another advantage of the Process is that it does not generate waste products.

Commercial success of the Tung FGD Process relies upon the successful resolution of three potential difficulties. First, as a chemical process that uses an organic as the medium, organic stability is always a major concern. In the Tung FGD Process, the organic medium, under abusive conditions, tends to undergo some physical change resulting in some loss of their SO₂ extraction capacity. This change must be 100% arrested before the organic can be considered suitable for long-term use. Secondly, for any process that involves liquid-to-liquid extraction and especially for any employing mixer-settler as the extraction equipment, phase inversion could be a problem. If phase inversion cannot be brought under control, phase separation could require more time than what is acceptable for a continuous operation. Thirdly, because the organic solvent possesses relatively high viscosity, the equipment selected for process application must have the correct attributes to permit sufficient mass transfer needed for an effective overall system operation.

The result of the current Tung FGD Process demonstration test program, as documented in this report, confirmed that the organic medium, as selected for use in the Process, is quite stable. It also demonstrated that the phase inversion can be readily controlled simply by controlling the phase ratio. Furthermore, the mixer-settler can be suitably used for mass transfer in the extraction process step even though the design features of the particular mixer-settler used can perhaps be upgraded in the future. This possible future upgrading is of only minor importance to the overall development of the Tung FGD Process.

Some problems remain in the design of the sieve tray column for steam stripping. With regard to steam stripping of the loaded organic, the vapor pressure of the loaded organic was measured by Badger Engineers Inc. of Boston MA. several years ago. These measurements showed that the loaded organic could be easily stripped equilibrium wise. The chemical kinetics of the stripping decomposition reaction was also completely characterized with the help from the E.I. DuPont DE Nemours & Company of Newark, DE. This kinetic study showed that the loaded organic will decompose completely within five minutes at 100°C. This means that kinetically, the loaded organic can be completely decomposed during stripping if the stripping column has a five minute residence time. The demonstration stripping columns were designed to have a longer residence time. Furthermore, with a 2-inch diameter Oldershaw column, the loaded organic was successfully stripped at Foster Miller Inc. of Waltham, MA. The test yielded a 92% stripping efficiency. The same column was also used at Hazen Research Corporation of Golden CO. Hazen's test yielded a 97% stripping efficiency. In spite of all of these background experiences, the stripping columns performed relatively poorly in the present pilot plant demonstration.

Detailed analysis of the experimental data suggests that the problem lies with the scale-up i.e. the columns that were used in the pilot plant were not properly scaled up from the 2-inch-diameter Oldershaw column. Stripping of high-viscosity liquids is a relatively new field in chemical engineering technology. It appears that the mechanism of stripping a high-viscosity liquid is drastically different from that of stripping a low-viscosity liquid, with which extensive experience is available. Turbulence is important for effective stripping. But with high-viscosity liquid, the turbulence created during bubble formation appears to be much more effective than the turbulence induced by the high-velocity steam.

Based upon the demonstration data, a quantitative scale-up criterion for stripping high-viscosity liquid has been proposed in this report. It is recommended that a mathematical model be set up based upon this proposed criterion. The model should then be confirmed and improved upon by experimental data. After this steam stripper scale-up problem is resolved, opportunities should be sought to build a second Tung FGD Process pilot plant.

**INTRODUCTION
AND BACKGROUND**

SECTION 2

2.1 SUMMARY OF PREVIOUS WORK

Initial Work

Phase I Work Summary

- (1) Work Done at York
- (2) Initial Work Done at Hazen
- (3) Work Done at Foster Miller
- (4) Research at Raycon
- (5) Final Confirmation Test at Hazen

2.1 PHASE II WORK

SECTION 2

INTRODUCTION AND BACKGROUND

The work described in this report was performed under the Department of Energy Contract Number DE-AC22-86PC81011, entitled Tung FGD Test Facility (Phase II) by Raycon Research and Development Inc. (Raycon) and its subcontractors. The principal subcontractor for the project was Raytheon Engineers and Constructor (RE&C) of Denver, CO.

This section describes the Tung Flue Gas Desulfurization (FGD) Process, summarizes previous work (mostly Phase I work) on which the Process is based, and describes some facets of the Phase II demonstration.

2.1 SUMMARY OF PREVIOUS WORK

The Tung FGD Process consists of three major steps: wet scrubbing of the flue gas; liquid-to-liquid extraction of the bisulfite ion into an organic solvent; and steam stripping of the organic solvent to generate SO₂ gas. The Process also has two proprietary minor steps: removal of Na₂SO₄ formed during absorption; and solvent regeneration which prevents the solvent from losing its extraction capacity in long-term use.

Initial Work

Extensive laboratory experimentation which was conducted prior to the start of this project provides the basis for the Tung FGD Process. Initial research concentrated on identifying suitable solvents to use for the Process. After solvents were selected, systematic study of the Process variables was done to define workable operating conditions.

The Tung FGD Process was awarded a grant from the U.S. Department of Energy's (DOE) Invention and Small Scale Technology Branch in 1984. DOE's support has enabled Raycon, among other things, to attain two equilibrium curves of basic importance to the process, namely, (1) a liquid-to-liquid extraction equilibrium curve (Figure 6.5), and (2) a vapor pressure curve of SO₂ over loaded organics (Figure 6.3). These curves confirmed that both good extraction and good stripping can be easily achieved from an equilibrium viewpoint.

In late 1984, the Process received some support from Stone & Webster Engineering Corp. of Boston, MA. A preliminary cost estimate by Stone & Webster suggested that the Process could provide a low-cost FGD option for installation to a large number of coal-fired power plants.

Phase I Work Summary

In 1985, the project was selected for funding under DOE/Pittsburgh Energy Technology Center's (PETC) Acid Rain Precursor Control Technology Program. The Phase II work of the resulting contract forms the basis for the work reported herein. Prior to the present Phase II work, bench-scale tests were

conducted during Phase I at Otto H York (York) Corp. in Parsippany, NJ; Hazen Research Corp. in Golden, CO; Foster Miller, Inc. in Waltham, MA; and Raycon's own chemical research laboratory in Watertown, MA. The results from these tests will be briefly summarized here.

(1) Work Done at York: Under Phase I of this project, York first conducted bench-scale, liquid-liquid, counter-current extraction tests using five agitated glass column configurations. None of these tests gave acceptable column performance.

Test results suggested that the extraction efficiency is highly dependent upon mechanical energy input. This is shown in Figure 2.1-1:

Figure 2.1-1
Extraction Column Efficiency vs Mechanical Energy Input

Inspection of this curve showed that the targeted 90% extraction efficiency might be reached if we were permitted to operate the column at higher rotational speeds. Unfortunately, this was not possible because the column would flood under higher rpm conditions.

The reason that the extraction column did not work can be easily understood. Extraction is a diffusional process, with the extraction column performance directly related to the rate of diffusion. The diffusivity of the

diffusing species in a liquid medium is, in turn, related to the viscosity of the medium according to the Stokes - Einstein relation:

$$D_L = \frac{T}{\mu F} \dots\dots\dots (2.1)$$

where D_L is diffusivity, T is the absolute temperature of the liquid, μ is the viscosity of the liquid, and F is a constant. As the viscosity increases, the rate of diffusion may be expected to decrease rapidly, thus leading to a sharp deterioration in the extraction efficiency. Based upon the above test results, it was concluded that a mixer-settler should be used instead of a counter-current extraction column.

(2) Initial Work Done at Hazen: Hazen was hired to evaluate whether mixer-settlers were suitable for the Tung FGD Process. Hazen test results showed that mixer - settlers were indeed viable.

With mechanical stirring, the extraction at Hazen achieved very high (approaching theoretical) stage efficiency and separation rates of the system were within the range used in the mixer - settler equipment. Thus, Hazen's results had identified a workable equipment for the necessary extraction process.

(3) Work Done at Foster Miller: Since a counter-current extraction system with rotating shaft could not be used for extraction because mechanical energy input was not sufficient, it was not clear whether a conventional sieve tray column would be suitable for stripping. Conventional stripping columns depend on high-velocity steam to provide turbulence but it was unclear as to whether the mechanical energy input by steam would be sufficient to induce good mass transfer and stripping performance. Foster Miller was hired to evaluate the performance of the 2-inch Oldershaw column. The test results were excellent. Stripping efficiency was shown to exceed 92% at steam rates of 3-4 lb steam / lb SO_2 released.

With these test results it was concluded that a sieve tray could be used in the pilot plant to strip the loaded organic effectively. This is especially true since

it is known that an industrial column can be designed with a higher F-value or higher steam velocity than a lab column. Higher steam velocity will cause more turbulence and thus should promote even better stripping.

(4) Research at Raycon: Hazen's work also revealed that the organic solvent, when abused, can lose extraction capacity upon cyclic use. This induced Raycon to undertake a comprehensive laboratory program aimed at understanding and controlling changes in the organic. Raycon first found that changes in extraction capacity, induced by abuse, are physical and reversible rather than chemical and irreversible. It was further found that by modifying some process designs, and by requiring certain process variables to operate within predefined domains, the change in organic properties can be controlled or eliminated. Raycon has also developed some proprietary solvent refining or treatment steps to further assure that these physical changes in the organic do not occur during cyclic process application of the organic solvent.

(5) Final Confirmation Test at Hazen: Based upon the above test results, a new combined test loop was assembled at Hazen consisting of a six-stage mixer-settler system and a 2-inch Oldershaw stripping column. This new combined test demonstrated that: (1) a countercurrent mixer-settler system could be used effectively for extraction; (2) a 20-stage stripping column could be used for steam stripping (stripping efficiency was extremely high i.e. 97%); and (3) when operating within the operating domains of several operating variables, the organic should be completely stable.

2.2 PHASE II WORK

As stated in the Project Work Plan, the objectives of the DOE Proof-of-Concept test program are:

- Demonstrate:
 - Operation and efficiency of each process step
 - Suitability of equipment for the process operation
 - Equipment and materials integrity in long term operation
 - Chemical reagent integrity in long term operation
- Characterize:
 - Energy / Material balance
 - Waste streams and disposal requirements

These objectives can be recasted as follows:

- To demonstrate that the organic is stable in the cyclic operation.
- To demonstrate that the equipment can operate to meet the process needs, especially the mixer-settler for extraction and the sieve tray column for stripping.
- To gather adequate data so that operation efficiency of each process step and relevant material balances can be assessed.

For Phase II, RE&C of Denver CO was selected as a major subcontractor. The engineering work started on November 3, 1991 when the Raycon/ Raytheon subcontract was signed. Engineering design, equipment procurement, and demonstration plant construction were executed by RE&C.

During the design phase, Raycon and consultant contributed on the stripper design. From Raycon's perspective, stripper design is of great importance for two reasons. First, slow diffusivity due to high solvent viscosity must be overcome. Secondly, from Raycon's laboratory experience, good stripping is very important to assure solvent stability.

Because high F-value or high steam velocity was conventionally considered as the key to achieve high turbulence and therefore, high stripping efficiency, the diameter of the column selected was small (10-inch diameter). This small diameter was beyond the commercial experience of many tray designers. Only one designer, Glitsch Incorporated of Uxbridge, Ontario, Canada, could design the tray with confidence using their unique orbital flow tray design technology.

Beyond special emphasis on stripper tray design, Raycon had also done a number of other things to assure good stripping performance. Vapor pressure was determined previously to insure that stripping would be a relatively easy task from an equilibrium viewpoint. During Phase II, chemical kinetics of the loaded organic decomposition was determined using equipment rented from the Engineering Test Center of E.I. DuPont DeNemous Co., Inc. in Newark, DE. This kinetic study showed that decomposition of the loaded organic should be completed within five minutes at 100°C.

After the equipment was installed, with additional financial support from Niagara Mohawk Power Corp. (NMPC), Raycon had commissioned a system review and critique by Messieurs Edward Meilun and Stanley Wilderoter. Before his retirement, Mr Meilun was the engineering manager of the Petro-Chemical Division of Stone & Webster Engineering Corp. In that capacity, he had managed the construction of more than thirty ethylene plants all over the world. Mr. Wilderoter was a system engineer at Stone & Webster Engineering Corp. also and he had performed a number of system reviews of pilot plants and industrial plants after their design and construction but before their commencement of the plant operation.

The objectives of the review were: (1) to provide a critique of the Tung FGD Process pilot plant design based upon Mr. Meilun and Mr. Wilderoter's past experience, and (2) to point out or draw attention to the system components which might have potential operational problems. Overall, the report provided useful critiques on potential system operating problems, and it recommended some changes in actual plant design.

Knowing that a number of demonstrations similar to the one planned by Raycon were crippled because of mechanical failures, near the end of the initial plant operation but before the plant shut down for ductwork repair, Raycon / Raytheon jointly conducted a mechanical test to assess the pilot plant mechanical readiness. On-stream time was used as the evaluation criterion. The test identified a number of mechanical operational problems and a mechanical upgrading plan was put in place. This permitted the mechanical upgrading effort to proceed concurrent to the ductwork repair. This upgrading effort was funded by Niagara Mohawk outside of the base DOE contract.

The mechanical upgrading was very successful. When the pilot plant was restarted after the duct repair, the on-stream time achieved by the pilot plant was estimated to be between 85 and 95% when operating at the 0.5 - 1 MW level. The on-stream time decreased if the pilot plant was pushed to run at higher than 1MW because among other problems some magnetic pumps decoupled more frequently at capacities higher than 1MW. So even though the Pilot plant was designed to operate at 2MW, it never did reach that capacity.

Laboratory experiences suggested that to operate the demonstration properly, the residual loading after a number of process steps must meet certain specifications. For example, the residual organic sulfur loading after stripping should not exceed 10%, and preferably 5%. Similarly, the same residual loadings after some minor proprietary process steps should not exceed certain concentration limits. Since these concentration specifications can more readily be met with high on-stream time, it was planned to conduct the demonstration initially at a low pilot plant capacity factor, using one-stage extraction and a 1% bleed rate. After obtaining positive results under those limited operating conditions, it was planned to repeat the tests under higher plant capacity factor, with five extraction stages instead of one and with a lower organic bleed rate. Unfortunately, this was never achieved as a result of steam stripper problems, and insufficient funds.

In this report, the data gathered are presented in Section 7, and discussion of these data are presented in Section 8. Other facets of the project are addressed in other chapters.

PROCESS

DESCRIPTION/CHEMISTRY

SECTION 3

SECTION 3

PROCESS DESCRIPTION/CHEMISTRY

The Tung FGD Process utilizes liquid-to-liquid extraction, regeneration of reagents and yields, in a commercial process liquid SO₂ as a saleable by product. The Tung FGD Process represents a major improvement to the state-of-the-art regenerative FGD technology. Current available regenerative processes require: (1) large amounts of energy for regeneration; and (2) extensive waste treatment. The Tung FGD Process offers major improvements in both areas.

A simplified process schematic developed for the Tung FGD Process is shown in Figure 3.1. The process consists of three primary steps:

- **Scrubbing** SO₂ in the flue gas is scrubbed with a sodium sulfite solution yielding a sodium bisulfite solution:

$$\text{SO}_2 + \text{Na}_2\text{SO}_3 (\text{aq}) + \text{H}_2\text{O} \rightarrow 2\text{NaHSO}_3 (\text{aq})$$
- **Extraction** SO₂ in the scrubbing bisulfite solution is transferred to an organic solvent. The regenerated sulfite solution can be reused for scrubbing:

$$2\text{NaHSO}_3 (\text{aq}) + \text{Org} (\text{org}) \rightarrow \text{Org} \cdot \text{H}_2\text{SO}_3 (\text{org}) + \text{Na}_2\text{SO}_3 (\text{aq})$$
- **Steam Stripping** The loaded organic is steam stripped to separate SO₂ gas from the organic. The regenerated organic can be reused for extraction.

$$\text{Org} \cdot \text{H}_2\text{SO}_3 (\text{org}) \rightarrow \text{Org} (\text{org}) + \text{SO}_2 + \text{H}_2\text{O} (\text{aq})$$

Figure 3.1
Tung Flue Gas Desulfurization Schematic

The process also has two proprietary minor steps which will not be discussed here in detail.

In a conventional thermal regenerative FGD process, the aqueous absorption spent liquor (an alkaline bisulfite) is steam vaporized directly. In such a system, not only is SO₂ difficult to vaporize, but the solvent water is also more readily vaporized than the SO₂. Hence, regeneration of SO₂ is always accompanied by vaporization of solvent water in an amount far in excess of the amount of SO₂ regenerated. This makes the SO₂ regeneration a relatively inefficient process, since water vaporization necessitates a large energy requirement. In Tung's FGD Process, vaporization of water is avoided and the energy consumption is therefore drastically reduced.

Vapor pressure data attained by Badger Engineers Inc. shows that vapor pressure of loaded organic is relatively high. It is therefore easy to strip, providing that the stripper design has specific features that promote the mass transfer in a relative viscous liquid. Stripping efficiency averaging 97% was attained with an Oldershaw column in a bench-scale test at Hazen Research

demonstrating that high-efficiency, low-energy stripping can be achieved. More on stripping will be discussed further in Sections 6.4 and 7.3.

In a commercial plant, chemical interactions between ashes and other impurities normally present in the flue gas with the organics were not anticipated. This projection needs to be confirmed by the data generated in the pilot plant.

4.1 PILOT PLANT DESIGN CRITERIA

4.2 PILOT PLANT DESCRIPTION

- SO₂ Absorption**
- Sulfite Extraction**
- Sulfate Crystallization**
- Steam Stripping**

4.3 ANALYTICAL EQUIPMENT, ANALYTICAL MEASUREMENTS AND SAMPLING

- Wet Analysis**
- Ion Chromatograph**
- PH Measurements**
- Viscosity Measurement**
- Entrainment Measurement**
- Sampling**

4.4 DATA ACQUISITION AND REDUCTION

- Data Acquisition and Reduction Design**
- Data Acquisition Prior to Duct Work Repair**
- Data Acquisition After Duct Work Repair**
- Data Reduction**

SECTION 4 TUNG FGD PILOT PLANT

This section describes the test facility site, equipment for each processing step, and some of the major design conditions.

The Tung FGD Process Demonstration Facility was designed to remove 90 percent of the SO₂ contained in a demonstration flue gas slip-stream, which amounts to about 6,400 ACFM (equivalent to 2MW) of treated flue gas at 143°C (295°F). The slip-stream was diverted from the flue gas leaving the Dunkirk Generating Station Unit No. 4's I.D. fans by a pilot plant I.D. fan located downstream of the demonstration facility flue gas scrubber. A process flow diagram for the facility is shown in Appendix A. The following material describes the design criteria and the equipment components incorporated into the system design.

4.1 PILOT PLANT DESIGN CRITERIA

The Process Design Criteria for the pilot facility are shown in Table 4-1. The table is divided into the criteria for each primary unit operation. These criteria served as the design basis for each equipment component. A material balance was assembled for all process streams. This information served as the basis for all equipment sizing and selection of construction materials. The detailed equipment descriptions are provided in the equipment list presented in Appendix B.

TABLE 4-1

TUNG PROCESS DESIGN CRITERIAFlue Gas Handling Area Criteria

Slipstream Gas Flow Rate	9,600 ACFM
Venturi Inlet Gas Flow Rate	6,400 ACFM
Venturi Pressure Drop	13" W.G.
Absorber Pressure Drop	6" W.G.
Venturi Inlet Gas Temperature	295°F
Absorber Outlet Gas Temperature	125°F
Inlet Ash Particulate Loading	0.015 Gr/DSCF
Plant Coal Sulfur Content	2.15% By Wt.
Coal Heating Value	13,040 Btu/lb
Inlet SO ₂ Concentration	1,450 ppm

SO₂ Removal Area Criteria

SO ₂ Removal Efficiency	90%+
Venturi L/G	15 Gallons/1,000 ACF
Absorber L/G	20 Gallon/1,000 ACF
Bisulfite Solution Concentration (outlet)	2 Molar
Oxidation Percentage (Original estimate as percentage of total sulfite)	10%

Reagent Storage Area Criteria

Soda Ash Storage	30 Days
Soda Ash Makeup Rate (original estimate)	10 lbs/hr
Solvent Storage (system charge = 6,000 lbs)	12,000 lbs
Solvent Bleed Rate (to waste disposal)	4 lbs/hr
Lime (Neutralization)	30 Days
Lime Feed Rate	1 lb/hr

Solids Handling Area Criteria

Sodium Sulfate Waste Product Rate (original estimate)	6 lbs/hr
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TABLE 4-1 (CONT'D)
TUNG PROCESS DESIGN CRITERIA

Extraction Area Criteria

Solvent Sulfite Loading (maximum)	90%
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Steam Stripping Area Criteria

Steam Feed Rate (Original estimate)	90 lbs/hr
Steam Temperature to Stripper	120°C
Regenerator Steam Pressure	35 psi (Saturated)
Solvent Preheat Temperature	98-100°C
Residual Sulfite Loading (maximum)	10%

4.2 PILOT PLANT DESCRIPTION

SO₂ Absorption

A 3 MW equivalent slipstream of the flue gas is taken from the Unit No. 4 ductwork downstream from the existing plant I.D. fan. This gas flow (approximately 9,600 ACFM) is split, with two-thirds of the gas proceeding to the inlet of the venturi scrubber/saturator. The other one-third of the flue gas volume is bypassed around the absorber to serve as reheat for the scrubbed gases prior to entering the pilot plant I.D. fan.

The venturi prescrubber contacts the flue gas with recycled water. Fly ash, hydrogen chloride/fluoride and some SO₂ are removed in the quench operation, which also results in a reduction in the flue gas temperature from about 145°C (295°F) to the adiabatic saturation temperature, about 52°C (125°F). A purge stream from the venturi liquor recycle loop is pumped to the quench blowdown/neutralization tank where lime is added for pH adjustment before the purge stream is pumped to the Dunkirk Station wastewater treatment facility. Makeup water is added to the integral venturi recycle sump to replace the water lost both by evaporation in the quencher stage and through the wastewater purge stream.

Saturated flue gas from the venturi scrubber first flows through a mist eliminator to remove residual acidic droplets, then flows into and up through the absorber stack (a packed tower) where the flue gas is contacted by a countercurrent scrubber liquor stream. In the primary absorber tower, flue gas is contacted by the recycled scrubber liquor to maximize sodium bisulfite concentrations. A secondary absorber was added to the design to complete the conversion of available sulfite in the scrubber liquor to bisulfite. Two 100%-capacity recycle pumps are installed to allow continuous operation of the primary absorber and the venturi/scrubber. A portion of this primary absorber recycle liquor can be diverted either to the secondary absorber sump or through a bleed filter, then to the sodium bisulfite liquor tank. From there the absorber liquid is split between the bisulfite liquor cooler and the sulfate crystallizer. This liquor stream leaves the absorber at approximately 52°C (125°F) and is cooled to 24°C (75°F) by the cooler.

The scrubbed flue gas passes through a mist eliminator before exiting the primary absorber. Scrubbed flue gas mixes with the bypassed flue gas for reheat, and then passes through the I.D. fan. The concentrated (regenerated) SO₂ gas from the off-gas condenser is combined with the flue gas flow which returns to the Niagara Mohawk ductwork at a temperature of at least 68°C (155°F). The 68°C (155°F) flue gas is returned to the Niagara Mohawk Unit No. 4 flue gas duct system by the demonstration facility I.D. fan. The bypassed flue gas (at least 1/3 of the total flow) provides sufficient reheat to avoid condensation and corrosion problems in the flue gas duct and stack.

Sulfite Extraction

The bisulfite-rich spent scrubber liquor is pumped from the sodium bisulfite liquor tank to the mixer-settler extraction system where it is contacted with organic solvent. Up to five stages of liquid-liquid extraction were available for plant operation.

Following the solvent extraction of sulfite, the regenerated sulfite solution exiting the first mixer-settler is sent to a sulfite/solvent separator tank. Here, any residual solvent is separated from the aqueous solution. Any residual solvent is sent back to the solvent stream exiting the mixer-settler. The aqueous solution can be centrifuged to remove any contaminated organic, if present, and then returned to the sulfite storage tank. This regenerated sulfite solution then serves as the reagent feed supply for the scrubber. Sodium losses are made up in this tank by direct feed of either soda ash or sodium sulfite solution.

Regenerated and fresh organic solvent from the lean solvent storage tank is pumped to a cooler where the organic solvent is cooled to 24°C (75°F). The cooled lean solvent then goes to the mixer-settler. Bisulfite from the sodium bisulfite aqueous stream is extracted into the solvent to produce a loaded organic stream with the subsequent regeneration of a sodium sulfite-rich aqueous stream. The regenerated aqueous scrubber solution exits the mixer-settler into the separator tank as previously noted.

The loaded organic stream exiting the last mixer-settler was designed to be centrifuged to eliminate any aqueous/solvent emulsion. This solvent centrifuge was later taken out of service due to the lack of aqueous entrainment in the loaded organic

stream. Any aqueous fraction is returned to the sulfite/solvent separator tank. The rich solvent is then pumped to the rich solvent storage tank. From there the rich solvent is pumped to the top of the solvent stripper after it is preheated from 24°C (75°F) to 100°C (212°F). A nitrogen blanket is maintained in all solvent systems to prevent deterioration/oxidation of the loaded organic.

Sulfate Crystallization

The bisulfite bleed stream to the sulfate removal system enters the sulfate crystallizer tank. The solution is cooled to bring the solution into a supersaturated condition with respect to sodium sulfate. The cooling is accomplished by a glycol recycle system with a refrigeration unit. The sulfate solids crystallize out of the solution and are filtered out by a vacuum pan filter. These solids are sent to disposal off-site. Any filtrate from the vacuum filter is returned to the bisulfite storage tank. This crystallizer step reduces the buildup of sulfate in solution due to natural oxidation in the absorbers.

Steam Stripping

Steam is fed into the bottom of the stripping column to strip SO₂ from the sulfite-loaded organic solvent stream. The stripping column is a 30-tray, 10-inch diameter column constructed of alloy. Multiple inlets are provided in the column to allow evaluation of performance for columns with various number of trays in service.

The solvent flows through a shell and tube heat exchanger to preheat the liquid prior to steam stripping. The preheater is operated at a very close approach temperature, using a steam supply with a controlled inlet temperature not exceeding 216°F. The temperature of the outlet solvent temperature is monitored to ensure that the preheater is achieving the proper outlet temperature i.e. 208°F ±4°F.

Solvent flows down through the tower and collects in the bottom sump. The level is maintained by controlling the flow rate from the stripper to the stripper bleed separator tank. This tank acts as a horizontal settler to remove most of the entrained, condensed steam from the solvent prior to sending the solvent to the wash system.

Steam flow to the stripper comes from the 35 psi steam supply. Steam pressure is throttled down to a point at which its condensation temperature is below 216°F. The low steam temperature is necessary to protect the solvent from overheating.

The SO₂/steam gas stream exiting the top of the stripper is processed through the SO₂ off-gas condenser, which removes the condensed steam from the SO₂. The SO₂ gas at 52°C (125°F) is returned downstream of the I.D. fan for mixing with the scrubbed flue gas and bypassed flue gas.

Solvent Treatment

The hot stripped solvent from the stripper separator is sent through some proprietary solvent treatment steps in which the solvent is better conditioned for reuse. The treated solvent is sent to a settler and then to the solvent storage tank.

4.3 ANALYTICAL EQUIPMENT, ANALYTICAL MEASUREMENTS AND SAMPLING

Wet Analysis

A number of analytical methods are based upon chemical wet analysis. Items of analytical equipment used are listed in Table 4.3-1 and chemicals needed in Table 4.3-2.

Table 4.3-1
Analytical Equipment Needed
For Wet Analysis

Pipets - 1 ml (serological - large tip)
Beakers - 50 ml
Beakers - 400 ml
(Larger Beakers to make solution up)
Graduated Cylinders - 10 ml
Graduated Cylinders - 25 ml
Graduated Cylinders - 50 ml
Erlenmeyer Flasks - wide mouth - 250 ml
Double Buret Support
Burets - 50 ml (2 for each set up)
Pipets (50 ml)
Separatory Funnels - 60 ml
Bubb-Type Safety Pipet Filters
Corning Hotplate Stirrer - and stirring Bars
Balance (Analytical)
Balance (Table)
Nitrogen
Centrifuge (Table) & Centrifuge Tubes
Flow Meters
Gas Regulator for N₂ Cylinder
Test Tubes
Support Stand for Test Tubes
Iron Stands, Clamps, Holders
Rubber Stoppers
Glass Tubing
Latex Lab Tubing
Thermometer
Reagent Bottles
Storage Bottles
Round Bottom Flasks - 250 ml and 500 ml
Medicine Droppers

Table 4.3-2
Chemicals Needed For Wet Analysis

For General Use

Sodium Meta Bisulfite
Sodium Hydroxide
Iodine Solution - 0.1N
Sodium Thiosulfate Solution - 0.1N
Starch Indicator
Sulfuric Acid
Sodium Carbonate

For Ion Chromatograph

Sodium Sulfate for Standard
Sodium Sulfite for Standard
Formaldehyde (to stabilize the sulfite solution)
Sodium Carbonate - Eluent
Sodium Bicarbonate - Eluent
Sulfuric Acid - Regenerant
Sodium Hydroxide Solution

Volumetric Flasks

2L(preparation of Eluent & Regenerating Solution)

1L(preparation of stock)

100 ml - for dilution

50 ml - for dilution

25 ml - for dilution

Pipets - 1 ml

5ml } listed previously

10 ml

B.D. Brand Disposable Syringes (5 ml Luer - Lok)

Among these chemicals, formaldehyde is considered as a hazardous material. If the laboratory does not have a special cabinet for its storage, mannitol may be used instead.

Analytical measurements that need to be performed to support the demonstration program include the following:

Table 4.3-3 Wet Analyses Needed to Support the Demonstration Program
<u>Aqueous Solution</u> Aqueous Loading Analysis - Total sulfite Na ₂ SO ₃ analysis NaHSO ₃ analysis Na ₂ CO ₃ analysis NaOH analysis Stripper condensate (i.e. H ₂ SO ₃) analysis <u>Organic Analysis</u> Loaded organic analysis Organic Loading Analysis in Lean Organic Extraction Equilibrium Determination

For the aqueous phase, total sulfite and Na₂SO₃ used standard iodometric titration; Na₂CO₃, NaOH, NaHSO₃ and H₂SO₃ use standard acid - base titration. For the organic phase, the sulfite is first extracted from the organic phase to the aqueous phase and the aqueous phase is then titrated iodometrically for total sulfite. The equilibrium curve determinations are based upon the total sulfite determination in both the organic phase and aqueous phase when they are in equilibrium with each other.

Procedure for standard acid - base titration and iodometric titration can be found elsewhere. Procedures for (1) extracting the sulfite from the organic phase to the aqueous phase preceding the organic loading titration and (2) equilibrium determination are appended.

Ion Chromatograph

A DX-100 ion chromatograph was analyzed from Dionex Corporation, North Pointe Commons, 9 East Stow Road, Marlton, NJ, to handle total sulfite and sulfate analysis. When sulfite loading in the organic drops below 1%, iodometric titration becomes inaccurate and the ion chromatograph must be used for more accurate determination.

The component parts of the ion chromatograph order include the following:

Table 4.3-4
Component Parts of Ion Chromatograph Ordered

<u>QTY</u>	<u>P/N</u>	<u>Description</u>
1	43025	DX-100 Ion Chromatograph
1	43050	DX-100 Spare Parts Kit
1	43117	Thermal Stabilizer (TS-2)
1	43174	IonPac AS4A-SC 4mm Column
1	43175	IonPac AG4A-SC 4mm Guard Column
1	43189	ASRS-1 (4mm) Anion Suppressor
1	38018	Installation Kit, AMMS or CMMS
1	36802	System Installation (extends warranty to 6 months)
1	42096	Model 4400 Integrator (1 Channel)
1	42819	Start up Kit, 4400
1	39534	Automated Autosampler (ASM)
		Please specify 5.0 or 0.5 ML cassettes for autosampler
1	43051	Automation Cable, DX-100 to Integrator to ASM

The ion chromatograph was found to be relatively easy to use and yielded accurate results. Several calibration curves for that equipment are appended.

PH Measurement

Standard pH probes are used for pH determination.

It should be cautioned that when pH measurement is used for sulfur loading determination, there is a discrepancy between the theoretical relationship and the measured relationship. The discrepancy is shown in Figure 8-1.2. Therefore before a pH probe is used to determine the sulfur loading, an experimental curve such as the one shown in Figure 8-1.2 for that particular probe must be first determined.

Viscosity Measurement

The viscosity measurements were made with a Brookfield dial viscometer, using the LV1 and LV2 spindles. Since the revolutions per minute of the spindle could vary, the instrument came with a slide table with factors that were to be multiplied to the reading on the dial. For example, spindle LV2 at 60 rpm had a multiplication factor of 5. Therefore, to get the viscosity in centipoise at a dial reading of 50 using spindle LV2 and 60 rpm, one would simply multiply 50 times 5 (250 centipoise).

Normally a 600ml cap is used for viscosity measurement. To use less sample for the measurements, a small 75ml cup was used. The viscometer manual specified that the instrument had to be calibrated if a cup smaller than 600 ml was used. Since we used a 75 ml cup, we had to calibrate it using 968 and 48 centipoise standards bought from Brookfield Labs. The calibration determined that spindle LV1 and LV2 had second correction factors of 0.818 and 0.793, respectively. This correction factor was multiplied to the result explained above (i.e., for LV2: $250 \times 0.793 = 198$ cp).

A typical experimental measurement is appended.

Entrainment Measurement

A benchtop General - purpose centrifuge was used to determine either aqueous entrainment in organic solution or organic entrainment in aqueous solution.

The procedure for the entrainment determination is appended.

Sampling

There are all together 4 gas sample ports (G1 to G4), 19 aqueous solution sample ports (A1 to A19) and 10 organic sample ports (S1 to S10). Several (A1, A5 A11, S3 & S6) have since been eliminated. The locations of the active sample ports are given in Figure 4.3-1. Table 4.3-5 gives the designation of these sample ports and the analyses to be performed on these samples.

Frequencies for analysis varied depending on the objectives of the experiment that was being performed. They are specified as an instruction sheet to the operator such as the one shown in Table 4.3-6. This instruction sheet differed from experiment to experiment.

REV.		ENG. RECORD		DRAWING STATUS		PROCESS FLOW DIAGRAM	
NO.	DATE	NO.	DATE	NO.	DATE	TUNG FLUE GAS DESULFURIZATION	
1		1		1		TEST FACILITY NMPC DUNKIRK UNIT NO. 4	
2		2		2		RAYCON RESEARCH & DEVELOPMENT INC.	
3		3		3		RAYCON RESEARCH & DEVELOPMENT INC.	
4		4		4		RAYCON RESEARCH & DEVELOPMENT INC.	
5		5		5		RAYCON RESEARCH & DEVELOPMENT INC.	
6		6		6		RAYCON RESEARCH & DEVELOPMENT INC.	
7		7		7		RAYCON RESEARCH & DEVELOPMENT INC.	
8		8		8		RAYCON RESEARCH & DEVELOPMENT INC.	
9		9		9		RAYCON RESEARCH & DEVELOPMENT INC.	
10		10		10		RAYCON RESEARCH & DEVELOPMENT INC.	
11		11		11		RAYCON RESEARCH & DEVELOPMENT INC.	
12		12		12		RAYCON RESEARCH & DEVELOPMENT INC.	
13		13		13		RAYCON RESEARCH & DEVELOPMENT INC.	
14		14		14		RAYCON RESEARCH & DEVELOPMENT INC.	
15		15		15		RAYCON RESEARCH & DEVELOPMENT INC.	
16		16		16		RAYCON RESEARCH & DEVELOPMENT INC.	
17		17		17		RAYCON RESEARCH & DEVELOPMENT INC.	

TABLE 4.3-5 SUBSYSTEM CLASSIFICATION OF SAMPLES

Absorber Subsystem Gas Samples					
G1	Flue Gas Inlet to Venturi				
G2	Flue Gas Inlet to Primary Absorber				
G3	Flue Gas Exit from Primary Absorber				
G4	Flue Gas Exit from ID Fan (Includes the Flue Gas Bypass)				
Absorber Subsystem Aqueous Samples			Analyses to be performed *		
A2	Primary Absorber		1,	8	
A3	Secondary Absorber		1,	8	
A6	Sulfite Feed to Absorbers		1,	8	
A10	Neutralization Tank Outlet Stream		8		
A14	Condensate T-110 - Canceled: Health Risk				
A15	Venturi to Neutralizer		8		
A17	Carbonate Storage Tank		8		
Extraction Subsystem					
A4	Sulfite Solution: Q - 101		1,	5,	8
A16	Bisulfite Feed to Extraction		1,	8,	9
S2	Rich Solvent: Q-105		2,	5,	10
S8	Solvent Feed to Extraction: T-101		3,	4,	10
S10	Lean Solvent: S-107		3,	5,	10
Stripper / Solvent Treatment					
S1	Regenerated Lean Solvent: Q-109		3,	4,	5, 10
S4	Stripper Tray		3,	5,	10
S5	Stripper Bleed Separator Solvent		3,	5,	10
Crystallization Subsystem					
A7	Bisulfite to Sulfate Filter: T-115		1,	8,	9
A8	Solid Sulfate Sample from Filter		1,	8,	9
A9	Filtrate Sample from Sulfate Filter		1,	8,	9
Procedure	Name	Time Needed			
		To Analyze			
1	Aqueous Total Sulfite Analysis	25 min.			
2	Rich Organic Loading Analysis	25 min.			
3	Lean Organic Loading Analysis	25 min.			
4	Lean Organic Equilibrium Curve	2 hrs			
5	Aqueous & Organic Entrainment Measurement	15 min.			
6					
7	Gas Chromatography	unknown			
8	pH	5 min.			
9	Sulfate Analysis (Turbidimeter)	15 min.			
10	Viscosity	15 min.			

CHEMICAL TESTING SCHEDULE

TEST: _____

DATE: _____

TIME TAKEN		INITIAL	1 HR LATER	1 HR LATER	2 HR LATER	2 HR LATER	EVERY 2 HOURS UNTIL TEST CONCLUSION					
	A4		*	*	*	*	*	*	*	*	*	*
	A16	*		*	*	*	*	*	*	*	*	*
	S2		*	*	*	*	*	*	*	*	*	*
	S8	*		*	*	*	*	*	*	*	*	*
	A11		*	*	*	*	*	(1)	(1)	(1)	(1)	(1)
	S1			*	*	*	*	(1)	(1)	(1)	(1)	(1)
	S5		*	*	*	*	*	(1)	(1)	(1)	(1)	(1)
(1) AS REQUIRED; EVERY 2 HOURS UNTIL ALL SOLVENT IS STRIPPED												

Table 4.3-6 Example of Sampling Schedule

4.4 DATA ACQUISITION AND REDUCTION

Data Acquisition and Reduction Design

The basic philosophy of the data acquisition system (DAS) design was to continuously record plant operating data that would be critical to the performance of the FGD system. A Siemens Programmable Logic Controller (PLC) formed the central core of this DAS. Initially, a Piping and Instrumentation Diagram (P&ID) was assembled and instrumentation requirements were identified in each process stream. Instrumentation was selected based on the need to know a specific parameter for performance analysis or process control, along with an appropriate design and materials of construction that would be appropriate for the process conditions at that point. Discussions between Raycon and Raytheon helped to identify those conditions that would be deemed critical to a successful system operation. These instruments were then selected to have their signals sent to the PLC.

The PLC had the capability to both record the signals sent by the instrumentation and to inform the operating staff within the control room whenever an operating parameter exceeded the specific ranges that had been preset within the computer memory. A third capability of the PLC was as a motor and valve controller. Whenever a change in specific operating conditions was desired, the PLC could be used to modify valve positions, change motor speeds or turn on or off motors for various pumps, fans, etc. The PLC then recorded the operating conditions at the new setting.

As Raycon and Raytheon developed experience with the system operation, it became apparent that key data points needed to be readily available. A manual data log sheet was assembled to record these data, along with other local readings that were not continuously sent to the PLC. This log sheet then became the site personnel's source for day-by-day analysis of the system operating conditions and comparisons of system performance (based on the results obtained after the chemical analyses of samples) with the test conditions.

On a daily basis, the data recorded by the PLC were downloaded onto floppy disks. These disks contain the recorded data on a one-minute average basis. Reduction of this information would require compilation of these data into longer

averaging periods and then production of continuous curves to establish system operating conditions throughout the program. This historical record could then provide insight into why system performance might be different over time, even given that the apparent operating conditions were identical.

However, it should be noted that the analytical data from the laboratory was far more important to the evaluation of the system operation than the plant operating data. Laboratory analytical data were also recorded on log sheets, taking great care to accurately track manual samples of process chemicals, with a record of both the time it was taken and the location noted on each sample. These data proved to be useful when producing system analyses of performance, such as a material balance around the process. The data in the archive were all recorded against the same time line, so that the integrated system performance and operating conditions could be evaluated during specific periods.

Initially, it was hoped that the data reduction could be done continuously throughout the program. However, due to the time lag between operation and sample analyses in the lab, plus the complexity of the system operation, this was not possible on a continuous basis. At least one or two additional people would have been required to accomplish this task, and the operating budget did not allow these additional staff to be sent to the site.

Data Acquisition Prior To Ductwork Repair

Before duct work repair (11-28, 1993 to 1-18, 1994), the on-site experimental work was directed by Raytheon's personnel. Besides the data that were routinely logged into a computer, some data were also hand logged into a set of data sheets which include:

1. Absorber - Floor Readings (hourly samples)
2. Primary Absorber System - Control Room (hourly samples)
3. Absorber Subsystem Aqueous Samples - Sample Analysis Summary
4. Extraction System - (hourly samples)
5. Extraction System - Control Room (hourly samples)
6. Extraction System - Sample Analysis Summary

7. Stripper Process Reading - Control Room
8. Solvent Regeneration System - Floor Reading (hourly samples) - 4 sheets
9. Stripper / Regeneration / Subsystem Samples - Sample Analysis Summary
10. Crystallization Subsystem - Sample Analysis Summary

The floor readings were taken by an operator at the plant and the control room reading were taken by the control room operator directly from the computer screen both on an hourly basis. Because of the shortage of test site personnel, neither the data logged in the computer or the hand logged data in the data sheets was treated in a timely manner.

Data Acquisition After Duct Work Repair

There was a managerial realignment for the on-site test program after the test was resumed after ductwork repair. Raycon joined the test team and took over the supervision, testing planning and test evaluation on a daily basis. Raytheon technical personnel assisted Raycon in the test plan implementation.

Extensive review of the progress made before the duct repair revealed one shortcoming of the prior program i.e. data collected were merely recorded but not evaluated. This deprived each individual test from being compared to the results already attained from the previous tests.

A major reason that the test results were not analyzed in a timely fashion was because as stated earlier analyzing data was tedious and time consuming. Data logged in the computer needed to be averaged out and down loaded into a spreadsheet before they could be treated. Since extra time was not much available at the site, the site test engineer in charge of the test program would have had to make a choice between treating the data already at hand or proceeding with gathering more data according to a preplanned program outlined in the Operating Manual. At the early stage of the test program, it appeared to make sense to opt to gather more data rather than to attend to data reduction and evaluation.

In order to assure the timely data treatment a new set of data sheets aiming at taking data at certain points of time was designed. They include:

1. Daily Data sheets - both operational data and analytical data are included
2. Data sheet for Absorber Subsystem
3. Data sheet for Extraction Subsystem
4. Data sheet for Stripper Subsystem
5. Data sheet for Alkali Wash Subsystem
6. Data sheet for Bubbler Subsystem

These data sheets are used in Appendix B as shown in Tables B-1, B3a, B3b, B5, B7a, B7b and B9. It is understood that these snap-shop data are not as satisfactory as time-average data taken over a certain interval of time under steady-state operating conditions, but they are nevertheless still useful and valid for a number of assessments such as for material balance evaluation and performance evaluation of certain equipment component provided that steady state was attained for that component when the snap-shot data were being taken.

Data Reduction

How evaluations were done to achieve material balance and performance assement may be illustrated by how data are treated for the Extraction Subsystem: A sketch of the extractor is shown as follows: This is the same picture that will later be shown as Figure B-2 in Appendix B.

Based upon this sketch, the following Sulfur Balance equation should hold:

$$\begin{matrix} \text{(I)} & & \text{(II)} \\ \left[\begin{array}{l} \text{Lean solvent} \\ \text{Feed to M/S} \\ \text{(gpm)} \\ \text{(71)} \end{array} \right] \left[\begin{array}{l} \text{Composition} \\ \text{(lb/gal)} \end{array} \right] & + & \left[\begin{array}{l} \text{chilled M/S} \\ \text{aqueous feed} \\ \text{(gpm)} \\ \text{(27)} \end{array} \right] \left[\begin{array}{l} \text{Composition} \\ \text{(in lb/gal)} \end{array} \right] \end{matrix}$$

$$\begin{matrix} \text{(III)} & & \text{(IV)} \\ = & \left[\begin{array}{l} \text{Rich solvent} \\ \text{outlet from} \\ \text{M/S (gal/min)} \\ \text{(45)} \end{array} \right] \left[\begin{array}{l} \text{Composition} \\ \text{(lb/gal)} \end{array} \right] & + & \left[\begin{array}{l} \text{Regenerated} \\ \text{Sulfite to} \\ \text{Centrifuge} \\ \text{(gpm)} \\ \text{(47)} \end{array} \right] \left[\begin{array}{l} \text{Composition} \\ \text{(lb/gal)} \end{array} \right] \end{matrix}$$

Then,

$$\% \text{ close} = \frac{[(\text{III}) + (\text{IV})] - [(\text{I}) + (\text{II})]}{[(\text{I}) + (\text{II})]} \dots\dots\dots (4.4-1)$$

$$\text{and Performance \% SO}_2 \text{ transferred} = \frac{(\text{III}) - (\text{I})}{(\text{I})} \quad (4.4-2)$$

$$= \frac{(\text{II}) - (\text{IV})}{(\text{I})} \quad (4.4-3)$$

Material balances and performances for the various component equipment including the absorber, extractor, stripper are given in Appendix B.

FLUE GAS SYSTEM

ABSORBER SYSTEM

PIPING

PUMPS

TANKS

OTHER PROCESS EQUIPMENT

LESSONS LEARNED

SECTION 5 MATERIALS OF CONSTRUCTION

Materials of construction for each system component were selected based upon process chemistry requirements and operating conditions. The following criteria applied to all systems:

- No copper or copper alloy (such as brass) material was acceptable. The copper would react with the organic solvent.
- All components in contact with the organic had to be constructed of 316 stainless steel as a minimum, including all transfer piping.
- Polyethylene and polypropylene (without fillers) were acceptable for organic storage. (Fillers in plastics or FRP could potentially dissolve into the organic.)
- All gaskets had to be made of a chemically inert material such as teflon.
- Wastewater service was typically PVC, as were other nitrogen-blanketing feedlines and electrical conduit. Carbon steel was used for service water and steam lines.

Every effort was made not to introduce chemicals into the system that could interfere with the organic solvent chemistry. It should be noted that no materials evaluations were done after the test program, so conclusive statements on materials of construction cannot be presented.

The following provides a discussion of materials selection by subsystem, addressing specific process issues and materials problems where they occurred.

FLUE GAS SYSTEM

The flue gas system design included takeoff ductwork to bring "dirty" flue gas from the NMPC plant to the pilot plant, transfer gas between absorber modules and an I.D. fan to return the "cleaned" gases to the NMPC ductwork. Carbon steel ductwork lined with a vinyl ester resin was selected as the duct material based on cost and previous positive experience in similar applications. The I.D. fan was constructed of stainless steel due to operation downstream of the scrubbers after flue gases were mixed to provide reheat. No materials failures occurred in the fan system.

In operation, the inlet ductwork to the plant failed due to corrosion of the carbon steel behind the liner. Analysis showed that the duct liner had possibly been improperly applied (too thick and insufficient surface preparation). Cracks in the liner occurred, allowing flue gas and condensed acid solutions to directly contact the carbon steel, causing corrosion at high rates. However, the outlet duct liner did not experience these same problems. It was theorized that the multiple startups and shutdowns caused multiple thermal expansions in the duct on the inlet (70°F ambient to 300°F+ when hot gas was flowing). This thermal shock, along with the questionable application of the liner (thicker liner material has less flexibility, causing the cracking) resulted in the failure. The outlet ductwork seldom saw hot gas and therefore did not experience the range of thermal shock seen by the inlet duct. The inlet duct was replaced with a thinner-walled S.S. duct.

ABSORBER SYSTEM

The venturi scrubber was constructed of rubber-lined carbon steel. No materials failures occurred in this unit. The connecting ductwork between the venturi and the primary absorber was constructed of FRP. This section performed adequately.

The primary and secondary absorber vessels and their internal piping were constructed of 316L stainless steel. Polypropylene packing was used to enhance gas-liquid contact. FRP and rubber-lined carbon steel were not acceptable for the absorber units due to potential reaction with or contamination of trace concentrations of organic solvent in the recycled scrubber liquor. Polypropylene was also selected for the mist eliminators installed at each absorber outlet. No materials problems were identified in the FGD system during the approximately 11 months of intermittent operation.

PIPING

Almost all of the piping in the system was constructed out of 316 S.S. due to potential contamination of the organic solvent by steel or other materials. No materials failures occurred. Some leakage at pipe connections can be attributed to the requirement that only teflon sealant was used at all joints. Alternate pipe sealing compounds with better sealing characteristics at varying temperatures could not be used due to their unknown impacts on the organic.

Where organic contamination of process streams was not a concern, PVC piping was used as appropriate given the various operating temperatures. No failures occurred. Carbon steel pipe was used effectively for all steam and some water lines.

PUMPS

The Tung pilot plant system used approximately 40 pumps to move liquids from one component to another. Rubber-lined carbon steel pumps were installed only in the venturi recycle service due to the low pH, fly ash content and no potential for organic solvent contact. However, the rubber-lined pumps originally supplied were not designed for low pH operations due to mis-specification by the pump supplier. Carbon steel components were used in areas in contact with the recycle liquor, causing major corrosion problems. After replacement with stainless steel parts, these pumps performed adequately.

All other pumps in the system were constructed of 316 stainless steel with teflon seals. Low, closely controlled flow rates were required in many process streams, so magnetically driven chemical-feed gear pumps were used in many locations. Many of these pumps had constant maintenance problems due to the inability to maintain flow capacities when operating at process temperatures and viscosities. The supplier revised its materials selection for the drive magnets part-way through the test program due to lower torque capability at temperatures above 100°F with standard magnets. Rare earth magnets were installed, reducing operating problems. In any future installation, the use of these pumps is not recommended due to their intolerance to small changes in operating conditions. It is also recommended that cleanable strainers be installed upstream of all pumps in a future installation to eliminate the potential for solids scoring of the pump internal surfaces. In general, the 316 S.S. material performed adequately following replacement of some ryton drive gears

originally used in aqueous solution service. Small particles in the solutions and differential temperature expansion caused binding of the composite pump gears.

All 316 S.S. centrifugal pumps performed adequately in terms of materials and flow capacity.

TANKS

Tanks were constructed of stainless steel, high density polyethylene and FRP depending on service. No materials problems were experienced.

OTHER PROCESS EQUIPMENT

All heat exchangers were constructed of 316L S.S. or higher alloy. No materials problems occurred. Mixer - Settlers used in extractor also were constructed of 316L S.S. and had no problems related to materials of construction. The stripper column and trays were constructed of 904L alloy due to high temperature operation with potentially low pH conditions and presence of sulfurous acid/gaseous SO₂. No materials problems occurred.

The sulfate crystallizer was designed for operation at the low pH conditions required for the bisulfite solution. However, the selection of the scraper blade material was a potential problem. The normal blades were a plastic composite. This presented a possible source of contamination to the process. Three composite materials, namely phenolic-linear laminate, polyimid laminated with fiber glass and expoxy laminated with fiber glass were evaluated and phenolic-linear laminate was selected for use based on laboratory evaluation. No problems attributed to this material selection were identified during system operation.

The stripper gasket material was originally specified to be woven teflon. This material appeared to function correctly initially. After a period of time however, the teflon stiffened and no longer sealed properly. It was replaced with woven teflon-coated fiberglass, but the stripper performance still did not meet the process requirements, so operation of the metal column was discontinued although no material problems occurred. Borositicate glass was used as the material for the substituted stripper column.

LESSONS LEARNED

In general, the use of 316 stainless steel piping and ductwork is probably the best alternative for a future installation. Storage tanks should be constructed of high-density polyethylene except where process temperatures exceed 140°F, including the organic solvent. Depending on the expected length of operation, 316L is probably adequate for the absorber construction. 317LMN or higher alloy should be considered for a longer life. All pumps should be designed for no contamination of the solvent (316 S.S. and teflon seals) and with higher capacity to eliminate the nuisance maintenance shutdowns. The lack of an extended test period (only ran over an 11 month intermittent period) and no post-program testing of materials for pitting or general corrosion rates results in the limited number of recommendations and conclusions regarding materials of construction.

**OPERATING MANUAL
OPERATOR TRAINING**

6.1 STARTUP AND INITIAL PLANT OPERATION

Startup (7-12-93 through 8-20-93)

Initial Plant Operation (8-23-93 through 11-19-93)

Absorber Subsystem

Extractor Subsystem

Stripper Subsystem

Mechanical Test

6.2 PARAMETRIC TESTING

Absorber Subsystem

Extractor Subsystem

Stripper Subsystem

Crystallizer Subsystem

6.3 INTEGRATED TESTING

6.4 STEAM STRIPPER TESTING

Steady State Operation

Stripper Repair

Repair Column Testing

Design and Installation of a 4-inch Glass Column

Glass Column Testing

Glass Column Design Modification

Modified Glass Column Testing

SECTION 6 SYSTEM OPERATION AND TEST RESULTS

This section provides a detailed description of the test program and records the test results of the program.

OPERATING MANUAL

An operating manual was assembled for the plant. This document detailed all aspects of the system operation, including the following:

- Project scope and equipment
- Staff organization and participants
- System description
- Laboratory procedures
- Startup and shutdown operation
- Subsystem operating methods
- Test plan
- Data accumulation and reduction
- Materials receiving and control
- Equipment operating manuals
- Piping and Instrumentation, General Arrangement and Process Flow Drawings
- Site security
- Data sheets
- Instrumentation set points
- Process concerns

This manual served as the basis for plant operation and was incorporated into the operator training program discussed in the following subsection.

OPERATOR TRAINING

A detailed plant operator training program was provided to all locally hired site staff members. The program included safety training on all system hazards and proper operating methods including protective clothing, etc. A Safety Manual and Failure Analysis Manual were provided to each staff member and reviewed during the safety training program along with NMPC safety requirements and site security.

The plant operating manual was reviewed in detail, specifying the responsibilities of all staff members. Laboratory chemists were provided with additional instruction on analytical methods in a five-day program which included hands-on operation of laboratory systems. This program was followed by several weeks of additional training during the startup of the plant. As a result, the operating staff for the Tung facility were able to maintain operating conditions over extended periods while responding to upset conditions and maintenance problems.

6.1 STARTUP AND INITIAL PLANT OPERATION

The pilot plant startup and initial phases of operation are addressed by subsystem and major equipment items. The sequence of initial startup activities is outlined with emphasis on solutions to mechanical and process problems encountered. The initial operating phases of the project included a series of planned tests to establish the operating limits of equipment, process flexibility and a preliminary scoping of key process conditions on equipment performance.

Startup (7-12-93 through 8-20-93)

The absorbers were first started on water to test level and flow controls, and to check operation of the pumps and dampers. The I.D. and booster fans were initially operated with air. On 7-26-93, circulation of sodium carbonate (Na_2CO_3) solution in the absorbers began and flue gas flow from Unit No. 4 through the system was initiated. The primary objectives during startup of the absorbers were:

- Verify control of primary absorber inlet temperature to prevent high temperature gas from damaging the polypropylene packing material.
- Achieve pH levels in the primary and secondary absorbers of about 6.0 and 5.0 (or less), respectively.
- Show conversion of the 1 M (molar) sodium carbonate feed to 2 M sodium bisulfite.
- Gather preliminary flue gas data.

While feeding the absorbers with 1 M carbonate solution, it was shown that the primary absorber pH could be maintained in the range of 5.8 to 6.0 and that the secondary absorber pH could be brought down to between 4.9 and 5.2. However, during this initial period, the total sulfite concentration in the circulating liquor could not be increased above about 1.2 M because sodium sulfite and sodium bisulfite were oxidizing to sulfate when in contact with the flue gas.

The extraction system, including the regenerated sulfite return circuit, was initially operated on water to verify mechanical integrity. This water testing occurred while the absorbers were being started up on flue gas. Sodium bisulfite solution was first fed to the extraction system (Q-105) during the week of August 9, 1993. No organic was handled during startup. The bisulfite was pumped directly back to the absorber system without regeneration to sulfite.

The stripper was operated using steam and water feed during startup. As with the extractor, no organic was handled in the stripper during startup. The primary objectives during stripper startup were to verify:

- Steam flow control (FIC-0667).
- Stripper feed temperature control (TCV-0650).
- Stripper feed flow indication (FT-0549).
- Stripper sump level control (LIC-0600).

Startup of the stripper included initial operation of the column outlet condenser (E-107) and SO₂ product gas fan (C-103). Water pumped from the stripper sump during startup flowed to the stripper bleed separator (S-104).

Steam flow control was found to be very unstable in automatic mode. This was attributed to the erratic signal received from the steam flow transmitter. During startup and a portion of the subsequent initial operations, all steam flow to the stripper was manually checked using the sparger rotameters (FI-0666A-E).

In order to control the liquid feed preheat, it was found that occasional adjustments to the temperature control valve (TCV-0650) were required to maintain a liquid feed temperature below 220°F. The temperature typically cycled between about 160°F to 210°F, apparently due to lag times between the control valve control of the steam rate and the outlet temperature readings. While operating on water, the magnetic-type stripper feed flow indicator (FI-0549) gave unreliable readings; this unit required recalibration. Subsequent test showed that this meter was incompatible with the organic/aqueous stream and it was removed from service.

The stripper sump level control worked as installed with some manual tuning required.

Startup of the washing system (Q107/-108) went smoothly, first on water only to check system integrity, followed by the feed of sodium carbonate aqueous solution. The temperature control valve (TCV-0787) on the feed line operated as expected.

The crystallizer was operated under various conditions, using only water initially, to determine appropriate filtrate and vacuum pump seal water flow rates. Conclusions were the following:

- Liquid rate to filter pan must not exceed 1/2 to 3/4 gpm to allow the filtrate pump (P-108) to keep up. Receiver overflow would otherwise move through to vacuum pump, causing poor performance.
- With no water flow to the filter pan, seal water discharged from the vacuum pump air vent line. The arrangement of the seal vent piping was reviewed with the supplier and modified to allow proper operation.

During the startup period, the pilot plant laboratory was set up with the necessary equipment, chemists were trained and verification of analytical methods was completed. Analytical procedures initially in place included sodium carbonate, sodium bicarbonate, total sodium sulfite, sodium bisulfite, pH analyses and organic sulfite loading of process samples and viscosity measurement. Plans were made for installation of a gas chromatograph (G.C.) to determine very low organic sulfite loading levels. An ion chromatograph replaced the G.C. later during the program due to the failure of the G.C. to perform adequately. The startup test period for the initial system design/installation was essentially complete after the first six weeks of operation.

Initial Plant Operation (8-23-93 through 11-19-93)

Following completion of the startup/checkout period, the plant began the initial stages of operation. The primary objectives during this period were to demonstrate equipment reliability and steady operation at design conditions and to evaluate in a preliminary manner the effects of changes in process conditions on system performance. Plant performance and test conditions evaluated are presented in the following tables by subsystem.

Absorber Subsystem

The baseline operating conditions established for the absorbers were as follows:

- Total maximum gas rate to venturi 6470 ACFM
(2 MW equivalent)
- Gas flow rate to secondary absorber 3100 ACFM
(48% of total)
- Primary absorber circulation rate 110 gpm
L/G (gal/ 1000 ACFM) 17
- Secondary absorber circulation rate 53 gpm
L/G (gal/1000 ACFM) 17
- Primary absorber pH 6.0
- Secondary absorber pH 5.0

Note: Planned subsystem tests were not conducted in numerical order shown in the operating manual nor on consecutive dates. Schedule and conditions were modified due to the results obtained during preliminary tests and the operational status of plant equipment and subsystems. Test results were accumulated by subsystem.

Table 6.1-1 Summary of Absorber Initial Operation	
Test AB-1 8-27-93	
Objective	Operate at base 2 MW gas rate.
Operating Conditions	6470 ACFM venturi inlet flow. Absorber circulation at baseline conditions
Results	Erratic gas flow indication (pulsation) at venturi Prescrubber inlet forced the reduction of gas flow to the 1 MW rate. Test AB-1 aborted after 2 hours.

Table 6.1-2 Summary of Absorber Initial Operation	
Test AB-6 8-27-93	
Objective	Operate at 1 MW gas rate.
Operating Conditions	3235 ACFM venturi inlet flow. 1550 ACFM flow to secondary absorber. L/G at baseline condition.
Results	SO ₂ removal 84%. Experienced high venturi outlet temperatures; example at 6:40 pm: venturi inlet (FI-0103) 3180 ACFM Bypass (FI-0102) 3680 ACFM Venturi outlet Temperature (TE-0104) 149°F (rising) Stabilized flows and temperatures and ran until normal shutdown at 8:30 PM. venturi inlet flow was stabilized at about 2700 ACFM; below the 1 MW target of 3235 ACFM.

Table 6.1-3 Summary of Absorber Initial Operation	
Test AB-6A 9-3-93	
Objective	The objective of this test was to operate the system at 1 MW equivalent flue gas flow. This is a repeat of AB-6 (8/27) which was not successfully performed because high venturi pre-scrubber outlet temperatures prevented establishing 1-MW equivalent gas flow rate.
Operating Conditions	3235 ACFM venturi inlet flow. 1550 ACFM flow to secondary absorber. L/G at baseline condition.
Results	The 1 MW flow rate of 3235 ACFM was easily maintained. Flue gas flows as high as 6300 ACFM (near 2 MW) were achieved at the end of this test. Total sulfite concentration of 1.14% was observed at secondary absorber pH=4.9.

Table 6.1-4 Summary of Absorber Initial Operation	
Test AB-6B 9-17-93	
Objective	The objectives of this test were to operate the system at 1 MW equivalent flue gas flow and produce 2 M sodium bisulfite solution. This is a repeat of AB-6A (9/3)
Operating Conditions	3235 ACFM venturi inlet flow. 1550 ACFM flow to secondary absorber. L/G at baseline condition.
Results	Flue gas flows of about 3200 ACFM (1 MW) were held steady during this test. Total sulfite concentration of 1.11% was observed at secondary absorber pH=5.0. The test was stopped to prepare for Test AB-1A which followed.

Table 6.1-5 Summary of Absorber Initial Operation	
Test AB-1A	
Objective	Operate at base 2 MW gas rate and produce 2 M sodium bisulfite solution.
Operating Conditions	6470 ACFM venturi inlet flow. Absorber circulation at baseline conditions.
Results	Flue gas flows as high as 6300 ACFM (near 2 MW) were achieved during this test. Total sulfite concentration of 1.14% was observed at secondary absorber pH=4.9. Oxidation rates at this low pH continued to keep the total sulfite concentration below the levels desired.

Table 6.1-6 Summary of Absorber Initial Operation	
Test AB-2 9-20-93	
Objective	Reduce the rate of gas to the secondary absorber from the baseline rate of 48% of total inlet flow to 30%.
Operating Conditions	6470 ACFM venturi inlet flow. Absorber circulation at baseline conditions.
Results	The total inlet rate was specified to be 2 MW equivalent flue gas flow, approximately 6470 ACFM, but only about 5100 ACFM was achieved due to fan limitations. NMPC plant data was not available for correlation. Low total sulfite concentration was reported from secondary absorber; 0.56% (one sample) at pH=4.6. This could be due to low carbonate concentration and the oxidation rates at this pH. This test was stopped to change conditions for Test AB-5. <u>Note:</u> The gas rate to the secondary absorber was inadvertently run at a lower rate than planned. This test will be repeated.

Table 6.1-7

Summary of Absorber Initial Operation

Test AB-5 9-20-93

Objective	The objective of Test AB-5 was to operate the Absorbers at increased L/G.
Operating Conditions	<p>6470 ACFM venturi inlet flow.</p> <p>The circulation in the primary absorber was increased from 110 gpm to 138 gpm. The secondary circulation was increased from 53 gpm to 66 gpm</p>
Results	<p>No flow reading was available from FT-0120C for the secondary absorber circulation flow because it was broken at this time. Previous valve settings were maintained at about 53 gpm.</p> <p>This 2 MW equivalent flow test could only get up to about 5150 ACFM (Unit No. 4 running at 100 to 140 MW). Fan capacity at plant conditions was still limited.</p> <p>Low total sulfite concentration was reported from secondary absorber; 0.55% at pH=4.5. This was apparently due to low carbonate inlet concentration and the high oxidation rate.</p> <p>This test was voluntarily stopped</p>

Table 6.1-8

Summary of Absorber Initial Operation

Test AB-4 9-21-93	
Objective	The objective of Test AB-4 was to operate the absorbers at decreased L/G.
Operating Conditions	<p>6470 ACFM venturi inlet flow.</p> <p>The circulation in the primary was reduced from 110 gpm to 83 gpm. The secondary circulation was specified to decrease from 53 gpm to 40 gpm.</p>
Results	<p>No flow reading was again available from FT-0120C.</p> <p>Previous valve settings were maintained at about 53 gpm.</p> <p>This 2 MW equivalent test could only achieve about 5150 ACFM.</p> <p>Low total sulfite concentration from secondary absorber; 0.29% at pH=4.3. Again, low carbonate concentration is suspected. This indicates that the 1.0 M sodium carbonate solution should be increased in concentration in order to achieve 2.0 M bisulfite and attempts to reduce the oxidation rate are necessary.</p> <p>This test was stopped to change conditions for Test AB-3</p>

Table 6.1-9 Summary of Absorber Initial Operation	
Test AB-3 9-21-93	
Objective	The objective of Test AB-3 was to operate at an increased gas rate to the secondary absorber to 60% of total inlet flow; baseline rate set at 48%.
Operating Conditions	5200 ACFM venturi inlet flow (fan limit taken into account). 3880 ACFM to the secondary absorber. L/G baseline conditions targeted.
Results	This 2 MW test could only achieve about 5000 ACFM total inlet flow; Unit No. 4 running at 194 MW. Plant load was apparently not responsible for reduced fan capacity. Low total sulfite concentration was reported from secondary absorber; 0.29% at pH=4.0. (See results of AB-4 above.) This test was voluntarily stopped

Table 6.1-10 Summary of Absorber Initial Operation	
Test AB-2A 9-24-93	
Objective	The objective of Test AB-2A was to reduce the rate of gas to the secondary absorber from the baseline rate of 48% of total inlet flow to 30%. This repeat test was conducted because the previous test was run at too low a gas rate to the secondary absorber; see note on 9/20/93.
Operating Conditions	6470 ACFM venturi inlet flow. Absorber circulation at baseline conditions.
Results	This 2 MW equivalent test could only achieve about 5100 ACFM; Unit No. 4 running at 192 MW. Low total sulfite concentration was reported from secondary absorber; 0.56% at pH=4.6. This test was voluntarily stopped

Table 6.1-11 Summary of Absorber Initial Operation	
Test AB-6C 9-27-93	
Objective	<p>The objective of Test AB-6C was to gather samples for GC analyses.</p> <p>The Perkin-Elmer gas chromatograph was installed and chemist training completed by this date.</p>
Operating Conditions	<p>3235 ACFM venturi inlet flow.</p> <p>1550 ACFM flow to secondary absorber.</p> <p>L/G at baseline condition.</p>
Results	Samples of primary and secondary absorber samples were obtained.

The startup program for the Tung Pilot Plant SO₂ absorption system identified some fundamental operating conditions that must be modified in the upcoming test program:

- Part of the sulfite was oxidized to sulfate. To compensate for this oxidation, the concentration of the reagent feed solution to the absorbers must be increased to allow production of a 2M bisulfite bleed stream. The 2M concentration of bisulfite is required for efficient sulfite extraction into the organic.
- The primary I.D. fan was limited to less than 2MW equivalent gas flows shortly after startup. Future tests were limited to approximately 80% of the design rate. Correlation to Unit No. 4 operating conditions was not completed. Recalibration of gas flow instrumentation did not indicate any problems with the measurement devices.

Extractor Subsystem

The baseline operating conditions established for the mixer-settler extraction

system were the following:

- One stage of extraction Q-105
- Lean organic flow 0.95 gpm
- Sodium bisulfite feed flow 1.20 gpm
- Aqueous/organic (A/O) volumetric ratio 1.27
- Extractor recycle flow 0 gpm
- Mixer speed 100% (360 RPM)

Table 6.1-12 Summary of Extractor Initial Operation	
Test EX-1	9-13-93
Objective	Operate single stage extractor (Q-105) at baseline process conditions.
Operating Conditions	0.95 gpm lean organic flow. 1.20 gpm sodium bisulfite flow. A/O of 1.27. 100% mixer speed (360 RPM).
Results	<p>During this test solvent carry over from the solvent centrifuge (S-108) to the bisulfite pump tank (T-202) was again experienced, as during the start up tests. Solvent was recycled back to Q-105 from T-202. Rich Solvent was accumulated in the rich solvent tank (T-109).</p> <p>Flow rates achieved were 0.94 gpm organic and 1.2 gpm aqueous. Higher rates were not attainable without overflowing the Q-105 organic drop box.</p> <p>Rich organic was loaded to 15% in the extraction system with an outlet loading of 5% achieved for stripped organic.</p>

Table 6.1-13 Summary of Extractor Initial Operation	
Test EX-1A 9-15-93	
Objective	This test repeats EX-1 to achieve higher loading.
Operating Conditions	0.95 gpm lean organic flow. 1.20 gpm sodium bisulfite flow. A/O of 1.27. 100% mixer speed (360 RPM).
Results	Solvent centrifuge (S-108) was bypassed until proper operating conditions are established which prevent solvent carryover to bisulfite pump tank (T-202). Rich solvent, loaded to 21%, was pumped from Q-105 directly to the Stripper.

Table 6.1-14 Summary of Extractor Initial Operation	
Test EX-2 9-16-93	
Objective	Operate at high aqueous/organic ratio in the Q-105 mixer.
Operating Conditions	0.95 gpm lean organic flow. 1.20 gpm sodium bisulfite flow. A/O of 1.41. 100% mixer speed (360 RPM).
Results	The A/O is increased from 1.27 to 1.41 by adjusting the aqueous recycle flow from the settling chamber of Q-105 to the mix tank. Only 11% rich organic loading was achieved which is attributable to low concentration of bisulfite in the aqueous feed.

Table 6.1-15 Summary of Extractor Initial Operation	
Test EX-3 9-22-93	
Objective	Operate at low mixer speed. <u>Note:</u> This test was planned to be run with stripper test ST-4 but stripper was not fully operational at that time.
Operating Conditions	0.95 gpm lean organic flow. 1.20 gpm sodium bisulfite flow. A/O of 1.41. 20% mixer speed (60 RPM).
Results	Actual solvent and aqueous feed flows were 0.8 gpm and 1.7 gpm, respectively. High aqueous flow was required to achieve the target A/O ratio of 1.27. 15% loaded organic was reported with an aqueous feed concentration of 0.43% total sulfite. This test was interrupted by high level in the neutralization tank (T-108).

Table 6.1-16 Summary of Extractor Initial Operation	
Test EX-1B 9-28-93	
Objective	Operate at low organic and aqueous flows to the extractor, and medium mixer speed. Gather samples for analysis by the gas chromatograph. <u>Note:</u> This test was run with stripper test ST-4A
Operating Conditions	0.48 gpm lean organic flow. 0.60 gpm sodium bisulfite flow. A/O of 1.27. 33% mixer speed (210 RPM).
Results	Low rich organic loading (5%) was reported with an aqueous feed concentration of 0.27 M total sulfite. Very low regenerated organic loadings were reported: 3% stripped.

Table 6.1-17

Summary of Extractor Initial Operation

Test EX-5 and -5A 9-30-93	
Objective	Test reduced organic flow impact - compare performance of tests EX-5 and EX-5A with the aqueous feed rate remaining the same for both tests.
Operating Conditions	<u>EX-5:</u> 0.67 gpm lean organic flow. 0.88 gpm sodium bisulfite flow. A/O of 1.27. 100% mixer speed (60 RPM). <u>EX-5A:</u> 0.35 gpm lean organic flow. 0.88 gpm sodium bisulfite flow. A/O of 2.43. 100% mixer speed (60 RPM).
Results	While the organic loading doubled with increased A/O, the organic loadings reported were very low; 2.0% during EX-5 and 4.0% during EX-5A.

Table 6.1-18

Summary of Extractor Initial Operation

Test EX-1C 10-11-93

Objective	Repeat baseline condition. <u>Note:</u> This test was run with stripper test ST-1B.
Operating Conditions	0.95 gpm lean organic flow. 1.20 gpm sodium bisulfite flow. A/O of 1.27. 100% mixer speed (360 RPM).
Results	<p>The target organic flow to the Extractor (Q-105) of 0.95 gpm was not achieved during this test. The test was run at 0.75 gpm organic flow to the extractor due to pump capacity limitations on the organic and aqueous overflow streams.</p> <p>Rich organic loading decreased during the test from 27% to about 2%. Bisulfite feed concentration remained at 1.5 M. This may have been due to the inadvertent reduction in mixer speed during the test.</p> <p>Mixer speed was inadvertently turned down during the test</p>

Table 6.1-19 Summary of Extractor Initial Operation	
Test EX-2A 10-14-93	
Objective	Test the effect of aqueous recycle rate on extraction.
Operating Conditions	0.95 gpm lean organic flow. 1.20 gpm sodium bisulfite flow. A/O of 1.41. 100% mixer speed (360 RPM).
Results	The aqueous recycle was adjusted to achieve an aqueous to organic ratio in the Q-105 mix tank equal to 1.41. Achieved flow rates were 0.48 gpm for aqueous and 0.38 gpm for organic (hydraulic restrictions in the system continue to be investigated - as the organic loading increased, the viscosity of the organic also increased). The average organic loading was 30% with stripped organic loading observed as low as 3%. Stripper column samples at trays 10 and 20 showed organic loadings of 4% and 5%, respectively. (Note: Tray 10 is the tenth tray from the bottom of the stripper. Organic enters at the top.)

Stripper Subsystem

The baseline operating conditions established for the stripper (V-104) are:

- Rich organic feed rate 0.95 gpm
(1.0 MW equivalent)
- Steam rate 90 lb/hr
- Number of trays in column 30
- Wet stripping A/O ratio No water added

Table 6.1-20 Summary of Stripper Initial Operation	
Test ST-4A 9-28-93	
Objective	Operate stripper at low organic feed rate (0.5 MW equivalent) and 90 lb/hr steam rate.
Operating Conditions	0.48 gpm rich organic rate. 90 lb/hr steam rate. 30 trays.
Results	Rich organic feed loading was low; 5%. 3% stripped organic loading was achieved. Only 40% efficient.

Table 6.1-21 Summary of Stripper Initial Operation	
Test ST-1B 10-11-93	
Objective	Operate stripper at increased organic feed rate (1.0 MW equivalent) and increased L/G.
Operating Conditions	0.95 gpm rich organic rate. 90 lb/hr steam rate. 30 trays.
Results	Rich organic feed rate achieved was 0.75 gpm due to pump capacity limitations. Rich organic feed loading decreased during the test from 27% to about 2%. Stripping efficiency was 50% or less.

Table 6.1-22

Summary of Stripper Initial Operation

Test ST-3 10-14-93	
Objective	Operate stripper at increased organic feed rate (1.0 MW equivalent) and decreased L/G (8 lb steam/lb SO ₂ steam rate).
Operating Conditions	0.95 gpm rich organic rate. 180 lb/hr steam rate. 30 trays.
Results	Rich organic feed rate achieved was 0.38 gpm (pump capacity limits caused extractor overflow at higher flow rates). Rich organic feed loading during the test averaged 30%. Stripped organic loading also decreased during the test from 6.1% to 2.8%. Stripper column samples at trays 10 and 20 showed organic loadings of 4.0% and 5.0%, respectively while operating at 2.8% loaded on the outlet.

Beginning during the week of 10-25-93, the absorbers were operated with sodium sulfite feed (replacing sodium carbonate as reagent makeup) to prepare bisulfite solution. The existing solution in the absorber system was purged and replaced with 1.0 M sodium sulfite. This change was made in an attempt to reduce the oxidation rate of sulfite/bisulfite to sulfate. It was theorized that the carbonate-based solution took a longer time to convert to sodium bisulfite and therefore had a higher rate of oxidation and was inhibiting the production of concentrated (2 M) bisulfite solution. Data from the sodium sulfite system would be more easy to interpret than that from a sodium carbonate system because less chemical species would be present in the absorption liquor.

Mechanical Test

Our pilot plant test schedule called for the initial test program to be completed by mid-October 1993. In order to make sure that the pilot plant was indeed ready for process evaluation, Raycon proposed to conduct a Raycon / Raytheon joint eight day mechanical test program scheduled for November 3 to November 12, 1993. The test would be witnessed and evaluated by both Raycon and Raytheon personnel. On - stream time was proposed as the main evaluation criterion.

While the preparations for the mechanical test were underway on October 28, 1993, RE&C notified Raycon by telephone that the pilot plant had encountered a serious problem of duct corrosion. As a consequence, some part of the duct had been taken down for inspection and flue gas would not be available for the absorbers. To circumvent this duct problem, it was decided that the mechanical performance test would exclude the test of absorption section. The organic and sodium sulfite / sodium bisulfite solutions already in the system would be used to conduct the test. When the sodium bisulfite solution in the feed tank was exhausted, the used solution in the sulfite tank would be pumped back for reuse in the test.

Test results indicated that the mechanical performance of the systems in terms of on stream time actually achieved was low - generally below 50%. A principal reason for the poor mechanical performance was attributable to the poor pump performance. Magnetic pumps decoupled excessively and demanded repair. Another reason for poor mechanical performance was leaks. Some leaks occurred at valves and others at junctions between the steel pipe and PVC pipe. Still another reason for

poor mechanical performance was that a number of flow meters plugged too often and required cleaning.

Concerning pump failure, the small flow rates required for the 2MW equivalent pilot plant and the range of operating conditions to be tested resulted in the selection of magnetically-driven chemical feed pumps. These pumps offered a 10X range of operating flows, with the maximum design flow typically falling in the middle of the specification sheet flow range. However, even though the expected viscosities and operating temperatures were provided (and not exceeded during actual operation), many of these pumps failed to perform adequately. Drive magnets did not provide sufficient torque at process operating temperatures due to incorrect specification sheet data. Also, the pump vendors did not provide the correct materials of construction in some cases. Gear materials expanded, causing decoupling and pump shutdowns initially. After replacement of gears and magnets with upgraded materials, the pumps were able to function smoothly, but were still not able to meet the necessary flow rates in many cases.

While the mechanical performance during this test was not satisfactory, it was realized that better performance in terms of achieving a higher on - stream time was absolutely essential to conduct meaningful tests to evaluate the process. Process performance could not even be measured if the mechanical equipment could not provide stable operation with relatively high on-stream time.

Because the pilot plant was scheduled to shut down from November 19 to December 30 for duct repair, the opportunity to upgrade our pump performance as well as make other necessary alterations to attain a good quality plant with acceptable on-stream time was available. Accordingly, a mechanical up-grading plan was put in place. It was emphasized that all these up-grading efforts must be completed while duct repair work was taking place, thus assuring that alterations have no additional impact on the program schedule. After upgrading, the plant ran satisfactorily mechanically after the duct repair with an on-stream time estimated at 85% to 95%.

6.2 PARAMETRIC TESTING

In the preceding subsection, mechanical upgrading during duct repair was discussed. During the time when duct repair and plant mechanical upgrading were in progress, Raycon and Raytheon also reviewed the effectiveness of the on-site test program and discussed how it might be strengthened. The attributes of project personnel were carefully reviewed. After much discussion, it was generally concluded that Raycon's direct involvement in the on-site test program would be essential. Accordingly, the on-site team was reorganized with Raycon in charge especially with respect to day-to-day test planning and test data evaluation. Raycon would be assisted by Raytheon's test engineer who would execute the test plan and by Raytheon's site manager whose duty is to keep equipment mechanically proficient so that the test program could be carried out effectively.

Following installation of the new stainless steel duct on the pilot plant inlet flue gas stream, the facility was restarted. After eliminating typical startup problems (line pluggage, etc.), the test program moved on to the parametric testing of plant subsystems. New organic solvent was added to the system to eliminate any possible performance deterioration due to operation outside of specified limits during the initial phase of testing. One of the primary objectives of this phase of testing was to produce a 2M bisulfite solution as a feed stock to the extraction system. Having achieved this specified bisulfite concentration that is required to operate the system, other parametric tests could be studied to evaluate their impacts on equipment component performance as well as integrated system performance.

Absorber Subsystem

The objectives for absorber subsystem parametric tests were to:

- Determine the sodium sulfite absorber feed concentration required to produce 2 M sodium bisulfite solution for feed to the extraction subsystem.
- Test the effect of one- and two-absorber operations on the production of 2 M sodium bisulfite solution.
- Test the effect of absorber L/G on the production of 2 M sodium bisulfite solution.

The baseline operating conditions established for the absorber parametric tests were:

• Total gas rate to venturi	3230 ACFM
• Gas rate to secondary absorber
(48% of total)	1550 ACFM
• Primary absorber circulation rate	110 gpm
L/G (gal/ 1000 ACFM)	34
• Secondary absorber circulation rate	53 gpm
L/G (gal/1000 ACFM)	34
• Primary absorber pH
(one absorber operation)	5.0
• Primary absorber pH
(two Absorber operation)	6.0
• Secondary absorber pH	5.0

Table 6.2-1

Summary of Absorber Parametric Test

Absorber Test No.1		1-27-94	
Objectives	Determine the rate of oxidation of sulfite to sulfate in the absorption process with one absorber during the transient absorption stage		
Operating Conditions	3230 ACFM venturi inlet flow (1MW equivalent) 0.8M total sulfite in the feed solution Absorber circulation at baseline conditions		
Results	<u>Time</u>	<u>Total Sulfite (ML)</u>	<u>pH</u>
	5:00pm	0.84	7.3
	7:40pm	1.19	4.9
	8:40pm	1.21	4.8
	9:15pm	1.05	4.8
	10:30pm	0.99	4.8

Table 6.2-2 Summary of Absorber Parametric Test				
Absorber Test No.2 1-28-94				
Objectives	Determine that rate of oxidation of sulfite to sulfate in the absorption process with one absorber during the transient absorption stage as well as the feed and bleed stage			
Operating conditions	Same conditions as Absorber Test No. 1			
Results	<u>Time</u>	<u>Total Sulfite (ML)</u>	<u>pH</u>	Note before flue gas admission
	10:30am	0.92	7.1	
	10:35am	0.94	6.8	
	10:40am	0.95	6.7	
	10:50am	1.02	6.4	
	11:05am	1.11	6.2	
	11:20am	1.18	6.0	
	11:35am	1.31	5.7	
	12:35am	1.44	5.0	
	1:35am	1.47	4.9	
	2:50am	1.38	4.9	
	3:30am	1.37	4.8	
	4:50am	1.32	5.0	
	5:30am	1.32	5.0	

Table 6.2-3

Summary of Absorber Parametric Test

Absorber Test No.3 2-3-94			
Objectives	Test the conversion of a more concentrated sodium sulfite solution (1.34M) to bisulfite solution at pH of 4.9, using one absorber		
Operating conditions	<p>3230 ACFM venturi inlet flow (1MW equivalent)</p> <p>One absorber operation</p> <p>Absorber circulating at base line conditions</p> <p>The test was conducted in two parts: Part 1, "Transient" and steady state and Part 2 "Feed/Bleed". In Part 1, feed solution was circulated in absorber for about 4 hours. In Part 2, continuous sulfite feed to the absorber was maintained at 0.83 gpm (approximately 1MW equivalent)</p>		
Results	<u>Time</u> Transient/steady state 9:23 am 9:28 9:38 9:48 10:08 10:28 10:58 11:28 11:43 2:08 12:38 1:08 1:28 1:58 Feed & Bleed 2:30 3:00 3:30 4:00 4:30 5:00 5:30	<u>Total Sulfite (M/L)</u> 1.35 -- 1.37 1.44 1.48 1.54 1.73 1.80 1.88 1.93 2.02 2.20 2.27 2.19 2.12 2.06 2.05 2.08 2.05 2.02 2.02	<u>pH</u> 7.6 -- 7.1 7.0 6.6 6.3 6.0 5.7 5.6 5.8 5.5 5.2 5.2 5.2 5.3 5.45 5.6 5.35 5.40 5.50 5.55

Table 6.2-4 Summary of Absorber Parametric Test			
Absorber Test No.4 2-4-94			
Objectives	Test the conversion of a less concentrated sodium sulfite feed solution (1.25 M) to bisulfite solution at pH of 4.9, using one absorber (V102), as was done in Absorber Test No. 3		
Operating conditions	3230 ACFM venturi inlet flow (1MW equivalent) One absorber operation Absorber circulating at base line conditions		
Results	<u>Time</u>	<u>Total Sulfite (M/L)</u>	<u>pH</u>
	Transient/steady state		
	11:14am		
	11:16	1.31	7.0
	11:26	1.34	6.9
	11:36	1.39	6.7
	11:56	1.42	6.4
	12:16pm	1.52	6.3
	12:46	1.67	6.1
	1:16	1.77	5.9
	1:46	1.82	5.7
	2:16	1.90	5.4
	2:45	1.85	5.3
	3:15	1.88	5.2
	Feed & Bleed		
	3:45	1.92	5.2
	4:05	1.80	5.4
	4:25	1.80	5.7
	4:45	1.60	6.0
	5:00	1.70	6.1
	7:50	1.88	5.5

Table 6.2-6		Summary of Absorber Parametric Test				
Absorber Test No.6 2-8-94						
Objectives		Operate using two absorbers				
Operating conditions		3230 ACFM venturi inlet flow (1MW equivalent) Two absorber operation 1.21 to 1.25 sodium sulfite feed solution Absorber circulation at baseline conditions The test was conducted in two parts as described for absorber Test No. 3 above				
Results		Primary Absorber			Secondary Absorber	
		<u>Time</u>	<u>Total Sulfite (M/L)</u>	<u>pH</u>	<u>Total Sulfite (M/L)</u>	<u>pH</u>
		Transient/steady state				
		10:56 am	1.23	7.3	1.23	6.8
		11:18	1.31	7.0	1.28	6.5
		11:38	1.33	6.8	1.38	6.4
		11:58	1.31	6.4	1.47	6.1
		12:58	1.36	6.3	1.76	5.6
		2:00	1.6	6.0	1.85	5.3
		3:00	1.7	5.5	2.02	5.2
		4:00	1.75	5.2	2.02	5.2
		Feed & Bleed				
		5:00	1.85	5.5	1.95	5.2
		5:30	1.65	5.4	1.85	5.2
		6:00	1.75	5.3	1.88	5.1
		7:00	1.80	5.4	1.85	5.8
		7:30	1.60	5.8	1.80	5.4

Table 6.2-7

Summary of Absorber Parametric Test

Absorber Test No.7 2-9-94

Objectives

Test the effect of L/G on the conversion of a 1.21M to 1.25M sulfite solution to a 2.00M bisulfite solution two absorber operation

Operating conditions

3230 ACFM venturi inlet flow (1MW equivalent)

Absorber solution was circulated at the following target rate:

- Primary 55 gpm
- Secondary 26 gpm

These circulation rates are about one half the rate used in previous tests and result in lower L/G of 17

Results

Primary Absorber

Secondary Absorber

<u>Time</u>	<u>Total Sulfite (M/L)</u>	<u>pH</u>	<u>Total Sulfite (M/L)</u>	<u>pH</u>
Transient/steady state				
9:00am	1.27	7.6	1.23	7.0
9:30	1.27	7.2	1.29	6.8
9:45	1.31	7.1	1.44	6.8
10:00	1.31	6.8	1.63	6.0
11:00	1.60	6.1	1.80	5.1
12:00	1.84	5.3	1.95	5.0
Feed & Bleed				
12:45	1.84	5.2	1.97	5.1
11:15	1.88	5.3	1.73	5.1
1:45	1.75	5.3	1.90	5.1
2:45	1.73	5.4	1.93	5.1
3:45	1.78	5.4	1.88	5.1
4:45	1.68	5.4	1.83	5.1
5:15	1.73	5.2	1.83	5.0

Extractor Subsystem

The objectives for extractor subsystem parametric test were to:

- Determine the effect of extractor mixer speed on extraction performance.
- Determine achievable rich organic loading.
- Observe sodium sulfite regeneration performance.
- Observe entrainment of aqueous fraction in rich organic and organic in regenerated sodium sulfite aqueous solution.

The baseline operating conditions established for the extractor parametric tests were:

• One stage of extraction	Q-105 mixer/settler
• Lean organic flow	0.95 gpm (1MW equivalent)
• Sodium bisulfite feed flow	1.20 gpm
• Aqueous/organic ratio	1.27
• Extractor recycle flow	0 gpm
• Mixer speed	100% (360 RPM)

Table 6.2-8

Summary of Extractor Parametric Test

Extractor Test No. 1 2-15-94	
Objective	Test the effect of mixer (Q-105) RPM on extraction performance; percent loading, percent entrainment and sulfite regeneration.
Operating Conditions	0.50 gpm lean organic flow. Sulfite loading in lean solvent 0.10% 0.64 gpm sodium bisulfite flow. Sodium bisulfite feed pH = 5.15 Total sulfite 1.83 A/O of 1.27. The test was run at three mixer speeds: - 60 RPM - 200 RPM - 360 RPM
Results	Low RPM (60): Rich organic loading 28% Aqueous entrainment in rich organic <1.0% Regenerated sulfite - pH 5.6 total sulfite 1.6 M bisulfite 1.1 M
	Medium RPM (200): Rich organic loading 36% Aqueous entrainment in rich organic 0.1% Regenerated sulfite - pH 5.9 total sulfite 1.5 M bisulfite 0.83 M
	High RPM (360): Rich organic loading 35% Aqueous entrainment in rich organic <0.1% Regenerated sulfite - pH 6.0 total sulfite 1.5 M bisulfite 0.79 M

Stripper Subsystem

The objectives for stripper subsystem parametric test were to:

- Observe the difference in stripping performance between dry and wet stripping.
- Determine the effect of liquid/gas ratio (L/G) in wet stripping operation by changing steam rate to the stripper column.
- Determine the effect of rich organic feed rate to the stripping column.

The baseline operating conditions established for the stripper parametric tests were:

- Rich organic feed rate 0.95 gpm
- Steam rate 90 lb/hr
- Number of trays 30
- Wet stripping A/O ratio 0

Table 6.2-9 .		Summary of Stripper Parametric Test																		
Stripper Test No. 1 2-15-94																				
Objective	Test the effect on L/G on dry stripping performance and percent loading of stripped organic.																			
Operating Conditions	This test was run at three steam rates: Low L/G 180 lb/hr Medium L/G 90 lb/hr High L/G 45 lb/hr 0.50 gpm rich organic rate. 30 trays. Wet stripping A/O = 0																			
Results	Rich and stripped organic loadings for each steam rate were: <table><tr><td></td><td><u>Rich</u></td><td><u>Stripped</u></td><td><u>Stripping Efficiency</u></td></tr><tr><td>Low L/G (180 lb/hr)</td><td>36%</td><td>3.9%</td><td>89.2%</td></tr><tr><td>Medium L/G (90 lb.hr)</td><td>41%</td><td>12%</td><td>70.7%</td></tr><tr><td>High L/G (45 lb/hr)</td><td>37%</td><td>14%</td><td>62.2%</td></tr></table> At the low L/G feed rate, stripper performance was acceptable.					<u>Rich</u>	<u>Stripped</u>	<u>Stripping Efficiency</u>	Low L/G (180 lb/hr)	36%	3.9%	89.2%	Medium L/G (90 lb.hr)	41%	12%	70.7%	High L/G (45 lb/hr)	37%	14%	62.2%
	<u>Rich</u>	<u>Stripped</u>	<u>Stripping Efficiency</u>																	
Low L/G (180 lb/hr)	36%	3.9%	89.2%																	
Medium L/G (90 lb.hr)	41%	12%	70.7%																	
High L/G (45 lb/hr)	37%	14%	62.2%																	

Table 6.2-10

Summary of Stripper Parametric Test

Stripper Test No. 2 2-16-94

Objective	Test the effect of L/G on wet stripping performance and percent loading of stripped organic. The aqueous/organic ratio tested was 0.5.																						
Operating Conditions	<p>This test was run at four steam rates:</p> <table><tr><td>Low L/G</td><td>180 lb/hr</td></tr><tr><td>Medium low L/G</td><td>135 lb/hr</td></tr><tr><td>Medium high L/G</td><td>90 lb/hr</td></tr><tr><td>High L/G</td><td>45 lb/hr</td></tr></table> <p>0.50 gpm rich organic rate (0.5 MW). 30 trays. Wet stripping A/O = 0.5 (0.25 gpm water added)</p>			Low L/G	180 lb/hr	Medium low L/G	135 lb/hr	Medium high L/G	90 lb/hr	High L/G	45 lb/hr												
Low L/G	180 lb/hr																						
Medium low L/G	135 lb/hr																						
Medium high L/G	90 lb/hr																						
High L/G	45 lb/hr																						
Results	<p>Rich and Stripped organic loadings for each steam rate were:</p> <table><tr><td></td><td><u>Rich</u></td><td><u>Stripped</u></td><td><u>Stripping Efficiency</u></td></tr><tr><td>Low L/G (180 lb/hr)</td><td>38%</td><td>6.5%</td><td>82.9%</td></tr><tr><td>Medium low L/G (135 lb/hr)</td><td>41%</td><td>6.6%</td><td>83.9%</td></tr><tr><td>Medium high L/G (90 lb/hr)</td><td>36%</td><td>11%</td><td>69.4%</td></tr><tr><td>High L/G (45 lb/hr)</td><td>37%</td><td>19%</td><td>48.7%</td></tr></table> <p>Once again, performance was acceptable at the Low and Medium Low conditions.</p>				<u>Rich</u>	<u>Stripped</u>	<u>Stripping Efficiency</u>	Low L/G (180 lb/hr)	38%	6.5%	82.9%	Medium low L/G (135 lb/hr)	41%	6.6%	83.9%	Medium high L/G (90 lb/hr)	36%	11%	69.4%	High L/G (45 lb/hr)	37%	19%	48.7%
	<u>Rich</u>	<u>Stripped</u>	<u>Stripping Efficiency</u>																				
Low L/G (180 lb/hr)	38%	6.5%	82.9%																				
Medium low L/G (135 lb/hr)	41%	6.6%	83.9%																				
Medium high L/G (90 lb/hr)	36%	11%	69.4%																				
High L/G (45 lb/hr)	37%	19%	48.7%																				

Table 6.2-11

Summary of Stripper Parametric Test

Stripper Test No. 3 2-17-94

Objective	Test the effect on L/G on wet stripping performance and percent loading of stripped organic. The aqueous/organic ratio tested was 0.75.																										
Operating Conditions	<p>This test was run at four steam rates with the low L/G repeated:</p> <table><tr><td>Low L/G</td><td>180 lb/hr</td></tr><tr><td>Low L/G (Repeat)</td><td>180 lb/hr</td></tr><tr><td>Medium low L/G</td><td>135 lb/hr</td></tr><tr><td>Medium high L/G</td><td>90 lb/hr</td></tr><tr><td>High L/G</td><td>45 lb/hr</td></tr></table> <p>0.50 gpm rich organic rate.</p> <p>30 trays.</p> <p>Wet stripping A/O = 0.75 (0.38 gpm water added)</p>			Low L/G	180 lb/hr	Low L/G (Repeat)	180 lb/hr	Medium low L/G	135 lb/hr	Medium high L/G	90 lb/hr	High L/G	45 lb/hr														
Low L/G	180 lb/hr																										
Low L/G (Repeat)	180 lb/hr																										
Medium low L/G	135 lb/hr																										
Medium high L/G	90 lb/hr																										
High L/G	45 lb/hr																										
Results	<p>Rich and stripped organic loadings for each steam rate were:</p> <table><tr><td></td><td><u>Rich</u></td><td><u>Stripped</u></td><td><u>Stripping Efficiency</u></td></tr><tr><td>Low L/G (180 lb/hr)</td><td>39%</td><td>17%</td><td>56.4%</td></tr><tr><td>Low L/G (180 lb/hr - repeat)</td><td>36%</td><td>22%</td><td>38.9%</td></tr><tr><td>Medium low L/G (135 lb/hr)</td><td>44%</td><td>33%</td><td>25.0%</td></tr><tr><td>Medium high L/G (90 lb/hr)</td><td>40%</td><td>29%</td><td>27.5%</td></tr><tr><td>High L/G (45 lb/hr)</td><td>34%</td><td>33%</td><td>2.9%</td></tr></table> <p>Performance was unacceptable at all conditions.</p>				<u>Rich</u>	<u>Stripped</u>	<u>Stripping Efficiency</u>	Low L/G (180 lb/hr)	39%	17%	56.4%	Low L/G (180 lb/hr - repeat)	36%	22%	38.9%	Medium low L/G (135 lb/hr)	44%	33%	25.0%	Medium high L/G (90 lb/hr)	40%	29%	27.5%	High L/G (45 lb/hr)	34%	33%	2.9%
	<u>Rich</u>	<u>Stripped</u>	<u>Stripping Efficiency</u>																								
Low L/G (180 lb/hr)	39%	17%	56.4%																								
Low L/G (180 lb/hr - repeat)	36%	22%	38.9%																								
Medium low L/G (135 lb/hr)	44%	33%	25.0%																								
Medium high L/G (90 lb/hr)	40%	29%	27.5%																								
High L/G (45 lb/hr)	34%	33%	2.9%																								

Table 6.2-12		Summary of Stripper Parametric Test																	
Stripper Test No. 5 3-10-94																			
Objective		Testing the effect on L/G on wet stripping performance and percent loading of stripped organic. The aqueous/organic ratio tested was 0.25.																	
Operating Conditions		This test was run at three steam rates: High L/G 45 lb/hr Medium L/G 115 lb/hr Low L/G 200 lb/hr 0.7 gpm rich organic rate (0.7MW). 30 trays. Wet stripping A/O = 0.25 (0.18 gpm of water added)																	
Results		Rich and stripped organic loadings for each steam rate were: <table><tr><td></td><td><u>Rich</u></td><td><u>Stripped</u></td><td><u>Stripping Efficiency</u></td></tr><tr><td>High L/G (45 lb/hr)</td><td>44%</td><td>24%</td><td>45.5%</td></tr><tr><td>Medium L/G (115 lb/hr)</td><td>41%</td><td>14%</td><td>65.9%</td></tr><tr><td>Low L/G (200 lb/hr)</td><td>38%</td><td>5%</td><td>86.8%</td></tr></table> Performance was acceptable only at the Low L/G level.			<u>Rich</u>	<u>Stripped</u>	<u>Stripping Efficiency</u>	High L/G (45 lb/hr)	44%	24%	45.5%	Medium L/G (115 lb/hr)	41%	14%	65.9%	Low L/G (200 lb/hr)	38%	5%	86.8%
	<u>Rich</u>	<u>Stripped</u>	<u>Stripping Efficiency</u>																
High L/G (45 lb/hr)	44%	24%	45.5%																
Medium L/G (115 lb/hr)	41%	14%	65.9%																
Low L/G (200 lb/hr)	38%	5%	86.8%																

Crystallizer Subsystem

The objectives for crystallizer subsystem parametric tests were to:

- Determine the reduction on total sulfite concentration between the filter feed and filtrate solutions.
- Measure the quantity of sulfate crystals produced from a filtration run.
- Demonstrate steady state crystallizer and vacuum filter operation with continuous bisulfite feed to the crystallizer circulation tank.

The baseline operating conditions established for the crystallizer parametric tests were:

• Circulation tank feed rate	0.57 gpm
• Vacuum filter feed rate	0.57 gpm
• crystallizer temperature	41°F
• Vacuum pump receiver pressure	-2.0 psig

Crystallizer Tests No. 1 and 2 were conducted on 2-3-94 and 2-4-94 to determine change in total sulfite in the filtration step between the crystallizer bisulfite feed and filtrate solutions. It is suspected that the total sulfite decreases due to sulfite oxidation.

The following table summarizes the results of Crystallizer Tests 1 and 2:

Table 6.2-13		Summary of Crystallizer Test	
Crystallizer Tests No. 1 and 2		2-3-94 and 2-4-94	
Objective	Determine reduction in total Sulfite concentration. Measure Sulfate crystal produced. Demonstrate vacuum filter operation.		
Operating Conditions		<u>Test 1</u>	<u>Test 2</u>
	Bisulfite feed rate to pan filter	0.62	0.63
	Filter feed temperature (°F)	41.00	42.00
	Sulfate crystal produced (lb)	68.00	30.00
	Filter feed total sulfite (M)	1.74	1.72
Results		<u>Test 1</u>	<u>Test 2</u>
	Filtrate total sulfite (M)	1.46	1.58
	Change in total sulfite (ΔM)	-0.28	-0.14
	Note: The total sulfite concentration are average of several samples taken during the test		

Crystallizer Test No. 3 was conducted on 2-18-94 in two parts: Part 3A and Part 3B. Test No. 3A was run in normal batch mode. The crystallizer circulating tank (T-115) was filled to a starting level, chilled and then fed to the pan filter for a period of time. Samples of the circulating tank contents and filtrate were taken at the beginning and end of the test.

Test No. 3B was run to investigate the feasibility of operating the filter system with a constant feed to the circulating tank and pan filter while maintaining system temperature at about 41°F. This test was run for about 5 hours with little difficulty. Temperature held in the range 41°F to 48°F.

Table 6.2-14

Crystallizer Test Results

Crystallizer tests No. 3 and 4

2-18-94

Objective	Determine reduction in total sulfite concentration. Measure sulfate crystal produced. Demonstrate vacuum filter operation.		
Operating Conditions		<u>Test 3</u>	<u>Test 4</u>
	Bisulfite feed rate to pan filter (gpm)	0.51	1.0
	Filter feed temperature (°F)	43	41.48
	Sulfate crystal produced (lb)	211	457
Results	Time	<u>Test 3</u>	<u>Test 4</u>
	7:30pm Total sulfite before filtration		1.85
	Total sulfite after filtration		1.50
	8:00pm Total sulfite before filtration	1.73	
	Total sulfite after filtration	1.18	
	8:00pm Total sulfite before filtration		1.65
	Total sulfite after filtration		1.65
	8:30pm Total sulfite before filtration		1.73
	Total sulfite before filtration		1.60
	8:45pm Total sulfite before filtration	1.75	
	Total sulfite after filtration	1.73	

6.3 INTEGRATED TESTING

Integrated testing covers the period of operation between February 24 through March 17, 1994. During this period, the pilot plant subsystems operated continuously, with several periods of maintenance down time. The process operated in an integrated manner and process conditions were controlled as close to the target conditions as possible. With the exception of the last several days, the steam stripping operated relative satisfactorily, i.e. it could strip the organic down to less than 10% in the residual organic loading -- a condition that must be met to prevent organic solvent from losing its extraction capacity in long-term integrated operation. Because the steam stripper performance would unfortunately soon be found to degenerate after this testing period, the test results achieved during this period became very important to the entire test program.

During this period, it has been demonstrated that:

- Organic remained stable
- Phase inversion was controlled
- No interfacial crud was observed

These are three major conditions important to the demonstration of the process operability.

A number of "parametric tests" were also conducted during this period. Results attained for these parametric tests were discussed in Section 6.2.

Data Summary

The day-by-day process operating conditions and laboratory analyses for the three major process steps during the integrated test period are summarized on a daily basis in Table 6.3-1. The absorbers were operated as required to maintain sufficient inventory of sodium bisulfite solution for extraction subsystem operation. The extraction subsystem downstream equipment operated continuously for most of this period.

Note: The plant was down for maintenance on 2-28 and 3-4, -5, -14, -15 & -16 due to equipment failures, specifically multiple pumps off-line.

Table 6.3-1 Daily Operating Data Summary
FEBRUARY 24 – MARCH 17, 1994 TEST PERIOD

SYSTEM		2-24		2-25				2-26	
		9:00A	4:00P	6:30A	10:45A	12:10P	1:45P	2:00A	10:30A
ABSORPTION - GAS									
1. Bypass Gas (2)	a) Rate - ACFM, FE-0102	1130	1103	1119	1089	1068	1026	1004	1036
2. Venturi Inlet Gas (3)	a) Rate - ACFM, FE-0103	3502	3340	3954	4021	3962	4240	4552	4666
Sample Point G1	b) SO ₂ - ppm								
	c) O ₂ - %								
3. Venturi Outlet Gas (4)	a) SO ₂								
Sample Point G2	b) % O ₂								
4. Primary Absorber Outlet Gas (5)	a) SO ₂								
Sample Point G3	b) % O ₂								
5. Secondary Absorber Outlet Gas (6A)	a) Rate - ACFM, FT-0106A	1321	1322	1338	1349	1345	1328	1340	1324
ABSORPTION - LIQUID									
6. Sulfox Absorber Feed (30)	a) Rate - GPM, FE-1030	21%	30%	1.14	1.1	1.06	1.06	0.98	1.36
7. Primary Absorber Bleed (9A)	a) Rate - GPM, FT-0109C	0-5	0-5	0-6	0-6	0-6	0-6	0-6	0-6
8. Secondary Absorber Bleed (20)	a) Rate - GPM, FT-0120	0	0	0-6	0-6	0-6	0-6	0-6	0-6
EXTRACTION									
9. Lean Organic Feed to M/S (44 = 35 = 71)	a) Rate - GPM, FI-0571C	0.65	0.7	0.6	0.7	0.7	0.75	0.9	0.83
Sample Point S8	b) Temp. - F, TI-0935	83.9	81.7	77.3	81	81.1	80.3	79.4	76.7
10 Chilled Blueite Feed to M/S (27)	a) Rate - GPM, FT-0526	0.83	0.89	0.86	0.89	0.89	0.95	1.15	1.13
Sample Point A16	b) Temp. - F, TI-0527	72	76	69	65	64	70	74	70
11 Aqueous/Organic Volumetric Ratio	Measured								
	Calculated	1.28	1.27	1.43	1.27	1.27	1.27	1.28	1.36
12 Settling Time	Measured								
	Calculated	24	22.3	26	22.3	22.3	20.8	17.3	18.8
STRIPPER									
13 Condensate Feed from NiMo (A)	a) Rate - GPM, FI-2098	0.15	0.18	0	0.18	0.18	0.19	0.23	0.23
14 Stripper Steam Feed (67)	a) Rate - k/hr, FE-0667	142	154	140	130	139	140	140	140
	b) Press. - psi, PI-0667	0	0	0	0	0	0	0	0
15 Rich Organic Feed (51)	b) Temp. - F, TI-0651	168	174	168	174	170	174	172	171
16 Stripper Pressure	psi								

	2-27		3-1				3-2				3-3			3-6			
	2:00A	2:30P	4:15P	9:00P	11:30P	11:45P	8:30A	10:00A	4:00P	2:00A	10:15A	1:15P	3:30P	3:00A	2:30P	4:00P	8:00P
30	947	921	1305	1354	45	23	2787	2654	2775	DOWN	1361	1244	1354	1384	1306		0
19	4741	4719	3745	3487	2747	1944	3936	3732	3711	DOWN	2909	3195	2747	1630	2124		1554
19	1350	1255					1701	1639	1632	DOWN	1649	1662	1666	1775	1671		1268
27	1.1	1.09	0.86	1.04	0.87	0.93					0.8	0.69	0.68	0.55	0.58		0.88
6	0-6	0-6	0-6	0-6	0-6	0-6	0-6	0-6	0-6	0-6	0-6	0-6	0-6	0-6	0-6	0-6	0-6
6	0-6	0-6	0-6	0-6	0-6	0-6	0-6	0-6	0-6	0-6	0-6	0-6	0-6	0-6	0-6	0-6	0-6
4	0.9	0.9	0.5	0.6	0.6	0.6	0.6	0.6	0.6	0.5	0.5	0.5	0.5	0.6	0.55	0.5	0.5
5.7	76.5	77.8	79	122.5	117.4	118	115	110	106	111	98.9	92.3	105.1	75.7	82.9		87.4
02	1.13	1.11	0.73	0.79	0.8	0.85	0.75	0.75	0.76	0.55	0.64	0.64	0.64	0.77	0.62		0.62
70	72	72	76	85	77	74	70	82	64	68	72	75	75	82	75	88	95
28	1.26	1.23	1.46	1.32	1.33	1.42	1.25	1.25	1.27	1.1	1.28	1.28	1.28	1.28	1.13		1.24
9.5	17.3	17.3	31.2	26	26	26	26	26	26	31.2	31.2	31.2	31.2	26	28.3	31.2	31.2
0.2	0.2	0.25	0.13	0.15	0.15	0.15	0.15	0.15	0.1	0.13	0.13	0.13	0.13	0.15	0.14	0.13	0.13
40		142	99	133	107	110	161	217	178	103	60	118	180	155	85		165
0		0	0	0	0	0	0	0	0	0	0	0	0	0	0		0
14	172	173	180	102	96	92	190	206	196	210	218	208	218	186	181	188	210

Table 6.3-1 Continued

Daily Operating Data Summary

FEBRUARY 24 - MARCH 17, 1994 TEST PERIOD

SYSTEM	ANALYSE	2-24		2-25				2-26		
		9:00A	4:00P	6:30A	10:45A	12:10P	1:45P	2:00A	10:30A	12:30P
ABSORPTION										
1 Sulfite Absorber Feed (30)	a) pH									
Sample Point A6	b) Total Sulfite moles/Liter									
	c) NaHSO3 - moles/Liter									
2 Chilled Bisulfite Feed to MS (27)	a) pH		4.6		4.7				4.8	
Sample Point A16	b) Total Sulfite moles/Liter		1.28		1.24				1.2	
	c) NaHSO3 - moles/Liter									
EXTRACTION										
3 Lean Organic Feed to MS (44 = 35 = 71)	a) % Organic Loading				0.56				0.81	
Sample Point S8	b) % Aqueous Entrainment				0.5				0.5	
4 Rich Organic Outlet from M/S (45)	a) % Organic Loading	40	34	41.0		33.1		38.0		
Sample Point S2	b) % Aqueous Entrainment	0	0.1	0.5		0		0		
5 Regenerated Sulfite to Centrifuge (47)	a) pH		6.4		6.4				6.4	
Sample Point A4	b) Total Sulfite moles/Liter		0.86		0.83				0.83	
	c) NaHSO3 - moles/Liter									
STRIPPING										
6 Stripped Lean Solvent (60)	a) % Organic Loading	4.2	5.67	4.9		6		9.7		
Sample Point S5	b) % Aqueous Entrainment	1	0.9	1.5		2		1		

2-27	3-1				3-2				3-3			3-6			
2:00A 2:30P	4:15P	9:00P	11:30P	11:45P	8:30A	10:00A	4:00P	2:00A	10:15A	1:15P	3:30P	3:00A	2:30P	4:00P	8:00P
4.65 1.18					4.65 1.03				4.5 0.97			4.9 1.58			
					1.05 0.4				0.49 1					3.48 0.8	
35.1 <0.1	32.3 <0.1		28.3 0.1		32.3 0		20.2 0.7	20.2 0	20.2 0.5	18.2 1	17.4 0	36.5 0.5	36.1 0.4	31.6 0.4	
					6.2 0.73				6.3 0.67					6.1 1.26	
8.87 0.2	7.74 1.3		4.29 0.2		2.1 1		2.67 0.3	2.6 0.3	2.2 0.5	2.75 1	2.75 0.5	6.48 0.5	6.72 8	5.02 0.7	

Daily Operating Data Summary

FEBRUARY 24 - MARCH 17, 1994 TEST PERIOD

[illegible]

5:00P 7:30P		3-9 11:30A 5:00P		3-10 6:00A 2:00P 4:00P 5:00P				3-11 9:00A 10:00A 11:00A			3-13 8:00P 9:15P 10:30P			3-17 1:30P 3:45P	
1189	1201	1102	1106	1232 BYPASS BYPASS BYPASS				7590	7994	7901	7855	7768	7681	BYPASSBYPASS	
4372	4491	4700	4691	3664				3189	3252	3560	3495	3856	4217		
1327	1322	1345	1349	1333 BYPASS BYPASS BYPASS				12208	12357	12379	11	12	13	BYPASSBYPASS	
1.52	1.97	1.3	0.82	1.17 BYPASS BYPASS BYPASS				1.57	1.58	1.54	1.1	1	1.08	BYPASSBYPASS	
0-6	0-6	0-6	0-6	0-6BYPASS BYPASS BYPASS				0-6	0-6	0-6	0-6	0-6	0-6	BYPASSBYPASS	
0-6	0-6	0-6	0-6	0-6BYPASS BYPASS BYPASS				0-6	0-6	0-6	0-6	0-6	0-6	BYPASSBYPASS	
1.2	1.2	1	1	0.6	0.7	0.7	0.7	1	1	1	1	1	1	0.5	0.5
77.2	75.7	71.6	71.9	67.4	65.9	65.9	67.4	60	70.1	68.7	120.8	108	95.8	72.8	70.9
1.34	1.25	1.27	1.28	0.84	0.89	0.89	1.02	1.4	1.4	1.41	1.36	1	0.94	0.64	0.63
102	102	102	99	82	94	92	92	96	98	96	97	98	96	77	80
1.12	1.04	1.27	1.28	1.4	1.27	1.27	1.46	1.4	1.4	1.41	1.36	1	0.94	1.28	1.26
13	13	15.6	15.6	26	22.3	22.3	22.3	15.6	15.6	15.6	15.6	15.6	15.6	31.2	31.2
0.3	0.3	0.25	0.25	0.05	0.18	0.18	0.18	0.27	0.27	0.27	0.25	0.25	0.25	0.12	0.12
		28.3	23.8	52.38	0	116	114	127	87	126	0	53	105	98.8	175
		0	0	0	0	0	0	0	0	0	0	0	0	0	0
190	192	166	186	204	204	188	206	186	186	190	192	188	190	211	214

Table 6.3-1 Continued

Daily Operating Data Summary

FEBRUARY 24 - MARCH 17, 1994 TEST PERIOD

SYSTEM	ANALYSIS	3-7						3-		
		9:30A	4:00P	9:00P	12:00A	2:00A	5:00A	6:45A	9:30A	4:30P
ABSORPTION										
1 Sulfite Absorber Feed (30)	a) pH								6.15	
Sample Point A6	b) Total Sulfite moles/Liter								1.21	
	c) NaHSO3 - moles/Liter									
2 Chilled Bisulfite Feed to M/S (27)	a) pH									
Sample Point A16	b) Total Sulfite moles/Liter									
	c) NaHSO3 - moles/Liter									
EXTRACTION										
3 Lean Organic Feed to M/S (44 + 35 = 71)	a) % Organic Loading									
Sample Point S8	b) % Aqueous Entrainment									
4 Rich Organic Outlet from M/S (45)	a) % Organic Loading			7.2				36.1		
Sample Point S2	b) % Aqueous Entrainment			0.2				0.7		
5 Regenerated Sulfite to Centrifuge (47)	a) pH			6.1						
Sample Point A4	b) Total Sulfite moles/Liter			1.25						
	c) NaHSO3 - moles/Liter									
STRIPPING										
6 Stripped Lean Solvent (60)	a) % Organic Loading			5.35	15.5	7.29	5.18			
Sample Point S5	b) % Aqueous Entrainment			0.8	0.5	0.3	0.2			26.

5:00P 7:30P	16.69	1	3.8	33.7	5.1	23.5	11.5	0.8	1.1	5
	1.46	6.2	1.63	5.6	1.56					
	41.3	1	0.8	51.4	7.6	43.7	7.7	41.3	2.3	38.0
			1.5	12.6	1					
	1.95	5.2	1.85	4.8						
	6.3	1.33	1.61	1.60	5.9	1.390				
3-9	11:30A 5:00P									
3-10	6:00A 2:00P 4:00P 5:00P									
3-11	9:00A 10:00A 11:00A									
3-13	8:00P 9:15P 10:30P									
3-17	1:30P 3:45P									

The flow of lean organic to the extraction subsystem was controlled between 0.5 and 1.0 gpm, as required for parametric tests. Rich organic loadings as high as 51% were achieved with sodium bisulfite concentrations in the range of 1.85 to 1.95 M. However, at that time the lean organic feed to the extraction system loading was also high, between 7.4% and 12.6%. This occurred during a period when high exit loadings were experienced in the solvent regeneration.

When organic loadings in the range of 35% to 40% were achieved with sodium bisulfite concentrations in the range of 1.2 M, 0.6% lean organic loading were measured.

Stripped organic loadings remained below 10% for most of the integrated test. The lowest stripped organic loading achieved was 2.1% (3-2-94) at the following conditions:

- Rich organic feed loading 32%
- Rich organic feed rate 0.6 gpm
- Steam rate to Stripper 160 lb/hr

Stripped organic loadings in the range of 13% to 34% were observed for a period of three days which coincided with low stripper steam rates. The high rich organic loading of 51% was produced at the same time.

6.4 STEAM STRIPPER TEST

On March 18, the pilot plant started a period of steady-state operation with a planned duration of one month. The objective of the steady-state operation was to reaffirm the results obtained during the integrated test period concerning the process operability. Unfortunately, the stripper steel column packing leaked, preventing the test from being completed as planned. Column repacking was not successful. To circumvent the leak problem, a 4 inch diameter glass column was ordered to replace the steel column.

While a 2-inch glass column used in Phase I of our bench-scale testing yielded a 97% stripping efficiency, the stripping efficiency of the 4-inch glass column used in the pilot plant which has different design features was much lower. Test data suggested that the 4-inch column was not scaled up from the 2-inch column correctly. The tray design of two sections of the 3-section column was modified. Data gathered from the glass column with the modified tray design appeared to confirm that to strip gas off a high-viscosity liquid, turbulence introduced during the initial bubble formation is more effective in enhancing the mass transfer than the turbulence induced by high steam velocity.

Steady State Operation

The steady state test started on March 18, 1994. On March 16, the organic solution in the pilot plant system was replaced. The following process conditions were set for the duration of this test:

Table 6.4-1 Steady State Test Process Operating Conditions	
Gas rate (as needed for meeting bisulfite inventory requirements)	3,560 ACFM (1.1 MW equ.)
Organic rate to extractor (Q - 105)	1.1 gpm (1.1 MW equ.)
Aqueous/ Organic phase ratio in extractor	1.27
Trays used in stripper	30
Steam rate to stripper	150-180 lb/hr (total)
Aqueous/Organic phase ratio in stripper	0-0.25
Organic bleed	1% per cycle

The test was characterized by very low stripping efficiency. The rich organic loading remained in the range of 33% to 41% with several data points showing lower values.

On 3-28-94, operations were briefly interrupted because it was suspected that the stripper was flooding. The system was shut down to allow the stripper to drain. The stripper was then restarted without condensate addition and the stripper was operated in dry mode for the remainder of this test. The performance of the stripper did not improve after the tests were renewed.

Close inspection showed that the stripping data have the following characteristics:

- (1) The stripping by the pilot plant 10-inch steel column was much less efficient (13% - 25%) than stripping achieved by a 2-inch column in Phase 1 bench-scale test (97%).
- (2) The pilot plant stripper performance decreased with time. Two similar strippings conducted on different dates yielded different results (e.g. 2/15,94 residual loading 3.9%; 4/2, 94 residual loading 13.8%)

- (3) The daily stripping performances were inconsistent.

In searching for a cause of the low stripping efficiency, the process aspects of the stripping including (1) feed quality (2) feed preheat temperature (3) stripping temperature and (4) stripping pressure as well as the hydraulic aspects of stripping including (1) differential pressure across the column (2) variation of sump level height (3) liquid rate and (4) steam rate were examined in great detail. None of them appeared to be responsible to the poor column performance.

The best explanation appeared to be:

- (1) The packing between the tray and column wall was loose and leaking.
- (2) Once packing leakage started, steam occasionally passed through it causing the leakage to be increased. Thus, the performance degenerated with time.
- (3) Because performance was poor due to packing leakage, large amounts of steam were used to try to improve the stripping efficiency. The use of large amounts of steam caused partial flooding. Occasional dumping, etc., could then introduce observed inconsistencies.

On April 6, another set of stripping data was taken. These data are summarized in Table 6.4-2

Table 6.4-2 Stripping Test with Steel Column Before Repair			
Steam Rate (lb/hr)	Rich Organic Loading (%)	Stripped Organic Loading (%)	Stripping Efficiency (%)
180	33.2	16.2	51.2
150	27.4	23.5	14.2
100	33	33	0
150	30.8	27.5	10.7
180	29.2	24.5	16.1
200	31.6	14.6	53.8
180	32.4	18.2	43.8

The data showed that the 10-inch steel column performance had degenerated to such an extent that at a 100 lb/hr steam rate, the stripping performance was zero percent (0%). The data suggested that the column packing was leaking badly and that meaningful tests could not continue. On April 6, 1994, the pilot plant was shut down for column packing repair.

Stripper Repair

The column was disassembled and taken to the Quackenbuch warehouse in Buffalo NY for repair. A representative from the column tray manufacturer, Glitsch Inc., was present to supervise the repair.

As the column was disassembled, it was noted that the tray cartridges pulled out easily. Gaps of about 1/16th to 1/8th of an inch existed around practically every tray. It was recalled that at the time of original installation, the cartridges were tight and difficult to pull into place. Relevant dimensions, as measured are as follows:

Tower vessel inside diameter	9 7/8" to 10" (depends on orientation)
Tray outside diameter	9 5/8"

Therefore, the clearance between the trays and the tower vessel wall was about 1/8" to 3/16". Clearly, dimension wise, the original teflon packing, which was 1/8" in thickness, did not completely fill the gap. A 1/8" thick packing material, was used because Glitsch advised us that in tray packing, complete filling of the gaps is not necessary. Our experience now suggests that Glitsch's advice may not be valid.

While it is possible that complete gap fill may not be necessary in most of the industrial stripping applications, it is nevertheless important in our case where the liquid flow rate is extremely low.

Instead of reusing the pure teflon packing, fiberglass impregnated with teflon packing was used. Unfortunately, packing material thicker than 1/8" was not available. In order to fill the gap completely, the packing was lapped to cover the edge of the tray twice, giving a double thickness (see Figure 6.4-1b) compared to a single thickness with the previous packing (see Figure 6.4-1a). No gaps were observed when the tower was replaced. When cartridges were slid into the column, it was reasonably tight. The repaired tower was shipped back to the pilot plant on April 18, 1994.

Original
Figure 6.4-1a

Replacement
Figure 6.4-1b

Repaired Column Testing

During the week of 4-18-94, the stripper column was reinstalled and the pilot plant was started up on 4-21-94. During startup, the steam rate to the column was increased in increments over several hours to a maximum rate of 200 lb/hr.

A series of tests were performed on 4-22-94 to test the repaired stripper column performance at an organic feed rate of 1.1 gpm. The resulting stripping performance given in Table 6.4-3 did not show much improvement over previous tests prior to the column repair.

Table 6.4-3 Stripping Test of the Repaired Steel Column			
Steam Rate (lb/hr)	Rich Organic Loading (%)	Stripped Organic Loading (%)	Stripping Efficiency (%)
100	59.5	38.8	34.8
150	53.8	47.7	11.3
180	57.0	41.7	26.8

While the stripping efficiency had improved from 0% to 34.8% at a steam rate of 100lb/hr steam rate, stripping efficiency did not further improve when the steam rate was increased. Another sign which showed that the column did not perform well is that the aqueous solution coming down to the tower bottom was acidic -- a phenomenon which had not been observed before. The test was interrupted because Dunkirk Unit No. 4 went down on 4-22-94 to repair a tube leak, forcing the pilot plant to shutdown.

The plant was restarted on 4-26-94 and stripper performance was tested at a rich organic feed rate of 1.0 gpm and over a range steam rates.

Table 6.4-4 Stripping Test of the Repaired Steel Column			
Steam Rate (lb/hr)	Rich Organic Loading (%)	Stripped Organic Loading (%)	Stripping Efficiency (%)
50	39.3	36.9	6.18
75	39.7	35.6	10.20
100	42.5	30.8	27.63
150	39.3	36.9	6.18
180	37.3	35.6	4.35
200	38.1	32.0	15.90

On 4-27-94, a series of stripper tests were conducted to continue investigating poor stripping performance. Under wet stripping conditions ($A/O = .25$), at a rich organic rate of 1.0 gpm, the following stripping efficiency was attained.

Table 6.4-5 Stripping Test of the Repaired Steel Column			
Steam Rate (lb/hr)	Rich Organic Loading (%)	Stripped Organic Loading (%)	Stripping Efficiency (%)
100	46.1	35.6	22.8
100	45.3	35.2	22.3

Under dry stripping condition, the following results were observed at a rich organic feed rate of 1.0 gpm.

Table 6.4-6 Stripping Test of the Repaired Steel Column			
Steam Rate (lb/hr)	Rich Organic Loading (%)	Stripped Organic Loading (%)	Stripping Efficiency (%)
100	51.4	39.2	23.7
150	53.0	37.2	29.8
180	51.0	37.6	27.4

The rich organic flow was reduced to 0.5 gpm and the previous test was repeated with improved results.

Table 6.4-7 Stripping Test of the Repaired Steel Column			
Steam Rate (lb/hr)	Rich Organic Loading (%)	Stripped Organic Loading (%)	Stripping Efficiency (%)
100	52.2	36.0	31.0
150	50.6	30.3	40.1
180	50.2	27.0	46.2

Is the column still leadking? In discussing this problem with a consultant, it was pointed out that a new leakage was most probably occurring in the junctions between

the two packings, i.e., (a) packing between the tray and the column wall and, (b) packing between a baffle and the column wall.

As is shown in Fig. 6.4-2a, the tray we used has an orbital flow design.

Orbital Flow Tray Design

Packing Between Baffle and Column Wall

Figure 6.4-2a

Figure 6.4-2b

The two downcomers are located on the same side of the tray with a baffle between them. The baffle is welded to the tray forcing the liquid coming down from the upper tray to flow around the baffle to the downcomer on the other side. There is a packing seal between the baffle and the wall as shown by area shaded by cross lines. Note that at the bottom side of this packing, there is no positive seal. In fact, there is a small void which is filled by packing material between tray and the wall which projects out above the tray surface. In the repaired column, when the packing was laped to cover the edge of the tray twice, there was no packing material projected out above the tray to fill the void (Figure 6.4-2b). In most instances, this void is big enough to constitute a leak, permitting the liquid from the tray above to flow directly to the downcomer on the other side of the baffle and then to the tray which is directly below.

Thus the liquid effectively short-circuited all effective stripping area on the plate. Aqueous solution also appeared to be able to leak through this type of void, and readily rendering the aqueous in the sump acidic.

The performance of the repaired column was not sufficient to proceed with the steady state test. When Dunkirk Unit No. 4 came down for tube leaks on 4-27-94 the test was terminated.

Design and Installation of a 4-inch Glass Column

At this point of time, it was thought that the only problem was a new packing leak which now occurred at the junctions of two packings. This was a tough problem and was not easy to fix. This was especially true because Glitsch could not furnish us with a thicker and wider packing material other than those of standard 1/8" in thickness and 2" in width.

In searching for a solution, it was recalled that in the Phase I bench scale results extremely good stripping efficiency with a 2" diameter glass column was achieved. That column routinely yielded 97% stripping efficiency at about 3-4 lb steam/pound SO₂ removed. The glass column manufacturer, could only furnish a 4" diameter column. It could be delivered in about 4-5 weeks. Since a glass column would not leak and since it could reasonably be expected to work well based upon Phase I experience, a glass column was ordered on 4-29-94 to continue the test rather than try to fix the leaking column for a second time.

The organic liquid we were using had relatively high viscosity (110 cp at room temperature and 6.6 cp at 100°C at 50% loading). In the Phase I bench scale Demonstration, it was observed that vigorous liquid stirring was extremely important to effect mass transfer. For instance, in extraction, the counter-current liquid-liquor extractor would not work but mixer settlers which can be effectively stirred mechanically worked well. Since stripping is also a mass transfer process, in scaling up the glass column diameter from 2 to 4 inches, the basic scale-up criterion used was to achieve high turbulence through the use of high-velocity steam (i.e. high F value where $F = \rho \sqrt{v}$ with ρ = gas density and v = superficial velocity). The design specifications thus attained are presented in Table 6.4-8 and the tray layout in Figure 6.4-3.

Table 6.4-8

4" Glass Column Design Specification

	Pilot Plant 10" 1.0	Proposed Glass 4" 1.0
Gross Tower Area, SQ. FT (AG)	0.545	0.087
Hole Area, SQ. FT (AH)	0.0121	0.00426 (2)
Downcomer I.D., IN.	2	1
Downcomer Area, SQ. FT. (A _{bc})	0.0218	0.00545
Tray Spacing, IN.	18	8
Weir Height, IN.	1.5	0.75
Design Vapor:		(1)
Steam, LB/HR	180	38 (Max)
Stripped SO ₂ , LB/HR	46	5
Total Vapor, LB/HR (W _V)	226	43
Vapor Density, LB/CF (P _V)	0.06	0.06
W _V /AG	414.7	494
FG ($U_V \times \rho_V^{1/2}$)	0.47	0.56
FS ($U_H \times \rho_V^{1/2}$)	21.2	11.4
Dry Press Drop, in liq (ΔP_d)	2.6	1.0
Design Liquid (Amine) LB/HR	810	154
Liq. Density, Lb/CF (ρ_L)	48	48
GPM	2.1	0.4 (Max)
GPM/AG	3.85	4.59
GPM/FT. cc Tower Diam.	2.52	1.2
Downcomer velocity, FT/SEC	0.21	0.21
Liq. Crest Over Downcomer, IN.	0.3 1/2 Pipe	0.25
Liq. Height on tray, IN. (H _L)	1.8	1.0
Total Tray Press. Drop, In. Liq. ($\Delta P_d + H_L$)	4.4	2.0
Total Tower Press Drop, PS1	3.6 (30 Trays)	1.1 (20 Trays)
Downcomer buildup, IN. ($\Delta P_T + H_L$)	5.4	3.0
D.C. Buildup % of tray SA	30%	2.4 (Norm) 37.5 % (Max) 30% (Norm)
(3) Normal Steam Rate = 32 LB/HR (4) Based on 50 - 1/8" ϕ Holes(Tray)		

Figure 6.4-3
4-Inch Glass Column Tray Layout

Glass Column Testing

The pilot plant was shut down on 4-28-94 for approximately two weeks for fabrication, delivery and installation of the 4", 20-tray column. The glass column was received on 5-16-94. It consisted of three sections. The top section has seven trays, the middle section seven trays and the bottom section six trays. Installation of the glass column started on the next day. In spite of the fact that extreme care was exercised during the installation, the top section was broken. While a replacement section was ordered, the plant startup proceeded with the 13-tray glass column on 5-20-94. A sketch of the glass column that was installed is shown in Figure 6.4-4.

In contrast to high expectations, the performance of this 13-tray column was disappointing. For instance, the effect of rich organic feed rate on stripping performance was tested on 5-23-94. The results showed an average stripping efficiency of only 25%.

The glass column was found to be difficult to operate. Steady state could not be attained without the operators' very close attention, and the data obtained were not reliable unless steady state operating conditions were maintained. It was also observed that the 4" glass column would begin to flood at an organic rate of about 0.4 gpm and a steam rate of 50 to 55 lb/hr.

The replacement top section of the column was installed on 5-24-94. In this test, an attempt was made to run the stripper column at very steady conditions to show repeatability of the stripping results. The test conditions were:

Organic rate	0.4 gpm
Steam rate	40 lb/hr
Column top pressure	0.0 psig

The results are summarized in Table 6.4-9.

Figure 6.4.4

A Sketch of 4" Glass Column

Table 6.4-9 Stripping Test of 4-Inch Glass Column			
Time	Rich Organic Loading (%)	Stripped Organic Loading (%)	Stripping Efficiency (%)
6:30 am	44.1	32.4	26.5
9:30am	41.5	31.1	25.4
10:30am	44.5	32.4	27.2

The average stripping efficiency was 26.4% with satisfactory repeatability. The stripping efficiency attainable was not sufficient to proceed with the steady state integrated test. The test was discontinued when the Dunkirk Unit No. 4 boiler went down for tube repair on 5-26-94.

Glass Column Design Modification

With the glass column, the trays in the glass column are directly fused onto the column wall. However, the column stripping efficiency was so low i.e. 25% compared to the 97% achieved with a 2" column during the bench scale test. As will be discussed in Chapter 7, several factors can account for part of the difference, but not all of it. Another explanation might be that the loaded organic in the pilot plant was less strippable than those used previously. This possibility was eliminated after a strippability evaluation which showed that the loaded pilot plant stock was just as easy to strip as stock derived from non-pilot plant sources.

Close examination of the problem indicated that bad performance was most probably attributed to the wrong scale-up criterion which was used in the design of the 4" column. The data showed that to effectively strip a highly viscous liquid, it is not only the turbulence that is important but also how this turbulence is being imparted to the system that is very important. It appeared that turbulence introduced by high F value was not as effective as the turbulence created when bubbles were formed at the hole. The more numerous the holes, the more turbulence created and the more effective will be the stripping. Based upon this preliminary analysis, two sections of 7 tray columns with 131 0.05" holes on each tray was ordered to replace the top and middle sections of the installed column, which only had 50, 1/8" holes. It was believed that when these two column sections were replaced, the comparison between the data from the original and the modified columns would reveal whether the analyses which

suggested that numerous small holes were more important than high F-value had merit and should be used as a basis for column scale-up.

Modified Glass Column Testing

The two new column sections were delivered to the pilot plant site on 6-7-94 and installation and check out was completed by 6-8-94. The test was resumed on 6-8-94 and 6-9-94 with the target conditions shown in Table 6.4-10.

Table 6.4-10 Steady State Operating Conditions		
Operating Conditions	Ranges	Targets
Gas rate (as needed for making bisulfite) (ACFM)	3,560 (1.1 MW)	3,560
Organic rate to extractor (gpm) Note: Achieved rate will be determined during startup	0.4	0.4
Aqueous/Organic phase ratio in extractor	1.27 - 1.45	1.27
Trays used in stripper column	20	20
Steam rate to stripper (lb/hr) (FIC - 0667)	20 - 80	38
Aqueous/Organic ratio in stripping	0 - 0.25	0

Stripper performance is summarized in Table 6.4-11 for tests conducted on 6-9-94 under various operating conditions. The stripping efficiencies reported are an average of three data sets (rich and stripped organics) at each operating condition. Good repeatabilities were attained.

Table 6.4-11 Stripping Test of a 4" Glass Column with Modified Trays				
Organic Feed Rate (gpm)	Steam Rate lb/hr	Rich Organic Loading (%)	Stripped Organic Loading (%)	Stripping Efficiency (%)
0.4	40	26.6	16.4	37.9%
0.4	32	28.6	18.3	36.0%
0.4	48	27.8	17.3	37.6%
0.15	48	29.4	14.0	52.1%
0.23	48	27.7	14.3	48.5%

Once again, it was observed that the operator's close attention was required to operate the glass column properly. Test operation must be repeated to assume reliability.

Data on the concentration profile along the column height were taken at different steam rates on 6-13-94 and 6-14-94. The data are presented in Table 6.4-12.

Table 6.4-12 Concentration Profile Along Stripping Column Height					
Organics Flow gpm	Steam Rate lb/hr	% Loading			
		Rich Organics Feed	Organics on Tray 15	Organics on Tray 10	Stripped Organics
0.4	32	38.5	32.0	31.2	24.7
0.1	32	36.5	18.6	19.4	13.8
0.1	40	32.4	21.4	18.6	15.4
0.1	48	34.4	19.8	19.0	12.1
0.1	23	38.9	22.7	20.2	24.3

While the data in Table 6.4-11 show that the modified column performed better (36% - 52.1%) than the original column before modification (17.7% - 29.8%), the improvement was not quite sufficient for a long-term integrated test in which the stripped organics should have a residual loading of less than 10%. The data are nevertheless important because they embody interesting features which appeared to

confirm the contention that for the stripping of a viscous liquid, trays with small and numerous holes performed better than trays with large holes. This scale up criterion, will be further discussed in Section 7.3.

**TEST RESULTS ANALYSES
AND INTERPRETATION**

SECTION 7

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- (1) Oxidation During Absorption
- (2) Initial Na_2SO_3 Concentration Needed to Produce a 2M NaHSO_3 Solution
- (3) Two Stage Absorption

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- (1) Repaired Steel Column Performance

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- (1) Modified 4-Inch Column Performance
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- (3) mass Transfer in Conventional Low-Viscosity liquid stripping
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SECTION 7

TEST RESULTS ANALYSES AND INTERPRETATION

Data collected under parametric testing are presented in Section 6.2. This section will provide some analysis and interpretation of the data. While the data are not disputable, readers may or may not agree with the author's analyses and interpretation. Interpretation is never the less important. For instance, interpretation of the stripper data led us to a stripper scale-up criterion, and this criterion or the improved version of it is the starting point of any future development work of this process.

7.1 PARAMETRIC TESTING

Absorber Subsystem

Seven experiments were conducted. Only the primary absorbers were used in the first five experiments. The major variable studied in the first five experiments was the initial Na_2SO_3 concentration to ascertain what initial Na_2SO_3 concentration was required to attain a 2M total sulfite concentration in the bisulfide product solution. These experiments were followed by two additional experiments in which both primary and secondary absorbers were used. Among others, the effect of L/G on absorption performance was studied in these two experiments.

(1) Oxidation During Absorption

The absorption experiment may be separated into two phases:

- (a) A transient absorption phase during which the Na_2SO_3 solution in the absorber sump was circulated to absorb SO_2 from the flue gas. During the absorption, Na_2SO_3 was converted to NaHSO_3 . Meanwhile part of the Na_2SO_3 or NaHSO_3 was oxidized by the oxygen in the flue gas to form sulfate:

Only a minimal amount of NaHSO_4 will be present in the prevailing pH range

- (b) A feed and bleed phase -- after Na_2SO_3 was almost fully converted to NaHSO_3 , the feed solution was added at a predetermined rate and the product solution was bled at the same rate as that to be used in the extractor.

During absorption, one mole of Na_2SO_3 will convert to two moles of NaHSO_3 . Therefore the total sulfite as well as the NaHSO_3 content will progressively increase while the Na_2SO_3 content will progressively decrease. This will be reflected as an increase in total sulfite concentration as well as a decrease in pH. Without oxidation, a 1M Na_2SO_3 solution (total sulfite = 1M/L, pH = 10.4) will eventually be converted to a 2M NaHSO_3 solution (total sulfite = 2, pH = 3.8).

Sulfur loading is defined as the percentage of sulfite that has been converted to bisulfite. The relationship between the sulfur loading and fraction of total sulfite that is bisulfite (i.e. $\text{NaHSO}_3/\text{total sulfite}$) for an initial 1M Na_2SO_3 solution is shown in Table 7.1-1 and is plotted in Figure 7.1-1.

Table 7.1-1 Sulfur Loading vs. Total Sulfite				
Sulfur loading (%)	Na_2SO_3 (M/L)	NaHSO_3 (M/L)	Total Sulfite (M/L)	$\frac{\text{NaHSO}_3}{\text{Total Sulfite}}$
0	1	0	1	0
20	0.8	0.4	1.2	0.33
40	0.6	0.8	1.4	0.57
60	0.4	1.2	1.6	0.75
80	0.2	1.6	1.8	0.89
100	0	2.0	2.0	1

Figure 7.1-1

Sulfur Loading vs $\frac{\text{NaHSO}_3}{\text{Total Sulfite}}$ Ratio
Initial Na_2SO_3 Concentration = 1M

It can be shown that sulfur loading can be determined by pH value of the solution alone by the following relationship:

$$S = \frac{\left\{2 + H^+ - \frac{10^{-14}}{H^+}\right\} \left\{1 + \frac{1.02 \times 10^{-7}}{H^+} + \frac{H^+}{1.54 \times 10^{-2}}\right\}}{1 + \frac{2 \times 1.02 \times 10^{-7}}{H^+}} \quad (7.4)$$

Where S is the total sulfur loading and H^+ is hydrogenion concentration. This theoretical relationship given by Equation (7.4) is plotted in Figure 7.1-2 in dotted lines. This relationship was confirmed experimentally and the experimental curve is shown as the solid line on the same plot. From curves in Figures 7.1-1 and 7.1-2 a curve which gives the relationship between the original Na_2SO_3 concentration per unit total sulfite concentration of an absorption solution with a given pH can be generated. This is shown in Figure 7.1-3. Either by curves in Figures 7.1-1 and 7.1-2 or by the curve in Figure 7.1-3, the percent of Na_2SO_3 that had been oxidized to Na_2SO_4 in the absorption solution at any time during the absorption process can be computed. This is illustrated by the following example.

Figure 7.1-2 pH vs. Sulfur Loading
(based upon pilot plant data)

Figure 7.1-3
PH VS Correlation Factor
(based upon pilot plant data)

Example: If a sodium sulfite feed solution has a total sulfur loading of .92 and pH of 7.1, what is the original Na_2SO_3 concentration? After an absorption process of 1 hour and 5 minutes, the total sulfite of the solution was determined to be 1.11 and pH was measured to be 5.7. How much Na_2SO_3 had been oxidized to Na_2SO_4 ?

Solution: (1) Sodium bisulfite feed solution: Total sulfite = .92 (mmole/ml)
From Figure 7.1-3 pH = 7.1

$$\text{Correlation factor} = \frac{\text{Na}_2\text{SO}_3 \text{ originally present}}{\text{Total Sulfite measured}} = .91$$

$$\therefore \text{Na}_2\text{SO}_3 \text{ originally present} = .92 \times .91 = .84 \frac{\text{mmol}}{\text{ml}}$$

(2) After 1 hour and 5 minutes absorption

$$\text{Total sulfite} = 1.31 \frac{\text{mmol}}{\text{ml}}$$

$$\text{pH} = 5.7$$

From Figure 7.1-3

$$\text{Correlation factor} = .60$$

$$\text{Na}_2\text{SO}_3 \text{ still present} = .79 \frac{\text{mmol}}{\text{ml}}$$

$$\text{Na}_2\text{SO}_3 \text{ oxidized} = .84 - .79 = .05 \frac{\text{mmol}}{\text{ml}}$$

$$\% \text{ Na}_2\text{SO}_3 \text{ oxidized} = \frac{.05}{.84} = 5.95\%$$

For one absorber absorption, the absorption data are presented in Tables 6-2.1 to 6-2.5. From these data points, the percentage of Na_2SO_3 that were oxidized to sulfate at any time after the commencement of absorption are computed as shown by the example. For Test No. 2, these computations are summarized in Table 7.1-2. In Figure 7.1-4a, the measured total sulfite and pH values of the absorption solution samples taken over time are plotted. In Figure 7.1-4b the percentages of sulfite that were oxidized to sulfate are plotted. The slope of that curve shows that the rate of oxidation during the transient absorption period is approximately 1.88% per hour. During feed and bleed, there was some initial oxidation at about the same rate as that attained during

the transient period, but it has leveled off when a steady state was reached after 5 hours. The total oxidation during these 5 hours was 9.4%.

The composition of flue gas used during that absorption process is summarized in Table 7.1-3. The SO₂ concentration was relatively low, averaging 388.5 ppm. The oxygen content was about 7.25%.

TABLE 7.1-2 Oxidation Rate Estimate For Absorption Test No. 2							
(1) Time	Data Measured			Computation			
	(2) Measured Total Sulfite (mmo1/ml)	(3) pH	(4) Correlation Factor Fig 7.1-3	(5) Na ₂ SO ₃ originally present	(6) Na ₂ SO ₃ still present	(7) ⁺ Na ₂ SO ₃ oxidized	(8) ⁺⁺ % Na ₂ SO ₃ oxidized
10:30AM 0min	.92	7.1	.91	.84	.84	.00	0.00%
10:35 5min	.94	6.8	.86	.84	.81	.03	3.57%
10:40 10min	.95	6.7	.85	.84	.81	.03	3.57%
10:50 20min	1.02	6.4	.76	.84	.78	.06	7.14%
11:05 35min	1.11	6.2	.72	.84	.79	.05	5.95%
11:20 50min	1.18	6.0	.66	.84	.78	.06	7.14%
11:35 1hr5min	1.31	5.7	.60	.84	.79	.05	5.95%
12:35 2hr5min	1.44	5.0	.53	.84	.76	.08	9.52%
1:35 3hr5min	1.47	4.9	.52	.84	.76	.08	9.52%
2:50 4hr5min	1.38	4.9	.52	.84	.72	.12	14.3%
3:30 5hr	1.37	4.8	.52	.84	.71	.13	15.5%
4:50 6hr20min	1.32	5.0	.52	.84	.69	.15	17.9%
5:30 7hr	1.32	5.0	.52	.84	.69	.15	17.9%
+ (7) = (5) - (6)		++ (8) = (7)/(5) x 100

TABLE 7.1-3		Flue Gas Analysis (2-3-94)												
Time	10:21	10:35	11:14	11:37	11:56	12:29	13:06	13:28	13:59	15:01	16:01	17:08	17:36	
T F	84	86	87	87	86	87	86	86	86	87	86	78	77	
Ox	7	7.5	7.1	7.4	7.2	7	7.3	7.1	7.2	7.2	7.4	7.1	7.8	average 7.25
CO	41	55	60	70	78	74	61	76	93	66	55	76	54	
NO	236	254	235	243	239	228	248	268	264	241	248	265	227	
SO ₂	0	798	50	98	167	358	639	768	903	348	347	319	255	average 388.5
CO ₂	11,5	11,1	11,4	11,2	11,4	11,5	11,3	11,4	11,4	11,4	11,2	11,4	10,9	
NO _x	248	267	247	255	251	240	261	282	277	253	261	278	238	

(2) Initial Na_2SO_3 Concentration Needed To Produce a 2M NaHSO_3 Solution.

The same data analyses were performed on absorption experiments other than Test No. 2. The total oxidation of sulfite to sulfate before a steady state was reached are summarized as follows:

Absorption Test No.1	17.9%
Absorption Test No.2	9.4 %
Absorption Test No.3	14.0%
Absorption Test No.4	14.4%
Absorption Test No.5	21.7%
Absorption Test No.6	23.5%
Average =	16.9%

Besides oxidation during absorption, there was oxidation during filtration since a pan filter was used in the pilot plant. From seven sets of data presented in Tables 6-2.13 and 7-2.14, the total sulfite retained in the filtrate was:

Test No. 1	80.8%
Test No. 2	91.1%
Test No. 3	68.2%
"	98.9%
Test No. 4	81.1%
"	100.0%
"	<u>92.5%</u>
Average	87.5%

Therefore, the oxidation during filtration was approximately 12.5%. Since only one-fourth of the side steam was going through the filter during the process operation, only 1/4 of the 12.5% or 3.1% of the sulfite will be oxidized in filtration during the process operation. Note that the oxidation loss due to sulfate removal in a commercial plant will be much less since a more advanced sulfate removal system rather than a pan filter will undoubtedly be used in a commercial plant.

Na_2SO_3 solution preparation will also incur some oxidation of the sulfite solution which we estimate to be 3% on average. Therefore, the sulfite oxidized during the process operation at the pilot plant may be estimated as follows:

• During preparation	3.0%
• During absorption including the initial stage of feed & bleed	16.9%
• During filtration	3.1 %
	<hr/>
Total	23.0%

In other words, in order to attain a 2M NaHSO_3 solution for process use, the initial Na_2SO_3 concentration in the absorber feed should be 1.23 M rather than 1M. This conclusion was verified in the subsequent absorber operation. In

our process operation, at least 1.8 M NaHSO_3 solution must be used as extractor feed solution.

(3) Two-Stage Absorption:

In Tests No. 5 and 6, both primary and secondary stages were used in absorption. Similar data-treatment was accorded to the absorption data. For Test No. 7, the data treatment is presented in Table 7.1-4, Figures 7.1-5a and 7.1-5b. Figure 7.1-5b shows that the secondary absorber had less oxidation than the primary absorber. That is somewhat unexpected since NaHSO_3 is normally regarded as easier to oxidize than Na_2SO_3 .

TABLE 7.1-4

Oxidation Rate Estimate for Absorption Test No. 7																
Primary Absorber									Secondary Absorber							
Measured Data				Computation					Measured Data		Computation					
		(2) Measured Total Sulfite (mmol/ml)	(3) pH	(4) Conversion Factor	(5) Na2SO3 Originally Present (mmol/ml)	(6) Na2SO3 Still Present (mmol/ml)	(7)=(5)-(6) Na2SO3 Oxidized (mmol/ml)	(8)=(7)/(5) x100 % Na2SO3 Oxidized	Measured Total Sulfite (mmol/ml)	pH	Conversion Factor	Na2SO3 originally present (mmol/ml)	Na2SO3 still present (mmol/ml)	Na2SO3 Oxidized (mmol/ml)	% Na2SO3 Oxidized	
9:00am	0 hr	1.27	7.60	0.95	1.20	1.20	0.00	0.00	1.23	7.00	0.90	1.11	1.11	0.00	0	
9:30	.5 hr	1.27	7.20	0.93	1.20	1.18	0.02	1.70	1.29	6.80	0.87	1.11	1.10	0.01	0.09%	
9:45	.75 hr	1.31	7.10	0.91	1.20	1.19	0.01	0.80	1.44	6.80	0.87	1.11	1.25	-	-	
10:00	1 hr	1.31	6.80	0.86	1.20	1.13	0.07	5.80	1.63	6.00	0.66	1.11	1.08	0.03	1.80%	
11:00	2 hr	1.60	6.10	0.69	1.20	1.10	0.10	8.30	1.80	5.10	0.54	1.11	.96	0.15	13.50%	
12:00pm	3 hr	1.84	5.30	0.55	1.20	1.01	0.19	13.20	1.95	5.00	0.52	1.11	1.01	0.10	9.00%	
Feed & bleed																
12:45	3.75 hr	1.84	5.20	0.55	1.20	1.00	0.20	16.70	1.97	5.10	0.53	1.11	1.04	0.07	6.30%	
1:45	4.75 hr	1.75	5.30	0.55	1.20	0.96	0.24	20.00	1.90	5.10	0.53	1.11	1.01	0.10	9.00%	
2:45	5.75 hr	1.73	5.40	0.57	1.20	0.98	0.22	18.30	1.93	5.10	0.53	1.11	1.02	0.09	8.10%	
3:45	6.75 hr	1.78	5.40	0.57	1.20	1.01	0.21	17.50	1.88	5.10	0.53	1.11	1.00	0.11	9.90%	
4:45	7.75 hr	1.68	5.40	0.57	1.20	0.95	0.25	20.80	1.83	5.10	0.53	1.11	0.97	0.14	12.60%	
5:15	8.75 hr	1.73	5.20	0.55	1.20	0.94	0.26	21.70	1.83	5.00	0.53	1.11	0.96	0.15	13.50%

Absorption efficiency varies with the pH of the absorbing solution. Some data taken during Test No. 3 are presented below in Table 7.1-5 and plotted in Figure 7.1-6

Table 7.1-5 Absorption Efficiency vs. pH of Absorbing Liquid in the primary absorber	
pH of the scrubbing solution	% Absorbed
6.9	100%
6.7	100%
6.4	100%
6.3	100%
6.1	94.3%
6.0	93.3%
5.9	82.0%
5.75	69.9%
5.7	57.6%
5.6	69.9%
5.5	4.0%

These data suggest that to get good absorption efficiency, a two-stage absorber must be used. If the pH in the primary absorber is maintained at 6.3, then very high absorption efficiency or nearly complete SO₂ removal can be attained. Part of that pH 6.3 liquid can then be sent to the secondary absorber to produce NaHSO₃ with a low pH and approximately 2M in NaHSO₃ concentration.

In tests No. 6 and No. 7, a venturi gas inlet flow of 3,230 ACFM was maintained. The liquid recirculation rate in the primary absorber in Test No. 6 was 110 gpm while in Test No. 7 was only 55 gpm. Hence, the L/G in Test No. 7 is only half as much as that in No. 6. Comparison of the total sulfite increase with time shows that oxidation in the primary absorber was much faster in Test

No. 7 with lower L/G as is shown in Figure 7.1-7a. That is to be expected. On the other hand, in the secondary absorber, there was not much difference in the oxidation rates between two tests as the total sulfite curves climb at nearly the same rate over time, as shown in Figure 7.1-7b.

Figure 7.1-6
Primary Absorber
pH of The Absorber Bleed vs Percent Absorbed

Figure 7.1-7a

Effect of L/G on Absorption
(Primary Absorber)

Figure 7.1-7b

Effect of L/G on Absorption
(Secondary Absorber)

Extractor Subsystem

One extraction test was conducted to test the effect of mixer speed with extraction performance in terms of percent extracted. Results are presented in Table 6.2-8 and plotted in Figure 7.1-8 below:

Figure 7.1-8
Percent Extracted vs Mixer Speed

These data indicate that a mixer rotor speed of 200 RPM was sufficient to attain the best result from the mixer.

It should be mentioned that our laboratory data has shown that the one-stage equilibrium concentration is 67% loading. The best results attained at the pilot plant was only 36%. That is to say the extractor can only attain a stage efficiency of 53.7% and that is to compare to the nearly theoretical extraction efficiency with the glassware mixer attained in Phase I at Hazen Research.

This relatively low extraction efficiency is perhaps a reflection of the design of the mixer-settler. On the other hand, low stage efficiency is not that important to the process operation as a whole because one always can use more mixer - settlers to attain higher loading.

Stripper Subsystem

Both dry and wet stripping were studied at the pilot plant. One of the parameters studied was therefore the aqueous to organic phase ratio. For dry stripping, this phase ratio was zero. The other phase ratios studied include 0.25, 0.5 and 0.75. Another variable studied was the steam rate. It varied from 50 lb/hr to 200 lb/hr with the liquid rate maintaining constant at 0.5 gpm (except in one instance, i.e., at aqueous to organic ratio of 0.25, the liquid rate used was 0.7 gpm rather than 0.5 gpm).

Data gathered are presented in Tables 6.2-9 to 6.2-12. These data are plotted in terms of stripping efficiency vs. steam rate in Figure 7.1-9 and in terms of residual sulfur loading vs. steam rate in Figure 7.1-10.

Inspection of these two figures reveal that dry stripping where $A/O = 0$ and wet stripping with $A/O = .5$ have comparable stripping performance. A high steam rate seems to favor dry stripping while low steam rate appears to favor wet stripping. The dry stripping and wet stripping have very different mass transfer mechanisms. In dry stripping, SO_2 is transferred from the organic phase to the gas phase directly. In wet stripping, the situation is more complex. Besides direct transfer from the organic phase to the gas phase, the SO_2 can also dissolve in the aqueous phase and then be stripped by steam from the aqueous phase. It appears that at low steam rates, the indirect stripping via an aqueous phase is favored.

Figure 7.1-9
Stripping Efficiency vs. Steam Rate

Figure 7.1-10
Residual Sulfur Loading vs. Steam Rate

At an A/O ratio of 0.25, the stripping efficiencies at various steam rates are shown to be lower rather than higher than those of dry stripping. To interpret these data, the general degeneration of the steam stripper performance during the testing period must be taken into consideration. As will be discussed in great detail under Section 7.3, the steam stripper performed relatively well up to 3-8-94. From 3-8 to 3-19, its performance was not consistent. It performed alright in some times but performed poorly in others. From 3-9 onward, the performance has degenerated. While the three experiments with phase ratios of 0, .5 and .75 were conducted during 2-15 to 2-17 when the stripper performed relatively well, the final set of data of stripping with a phase ratio of .25 were taken on 3-10-94 when the column performance had already degenerated. The data are therefore in suspect.

7.2 INTEGRATED TESTING

Key Operating Conditions

The daily operating conditions and test results are summarized in Section 6.3 Table 6-3.1. Besides these integrated tests aimed at confirming the operabilities of the equipment component as well as the process, several parametric tests were conducted, of which the results are discussed under section 6.2.

For a process that involves organics, organic stability is always a matter of concern. The organic stability of concern is not thermostability, because the organic solvent selected is highly thermostable and the highest temperature ever reached in the process operation was relatively low (i.e., 100°C). Rather, the concern with the organic stability is in regard to its extraction capacity. The organic extraction capacity tends to decrease if the process does not operate within a certain domain with respect to several key operating parameters.

A key operating parameter to maintain the organic extraction capability is organic loading. Our laboratory experience suggests that if we can keep the organic loading after stripping to less than 10% and preferably to under 5%, then the organics will be entirely stable, without losing its extraction capacity.

As the organic extraction capacity decreases, its viscosity tends to increase simultaneously according to our laboratory experience.

For a process which involves liquid-liquid extraction using a mixer-settler, phase inversion is, for many such systems, a major problem. In a mixer-settler system, mass transfer takes place in the mixer when two phases are intimately mixed. Depending upon the phase ratio, this mixture can be either an oil-in-water dispersion, or a water-in-oil dispersion. In the extraction step of the process, an organic-in-water dispersion will separate readily but a water-in-organic dispersion separates with greater difficulty. A water-in-organic dispersion is therefore to be avoided in the process operation.

The key process variable to avoid the formation of the wrong kind of dispersion is the phase ratio. For extraction, the aqueous-to-organic volumetric

phase ratio must be kept at greater than 1.2 in order to assure the formation of an organic-in-water dispersion rather than the undesirable inverse kind. Furthermore, during the operation, if the phase ratio is not carefully monitored, then "phase inversion" may occur.

Phase inversion means that the organic-in-water suspension is changed over to a water-in-organic suspension which is more difficult to separate. The change over from one kind of dispersion to another usually occurs suddenly. When it does occur, the two phases do not separate fast enough and mixer settler operation becomes upset. Some corrective action must then be taken to break the solvent-aqueous emulsion.

Phase inversion can occur wherever the two phases (aqueous and organic) are intimately mixed for some mass transfer purpose. In this process, it can occur in the extraction, stripping, alkali washing and bubbling unit operations. The aqueous-to-organic volumetric phase ratios that should be used to avoid phase inversion, according to pilot plant experience are:

Extraction	> 1.2
------------	-------

Stripping	no restriction
-----------	----------------

These important operating parameter restrictions, together with requirements on sulfur loading and pH of the bisulfite solution after absorption as well as organic sulfur loading after extraction, are summarized in Fig 7.2-1.

It was found that in the pilot plant the phase ratio control could be attained. Although during the initial operating period, due to lack of operating experience and inadequacy in some design features, a number of upsets did occur. But in each case, the upset conditions was corrected readily.

Figure 7.2-1
INTEGRATED OPERATION - KEY OPERATING VARIABLES TO BE
MAINTAINED

Many concentration specifications were not achieved during this operating period. Furthermore, they vary widely over time as illustrated by Figure 7.2-2 and 7.2-4. In these figures, all the available data points are plotted

In Figures 7.2-2, the data show that the concentration of the sodium bisulfite solution was lower than specification until March 8, 1994. On that date, the specified sodium bisulfite level of 1.8 was attained. This low sodium bisulfite concentration are also reflected in the low values of organic loadings after extraction as shown in Figure 7.2-3. This figure shows that on 3-8-94, when the total sulfite in the sodium bisulfite reached 1.8, the corresponding rich organic loading after extraction was about 40%. Stripping data plotted in Figure 7.2-4 show that with a few exceptions, the acceptable residual sulfur loading of not more than 10% was achieved, and in some instances, a level of less than 5% was attained.

Figure 7.2-2
Total Sulfite Concentration of Bisulfite Bleed After Absorption

7-28

Figure 7.2-3
Organic Sulfur Loading After Extraction

Figure 7.2-4
Residual Sulfur Loading After Stripping

7-30

Process Operability

This integrated test period offered an opportunity to evaluate the process operability. The process can be rendered nonoperable on the following three accounts:

1. The organic solvent degenerates with a decrease in extraction capacity over time. This degeneration will be accompanied by an increase in solvent viscosity.
2. Phase inversions occur and cannot be controlled readily by controlling some key variable(s) or a combination of variables. When phase inversion occurs, the dispersion between the two phases of an aqueous-solvent system separates with difficulty i.e. not fast enough to sustain a continuous operating system.
3. Crud or solid particle bearing mud cumulates in between aqueous and organic layers. This crud is hard to remove and interferes with the continuous operation of the process.

The solvent extraction capacity can best be measured by an extraction equilibrium curve as discussed in Appendix C. To gain accuracy, ten equilibrium curves were made for ten fresh organic solvents and four for solvent samples taken on 3-13-94. The results of these determinations are summarized in the following Table 7.2-1. The composite values from these determinations are plotted in Figure 7.2-5. Since the equilibrium curve for the 3-13-94 sample is essentially superimposed on the control equilibrium curve, it was concluded that there was no degeneration of organic extraction capacity upon usage. No sample was taken after 3-14-94.

Raycon's research showed that organic solvent degeneration or loss in extraction capacity is due to a physical change occurring during the process application of the solvent.

Table 7.2-1

Extraction Equilibrium Curves

	First Data Point		Second Data Point		Third Data Point	
	<u>Aqueous</u>	<u>Organic</u>	<u>Aqueous</u>	<u>Organic</u>	<u>Aqueous</u>	<u>Organic</u>
	moles SO ₃ =/mol Na+	moles SO ₃ /mol thso	mol SO ₃ =/mol Na+	mol SO ₃ =/mol theo	molSO ₃ =/mol Na+	molSO ₃ =/mol theo
Experiment No.						
Contol Sample						
Experiment No. 1	.69	.71	.88	.94	.93	1.05
Experiment No. 2	.69	.71	.89	.94	.92	1.05
Experiment No. 3	.69	.71	.88	.94	.93	1.04
Experiment No. 4	.69	.71	.89	.94	.93	1.04
Experiment No. 5	.69	.72	.88	.94	.93	1.05
Experiment No. 6	.70	.70	.88	.94	.94	1.05
Experiment No. 7	.71	.69	.88	.93	.95	1.03
Experiment No. 8	.69	.67	.88	.91	.94	1.04
Experiment No. 9	.70	.68	.87	.92	.95	1.03
Experiment No. 10	.70	.70	.88	.93	.94	1.03
Composite	.69	.70	.88	.93	.94	1.04
3-13-94 Sample						
Experiment No. 1	.71	.71	.90	.94	.94	1.04
Experiment No. 2	.73	.68	.88	.92	.93	1.02
Experiment No. 2	.72	.68	.88	.93	.94	1.02
Experiment No. 3	.73	.71	.88	.94	.94	1.02
Experiment No. 4	.71	.72	.98	.94	.94	1.02
Composite	.72	.71	.89	.94	.94	1.03

Figure 7.2-5
Extraction Equilibrium Curves

This degeneration reaction (a) must be controlled, diminished or fully stopped if the process is to operate within the prescribed operating conditions including the prescribed residual concentrations after each of the process steps. If the prescribed operating conditions cannot be met, some degeneration change may then occur. In that case, as a second layer of protection, the reversible reaction or the regeneration reaction (b) will initiate in the proprietary solvent treatment step. As a consequence, the organic should remain 100% intact after the completion of each cycle with zero loss in extraction capacity. Intermittent integrated operation from February 24 to March 13 demonstrated that this "zero loss" per cycle operation can be sustained.

Viscosities as determined by drainage time (as discussed in Appendix D), are plotted at the right lower corner of Figure 7.2-5. If there were degeneration of solvent extraction capacity, the viscosities of the 3-13-94 sample would be higher than the control. This was not the case. In fact the viscosities (which are proportional to the drainage time) for the 3-13-94 samples were lower than that of the fresh organic. However, the drainage times of the 3-13-94 samples were measured at 22°C which is 1° higher than when the control curve was measured. Therefore, with temperature correction, the viscosities of the two samples were more or less comparable.

In discussing the organic extraction capacity, two conditions should be recognized. First, during these integrated operations, the organic bleed rate was kept at 1% per cycle. This bleed rate was planned to be reduced to 0.1% in the subsequent test program. Secondly, as discussed earlier, during this period of the integrated test, the solvent treatment steps did not work properly. Without the solvent treatment steps working properly, the fact that the solvent nevertheless still maintained its integrity with respect to extraction capacity was indeed reassuring. It demonstrated that the solvent does not degenerate readily.

Phase inversion occurred rarely in the extractor and stripper. It could occur when the aqueous-to-organic (A/O) ratio in the solvent treatment steps were permitted to move outside the specified range. To avoid phase inversion, the phase ratios must be controlled through monitoring the organics and aqueous solution flow rate closely.

When phase inversion did occur, it could be corrected in several ways. In cases when it was detected soon after it began, immediate correction of flow rates was found to be effective to reverse the inversion and clear the system. When a more serious upset was encountered, it was necessary on occasions to stop the aqueous flow of the affected process step until the process cleared. This generally did not take longer than 15 to 20 minutes.

One quite desirable feature in phase inversion control should be mentioned. That is during the time when the upset condition was being corrected, some emulsion-like mass would flow into the lean solvent tank. It was found that these emulsions separated in the lean solvent tank quickly. The aqueous which was settled at the bottom can then be drained off and organics recycled to the operation circuit. In this manner, the system is to a large extent, self correcting.

A common problem which arises in the solvent extraction process using the mixer-settlers is the gradual build up of a "crud" layer at the aqueous / organic interface in the settler. From experience accumulated in hydrometallurgy, this crud layer is generally very hard to remove, but if not removed, it could disrupt the mixer-settler's operation as well as operation of the downstream equipment. Crud formation is often caused or exacerbated by the presence of suspended particulate matter in the system. Since our system does have particulate in the flue gas, it is quite possible that crud formation could have become an operating problem. Fortunately, this was not the case. During the integrated testing period, the formation of crud was not detected.

While good results were attained, steady state was not obtained during this initial integrated operating period. A number of operating variables/results oscillated rather widely, as is shown by Figures 7.2-6 to 7.2-16. In these figures, daily average data derived from Table 6-3.1 are plotted. In order to reconfirm

the encouraging results on process operability under steadier conditions, this day to day integrated test was terminated on 3-17-94. On that date, a new series of steady-state long term tests were initiated.

Extraction - Variation of Aqueous/Organic Phase Ratio Over Time

Figure 7.2-9

Stripping - Variation of Steam Flowrate Over Time

Figure 7.2-10

Stripping - Variation of Rich Organic Feed Temperature Over Time

Figure 7.2-11

7.3 STEAM STRIPPER TESTS

Performance of Pilot Plant Steel Column

(1) Steel Column Performance.

The stripping column performance was generally satisfactory in the integrated test periods (2-23 to 3-17-94). During that period it could generate a stripped organic with a residual concentration of less than 10%, enough to sustain an integrated test. But the performance degenerated after 3-17-94 as shown in Figure 7.2-4. In this figure, except for three analytical points at the very beginning of that period, all residual concentrations analyzed were above 10%. On March 18, column flooding was suspected, but drainage and restart of the column did not improve the performance. On April 6, another set of data was taken. These data are plotted in Figure 7.3-1

Figure 7.3-1
Steel Column Stripper
Performance Test
(Table 6-4.2)

This plot clearly suggested that the column was by then leaking so badly that at 100 lb/hr steam rate, the stripping efficiency was zero. Since residual organic concentrations were not high enough to sustain an integrated test, the operation stopped on 4-6-94 and the column was taken down for repair.

(2) Organic Stability

Inspection of Figure 7.2-4 suggests that between March 17 and 24 the residual concentration of the organic solvent after stripping met the less demanding specification (i.e. 10%) in about 50% of the time. During this period, solvent treatment step which are not reported here, were not performing. Organic Inventory was changed on March 16. After eight days of operation under these off-spec. rather abusive operating conditions, the organic solvent was still 100% active on March 24, 1994. This is shown by Figure 7.3-2 in which the equilibrium curve for the 3-24-94 sample superimposed on the control curve.

Degeneration was shown in the 3-28-94 sample taken after 12 days of operation. This can be seen by comparing curves A and B in Figure 7.3-3. But after this degenerated organic solvent was put through another cycle of operation in the laboratory, the solvent regained nearly half of the lost extraction capacity.

Figure 7.3-2
Extraction Equilibrium Curve of 3-24-94 Sample

Figure 7.3-3
Extraction Equilibrium Curve of 3-28 Sample

These data demonstrated several very important organic characteristics with regard to stability. First, good stripping is very important. Good stripping appears to be able to prevent or greatly diminish the degeneration reaction (a) (described under Section 7.2 Integrated Testing process operability subsection) from proceeding even under conditions when the stripping performance was far from ideal; and the organic treatment steps were mostly not performing. The latter means that there was no regenerative reaction (b) occurring to help maintain the organic stability.

These test results more than confirm the conclusion we reached from our previous laboratory studies. Our laboratory work suggested that if the stripping is sufficiently effective as to yield an organic residual concentration of less than 5% loading, then the organic solvent will be stable with or without organic treatment steps. Our pilot plant test results now suggest that as long as the organic residual concentration after stripping meeting the 10% loading specification more than 50% of the time, the organic will be stable with or without solvent treatment steps.

Secondly, the organic degeneration may be reversible. From 3-24 to 3-28-94, the organic degenerated because the stripping condition was grossly abusive. The lab-data which show that the organic was about 50% regenerated after being put through another cycle of operation means that the degeneration is temporary. It might gain back its extraction capacity soon after the abusive condition is removed and the stripping conditions return to normal.

Performance of Repaired Steel Column

(1) Repaired Steel Column Performance

Test results presented in Table 6.4-3 and 6.4-4 are plotted in Figures 7.3-4 and 7.3-5

Figure 7.3-4
Repaired Steel Column Performance
(Table 6.4-3 and 6.4-4)

Figure 7.3-5
Repaired Steel Column Performance
(Tables 6.4-6 and 6.4-7)

The curves in Figure 7.3-4 portrayed a rather strange relationship between stripping efficiency and steam rate. It is hard to explain because they (i.e. the curves) are probably heavily influenced by leakage dynamics. Data for the top curve were taken on 4-22-94 and the bottom curve on 4-26-94. Comparison between these two curves does show that the column performance before the packing repair was degenerating.

In spite of the complexity of the leakage dynamics, Figure 7.3-5 clearly shows that the low liquid flow rate yielded better stripping efficiency. Thus, at a low liquid rate (0.5 gpm), the column can achieve a 46.2% stripping efficiency at a 180 lb/hr steam rate. At a higher liquid flow rate (1gpm), the stripping efficiency was only about 27.4% at the same steam rate. In both instances, the stripped organic loading is over 10% -- too high to support an integrated steady-state test.

Performance of 4-inch Glass Column

(1) 4-inch Glass Column Performance

Up to this time, the problem we thought we had encountered was packing leakage. With the glass column, leakage was definitely eliminated. Test results, taken in triplicate to assure reliability, are presented in Table 6.4-9 and plotted in the following Figure 7.3-6

Figure 7.3-6
4-Inch Glass Column Performance
(Table 6.4-4)

The results were disappointing, with the stripping efficiency averaged only 26.4%. The residual sulfur loading after stripping was above 30%, which was too high to sustain a integrated steady-state test as was planned.

These under-performances were especially puzzling since a 2-inch diameter glass column had performed extremely well with an average of 97% stripping efficiency during the Phase I bench scale test at Hazen Research.

What could go wrong with the 4-inch column test? This important question will be discussed under the following several subsections: (2)

Strippability (3) Stripping efficiency versus initial loading (4) Number of trays used (5) Stripping efficiency versus design features.

(2) Strippability Evaluation

One question raised was whether the loaded organic produced in the pilot plant was as strippable as that produced at Hazen during the bench scale test, or that prepared in the laboratory. In other words, did some impurities present in the flue gas or impurities introduced from the stainless steel apparatus have some adverse effect on the organic strippability? To answer this question, a strippability evaluation apparatus was set up as shown in Figure 7.3-7. Using this apparatus, the results of the evaluation presented in Table 7.3-1 shows that the loaded organics prepared in the pilot plant had the same strippabilities as loaded organic prepared anywhere else. This ruled out strippability change as a cause of poor stripping performance.

Table 7.3-1 STRIPPABILITY EVALUATION		
Stock Stripped	Initial Loading Before Stripping	Final Loading After Stripping
Lean Organic Loaded In the lab	54.6%	16.2%
Plant Rich Organic	48.9%	15.0%
Plant Stripped Organic	29.9%	16.2%

Figure 7.3-7 Strippability Evaluation

APPARATUS

Procedure

1. Place 50 ml of 60/40 organic mixture into a 500 ml round bottom flask. Purge with nitrogen and insert a magnetic stirrer in the flask.
2. Load once with 2M sodium bisulfite solution. Mix for 10 minutes. Allow to settle and remove aqueous.
3. Analyze organic (Centrifuge first if possible)
4. Add 150 ml distilled water to flask. Place stripping stopper on flask. Keep nitrogen flow very low.
5. Bring solution to a boil (100°C).
6. Turn nitrogen flow up until it is bubbling rather vigorously into traps. Start mixer.
7. Strip for 15 minutes with nitrogen flowing and mixer going.
8. Remove remaining aqueous and analyze organic.

(3) Stripping Efficiency vs. Initial Organic Loading

When an amine is loaded with sulfurous acid or SO₂, it first forms normal sulfite. The reactions may be typically written as follows with amine symbolically written as RNH₂.

These reactions can proceed until the loading exceeds 50%. Beyond 50%, no more excess amine is present and further loading must proceed with the generation of amine bisulfite according to the following reaction.

Since amine bisulfite RNH₃•HSO₃ decomposes more readily than normal diamine sulfite, loading from 50% to 100% is easier to strip than that from 0% to 50%. In fact, because stripping produces amine which is basic, the more the loaded amine is stripped, the more basic the solvent will become, and further stripping will consequently become increasingly difficult.

The relationship between initial loading and strippability had been evaluated in our laboratory using the apparatus shown in Figure 7.3-7. The results are summarized in Table 7.3-2.

Table 7.3-2 Stripping of Organic With Different Initial Loading		
Initial Loading (%)	Final Loading (%)	Stripping Efficiency
23.0%	14.0%	39.0%
24.0%	14.0%	42.0%
29.0%	13.0%	55%
32.0%	13.0%	59%
40.6%	13.0%	68%
41.3%	14.0%	66%
49.6%	14.5%	71.0%
50.0%	14.3%	71.0%
58.0%	15.0%	74.0%
58.0%	15.0%	74.0%
85.5%	16.0%	77.0%
68.7%	16.0%	77.0%
77.0%	18.0%	77.0%
79.7%	16.7%	79.0%
93.0%	17.0%	82.0%
98.0%	18.8%	81.0%
106.0%	19.0%	82.0%
110.0%	21.0%	81.0%

These data are plotted in Figure 7.3-8. During the bench scale test at Hazen, the initial loading averaged 86%. The relationship given in Figure 7.3-8 can be used to estimate the performance of the 4-inch column if the initial loading was 86% so as to render the data from the 4-inch column and that from the 2-inch column more comparable. This can be done through the construction of a correction curve given in Figure 7.3-9. Using this curve, the stripping efficiency of 26.4% at an average initial concentration of 43.4% will be corrected to $1.17 \times 26.4\% = 30.9\%$ at an initial loading of 86%.

Figure 7.3-8
Stripping Efficiency
vs, Sulfite Loading
in the Organic Feed

The number of stripping stages used definitely has an influence on the overall stripping efficiency. To estimate that influence, the sulfur loading distribution of the solvent along the column height is important. These distributions had been measured for the steel column on 4-6-94 and for the glass column on 6-12-94. They are presented in the following Table 7.3-3.

Table 7.3-3		Concentration Distribution Along Column Height	
9-inch Steel Column		4-inch Steel Column	
Tray No.	Sulfur Loading	Tray No.	Sulfur Loading
	(%)		(%)
Rich solvent	38.4	Rich solvent	35.7
24	29.1		
20			
19	26.7		
		15	21.5
14	25.9		
		10	19
9	27.5		
5	23.5		
Stripped solvent	21.8	Stripped Solvent	14.5

Data in Table 7.3-3 are plotted in Figures 7.3-10 and 7.3-11. The curves in these two Figures are quite similar. They show that a great deal of stripping was taken place in the top five stages. After the top five stages, the concentration drops nearly linearly with the stripping stages. In the present instance, no correction is needed since the 4-inch column and the 2-inch column both have 20 stages.

(5) Stripping Efficiency vs. Design Features

While part of the low efficiency for the 4-inch column can be attributed to low initial sulfur loading, after a correction was made the performance of the 4-inch column (30.9%) was still far below the performance achieved by the 2-inch column (97%). To explain this wide difference, we must now look toward the variation in design and operating features between these two columns.

The major differences in the design and operating features which might contribute to the differences in performance of the two columns are contrasted in the following Table 7.3-4.

Table 7.3-4 Stripping Column Characteristics		
	2-inch column	4-inch column
<u>Design Characteristics</u>		
Hole diameter	.05"	$\frac{1}{8} = .125"$
No. of holes per tray	87	50
Weir height	5 mm	.75" = 19.1 mm
<u>Operating Characteristics</u>		
Steam rate	14-36 gm/min = .85 - 4.76 lb/hr	38 lb/hr
Liquid rate	35-101 gm/min = .011 - .032 gpm (.022 av)	0.49pm

Figure 7.3-10
Concentration Distribution
Along a 9 inch Steel Column

Figure 7.3-11
Concentration Distribution
Along a 4 inch Glass Column

Inspection of this table suggests that the presence of a large number of small holes could be of major importance if mass transfer in a viscous liquid depends largely on the turbulence imparted to the system during the initial bubble formation. The effect of weir height is hard to assess; we are assuming that the difference in weir height does not have any major impact on performance.

The table further shows that the 4-inch column was operating at a much higher gas velocity and liquid velocity. If the bubble formation is of key importance, then perhaps the turbulence induced by the bubble formation may not much depend on the steam velocity. On the other hand, liquid velocity could be quite significant because it may be not the number of small holes per se, but rather the number of small holes per unit liquid flow rate that is important. The latter measures the turbulence input or effective energy input to the unit amount of liquid being stripped.

Thus, stripping liquid of high viscosity could be radically different from stripping liquid of relatively low viscosity such as hydrocarbons, or alcohols, etc. Unfortunately, stripping of high-viscosity liquid is a nascent field in chemical engineering technology. Instead of drawing from past experiences from the field, we are now finding ourselves pioneering an effort in new engineering development.

Nevertheless, our own engineering analyses based upon our meager pilot plant data led us to believe that in stripping a liquid of high viscosity the presence of a large number of holes on trays in tray design and running the column at relatively low liquid velocity during column operation could be two important factors in attaining high stripping efficiency. Accordingly, two 7-tray sections of small holes were ordered to test out the validity of our analysis.

Performance of 4-inch Glass Column With Modified Tray Design

(1) Modified 4-inch Column Performance

The performance data of the modified glass column taken in duplicates are recorded in Table 6.4-11 and plotted in Figures 7.3-12 and 7.3-13

Figure 7.3-12
Stripping Efficiency vs Steam Rate
(Table 6.4-11)

Figure 7.3-13
Stripping Efficiency vs Liquid Rate
(Table 6.4-11)

Figure 7.3-12 shows that the stripping efficiency is independent of the steam rate. This is in line with our engineering analysis, namely, that turbulence created during bubble formation promotes mass transfer but whether the bubble is created by high-velocity steam or low-velocity steam does not make much difference.

At a liquid rate of 0.4 gpm, an average stripping efficiency of 37% was attained on 6-9-94. This is definitely better than 26.4% stripping efficiency attained on 5-25-94 before tray modification. It should be taken into consideration that a new half-batch of organics was charged to the system on 5-20-94, the test of the 4-inch original column took place on 5-25-94 when the organic was relatively fresh. On the other hand, the test on the modified column was conducted on 6-9-94 using organic which was already much abused (due to under performance of stripper, etc.) and degenerated. If organics used were of the same quality, the differential in performances would be much wider. The organic degeneration can be seen in Figure 7.3-14

Figure 7.3-13 shows that as the liquid rate decreased, the performance increased. This is also in conformity with our engineering analysis which suggests that it is not the presence of small holes alone but rather the number of small holes present per unit liquid flow rate that is of key significance. The effect of liquid rate shown in Figure 7.3-13 is corroborated by the repaired steel column data shown in Figure 7.3-5.

The modified 4-inch column has 313 holes per plate in comparison to 87 holes per plate in the 2-inch column used at Hazen. Table 7.3-4 shows that at Hazen, the average liquid flow rate was 0.022 gpm. The corresponding liquid flow rate for the 4-inch column therefore should be $.022 \times \frac{313}{87} = .08$ gpm. In other words, to attain a 97% stripping efficiency, the organic should be loaded up to 86% and stripped at a liquid flow rate of 0.08 gpm.

Figure 7.3-14
Extraction Equilibrium
Curve of 6-13-94 Sample

(2) Comparison of Performances Between the 4-inch and 2-inch Columns

To compare the performances of the two columns, they would have to be put onto comparable bases. For that purpose the performance of the 4-inch column is adjusted as follows:

Performance of the 4-inch column

- Liquid rate = .15 gpm
- Initial loading = 29.4%
- Number of modified trays = 14
- Stripping efficiency 52.1 %

- Correction for liquid flow rate

• by Figure 7-3.13

Performance of the 4-inch Column

- liquid rate = .08 gpm
- Initial loading = 29.4%
- Number of Modified trays = 14
- Stripping efficiency = 58%

- Correction for Initial Loading

by Figure 7-3.9

Performance of the 4-inch Column

- Liquid rate = .08 gpm
- Initial Loading = 86%
- Number of modified trays = 14
- Stripping efficiency =
 $1.49 \times 58\% = 86.4\%$

- Correction for number of modified trays

by Figure 7.3.11

Performance of the 4-inch Column

- Liquid rate = .08 gpm
- Initial Loading = 86%
- Number of modified trays = 20
- Stripping efficiency =
 $86.4 + (.47 \times 6) = 89.2\%$

With these adjustments, the performance of the 4-inch column becomes 89.2% in comparison to 97% attained with the 2-inch column. The check is within the range of reasonableness considering the many approximations used adjustments.

Major assumptions used in adjustments include the following:

- (1) Figure 7.3.13 is valid for liquid flow rate correction.
- (2) Figure 7.3-9 derived from laboratory data can be used to approximate the stripping efficiency at a higher sulfur loading.
- (3) The bottom section of the modified column with six trays with large holes was not contributing to the stripping at all.

In spite of these assumptions, the fact that the performances of the two columns (when put on comparable bases) do check with each other within a reasonable range is significant because it renders some creditability to our engineering analysis. In other words, subject to future confirmation, our engineering analysis on stripping high-viscosity liquid could quite likely be valid.

With the 4-inch column, when operated at the highest efficiency attainable or 52.1%, the residual loading was 14%, which was still too high to sustain an integrated steady-state test as planned. Besides, to operate at 0.15 gpm is too low a liquid flow rate to make the integrated test meaningful. The test was therefore terminated, since to test at a higher rate would require a new and larger column which was beyond our budget constraints.

(3) Mass Transfer in Conventional Low-Viscosity Liquid Stripping

To discuss mass transfer in a system such as a water-air system, one first has to recognize that regimes of operation exist. At low air rates and low hydrostatic heads on a perforated tray, the material on the tray is a large-bubble mass or bubble foam. As the air rate or head is increased, discontinuities (large gas cavities) appear in the foam. At low heads, these cavities act as

chimneys, vertical open passes through the froth, which rapidly form and disappear. At higher heads, these cavities are large bubbles called "slugs". The slugs rise rapidly through the foam and generate considerable turbulence in it. The combination of foam and cavities, whether chimneys or slugs, is designated froth. Most of the gas traverses froth via the gas cavities. At high air rates and very low heads, the froth breaks down and the liquid traverses the plate as coarse spray.

There is a progressive transformation vertically in the character of the liquid-gas structure, which is a function primarily of the total depth of the structures and of the gas rate. This structure may be divided into three regions: a bottom water layer, a turbulent constant density zone, and a decreasing density "foam drainage" region.

The presence of a large mass of gas bubbles in the gas-liquid mixture on a tray over most operating conditions is readily apparent to anyone observing a tray in action. A natural consequence has been the postulation of gas liquid contacting on a bubble plate to be representable by contact between rising gas bubbles/slugs and the liquid films. In the froth regime, the mass transfer data can be well correlated by a linear combination of the buoyant power factor, representing the rate of energy loss suffered by the air in traversing the froth and a function of the group.

$$(A_H/A_P) (V_H^2 / 2 g c h_o) \dots\dots\dots (7.3-3)$$

where: A_H = Area of the holes
 A_P = Plate area
 V_H = Hole velocity, ft/sec
 H_o = Hydrostatic liquid head, cm of water

It is believed that the frequency of the formation of the slug, the size, and the initial rate of rise of these gas slugs would all be influenced by the magnitude of this group.

Equation (7.3-3) indicates that for a conventional low-viscosity liquid system such as water-and-air system, mass transfer depends greatly on the gas velocity. High gas velocity or high F value (i.e. $V_h \sqrt{\rho}$ where ρ is gas density) is normally used as the key design factor. It was the criterion we used in scaling up the 4-inch glass column from the 2-inch column. As it turned out, it is incorrect.

(4) Mass Transfer in Stripping High-Viscosity Liquid

From the visual observation of the glass column operation, we noticed that our system differs from the low-viscosity water-air system in at least two significant respects:

- (1) In the water-air system, the bubble-froth material usually has a height which is approximate 3 times weir height. In our system, the bubble-froth material height is much less and was approximately only 0.75 x weir height. Thus, the chance for mass transfer in the bubble-froth region is much less because of the reduced height alone.
- (2) The viscosity of our liquid is much higher. For the 50% loaded stock, viscosity at 100°C is about 8 c.p. Therefore, the diffusional resistance for mass transfer (see equation 2.1) in the froth region is much higher than that in a liquid of lower viscosity.

Because of these two factors, a high steam rate is a relatively ineffective way to induce high stripping efficiency.

As stated earlier from experience in our pilot plant test, it appears to us that the mass transfer mechanism in stripping a liquid of high viscosity is drastically different than that for conventional stripping with a low-viscosity liquid. For stripping high-viscosity liquid, turbulence created during the initial bubble formation is much more important than turbulence imparted by high-velocity steam. It is our preliminary conclusion, subject to future confirmation, that the scale-up criterion for the stripper should be the number of holes per unit liquid flow rate rather than high F value or high steam velocity.

We have previously estimated that using the 4-inch modified column, to attain a 97% stripping efficiency, the organic should be loaded up to 86% and stripped at a liquid flow rate of 0.08 gpm. An efficiency of 86% can be attained by using multiple stages of our extraction system. A larger column should be used to strip at higher flow rates.

Thus to operate at 1 gpm (1 MW), a 14-inch ($4\sqrt{1/0.08} = 14$) diameter column should be used. To operate at 2 gpm (2 MW), a column 20-inch in diameter ($4\sqrt{2/0.08} = 20$) should be used.

8.1 PILOT PLANT CAPACITY

8.2 PILOT PLANT SHUTDOWNS

Duct Repair

Stripping Column Leak Repair (4-6-94 - 4-21-94)

Glass Column Fabrication and Installation (4-28-94 - 5-16-94)

Glass Column Design Modification (5-26-94 - 6-14-94)

8.3 SUGGESTED PLANT MODIFICATIONS

Component Equipment Related Improvements

Pump Related improvements

Instrument & Analysis Related Improvements

Material Related Improvements

System Related Improvements

SECTION 8

PILOT PLANT OPERABILITY

8.1 PILOT PLANT CAPACITY

After the pilot plant was built, a major concern was its mechanical operability. To assure that all the equipment components were mechanically fit, Raycon commissioned a system study by two consultants in March 1993 and a mechanical test in November 1993. The plant was substantially upgraded mechanically as a result of these two studies.

While the plant's designed capacity was 2MW, it was revealed during the mechanical test that the capacities of several equipment components were less than the rated plant capacity. Among them, the primary I.D. fan was limited to approximately 80% of the designed capacity. The capacities of a number of magnetically driven chemical-feed pumps were also limited to various degrees. These magnetically driven pumps were selected because they were completely enclosed, thus precluding any chance of feed contamination. In practice these pumps were found to be quite intolerant to small changes in operating conditions. Furthermore, for pumping relatively viscous liquid, the magnetic torque required appeared to be higher than estimated. These factors made the pump capacities more limited than anticipated. Some of the pumps could reach a capacity of only about 60% of the designed capacity. The capacity of the other pumps were higher, varying from 60% - 100% of the designed capacity.

The plant upgrading could then proceed consistent with the following two goals; (1) to emphasize equipment operability over capacity, i.e. to upgrade all equipment components so that they could all operate reliably, e.g., to achieve a 90% on-stream time; (2) to emphasize equipment capacity over operability, i.e. to replace these equipment components of lesser capacities with the increased capacities so that the pilot plant as a whole could operate at the designed capacity level. Because of our budget constraint, we were forced to select only one of these two goals. We opted the first goal. The rationale was that after the demonstration was achieved at a lower-than-designed capacity, it could always be repeated at the rated capacity if funds were available to replace those components that needed to be replaced.

While a major reason for not operating the pilot plant at the rated capacity was economic, it was also found that the plant could be operated more steadily at lower capacities thereby generating more reliable information. This was especially true before March 17, 1994, when several modifications in the design of equipment of the solvent treatment steps were still being defined and implemented. After March 17, 1994 while the solvent treatment steps were operating relatively acceptably, the stripping column soon leaked, and from then onward, the demonstration mainly focused upon gathering data for developing a scale-up criterion for the design of the pilot/commercial columns so that the organic medium could be stripped effectively. Thus, although the plant's rated capacity was 2MW, this capacity was never reached. Most of the demonstration experiments were conducted at 0.5-1.2 MW levels.

8.2 PILOT PLANT SHUTDOWNS

The mechanical test conducted in early November 1993 test concluded that on stream time was generally low --- below 50%. The low on stream time were attributed to, among others: (1) low magnetic pump performance; (2) leaks at valves and other junctions; and (3) flow meter plugging. These mechanical conditions prevailed over the initial operating period and shut downs for equipment maintenance occurred frequently. However, during that period, the operation was on an eight-hour per day basis. Furthermore, each test scheduled during that period took a relatively short amount of time to complete. The test, therefore, could usually be scheduled around those shutdowns such that these shutdowns would not impede too much to the overall progress of the test program. Besides, a number of shutdowns were also induced by boiler operations. Dunkirk station's No. 4 boiler from which we derived the flue gas and steam supply was shutdown occasionally for a number of reasons including: mechanical problems such as tube leaks, low electricity demand etc. Sometimes, even when the boiler was operating, the steam pressure from the station was too low for pilot plant operation.

Initially the pilot plant steam supply came from a steam header. This header derived steam from the de-aerator for Dunkirk station's No. 1 and No. 2 boilers. The supply of steam from this de-aerator was limited. This same steam header also supplied steam for space heating, etc. for the Dunkirk station. As a

consequence, during the severely cold winter months, the steam pressure at the pilot plant was frequently not sufficient for pilot plant operation. By the end of January 1994, a dual steam supply line to the pilot plant was installed. Since this did not quite solve the problem, NMPC's Dunkirk Station management permitted the pilot plant to derive its steam directly from the No. 4 boiler steam drum (2400 psi). After that time, the pilot plant did not have any steam supply problem.

In addition to these occasional shut-downs, the pilot plant had also experienced four major shutdowns. These are summarized as follows:

Duct Repair

A major shut down due to ductwork repair occurred from October 28, 1993 to January 18, 1993.

On October 28, 1993, RE&C notified Raycon that the pilot plant had encountered a serious problem in duct corrosion, and that part of the ductwork was taken down for repair. The ductwork was made of carbon steel coated with a protective layer of resin. When properly installed, the resin had been demonstrated to last for years in industrial applications. However, to apply the coating correctly is a delicate operation. Before coating, the steel substrate surface must be prepared to meet stringent roughness specification. The coating itself must not be too thin. Nor too thick. When it is too thick, it tends to lose the desired flexibility and cracks. Since the flue gas ductwork accumulated strongly acidic liquid ($\text{pH} < 1$), once a crack occurs, the acid solution will penetrate through the coating to directly contact the steel. It can then corrode the carbon steel at very fast rates. Coating failure analysis conducted by Plasite Laboratory concluded that in our case, the substrate surface had shown evidence of a blast profile, but it lacked depth and density. The resin coating itself was somewhat too thick, and it exceeded the recommended range of 35 - 43 mils.

The corrosion attacks occurred on the inlet side of the duct upstream of the ID fan. This is probably because the multiple startups and shutdowns had imposed thermal stress in the duct on the inlet side (70°F ambient to 300°F

when the gas was flowing). On the outlet portion of the ductwork, the gas has already been cooled down by the venturi prescrubber. The temperature variation was less severe.

Recognizing that the delicate coating operation is very difficult to execute in the relatively small duct, it was decided to install a stainless duct to replace the corroded steel duct. The stainless duct when installed operated satisfactorily for the remainder of the test program.

Stripping Column Leak Repair (4-6-94 to 4-21-94)

To repair the packing leak of the 10-inch diameter steel column, the column was disassembled and taken to the Quakenbush warehouse in Buffalo, NY for repair. Refer to discussion in 6.4 Steam Stripper Test - Subsection Stripper Repair.

Glass Column Fabrication and Installation (4-28-94 to 5-16-94)

Because the repaired steel column did not perform well, a 4-inch diameter glass column was ordered to take its place. The column was fabricated by Lab-Glass of Kingsport, TN. The fabrication took two weeks. Refer to discussion in 6.4 Steam Stripper Test - Subsection Design and Installation of 4-inch Glass Column.

Glass Column Design Modification (5-26-94 to 6-14-94)

Two sections of the 4-inch glass column's tray design were subsequently modified to improve the stripping efficiency. Refer to discussion in Section 6.4 Steam Stripper Test - Subsection Glass Column Design Modification.

8.3 SUGGESTED PLANT MODIFICATIONS

Based on the experience gained during the operation of this pilot plant, the following is an attempt to identify how the system design should be modified to improve the plant operation. The improvements are classified into: (1) component equipment related improvements; (2) pump related improvements; (3) instrument / analysis related improvements; (4) material related improvements; and (5) system related improvements.

Component Equipment Related Improvements

- Absorber: Stilling wells should be installed on some components, such as venturi and absorber sumps to "smooth" the liquid level signal transmitted to the data acquisition system.
- Extractor, Solvent treatments: Control of the organic / aqueous phase ratio was typically set manually. A method to automatically measure and control the phase ratio entering various components would greatly simplify and improve operation of the system. New two-phase control instrumentation capable of measuring volume percentages of oil and water mixtures, although expensive, could prove to be very useful.
- Extractor: Investigate whether extractor performance can be improved by improvement in design features.
- Stripper: Provide stripper pressure control and measurement.
- Waste Tank: pH control of the waste tank might more easily be accomplished by feeding 50% a caustic solution instead of using lime.

Pump Related Improvements:

- Do not use magnetic drive pumps if they can be avoided.
- Install strainers upstream of, and bypasses around, all pumps. Solids present in the various streams caused plugging.
- All pumps should have outlet pressure gauges. These local pressure gauges would allow on-line tracking of pump performance, enabling the operators to identify potential failures.
- Pumps should be sized to empty tanks within reasonably short periods of time.
- Install spare pumps.

Instrument and Analysis Related Improvements:

- Install a data acquisition system with more user-friendly operating modes, allowing report production and data manipulation while on-line.
- An extractive-type continuous emission monitoring system is required for a future installation. In-situ monitors are not well suited for the cycling/unstable operation typical of a pilot plant. It should have multiple sample point capability. The cost will be much higher.
- Add local indicators at all data transmitters that send data to the data acquisition system. Calibration of flow, temperature, etc., instrumentation would be much easier with local readouts and would also allow easier checks of questionable data.
- Install strainers upstream of, and bypasses around, all flow meters.
- Provide sample points in all mixer tank sections of the mixer / settler components. This provides direct access for analysis of tank phase ratios, a critical control parameter to avoid phase inversion.

- Due to the critical nature of the steam flow rate into and out of the stripper tower, better methods of measurement are required, especially where steam conditions are widely variable. Do not use steam rotameters for steam flow measurement.
- Magnetic flow meters are not acceptable for organic stream flow measurements due to the electrical conductance characteristics of the organic solvent. Small quantities of water can give false readings. Rotameters are also not acceptable for organic flow measurements due to varying organic viscosity depending on sulfite loading, etc.
- Provide direct temperature control for the rich organic feed to the stripper. The methodology to obtain this control is not clear at this time.
- Provide level control instrumentation on the bubbler / regenerator.
- Automatic on-line analyzer may be developed for organic and aqueously loading analysis.
- Alternate sampling methods for the organic should be developed to eliminate waste, spills and air contact.

Material Related Improvements

- Stainless steel should be used for ductwork handling flue gases.
- Materials of construction for the instrumentation in the sodium bisulfite bleed stream should be upgraded to avoid corrosion.

System Related Improvements

- Provide floor sumps or trenches for pump seal bleeds, spills and wash downs. Isolation of these sumps will be necessary to maintain organic isolation from the rest of the plant.
- Add site glasses to all storage tanks, extending to the bottom of each tank.

- Eliminate centrifuge separators from the system design. The organic and aqueous phases separate quickly and separator will therefore probably not be required.

CONCLUSIONS

SECTION 9

SECTION 9

CONCLUSIONS

From the test results obtained at the pilot plant, the following conclusions can be reached.

- 1) The organic solvent appears stable, and no degeneration in extraction capabilities or increase in viscosity were observed during the intermittent integrated test period from February 24 to March 13, 1994.
- 2) The organic stability is further enhanced by the observation that the stripping, and solvent treatment are providing multi-layer protection to organic solvent stability.
- 3) After the integrated test period, during the first eight days in the attempted steady-state test period when steam stripping was performing acceptably only half of the time and solvent treatment steps were not working at all, the organic was still fully active after eight days of operation indicates that the organic is stable under even more adverse operational conditions than those that prevailed during the period of integrated testing.
- 4) Organic degeneration, with regard to temporary loss in extraction capability, if and when occurred due to exposure to abusive operating conditions, is likely to recover after the abusive operating conditions are removed and the operation returns to normal, based upon the limited data obtained from the pilot plant operations.
- 5) Phase inversion, which may occur during extraction, and solvent treatment can be avoided if the aqueous-to-organic phase ratio for each of these stages is kept within specific operating limits.
- 6) Phase inversion, if encountered can be corrected simply by adjusting the phase ratio back to within the acceptable ranges.

- 7) No crud formation was observed. Crud means muddy solid laden materials existing at the aqueous - organic interface. It is usually very difficult to remove.
- 8) Pilot plant experience suggests that a two-stage absorption should be used. The pH of the first stage should be maintained above 6.3 to obtain good SO₂ removal.
- 9) Mixer - settlers can be used for the extraction process. However mixer - settlers of our selection yielded relative low extraction efficiency in the case of the pilot plant.
- 10) Recognizing that stripping a liquid of high viscosity is radically different from stripping one of low viscosity, the design of an effective stripper should be based upon different engineering principles than those being used conventionally and at the pilot plant. Engineering analysis of the pilot plant data suggested that the number of tray holes per unit liquid flow rate could be a valid scale up criterion.
- 11) The stripper must have a pressure control system at its top.
- 12) The separator for stripped organic should be designed carefully to separate the acidic aqueous from the organic cleanly.

RECOMMENDATIONS

SECTION 10

SECTION 10

RECOMMENDATIONS

Following future works are recommended:

1. The scale-up criterion proposed in this report, i.e., the stripping column should be scaled up by holding the number of holes per unit liquid flow rate constant, should be verified and further improved by laboratory experimental data.
2. Once the stripper scale-up problem is sufficiently resolved, opportunities should be sought to build a second demonstration plant to demonstrate the process.
3. In the next demonstration plant:
 - An effective separator should be installed to separate the acid aqueous from the stripped organic effectively.
 - Automatic phase control options for the extractors and solvent treatment units should be provided.
 - Aqueous and organic loading analysis should be automatic and on-line.
 - Pumps and gas analyses instrumentation should be selected carefully so that they will function dependably.
 - A user-friendly computer system should be selected for operational control and data logging.
 - The stripper column should have an automatic pressure control at the column top. Pressure gages should be installed along the column height.
 - The Duckwork should be made of stainless steel.
4. Search for alternate amines should continue. Material Safety Data Sheets should be developed for the promising amines.

APPENDICES

- A PROCESS FLOW DIAGRAM
- B MATERIAL BALANCES AND COMPONENT EQUIPMENT
PERFORMANCES
- C EQUIPMENT LIST
- D ANALYTICAL PROCEDURES

APPENDIX A
PROCESS FLOW DIAGRAM

[illegible]

APPENDIX B

**MATERIAL BALANCE & EQUIPMENT
PERFORMANCE**

APPENDIX B

MATERIAL BALANCE & EQUIPMENT PERFORMANCE

Absorption Subsystem

A schematic diagram of the Absorption Subsystem is given in Figure B-1.

Test Data

Test data taken around the Absorption subsystem on June 13, 1994, 3:30pm, are presented in Table B-1. These data are snap shot data taken around the absorber nearly simultaneously after a steady state of absorber operation was attained.

Unit Conversion

Data around the Absorber Subsystem were collected on June 13, 1994 and presented in Table B-1. To make calculation, the units of these data are converted to those which can be directly used in the calculations. i.e. to convert:

Table B-1

ABSORBER SUBSYSTEM

6-13-94

1. TEST CONDITIONS AND ANALYSIS

GAS (1.5 MW EQUIVALENT)

3:30pm

1. Venturi Pressure Drop/Damper Setting	in H2O	PDT-0101	10.9
2. Flue Gas from NiMo (1)	a) Rate - ACFM	None	4798
	b) SO2 - ppm		1346
	c) NOx - ppm		231
	d) O2 - %		7.2
3. Bypass Gas (2)	Rate - ACFM	FE-0102	1430
4. Venturi Inlet Gas (3) Sample Point G1	a) Rate - ACFM	FE-0103	3368
	b) Temperature - F	TE-0103	293
	c) Pressure - in H2O	PT-0103	0.35
	d) SO2 - ppm		1346
	e) NOx - ppm		231
	f) O2 - %		7.2
5. Primary Absorber Outlet Gas (3) Sample Point G3	a) Rate - ACFM	None	
	b) Temperature - F	TE-0105	124.8
	c) Pressure - in H2O	PT-0105	-16
	d) SO2 - ppm		430
	e) NOx - ppm		229
	f) O2 - %		8.3

LIQUID

6. Venturi Recycle (8)	Rate - GPM	FT-0108	Inoperative
7. Venturi Bleed (17)	Rate - GPM	FT-0217	Inoperative
8. Sulfite Absorber Feed (30) Sample Point A6	a) Rate - GPM	FT-1030	0.9
	b) Total Sulfite moles/Liter		1.29
	c) NaHSO3 moles/Liter		0.12
	d) Na2SO4 moles/Liter		0.1
	e) pH		7.6
9. Primary Absorber Recycle (9)	a) Rate - GPM	FT-0109	119
10. Secondary Absorber Recycle	a) Rate - GPM	FT-0120C	
11. Secondary Absorber Bleed (20) Sample Point A3	a) Rate - GPM	FT-0120	0.9
	b) Total Sulfite moles/Liter		1.85
	c) NaHSO3 moles/Liter		1.76
	d) Na2SO4 moles/Liter		0.57
	e) pH	AT-0120	5.1

(1) gas rate from ACFM to lb mole gas / min, (2) gas composition from ppm SO₂ to percent (%) SO₂ and (3) liquid composition from Molar total sulfite to lb SO₂ / gal. These conversions for relevant streams are attained as follows:

1. Gas inlet - Stream 3

Assuming gas law applies
$$\frac{V}{N} = \frac{RT}{P} \quad (9-3.1)$$

Where P, V, T designate gas pressure, volume and temperature respectively. N designates gas flow rate in lb mol gas / min and R is the gas constant.

Hence:
$$\text{Gas rate} = N = \frac{VP}{RT} = \frac{[3368 \times \frac{492}{T} \times \frac{14.7}{P}]P}{RT}$$

$$= \frac{3368 \times 492 \times 14.7}{10.73[460 + 295]} = 3.98 \text{ lb mol gas / min}$$

and
$$\text{Gas composition} = 1346 \text{ ppm} = \frac{1346}{10^6} \times 100 = 0.1346\%$$

2. Gas outlet - Stream 5

Assuming the venturi inlet flue gas (stream 3) contains 7 mole per cent water and has an average molecular weight of 29.5 (note: These assumed values can be precisely determined if the approximate analysis of coal used in 6-13-94 and the flue gas analysis at the time when data were taken are known). From the humidity chart in P20 - 6 Chemical Engineering Handbook - 5th edition by Robert H Perry & Cecil H. Chilton, we will have:

Since initial conditions:

7 mole percent (0.046 lb water per lb dry flue gas)

295°C

Therefore from the humidity chart final conditions:

9 lb water per 100 lb dry flue gas (14.75 mole percent)

Flow rate of the saturated flue gas at stream 5

$$= 3,368 \frac{(100 - 7)}{(100 - 14.7)} = 3,674 \text{ ACFM}$$

$$\therefore \text{Gas rate} = 3,674 \text{ ACFM} = \frac{3674}{3368} \times 3.983 = 4.345 \text{ lb mole gas / min}$$

$$\text{Gas composition} = 450 \text{ ppm} = \frac{450}{10^6} \times 100 = 0.045\%$$

3. Aqueous Inlet - Stream 30

Aqueous rate = 0.9 gpm

Aqueous Composition = 1.29 M

$$= 1.29 \times 3.7854 \times 64.063 \times \frac{2.2}{1000} = .6882 \text{ lb SO}_2 / \text{gal}$$

$$\left(\frac{\text{moles}}{\text{L}} \right) \quad \left(\frac{\text{L}}{\text{gal}} \right) \quad \left(\frac{\text{gSO}_2}{\text{mol}} \right) \quad \left(\frac{\text{lb}}{\text{g}} \right)$$

4. Aqueous outlet - Stream 20

Aqueous rate = 0.9 gpm

Aqueous Composition = 1.85 M

$$= 1.85 \times 3.7854 \times 64.063 \times \frac{2.2}{1000} = .9887 \text{ lb SO}_2 / \text{gal}$$

Data with converted units are summarized in Table B-2.

Table B-2 Data for Absorption System			
Stream		Original Units	Converted Units
3. Venturi Inlet Gas, SP (G1)	Rate	3368 ACFM	3.983 lb mol gas / min
	SO ₂	1346 ppm	0.1346 % SO ₂
5. Primary Absorber Outlet, SP (G3)	Rate	3674 ACFM	4.345 lb mol gas / min
	SO ₂	450 ppm	0.045 % SO ₂
30. Sulfite Absorber Feed, SP (A6)	Rate	0.9 gpm	0.9 gpm
	Total Sulfite	1.29 M	0.6882 lb SO ₂ / gal
	Sulfate	0.1 M	0.054 lb SO ₂ / gal
20. Secondary Absorber Bleed, SP (A3)	Rate	0.9 M	0.9 gpm
	Total Sulfite	1.85 M	0.9870 lb SO ₂ / gal
	Sulfate	0.57 M	0.3041 lb SO ₂ / gal

SO₂ Balance

Let:

$$I = [\text{Venturi Inlet Gas Rate, Stream 3}] \times [\text{Composition}]$$

$$= 3.983 \frac{\text{lb mol gas}}{\text{min}} \times 0.1346 (\% \text{SO}_2) \times 64.063 \frac{\text{lb SO}_2}{\text{lb mol SO}_2} = 0.3434 \frac{\text{lb SO}_2}{\text{min}}$$

$$II = [\text{Sulfite Absorber Feed Rate, Stream 30}] \times [\text{Composition}]$$

$$= 0.9 \frac{\text{gpm}}{\text{min}} \times (0.6882 + 0.0534) \frac{\text{lb SO}_2}{\text{min}} = 0.6674 \frac{\text{lb SO}_2}{\text{min}}$$

$$\text{III} = [\text{Primary Absorber Outlet Rate, Stream 5}] \times [\text{Composition}]$$

$$= 4.345 \frac{\text{lb mol gas}}{\text{min}} \times 0.045 \frac{\% \text{SO}_2}{\text{lb SO}_2 / \text{lb mol SO}_2} \times 64.063 \frac{\text{lb SO}_2}{\text{lb mol SO}_2} = 0.1256 \frac{\text{lb SO}_2}{\text{min}}$$

$$\text{IV} = [\text{Absorber Bisulfite Bleed Rate, Stream 20}] \times [\text{Composition}]$$

$$= 0.9 \frac{\text{gpm}}{\text{min}} \times (0.9870 + 0.3041) \frac{\text{lb SO}_2}{\text{gal}} = 1.162 \frac{\text{lb SO}_2}{\text{min}}$$

Around the system given in Figure B-1, the following SO₂ balance equation shall hold:

$$\text{I} + \text{II} = \text{III} + \text{IV} \quad \text{and}$$

$$\begin{aligned} \% \text{ close} &= \frac{[\text{III} + \text{IV}] - [\text{I} + \text{II}]}{[\text{I} + \text{II}]} \times 100\% \\ &= \frac{[0.1256 + 1.162] - [0.3434 + 0.6674]}{[0.3434 + 0.6674]} \\ &= 28.8\% \end{aligned}$$

Performance

Assuming the Venturi absorber did not remove any SO₂, the absorption efficiency may be calculated as follows:

$$\text{Absorption Performance} = \frac{\text{I} - \text{III}}{\text{I}} = \frac{.3434 - .1256}{.3434} = 63.4\%$$

Percent (%) SO₂ oxidized can be calculated from sulfate formation

$$\% \text{ SO}_2 \text{ oxidized} = \frac{[0.57 - 0.1] \left[\frac{80}{96} \right]}{1.29} = 30.4\%$$

This is to compare with 23% oxidation concluded during the parametric study (see 9.2 Parametric Testing Absorber Subsystem).

Extraction Subsystem

A schematic diagram of the extraction subsystem is given in Figure B-2.

FIGURE B-2 :: EXTRACTION SUBSYSTEM

Test Data

Test data taken around the extraction system on May 26, 8:00pm are presented in Table B3a and on 11:00am in Table B3b. These data are snap shot data taken almost simultaneously after a steady state of extraction operation was attained.

Unit Conversion

1ml organic = 1.236 mmols active organic

Relevant equation used include;

$$\begin{aligned} \text{Organic Composition (\%)} &= \% \text{ Loading} \times \frac{1.236}{100} \left(\frac{\text{mmols}}{\text{ml}} \right) \times 3.7854 \left(\frac{\text{L}}{\text{gal}} \right) \times 32 \left(\frac{\text{gms}}{\text{mol}} \right) \times \frac{2.2}{1000} \left(\frac{\text{lbs}}{\text{g}} \right) = \\ &= .003294 \times (\% \text{ Loading}) \text{ in lbs/gal S} \end{aligned}$$

Where % organic loading is defined as the total sulfite molarity actually found in the active organic divided by the theoretical maximal total sulfite molarity of active organic (1.2336).

$$\begin{aligned} \text{Aqueous Composition} &= 2.05 \left(\frac{\text{mol}}{\text{L}} \right) \times 3.7854 \left(\frac{\text{L}}{\text{gal}} \right) \times 32 \left(\frac{\text{gms}}{\text{mol}} \right) \times \frac{2.2}{1000} \left(\frac{\text{lbs}}{\text{g}} \right) = .2665 \times \left(\frac{\text{mols}}{\text{L}} \right) \text{ in } \frac{\text{lbs}}{\text{gal}} \end{aligned}$$

Table B-3a

EXTRACTION SUBSYSTEM 5-26-94

I. TEST CONDITIONS AND ANALYSIS

8:00 pm

GAS (0.4 MW EQUIVALENT)

1. Lean Organic Feed to M/S (44 = 35 = 71) Sample Point S8	a) Rate - GPM b) Temperature - F c) % Organic Loading d) % Aqueous Entrainment e) Viscosity - cP	FI-8571C TI-0935	0.4 82.1 0.08 0
2. Rich Organic Outlet from M/S (45) Sample Point S2	a) Rate - GPM b) Temperature - F c) % Organic Loading d) % Aqueous Entrainment e) Viscosity - cP	None None	0.4 39.2 0
3. Chilled Bisulfite Feed to M/S (27) Sample Point A16	a) Rate - GPM b) Temperature - F c) Total Sulfite moles/Liter d) NaHSO ₃ - moles/Liter e) Na ₂ SO ₄ moles/Liter f) % Organic Entrainment g) pH	FT-8526 TI-0527	0.77 76 2.05 1.76 1.06 0 5.3
4. Regenerated Sulfite to Centrifuge (47) Sample Point A4	a) Rate - GPM b) Temperature - F c) Total Sulfite moles/Liter d) NaHSO ₃ - moles/Liter e) Na ₂ SO ₄ moles/Liter f) % Organic Entrainment g) pH	None None	0.77 76 1.54 0.86 1.01 0 6.2
5. Regenerated Sulfite (85) Sample Point A5	a) Rate - GPM b) Temperature - F c) Total Sulfite moles/Liter d) Na ₂ SO ₄ moles/Liter e) % Organic Entrainment f) pH	None None	0.77 76 1.54 1.01 0 6.2
6. Aqueous/Organic Volumetric Ratio			1.93
7. Mixing Speed (Q-105)	RPM		480
8. Weir Height	inches		10.25

Table B-3b

EXTRACTION SUBSYSTEM

5-26-94

1. TEST CONDITIONS AND ANALYSIS

GAS (0.4 MW EQUIVALENT)				11:00am
1. Lean Organic Feed to M/S (44 = 35 = 71) Sample Point S8	a) Rate - GPM b) Temperature - F c) % Organic Loading d) % Aqueous Entrainment e) Viscosity - cP	FI-0571C TI-0935	0.4 81.2 0.37 6	
2. Rich Organic Outlet from M/S (45) Sample Point S2	a) Rate - GPM b) Temperature - F c) % Organic Loading d) % Aqueous Entrainment e) Viscosity - cP	None None	0.4 44.5 0	
3. Chilled Bisulfite Feed to M/S (27) Sample Point A16	a) Rate - GPM b) Temperature - F c) Total Sulfite moles/Liter d) NaHSO ₃ - moles/Liter e) Na ₂ SO ₄ moles/Liter f) % Organic Entrainment g) pH	FT-0526 TI-0527	0.7 80 2.05 1.79 1.02 0 3.2	
4. Regenerated Sulfite to Centrifuge (47) Sample Point A4	a) Rate - GPM b) Temperature - F c) Total Sulfite moles/Liter d) NaHSO ₃ - moles/Liter e) Na ₂ SO ₄ moles/Liter f) % Organic Entrainment g) pH	None None	0.7 80 1.64 0.92 1.05 0 6.2	
5. Regenerated Sulfite (85) Sample Point A5	a) Rate - GPM b) Temperature - F c) Total Sulfite moles/Liter d) Na ₂ SO ₄ moles/Liter e) % Organic Entrainment f) pH	None None	0.7 80 1.64 1.05 0 6.2	
6. Aqueous/Organic Volumetric Ratio			1.75	
7. Mixing Speed (Q-105)	RPM		480	
8. Weir Height	inches		10.25	

The units of data presented in Table B-3a are converted. Results are as follows:

Table B-4: DATA FOR EXTRACTION SYSTEM			
Stream		Original Units	Converted Units
71.	Lean Organic Feed to Q-105, Rate SP (S8) % Organic Loading	0.4 gpm 0.08	0.24 gpm, AO 2.6×10^{-4} lb S/gal, AO
27.	Bisulfite Feed to Q-105, Rate SP (A16) Total Sulfite Sodium Bisulfite Sodium Sulfate	0.77 gpm 2.05 M 1.76 M 1.06 M	N/A 0.546 lb S/gal, AO N/A 0.282 lb S/gal, AO
45.	Rich Organic from Q-105, Rate SP (S2) % Organic Loading	0.4 gpm 39.2 %	0.24 gpm, AO 0.129 lbs/gal, AO
47.	Regenerated Sulfite from Q-105, Rate SP (A4) Total Sulfite Sodium Bisulfite Total Sulfate	0.77 gpm 1.54 M 0.86 M 1.01 M	N/A 0.410 lb S/gal, AO N/A 0.269 lb S/gal, AO
Where AO = Active Organic			

Sulfur Balance

Let:

$$I = [\text{Lean Solvent Feed to Q-105, Rate}] \times [\text{Composition}]$$

$$= 0.24 \text{ (gpm, AO)} \times (2.6 \times 10^{-4} \text{ (lb S/gal, AO)}) = 6.24 \times 10^{-5} \text{ (lb S/min)}$$

$$II = [\text{Bisulfite Feed to Q-105, Rate}] \times [\text{Composition}]$$

$$= 0.77 \text{ (gpm)} \times (0.546 + 0.282 \text{ (lb S/gal)}) = 0.6376 \text{ (lb S/min)}$$

$$\text{III} = [\text{Rich Solvent from Q-105, Rate}] \times [\text{Composition}]$$

$$= 0.24 \quad \times \quad 0.129 \quad = 3.096 \times 10^{-2}$$

(gpm, AO) (lb S/gal, AO) (lb S/min)

$$\text{IV} = [\text{Regenerated Sulfite from Q-105, Rate}] \times [\text{Composition}]$$

$$= 0.77 \quad \times \quad (0.410 + 0.296) \quad = 0.5228$$

(gpm) (lb S/gal) (lb S/min)

The following Material Balance equation shall hold;

$$\text{I} + \text{II} = \text{III} + \text{IV}$$

$$\% \text{ Close} = \frac{[\text{III} + \text{IV}] - [\text{I} + \text{II}]}{[\text{I} + \text{II}]} \times 100\%$$

$$= 100 \times \frac{[3.096 \times 10^{-2} + 0.5228] - [6.24 \times 10^{-5} + 0.6376]}{[6.24 \times 10^{-5} + 0.6376]}$$

$$= 13.16\%$$

Performance

The performance can be calculated from both the organic data and the aqueous data.

From the organic data; performance is defined as the net loading of the organic after the extraction process divided by the theoretical net loading possible (i.e. 67%). Since the inlet and outlet flowrates are assumed to be the same, the performance can be calculated from the % organic loading data. Therefore;

$$\text{Performance} = \frac{[\text{Outlet} - \text{Inlet}] \% \text{ Organic Loading}}{67\%} \times 100 \quad (4)$$

$$= \frac{39.2 - 0.08}{67} \times 100 \quad (5)$$

$$= 58.4\% \quad (6)$$

From Aqueous data; performance is defined as the change in the amount of sodium bisulfite through the extraction system divided by the theoretical amount of bisulfite required to load the organic to 67% (i.e. 1.66 moles). Since the inlet and outlet flowrates of the aqueous solutions are assumed to be equal, the performance can be calculated from the bisulfite molarities given in Table I, with a basis of one liter.

$$\begin{aligned}
 \text{Performance} &= \frac{[\text{Outlet} - \text{Inlet}] \text{ moles of bisulfite}}{1.66} \times 100 \\
 &= \frac{1.76 - 0.86}{1.66} \times 100 \\
 &= 54.2\%
 \end{aligned}$$

Stripping Subsystem

A schematic diagram of the stripper subsystem is given in Figure 9-3.3.

Stripping Data

Test data taken on March 10, 1994 at 5:00pm are recorded in Table B5. These data are snap shot data taken around the stripper almost simultaneously after a steady state of the stripping system was attained.

Unit Conversion

Table B-6 presents stripping data of B-5 collected on March 10 at 5:00pm in converted units.

Table B-5

STRIPPER SUBSYSTEM

3-10-94

I. TEST CONDITIONS AND ANALYSIS

GAS (0.7 MW EQUIVALENT)			5:00p
1. Number of Stages			30
2. Stripper Steam Feed (67)	a) Rate - lb/hr b) Pressure - psi	FE-0667 PI-0667	114.2 0
3. Stripper Overhead Gas Return to NiMo (58)	a) Rate - ACFM b) Temperature - F c) Pressure - in. H ₂ O	None TI-0657	183
4. Rich Organic Feed (51) Sample Point S2	a) Rate - GPM b) Temperature - F c) % Organic Loading d) % Aqueous Entrainment e) Viscosity - cP	None TI-0651	0.7 206 38 2.3
5. Stripped Lean Solvent, V-104 (60) Sample Point S5	a) Rate - GPM b) Temperature - F c) % Organic Loading d) Na ₂ SO ₄ - moles/Liter e) % Aqueous Entrainment f) Viscosity - cP	TI-1226-1	0.7 140 5 1.1
6. Condensate Feed from NiMo (A)	a) Rate - GPM	FI-2098	0.18
7. Stripper Condensate Feed (35)	a) Rate - GPM b) Temperature - F c) H ₂ SO ₃ moles/Liter d) % Organic Entrainment	None None	
8. Stripper Overhead Gas Return to NiMo (58)	a) Rate - ACFM b) SO ₂ - ppm		
9. Aqueous Waste from Stripper Separator (61) Sample Point A11	a) Rate - GPM b) Total Sulfite - moles/Liter c) % Organic Entrainment	None	
10. Emulsion Formation			
11. Foam Formation			

Table B-6 DATA FOR STRIPPER SYSTEM

Stream		Original Units	Converted Units
Aqueous Feed to Stripper	Rate	0.2 gpm	N/A
51. Organic Feed to Stripper SP (S2)	Rate % loading	0.7 gpm, TO 38	0.42 gpm, AO 0.125 lb S/gal AO
59. Organic Outlet from Stripper SP (S5)	Rate % Loading	0.7 gpm, TO 5	0.42 gpm, AO 0.0165 lb S/gal AO
67. Steam Feed to Stripper	Rate	114.2 lb/hr	N/A

Material Balance and Others

For Stripper, material balance can't be examined because SO₂ emitted with steam at the top of the stripper was not measured. Other items of interest include: (1) Aqueous / Organic volumetric ratio to stripper and (2) Pounds of steam used per pound of SO₂ stripped.

The Aqueous / Organic volumetric ratio fed to the stripper was targeted at 0.25. Actual A/O ratio is:

$$\begin{aligned} A/O &= \frac{0.18 \text{ gpm water}}{0.7 \text{ gpm organic}} \\ A/O &= 0.26 \end{aligned}$$

It is of special interest to know how much steam is needed to strip SO₂ from the organic. To compute that steam requirement, the following data have been used.

$$\begin{aligned} \text{Steam used} &= \frac{114.2}{60} = 1.90 \text{ lb/min} \\ \text{SO}_2 \text{ stripped} &= .42 \left(\frac{\text{gal}}{\text{min}} \right) (1.25 - 0.165) \left(\frac{\text{lbs}}{\text{gal}} \right) \frac{64}{32} = .091 \text{ lb/min} \\ &\quad \left(\frac{\text{lb SO}_2}{\text{lb S}} \right) \end{aligned}$$

$$\therefore \text{lb steam / lbSO}_2 = \frac{1.16}{.091} = 12.7$$

Performance & Others

% SO₂ stripped can be calculated from organic loading decrease

$$\begin{aligned} \% \text{ SO}_2 \text{ Stripped} &= \frac{\text{Organic Loading (Inlet - Outlet)}}{\text{Organic Loading Inlet}} \times 100 \\ &= \frac{38 - 5}{5.1} \times 100 = 86.8\% \end{aligned}$$

The residual sulfur loading is of great interest since integrated test requires that the residual loading be below 10%. The residual sulfur loading is simply the % loading remaining in the organic after going through the steam stripper. On March 10 (5:00 pm) the residual loading was 5%.

Summation of Results

Table B-11 MATERIAL BALANCE & PERFORMANCE -- SUMMATION OF RESULTS			
Operation	Sulfur Material Balance	Performance	Others
Absorption June 13, 1994	28.8% close	63.4% removal	% SO ₂ oxidized: 30.4% residual S loading = 5%
Extraction			
May 26, 1994 8:00pm	13.16% close	58.4%, 54.2%	A/O = .23
May 26, 1994 11:00pm	6.25% close	65.9%, 52.4%	A/O = .26
Stripping			
March 10, 1994 5:00pm	—	86.8%	A/O = .26, 12.1 lb steam/lb SO ₂ released residual S loading = 5%

The above table shows that the material balance closes vary from 6.25% to 28.8%. While 28.8% is relatively on the high side, it is believed that large deviations in these instances are attributable to incidental imperfections in sample taken, sample analysis etc. rather than a reflection of inherent

deficiencies in operation or in analytical techniques. The material balances will progressively improve as operators gain more operating experience.

On performance, the absorber system shows a removal efficiency of 63.4%. As is discussed in subsection 8.1 under two stage absorption, the percent removal of SO₂ depends on the pH of the primary absorber recirculating liquid. To gain high efficiency, this pH must be maintained below 6.3. If pH was maintained at 5.72 - 5.74 then absorption efficiency will not be any higher than 63 - 64%. This perhaps explains why the absorption efficiency was not any higher. Unfortunately, it can't be confirmed because pH value for the recycling liquid in that experiment was not recorded.

Extraction efficiency for Mixer - Settler has been found to be lower than expected. The design features of mixer - settlers should be carefully reviewed to see whether it can be upgraded in future installations.

Stripper data was taken early on, i.e. on March 19, 1994. At that time the stripper still functioned moderately well. It had a stripping efficiency of 86.8% with a residual loading of 5% at a steam rate of 12.1 lb steam/lb SO₂ stripped.

APPENDIX C
EQUIPMENT LIST

APPENDIX C

EQUIPMENT LIST

<u>Acct No.</u>	<u>Equipment Item and Description</u>	<u>Qty</u>
B-101	Stripper HP Regenerator Tray Tower - 3 Trays 316 L S.S. 12-3/4" O.D. x 13'-6"	1 Ea
C-101	I.D. Fan 50 HP Motor 316 S.S. Fan Blade Designed for 20" Pressure Drop/8,800 ACFM @ 180°F	1 Ea
C-102	Sulfate Filter Vacuum Pump Included in Sulfate Filter Package	1 Ea
C-103	SO ₂ Product Gas Fan Centrifugal Blower 10 ACFM 1/2 HP Motor	1 Ea
E-101	Bisulfite Solution Fd Cooler Plate Heat Exchanger 7.75 ft ² Surface 316 S.S. Plates, Nitrile Gaskets	1 Ea
E-102	Glycol Refrigeration Chiller 2-35 HP Compressors 200 gpm Cooling Water	1 Ea
E-103	Regen. Lean Solvent Cooler Plate Heat Exchanger 7.56 ft ² Surface 316 S.S. Plates, Nitrile Gaskets	1 Ea
E-104	Lean Solvent Feed Cooler Plate Heat Exchanger 4.3 ft ² Surface 316 S.S. with Nitrile Gaskets	1 Ea
E-105	Rich Solvent Feed Cooler Plate Heat Exchanger 4.3 ft ² Surface 316 S.S. with Nitrile Gaskets	1 Ea

EQUIPMENT LIST (CONTINUED)

<u>Acct No.</u>	<u>Equipment Item and Description</u>	<u>Qty</u>
E-106	Stripper Outlet Gas Precooler Plate Heat Exchanger 3.61 ft ² Surface 316 S.S. Plate with EPDM Gaskets	1 Ea
E-107	Stripper Outlet Gas Condenser Plate Heat Exchanger 9 ft ² Surface 316 S.S. Plate with Nitrile Gaskets	1 Ea
E-101	Mixer-Settler No. 1 Agitator Clamp-On Design 0.25 HP 316 Stainless Steel	1 Ea
M-102	Mixer-Settler No. 2 Agitator Clamp-On Design 0.25 HP 316 Stainless Steel	1 Ea
M-103	Mixer-Settler No. 3 Agitator Clamp-On Design 0.25 HP 316 Stainless Steel	1 Ea
M-104	Mixer-Settler No. 4 Agitator Clamp-On Design 0.33 HP 316 Stainless Steel	1 Ea
M-105	Mixer-Settler No. 5 Agitator Clamp-On Design 0.33 HP 316 Stainless Steel	1 Ea
M-106	Sulfate Crystallizer Tank Agitator Clamp-On Design 0.33 HP 316 Stainless Steel	1 Ea
M-107	Soda Ash Tank Agitator Clamp-On Design 1/2 HP 316 Stainless Steel	1 Ea

EQUIPMENT LIST (CONTINUED)

<u>Acct No.</u>	<u>Equipment Item and Description</u>	<u>Qty</u>
M-108	Purge Neutralization Tank Agitator Clamp-On Design 0.33 HP PVC Coated S.S. Shaft	1 Ea
P-101A&B	Venturi Recycle Pump 82 gpm 6-3/4" Diameter Impeller Worthite 20 Coated Steel 1.5 HP	2 Ea
P-102	Venturi Blowdown Pump 0.5 gpm 4-3/4" Diameter Impeller Worthite 20 Coated Steel 0.25 HP	1 Ea
P-103A&B	SO ₂ Absorber Recycle Pump 109 gpm 4-1/4" Diameter Impeller 316 S.S. 5 HP	2 Ea
P-104	Fly Ash Filter Wash Pump 0.002 gpm 316 S.S./TFE Air Operated Diaphragm Chemical Injection Pump	1 Ea
P-105	Bisulfite Solution Feed Pump 316 S.S. 5-1/4" Diameter Impeller 0.5 HP	1 Ea
P-106	Sulfate Crystallizer Feed Pump 316 S.S. 5-1/4" Diameter Impeller 0.5 HP	1 Ea
P-107	Sulfate Filter Feed Pump 316 S.S. 5-1/4" Diameter Impeller 0.5 HP	1 Ea

EQUIPMENT LIST (CONTINUED)

<u>Acct No.</u>	<u>Equipment Item and Description</u>	<u>Qty</u>
P-108	Sulfate Filtrate Pump 316 S.S. 5-1/4" Diameter Impeller 0.5 HP	1 Ea
P-109	Glycol Recycle Pump 316 S.S. 5-1/4" Diameter Impeller 0.5 HP	1 Ea
P-110	Glycol Chiller Feed Pump 316 S.S. 5-1/4" Diameter Impeller 0.5 HP	1 Ea
P-111	Regen. Sulfite Transfer Pump 316 S.S. 5-1/4" Diameter Impeller 0.5 HP	1 Ea
P-112	M/S Separator Solvent Return Pump 316 S.S. 5-1/4" Diameter Impeller 0.5 HP	1 Ea
P-113	M/S Separator Regen. Sulf. Pump 316 S.S. 5-1/4" Diameter Impeller 0.5 HP	1 Ea
P-114	Sulfite Absorber Feed Pump 316 S.S. 5-1/4" Diameter Impeller 0.5 HP	1 Ea
P-116	Regen. Lean Solvent Pump 316 S.S. 5-1/4" Diameter Impeller 0.5 HP	1 Ea

EQUIPMENT LIST (CONTINUED)

<u>Acct No.</u>	<u>Equipment Item and Description</u>	<u>Qty</u>
P-117	Lean Solvent Feed Pump 316 S.S. 5-1/4" Diameter Impeller 0.5 HP	1 Ea
P-118	Rich Solvent Transfer Pump 316 S.S. 5-1/4" Diameter Impeller 0.5 HP	1 Ea
P-119	Rich Solvent Stripper Feed Pump 2.6 gpm 4-1/4" Diameter Impeller 316 S.S. 0.5 HP	1 Ea
P-120	Lean Solvent Stripper Outlet Pump 316 S.S. 5-1/4" Diameter Impeller 0.5 HP	1 Ea
P-121	Stripper Regen. Bleed Pump 316 S.S. 5-1/4" Diameter Impeller 0.5 HP	1 Ea
P-123	Makeup Solvent Feed Pump 316 S.S. 5-1/4" Diameter Impeller 0.5 HP	1 Ea
P-124	Condensate Pump 316 S.S. 5-1/4" Diameter Impeller 0.5 HP	1 Ea

EQUIPMENT LIST (CONTINUED)

<u>Acct No.</u>	<u>Equipment Item and Description</u>	<u>Qty</u>
Q-101	Mixer-Settler No. 1 30" x 27" x 5' Epoxy Coated 316 S.S.	1 Ea
Q-102	Mixer-Settler No. 2 30" x 27" x 5' Epoxy Coated 316 S.S.	1 Ea
Q-103	Mixer-Settler No. 3 30" x 27" x 5' Epoxy Coated 316 S.S.	1 Ea
Q-104	Mixer-Settler No. 4 30" x 27" x 5' Epoxy Coated 316 S.S.	1 Ea
Q-105	Mixer-Settler No. 5 30" x 27" x 8' Epoxy Coated 316 S.S.	1 Ea
Q-106	Sulfate Crystallizer Tank Covered Tank Multiple Bleed Points 3.9' Diameter x 5' High 316 S.S., 11 Ga	1 Ea
S-101	Absorber Bisulfite Bleed Filter Duo/Fabric Basket Filter On-Line Washing Capability Polypropylene Baskets 316 S.S. Container	1 Ea
S-103	Sulfate Filter Pan Vacuum Filter Includes: 3' x 3' S.S. Pan Polypropylene Cloth 3 HP Vacuum Pump 3/4 HP Filtrate Pump Receiver	1 Ea

EQUIPMENT LIST (CONTINUED)

<u>Acct No.</u>	<u>Equipment Item and Description</u>	<u>Qty</u>
S-104	Stripper Regenerator Bleed Separator 24" x 96" Column 316 L S.S. Includes 25" Sight Glass	1 Ea
S-105	Lean Solvent/Sulfite Separator 12" x 48" Column 316 L S.S. Includes 25" Sight Glass	1 Ea
S-106	Soda Wash Separator 36" x 144" Column 316 L S.S. Includes 25" Sight Glass	1 Ea
S-107	Sulfite Centrifuge High Speed Centrifuge 1 HP TEFC Drive Motor Liquid/Liquid Separator	1 Ea
S-108	Solvent Centrifuge High Speed Centrifuge 1 HP TEFC Drive Motor Liquid/Liquid Separator	1 Ea
S-109	Centrifuge Extractor (1 Month Rental) 12.5 HP Motors Batch or Continuous Operation 25-30 gpm Maximum Feed Rate	1 Ea
T-101	Lean Solvent Storage Tank 4.7' Diameter x 5' High Covered Tank 316 S.S., 11 Ga	1 Ea
T-102	Bisulfite Storage Tank 5.2' Diameter x 6' High Covered Tank 316 S.S., 11 Ga	1 Ea
T-103	Glycol Storage Tank 8' Diameter x 8' High Covered Tank 316 S.S., 10 Ga	1 Ea

EQUIPMENT LIST (CONTINUED)

<u>Acct No.</u>	<u>Equipment Item and Description</u>	<u>Qty</u>
T-104	M/S Sulfite/Solvent Separator Tank 5' Diameter x 5' High Covered Tank 316 S.S., 11 Ga	1 Ea
T-105	Sulfite Storage Tank 3.9' Diameter x 5' High Covered Tank 316 S.S., 11 Ga	1 Ea
T-106	Lean Solvent Soda Wash Tank 6.8' Diameter x 8' High Covered Tank 316 S.S., 11 Ga	1 Ea
T-107	Soda Ash Storage Tank 3.5' Diameter x 4' High Covered Tank 316 S.S., 11 Ga	1 Ea
T-108	Venturi Purge Neutralization Tank 2.6' Diameter x 3' High Covered Tank FRP, 3/16" Thick	1 Ea
T-109	Rich Solvent Storage Tank 5' Diameter x 5' high Covered Tank 316 S.S., 11 Ga	1 Ea
T-110	Condensate Tank 2.5' Diameter x 3' High Covered Tank 316 S.S., 11 Ga	1 Ea
T-111	N ₂ Tank & Distribution Manifold (Rental) Dewar Tanks (3) Liquid N ₂	1 Ea
V-101	Venturi Prescrubber Prescrubber/Quencher Rubber-Lined Carbon Steel Manually Adjustable Inconel Throat	1 Ea

EQUIPMENT LIST (CONTINUED)

<u>Acct No.</u>	<u>Equipment Item and Description</u>	<u>Qty</u>
V-102	SO ₂ Absorber 316 L S.S. Header and Packed Tower No. 2 Jaeger Tri-Paks 6" Mesh-Type Polypropylene M.E.	1 Ea
V-103	Sulfate Filtrate Receiver Included in Sulfate Filter Package	1 Ea
V-104	SO ₂ Steam Stripper Tray Tower - 30 Trays 316 L S.S. 12-3/4" O.D. x 53'-6" Column	1 Ea

APPENDIX D

ANALYTICAL PROCEDURES

APPENDIX D

ANALYTICAL PROCEDURES

- I Rich Organic Loading Analysis**
- II Organic Extraction Equilibrium Curve Determination**
- III Ion Chromatography - Sulfite and Sulfate Analysis**
- IV Viscosity Measurement**
- V Entrainment Measurement**

PROCEDURE 1 RICH ORGANIC LOADING ANALYSIS

SAMPLES: S2 AND S3

Reagents:

- 0.1N I₂ solution
- 0.1N Sodium thiosulfate solution
- 2N NaOH
- 2N H₂SO₄
- Starch Indicator
- Distilled H₂O

Procedure:

1. Centrifuge Sample for 15 min (under N₂)
2. Into a small wash bottle (30 ml) place 20 ml of 2N NaOH. Add a few mls of it into 60 ml separatory funnel.
3. Transfer 1 ml of sample into the separatory funnel and wash all of organic out of pipette with 2N NaOH in wash bottle. Add remaining NaOH to separatory funnel. Shake well for 1 min. Allow to settle 1 min.
4. Into an Erlenmeyer flask containing 100 ml distilled H₂O, drain 19 ml 0.1N I₂ solution (assuming that organic is approximately 70% loaded). Add 25 ml H₂SO₄ to the flask just prior to sample addition.
5. Drain all of the soda wash directly in the flask. If mixture is not amber color, add more I₂. Mix well.
6. Add several drops of starch indicator and titrate with 0.1N sodium thiosulfate until color disappears.

$$\text{mmols } \text{SO}_3^- = \frac{\text{Net ml } I_2 \text{ Soln} \times 0.1}{2}$$

Amine = 60/40 mixture

60% is active amine

2.06 X 60% = 1.236

correct for 1 ml of active amine

$$\text{mmols } \text{SO}_3^- = \frac{\text{Net ml } I_2 \times 0.1}{2 \times 1.236} \quad (1)$$

PROCEDURE II ORGANIC EXTRACTION
EQUILIBRIUM DETERMINATION

Reagents:

- 0.1N I_2 Solution
- 0.1N Sodium Thiosulfate Solution
- 2N NaOH
- 2N H_2SO_4
- Sodium Meta bisulfite ($Na_2S_2O_5$)
- Starch solution
- Distilled H_2O

Procedure:

1. Centrifuge about 15 ml of organic sample (under N_2) for 15 min (unless you are using fresh organic).
2. Into a N_2 purged 60 ml separatory funnel; transfer 7 ml of the centrifuged organic. Keep the N_2 purging during entire experiment whenever sample is exposed to atmosphere.
- 3.* Prepare a fresh 2N $NaHSO_3$ solution just prior to each extraction. Use 1.69 grams of sodium meta bisulfite, and 8.9 ml of distilled H_2O .
4. To a 250 ml erlenmeyer flask containing 100 ml distilled H_2O add by burette 22 ml 0.1N I_2 solution. Add 2 ml 2N H_2SO_4 to the Flask. Transfer 0.5 ml of the aqueous solution (From Step 3, or from Step 7). Solution should be amber in color.
5. Titrate with 0.1 N Sodium Thiosulfate (use starch indicator if desired).
6. Under N_2 purge add the aqueous solution remaining from step 3 to the organic and shake vigorously for 1 min. Allow to settle for 2-3 minutes.
7. Drain the aqueous off and analyze 0.5 ml as in Steps 4 and 5 (see chart on page 2 for approximate amount of I_2 to be used). Discard the remaining aqueous.

* Power Sorb must be used to analyze stripped samples.

8. Place 20 ml of 2N NaOH into a small wash bottle (30 ml bottle). Transfer about 2-3 ml of the NaOH into a 60 ml separatory funnel. Transfer 1 ml of the organic by pipette into the funnel and wash all of the organic out of it with the 2N NaOH in the wash bottle. Add the remaining NaOH to the separatory funnel. Shake well for 1 min. Allow to settle for 1 min.
9. Into an erlenmeyer flask containing 100 ml distilled H₂O, drain 0.1N I₂ solution. (See chart at end for approx. amount). Add 25 ml H₂SO₄ to the flask just prior to sample addition. Drain all of the soda wash directly into the flask. Solution should be amber color. Mix well.
10. Add several drops of starch indicator and titrate with 0.1N sodium thiosulfate until color disappears.
11. Repeat steps 3 - 10 two more times or until organic is fully loaded.
12. Plot the curve on graph paper.

$$X = \frac{\text{Aqueous Solution Sulphur Loading Concentration after extract}}{\text{Aqueous Solution Sulphur Loading Concentration before extract}}$$

Y = Organic Sulphur Loading

(See Calculations on next page)

Approximate amounts of I₂ to be placed in Erlenmeyer flask for each analysis after extraction.

Extraction	Aqueous (0.5 ml)	Organic (1 ml)
	0.1N I ₂	0.1N I ₂
1	15 ml	18 ml
2	18 ml	24 ml
3	19 ml	27 ml

PROCEDURE III ION CHROMATOGRAPHY - SULFITE AND SULFATE DETERMINATION

OPERATING INSTRUCTIONS

1. Turn on the nitrogen cylinder outside the trailer. Make sure the pressure gauge is between 90 and 120 psi.
2. Turn on the Ionic Chromatograph. Make sure the bottles are full with eluant. Set the High Limit Alarm off by pressing the 'Hi Limit' button once.
3. Open the front door and turn on the nitrogen switch to ON.
4. Set the pressure gauge between 5 and 10 psi, inclusive.
5. Open the bleed valve 3 turns.
6. Turn on the pump. Wait for 2 or 3 minutes.
7. Close the bleed valve. Press the 'Auto Offset' button to ON. Press the 'Low Limit' button once to ON. Press and hold the 'Hi Limit' button until the indicator is at about 3000. Check that the pressure is at about 1500 psi by pressing the 'psi' button. If the conductivity reading is not 0, then press the 'Auto Offset' button, wait a few seconds, and press it again. Repeat this until the reading is 0.
8. Inject the sample through the syringe port. Make sure the 'LOAD' indicator button is ON; otherwise, press the 'Sample' button once.
9. Type 'FI = X*' on the integrator, and hit the <ENTER> key.
10. Press the 'Sample' button on the IC, and the <Inj A> key on the integrator at the same time.

* The value of X depends on what is being analyzed

X = 1 for 0.1 to 3.0 ppm Sulfate
X = 3 for 1 to 20 ppm Sulfite

X = 2 for 1 to 20 ppm Sulfate
X = 4 for 0.1 to 3 ppm Sulfite

$$\% \text{ Organic Sulfite Loading} = \left(\frac{\text{ppm reading} \times DF}{1.236 \times 80 \times 1000} \right) \times 100$$

$$\text{Molarity} = \frac{\text{ppm reading} \times DF}{MW \times 1000}$$

Sulfite MW = 80
Sulfate MW = 96

Sulfite Analysis (Samples A11 and A13)

DF (Dilution Factor) = 500

FI = 4

Sulfate Analysis (All Samples)

DF (Dilution Factor) = 5000 (ie. 0.2 in 1000).

FI = 2

Table D-1 Sulfate Concentration vs. Area Count

Range = 3 (File = 1)

CONC	AREA
0.08	885046
0.2	1317988
0.4	1988392
1	3579093
2	7175764
3	10795116

CORRELATION COEFFICIENT FACTOR: 0.9989

Table D-2 Sulfate Concentration vs. Area Count

Range = 10 (File = 2)

CONC

AREA

CORRELATION COEFFICIENT FACTOR: 0.9997

1	1080367
2	2128218
3	3222031
4	4167970
10	10528987
20	22111910

Table D-3 Sulfate Concentration vs. Area Count

Range = 10 (File = 3)

CONC	AREA
1	860633
2	1147257
3	1681152
5	2768949
10	5542193
20	11362381

CORRELATION COEFFICIENT FACTOR: 0.9994

Table D-4 Sulfate Concentration vs. Area Count

Range = 3 (File = 4)

CONC	AREA
0.1	318097
0.2	339868
0.4	782498
1	1993962
2	3568236
3	5125584

CORRELATION COEFFICIENT FACTOR: 0.9985

PROCEDURE IV VISCOSITY MEASUREMENT
All Organic Samples

Viscosity by Brookfield Viscometers

1. Place a sample in a 75 ml cup
2. Get a reading from the viscometer using spindle No. 2. If reading is less than 10, get another reading using spindle No. 1.

Viscosity (centiPoise)

= Reading x "Factor in Slide Ruler" x "Spindle Correction Factor"

Spindle No. 2 Correction Factor = 0.793

Spindle No. 1 Correction Factor = 0.818

3. Results of viscosity measurements at site are presented in the following figure. They check fairly closely to the viscosities measured by Hazen Research Corporation, Golden, CO as shown in Figure 6.2.

Viscosity By Drainage

1. Fill a one millimeter pipet with the loaded organic of which the viscosity is to be determined.
2. Lift the pipet from the liquid and place it in touch with the wall of a beaker. Let the liquid drain down from the one millimeter mark to empty. Record the time duration. More viscous liquid will take longer to drain to empty.

PROCEDURE V ENTRAINMENT MEASUREMENT

Organic or aqueous samples may contain entrained aqueous or organic species, respectively. This entrainment may be related to process operating conditions. Therefore, these samples will periodically be analyzed for the percent by volume of the entrainment using the following procedure:

Reagent: None

Procedure:

1. Add 10 ml sample (either aqueous or organic) in a graduated centrifuge tube.
2. Centrifuge for 15 minutes (or longer if necessary, but if longer time is used, note the time duration).
3. Read the organic volume as well as the aqueous volume.

Computation:

Organic Volume = X ml Organic entrainment = $X \times 10\%$

Aqueous Volume = Y ml Aqueous entrainment = $Y \times 10\%$