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INTER-OFFICE MEMORANDUM

SAVANNAH RIVER PLANT

DPSPU-83-272-143

RECORDS ADMINISTRATION



AEKD

October 11, 1983

TO: J. T. BUCKNER, JR., 704-F

FROM: H. P. HOLCOMB, 772-F *HPH*

MINIMIZING SOLIDS IN FERROUS SULFAMATE SOLUTIONS

SUMMARY

Ferrous sulfamate solutions are used at SRP to selectively adjust the oxidation state of plutonium to +3 to facilitate its purification from other elements. A study by the Separations Technology Laboratory of the solids occurring in process ferrous sulfamate (FS) solutions and the mechanism for their formation has concluded with recommendations for minimization of these iron-bearing solids.

The solids are produced as a result of dissolved oxygen or added nitric acid oxidizing ferrous ion (Fe^{+2}) to ferric ion (Fe^{+3}). Ferric ion in the minimally acidic solution hydrolyzes and eventually precipitates as the jellylike hydrous ferric oxide. Other evidence indicates that over a longer term, insoluble complex salts containing ammonium, ferric, and sulfate ions are also forming. These ions are products of FS or sulfamic acid decomposition and crystallize in tankage as stoichiometric compounds over a longer time frame than that necessary for hydrous ferric oxide to form. The latter can appear in as little as 48 hours.

Recommendations offered to minimize solids formation are directed toward reducing the dissolved oxygen content of FS solutions and to maximize other conditions that increase FS stability. Suggestions include employing inert gas blankets over stored solutions, inert gas sparges of solutions used to prepare FS, solution temperature control, and improved filtration systems for removing solids. Examination of these recommendations for cost effectiveness and ease of implementation will be the next phase of this program.

J. T. BUCKNER, JR.

Page 2

October 11, 1983

INTRODUCTION

Solids occurring in cold feed FS cause process difficulties from time to time. Such problems are primarily hydraulic in nature and result in downtime because of plugged valves, lines, rotameters, and filters. Thus far, no evidence exists for the FS solids directly causing hydraulic problems in the "hot" portions of the process where FS is used as a plutonium reducing agent.

Samples of solids from both F-Area and H-Area FS cold feed tanks were obtained and analyzed. Also, the FS vendor, Southwestern Analytical Chemicals, Austin, TX, was visited. The results of that visit are described in the Appendix of this document. The FS shipment that arrived at SRP on 9/17/83 was examined on loading at the vendor on 9/15. Samples of the 50% FS solution were taken for analysis and experimentation. FS preparation, shipment, and the solids problem were discussed with Brian Hale of the vendor's staff. Although this shipment of FS was solids-free at the time of loading, hydrous ferric oxide solids were present in the tank trailer on arrival at SRP 48 hours later because of oxidation and hydrolysis.

This document discusses the results of the analyses and tests performed on the solids and solutions, explains the chemistry involved in the formation of the solids, and makes recommendations to minimize the solids and alleviate the problems they cause.

DISCUSSION

Types of Solids

Two distinct types of solids have been identified as occurring in cold feed FS solutions. Samples of solids taken from Tank 272 (FS receipt and adjustment tank) in F-Area and from Tank 13D (1AS cold feed) in H-Area were crystalline in nature. Samples of solids from vendor FS on receipt at SRP contained a different type of solid, an amorphous gel. The crystalline solids, being older, slowly ingrew as complex salts containing components resulting from FS and sulfamic acid (SA) degradation. The gel solid was formed during the 48-hour transport of FS from the vendor to SRP and, therefore, was much younger in age. However, the amorphous gel on aging and dehydration gives rise to one of the crystalline solids. Analyses and tests identifying these solids and leading to the foregoing conclusions follow.

J. T. BUCKNER, JR.

Page 3

October 11, 1983

Old Solids

Crystalline solids were found in FS samples taken from Tank 272 in F-Area and from Tank 13D in H-Area in August. These samples underwent testing and analysis to identify the solids.

Characteristics of the Solids. Tank 272 solids were a light yellow-brown color and composed of small particles. Those from Tank 13D were larger chunks of dark brown crystals. Neither solid was readily soluble in water or 6M nitric acid. However, 6M hydrochloric acid produced slow dissolution with the appearance of the characteristic yellow color of the ferric chloride complex.

Analyses of the Solids. A portion of each solid was thoroughly washed with deionized water and then dried at 60°C for about 6 hours. Each dried solid was divided into three portions: one for x-ray diffraction analysis, another for emission spectrographic analysis, and the third was equilibrated with deionized water with the subsequent solids-free supernate being submitted for ion chromatographic analysis for anions. Results of these analyses are summarized in Table 1.

TABLE 1

ANALYSIS OF OLD SOLIDS FROM COLD-FEED FS

	<u>Tank 272 Solids</u>	<u>Tank 13D Solids</u>
<u>X-Ray Diffraction</u>		
Major component	KFe ₃ (SO ₄) ₂ (OH) ₆ was best powder pattern fit, but instead of K ⁺ , cation could be NH ₄ ⁺ or Na ⁺ .	Fe ₂ O ₃ ·H ₂ O
Minor component	Difficult to identify	NH ₄ Fe ₃ (SO ₄) ₂ (OH) ₆
Elements detected by XRF	Fe, S	Fe, S
<u>Emission Spectroscopy</u>		
Major	Fe, Si	Fe
Minor	Mo	Cr, Mo, Si, Sn, Ti, Mn
<u>Ion Chromatography</u>		
Anions detected	NH ₂ SO ₃ ⁻ , NO ₃ ⁻ , SO ₄ ⁻²	NH ₂ SO ₃ ⁻ , NO ₃ ⁻ , SO ₄ ⁻²

J. T. BUCKNER, JR.

Page 4

October 11, 1983

The supernate over the Tank 272 solids sample was amber in color, unlike the emerald green of fresh, pure FS. However, the solution still maintained a high reducing normality that met specifications. Filtration of this supernate through a 0.45 μ m porosity membrane filter yielded no residue on the filter indicating that the discoloration, probably ferric ion in small concentration, is in solution and not a finely divided solid or gel therein.

Analysis and Test Conclusions. These data verify that the crystalline solids contain components of FS degradation as well as sulfamic acid decomposition. The solids, relatively insoluble in the FS solution, crystallize and grow into quantities that can be detected on solution sampling. Such crystalline growth requires time not only for formation but also for production of the precursory ionic components. Therefore, solids such as these old ones only appear in aged FS solutions or as ingrowths in equipment extensively exposed to FS solutions. It is unlikely that the old solids are significant contributors to the filter pluggage experienced in process operations. If they were, the frequency of hydraulic upsets in cold feed should have been on the increase.

New Solids

The new solids, identified as hydrous ferric oxide (or ferric hydroxide) are the prime cause of the hydraulic difficulties encountered with the cold feed FS solutions. These new solids are formed as a result of two chemical reactions. The first is oxidation of the ferrous ion to ferric. The second reaction is the hydrolysis of ferric ion producing the gel precipitate in the weakly acidic solution. On aging, the amorphous, jellylike hydrous ferric oxide undergoes partial dehydration to produce crystalline ferric oxide monohydrate, $\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$, identified as the major component of Tank 13D solids.

New Solids Occurrence. A description of the ferrous sulfamate logistics can be found in the Appendix of this document. Hydrous ferric oxide can form with relative rapidity in low acid solutions containing ferrous or ferric ions. For example, the shipment of 50% FS arriving SRP on 9/17, contained hydrous ferric oxide solids although it was solids-free 48 hours earlier on departure from the vendor. The pH of that solution was 1.60 with a free acidity of 0.11M. Similarly, a sample of Laboratories Department prepared reagent FS formed hydrous ferric oxide within 48 hours of makeup. The pH of this solution was 2.35 and free acidity only 0.02M. Addition of 50% nitric acid to a pH of 1.00 in either solution partially dissolved the solids. However, the gel solids can reform, albeit at a much slower rate as described in the chemistry section of this document. The reformed gel solids are causing pluggage of the H-Area cold feed filter.

J. T. BUCKNER, JR.

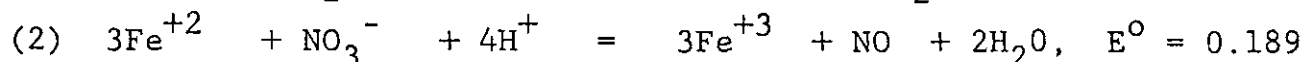
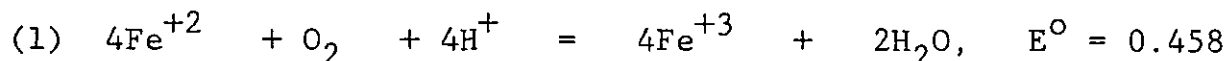
Page 5

October 11, 1983

SOLIDS FORMATION CHEMISTRY

Decomposition of Ferrous Ion - Oxidation to Ferric Ion

Ferrous ion is oxidized in these solutions by either dissolved oxygen or by nitric acid. The reactions involved are the sum of the respective half-cell reactions²:

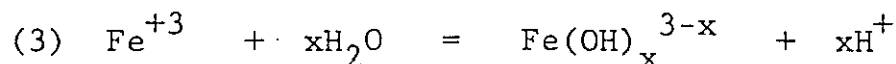


Once ferric ion is produced, it can hydrolyze and precipitate in solutions of low acidity as discussed in the following section. Kinetic data³ show that reactions (1) and (2) are very temperature and acid dependent. Both dependencies are first-order with relation to ferrous ion oxidation. These data indicate that a tripling of the reaction rate occurs with an increase in temperature from 25°C to 35°C at a given acidity. Similarly, increases in nitric acid concentration in the solution at constant temperature promote a marked increase in ferrous ion oxidation. Therefore, increased temperature and increased nitric acid concentration of FS solutions exacerbate FS decomposition and subsequent solids formation.

To put the effect of the dissolved oxygen into perspective, in water equilibrated with air⁴, there is 2.4×10^{-4} moles oxygen (STP) per liter. If the vendor-supplied 50% FS is considered to have the same dissolved oxygen capacity, then there is enough dissolved oxygen to produce 9.6×10^{-4} moles Fe^{+3} per liter of solution according to equation (1) above. From a laboratory experiment, it was determined that 10 mg of iron as Fe^{+3} produces 0.5 mL of settled hydrous ferric oxide. Therefore, the 725 g Fe^{+3} that could be produced by dissolved oxygen in a 45,000 lb shipment from the vendor would generate 36 liters of gel precipitate if all the dissolved oxygen reacted. Because of the equilibrium situation produced, it would not, but this exercise does provide some indication of the solids-forming capability of the oxygen in the FS solutions.

Hydrolysis of Ferric Ion - Precipitation

Once ferric ion is formed in solution, it can hydrolyze according to the following equation:



The hydrous ferric oxides produced are insoluble and precipitate from solution as an amorphous gel, $\text{Fe}(\text{OH})_3 \cdot x\text{H}_2\text{O}$. Ferric hydroxide is quite insoluble, $K_{sp} = 6 \times 10^{-38}$.⁵ Reactions (1) or (2) plus (3) thus give rise to the new solids.

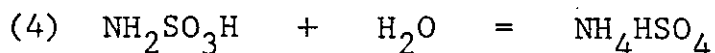
J. T. BUCKNER, JR.

Page 6

October 11, 1983

Formation of Old Solids - Hydrolysis of Sulfamic Acid

The old crystalline solids are produced from reactions (1), (2), and (3) as well as from the following reaction, the hydrolysis of sulfamic acid⁶:

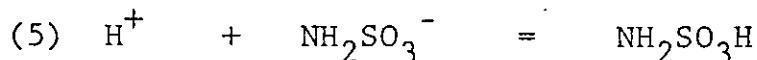


Kinetic data⁷ indicate that at 20°C, this reaction is almost negligible over an 8 month period. However, at 80°C, about 45% of a 1M sulfamic acid solution is hydrolyzed in 8 hours. Vendor supplied FS solutions contain varying quantities of excess sulfamic acid. The acid hydrolyzes on exposure of the solution to elevated temperatures as would occur in Tank 272 during summer. The bisulfate ion and ammonium ion result. Bisulfate ion, being a proton donor and therefore acidic, undergoes ionization or deprotonation, forming sulfate ion. Eventually, these ions, together with ferric ions, react to form the insoluble, crystalline complex compound identified in equipment dedicated to FS storage or use. The compound has the formula, $\text{NH}_4\text{Fe}_3(\text{SO}_4)_2(\text{OH})_6$.

The other old solid, ferric oxide monohydrate, has already been described as forming by gradual dehydration of the new solid, hydrous ferric oxide gel, over a period of time.¹

CHEMISTRY TO MINIMIZE SOLIDS FORMATIONAddition of Acid to FS Solutions

Equation (3) equilibrium can be shifted to the left to maintain ferric ion solubility by the addition of acid. However, the presence of acid, especially nitric, promotes oxidation of ferrous ion as evidenced by equations (1) and (2). Adding acid also drives the ionization of sulfamic acid ($K_i = 0.1^8$) toward the unionized form, as:



Sulfamic acid (SA) is the least soluble sulfamate except for stannous and basic mercury salts of sulfamic acid.⁹ Experiments in the Separations Technology Laboratory have shown that SA crystallization occurs between pH 1 and 0.5 on addition of nitric acid to 40% FS. At a pH of 0.5, extensive SA solidification occurs.

Therefore, the quantity of nitric acid added to FS to minimize hydrolysis must be judiciously chosen so as not to be detrimental from promoting both oxidation of ferrous ion and crystallization of SA. Vendor-added acid could reduce solids formation during transport.

J. T. BUCKNER, JR.

Page 7

October 11, 1983

Further experimentation has shown that excess SA can be added to FS solutions to lower pH and minimize hydrous oxide formation. In this manner, acidity can be increased without addition of an oxidant. For example, 5 mL of aqueous, saturated SA (2.3M) added to 25 mL of 50% FS, as received, lowered the pH from 1.60 to 1.05 and stabilized the solution toward gel formation for a period of at least 12 days. A 50% FS solution similarly adjusted with nitric acid to the same pH remained without solids during this period but had begun to change noticeably to a duller green during the same 12-day period. Lowering the pH even further, by use of either SA or nitric acid would be detrimental because of lowered SA solubility and its crystallization from solution.

Therefore, based on the foregoing experiments, the minimum pH to minimize solids formation in the FS should be in the 0.9-1.0 pH range.

Exclusion of Oxygen in FS Solutions

Dissolved oxygen is the primary oxidant causing ferrous ion decomposition. If oxygen can be minimized in FS solutions, then subsequent solids formation will be accordingly minimized. In order to accomplish this, aqueous solutions used to prepare FS should be sparged with an inert gas, such as nitrogen, to reduce the dissolved oxygen content. An alternative would be to thoroughly sparge, with inert gas, FS solutions immediately after preparation or adjustment. Caution must be maintained to prevent loss of water, the solvent, to the vapor phase if sparging is vigorous and/or lengthy. Such loss would cause decreased solute solubility, and solids would then form if solvent so lost were not replenished.

Current vendor shipping practice is to load approximately 3000 gal of 50% FS into a 6000-gal capacity tank trailer. During travel, sloshing of the tank contents occurs, keeping the FS at a maximum equilibrium level of dissolved oxygen. Such a condition promotes oxidation. Shipment should be made in a smaller vessel, or the FS shipment should be blanketed with inert gas prior to departing the vendor.

If SRP continues to adjust 50% FS on receipt from the vendor, then the adjustment solutions will also require sparging to reduce their oxygen content prior to their addition to FS. The FS could possibly be sparged with inert gas following their addition to accomplish the same purpose.

FS storage tanks at SRP have an air freeboard above the liquid level. Once oxygen content of the solution is lowered, an inert gas blanket (N_2 , CO_2) must be maintained over the solutions to ensure continued reduced oxygen levels. If the inert gas blanket is not maintained, the stored FS solutions will reequilibrate with the air freeboard, and the dissolved oxygen content will increase.

J. T. BUCKNER, JR.

Page 8

October 11, 1983

Temperature Control of Stored FS Solutions

Extensive data ^{3, 6} are available to verify the effect of increased temperature on FS and sulfamic acid decomposition. To minimize solids formation, the temperature of stored FS solutions should be maintained in the 20-25°C range. Low temperatures cannot be tolerated even though oxidation and hydrolysis rates would be lessened. Temperatures too cool will reduce the solubility of FS and SA, causing them to crystallize from solution. Tank 272, the primary cold feed FS storage tank in F-Area, is equipped with cooling coils, but they are not piped up for cooling service at present. This and other FS storage tanks exposed to the gamut of uncontrolled environmental temperatures would require some means of temperature control to prevent the effects of too high or too low thermal conditions.

RECOMMENDATIONS TO MINIMIZE SOLIDS FORMATION IN FERROUS SULFAMATE

Based on the information gained as a result of this study of the FS solids problem, the following are recommended as being positive actions to take to minimize FS solids:

- Inert gas sparge of solutions used to prepare or to adjust FS, or
- Inert gas sparge FS solutions immediately following preparation or adjustment.
- Employ inert gas blanket over stored FS solutions.
- Control temperature of stored FS solutions.
- Vendor adjust acid content prior to FS shipment.
- Optimize shipping conditions.
- Examine 222-F filtration system and modify if necessary.
- Conduct study to determine optimum nitric acid concentration in FS solutions of varying concentration. Determine feasibility of using only sulfamic acid for acidification purposes.

FUTURE PROGRAM

Each of the foregoing recommendations should be reviewed on the

J. T. BUCKNER, JR.

Page 9

October 11, 1983

bases of both cost effectiveness and production impact. Those that are deemed to provide a positive, workable solution to the solids problem should then be implemented.

ACKNOWLEDGMENTS

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J. T. BUCKNER, JR.

Page A-1

October 11, 1983

APPENDIX A

FERROUS SULFAMATE LOGISTICS

Figure 1-A is a block diagram of the way in which ferrous sulfamate solutions are received and used at SRP. In essence, it is a summary of the following discussion.

Vendor

50 wt% ferrous sulfamate solution is supplied to SRP by Southwestern Analytical Chemicals, Inc., of Austin, TX. Shipments of approximately 45,000 lb (3000-3500 gal) are delivered by tank trailer to SRP every 4-8 weeks, depending on production requirements. In 1982, 477,100 lb of 50% FS was received. The vendor was visited by C. W. Jenkins and the author on September 15, 1983. The results of that visit are described in Appendix B.

Receipt and Adjustment of 50% FS

The solution is unloaded at 222-F via pump. The solution passes through a filtering system (wound synthetic fiber cartridges) and then into Tank 272. During unloading, the solution is sampled downstream of the filtration system for essential materials specification analysis. After unloading and agitating in Tank 272, the solution is again sampled. Tank 272 has a maximum capacity of approximately 79,000 lb. Therefore, with no heel, a single shipment will fill the tank slightly over half full, leaving a significant air space over the solution.

The 50% solution is then adjusted in Tank 272 with process water and 50% nitric acid to yield a solution that is 40% FS and 2% nitric acid. Following adjustment, the solution is again sampled to ascertain if it meets operating specifications.

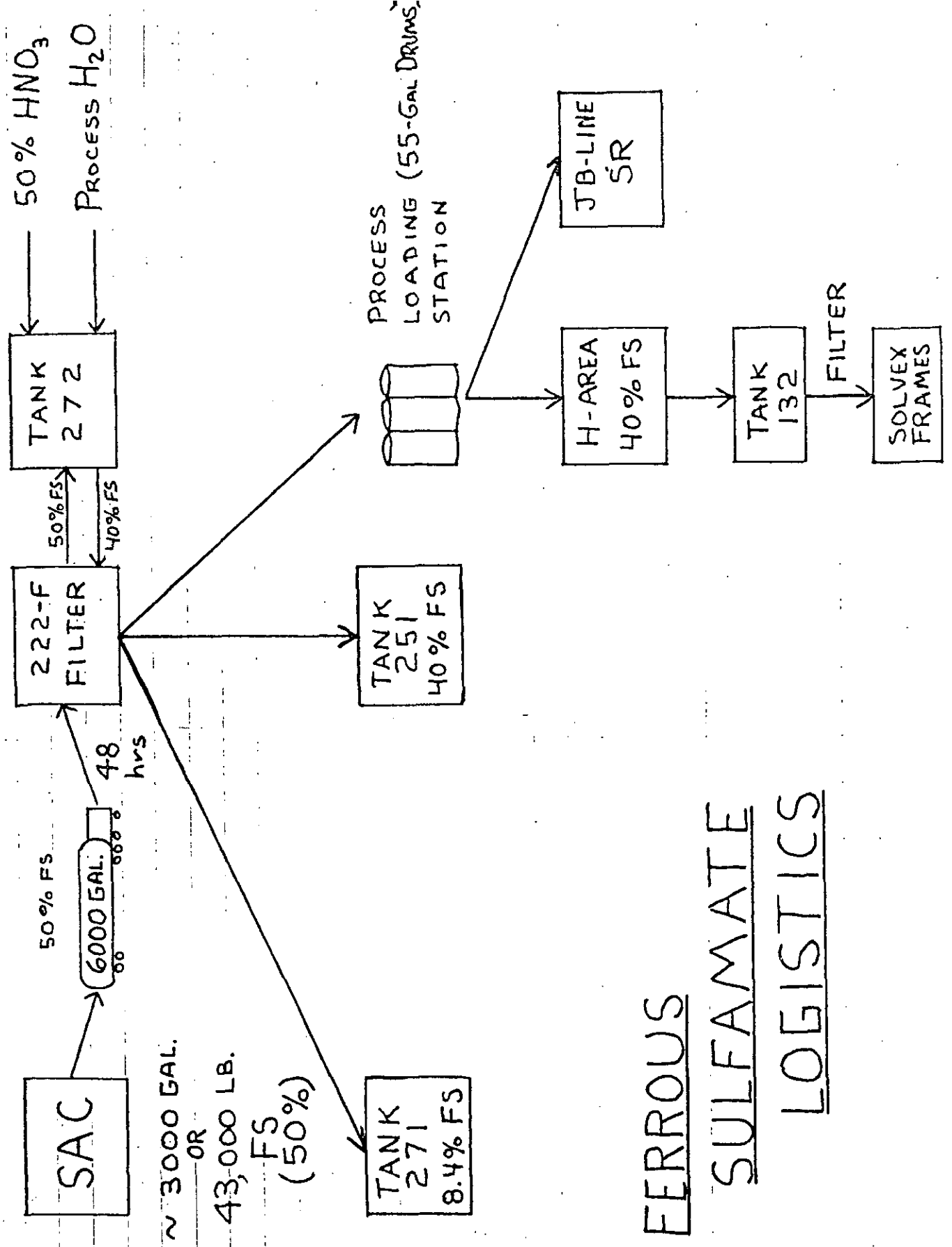
FS Uses

From Tank 272, 40% FS can go in three directions:

1. It can be transferred through the filtering system into tank 251 at 222-F. Tank 251 is the 40% FS hold tank for F-Area processing. From Tank 251 the solution is transferred without filtering to the canyon cold feed tanks.
2. The solution can be transferred, with filtration, to Tank 271 where the FS is adjusted to 8.4% for use in first cycle and second uranium cycle solvent extraction.

FERROUS SULFAMATE LOGISTICS

FIGURE 1-A



J. T. BUCKNER, JR.

Page A-3

October 11, 1983

3. With filtering, the solution can be transferred to the process loading station at 222-F for filling of 55-gal drums that go to JB-Line Special Recovery or to H-Area for use in solvent extraction and in frames processing. Tank 132 in H-Area is the primary 40% FS receipt tank. Tank 13D, where old solids were found in a sample, is downstream of this tank in the canyon.

Occurrence of Solids

New solids, hydrous ferric oxide, have been found in the tank trailer as received from the vendor and in Tank 272 after unloading and passing through the 222-F filtration system. These solids are also speculated to be the cause of filter pluggage in H-Area that resulted in two days processing downtime.

The very slightly soluble crystalline solids (old solids) have been detected only in samples from Tanks 272 (F-Area) and Tank 13D (H-Area). Their occurrence elsewhere in the FS system is quite probable.

APPENDIX B

VISIT TO SOUTHWESTERN ANALYTICAL CHEMICALS, FS VENDOR

Two Separations Technology personnel, C. W. Jenkins and the author, visited Southwestern Analytical Chemicals, Austin, TX, the FS vendor on September 15, 1983. While there, discussions were held with Mr. Brian Hale of their staff. We also toured their facility and observed the loading of the 50% FS shipment received at SRP on 9/17.

SAC was observed to be a competent firm concerned with the quality of their products. Their facility was relatively neat and orderly. Three tanks are dedicated to FS production: a 1200-gal fiberglass-lined S/S dissolver and two fiberglass storage tanks (3000-gal and 500 gal). Some 2+ dissolver batches are necessary to prepare a single 3000-gal shipment for SRP.

Their raw materials for FS preparation consist of 99.5+% iron powder from Amax or Hoegenese and 99+% sulfamic acid crystals from Nissan Chemical. They have tried both du Pont and Taiwanese supplied sulfamic acid, but these suffered from increased levels of impurities.

J. T. BUCKNER, JR.

Page A-4

October 11, 1983

Preparation of a batch of FS takes place over a 5-day period. The initial reaction is made by charging the dissolver with water and crystalline sulfamic acid. Before dissolution takes place, the first of three iron powder charges is made. In this manner, the exothermic iron-sulfamic acid reaction is countered by the endothermic dissolution of sulfamic acid. Temperature during reaction reaches a maximum of 45°C. The solution and solids are mechanically agitated. There is no sparge nor purge. Iron addition takes place over a 3-day period. Then the final solution is stirred and allowed to cool over a 2-day period. The batch is filtered through a Sparkler Leaf Filter (<10 µm porosity rating) using HiFlo (diatomaceous earth) as a filtration aid to remove the reaction solids, primarily carbon from the iron powder. The as-filtered FS is then kept in one of the two storage tanks until shipment. The storage tanks have no temperature control although they are inside the SAC building.

Loading for shipment consists of pumping the 50% FS through a PVC hose into a clean and dry 6000-gal tank trailer. On the end of the hose is clamped a 10 µm rated porosity bag filter. After loading, the bag filter was examined and found to contain an estimated 100-150 g of rusty red solids. The initial phase of loading was observed through the tank hatch. The FS was clear in appearance, emerald-green in color. A dip sample pulled following loading was normal in appearance except for a very slight haze that was probably due to entrained air from loading. Transport time for the tank trailer, slightly over half full, to SRP is 48 hours. A larger shipment is not possible due to highway weight constraints.

Discussions with Brian Hale of SAC indicated that the only possible area of concern was in the quantity of sulfamic acid used to prepare the FS. SAC may need to add more in order to increase acidity although their measurement of the 9/15 shipment showed a pH in the 0.9-1.0 range. Two independent SRP measurements on the same material agreed at pH 1.60. This pH measurement discrepancy is under mutual investigation as well as a cooperative effort to improve shipping conditions. Adjustment of acidity by SAC with sulfamic and/or nitric acid prior to FS shipment may alleviate hydrous ferric oxide formation during transport. The prime constraint for their doing this is their current lack of FS storage space (3500 gal maximum).

Contact with the vendor shall continue.

HPH/h