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Technical Note

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FINAL FEASIBILITY REPORT ON CHEMICAL TREATMENT OF SODIUM NITRITE WASTEWATER

ABSTRACT This report on the sodium nitrite wastewater treatment process discusses the results of 12 simulation runs and six test runs using the boiler hydroblasting wastewater from the Long Beach Naval Shipyard (LBNSY). Reproducible results were obtained showing the total destruction of sodium nitrite by sulfamic acid in Navy boiler hydroblasting wastewater. The removal of heavy metals was equally successful, an approach which resulted in reducing nearly all the ions to the discharge limits by EPA standards. The sludge contained 30 percent solids by weight and passed the TCLP test required for disposal. The estimated cost of treatment remains under \$0.30 per gallon compared with the 1990 contract haul cost of \$2.00 per gallon.

NAVAL CIVIL ENGINEERING LABORATORY PORT HUENEME CALIFORNIA 93043-5003

METRIC CONVERSION FACTORS

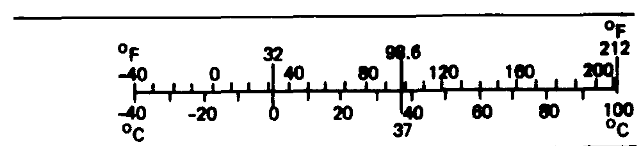
Approximate Conversions to Metric Measures

Symbol	When You Know	Multiply by	To Find	Symbol
		LENGTH		
in	inches	*2.5	centimeters	cm
ft	feet	30	centimeters	cm
yd	yards	0.9	meters	m
mi	miles	1.6	kilometers	km
		AREA		
in^2	square inches	6.5	square centimeters	cm^2
ft^2	square feet	0.09	square meters	m^2
yd^2	square yards	0.8	square meters	m^2
mi^2	square miles	2.6	square kilometers	km^2
	acres	0.4	hectares	ha
		MASS (weight)		
oz	ounces	28	grams	g
lb	pounds	0.45	kilograms	kg
	short tons (2,000 lb)	0.9	tonnes	t
		VOLUME		
tsp	teaspoons	5	milliliters	ml
Tbsp	tablespoons	15	milliliters	ml
fl oz	fluid ounces	30	milliliters	ml
c	cups	0.24	liters	l
pt	pints	0.47	liters	l
qt	quarts	0.95	liters	l
gal	gallons	3.8	liters	l
ft^3	cubic feet	0.03	cubic meters	m^3
yd^3	cubic yards	0.76	cubic meters	m^3
		TEMPERATURE (exact)		
°F	Fahrenheit temperature	5/9 (after subtracting 32)	Celsius temperature	°C

*1 in = 2.54 (exactly). For other exact conversions and more detailed tables, see NBS Misc. Publ. 286, Units of Weights and Measures, Price $2.25, SD Catalog No. C13.10:286.

Approximate Conversions from Metric Measures

Symbol	When You Know	Multiply by	To Find	Symbol
		LENGTH		
mm	millimeters	0.04	inches	in
cm	centimeters	0.4	inches	in
m	meters	3.3	feet	ft
m	meters	1.1	yards	yd
km	kilometers	0.6	miles	mi
		AREA		
cm^2	square centimeters	0.16	square inches	in^2
m^2	square meters	1.2	square yards	yd^2
km^2	square kilometers	0.4	square miles	mi^2
ha	hectares (10,000 m^2)	2.5	acres	
		MASS (weight)		
g	grams	0.035	ounces	oz
kg	kilograms	2.2	pounds	lb
t	tonnes (1,000 kg)	1.1	short tons	
		VOLUME		
ml	milliliters	0.03	fluid ounces	fl oz
l	liters	2.1	pints	pt
l	liters	1.06	quarts	qt
l	liters	0.26	gallons	gal
m^3	cubic meters	35	cubic feet	ft^3
m^3	cubic meters	1.3	cubic yards	yd^3
		TEMPERATURE (exact)		
°C	Celsius temperature	9/5 (then add 32)	Fahrenheit temperature	°F



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This report on the sodium nitrite wastewater treatment process discusses the results of 12 simulation runs and six test runs using the boiler hydroblasting wastewater from the Long Beach Naval Shipyard (LBNSY). Reproducible results were obtained showing the total destruction of sodium nitrite by sulfamic acid in Navy boiler hydroblasting wastewater. The removal of heavy metals was equally successful, an approach which resulted in reducing nearly all the ions to the discharge limits by EPA standards. The sludge contained 30 percent solids by weight and passed the TCLP test required for disposal. The estimated cost of treatment remains under $0.30 per gallon compared with the 1990 contract haul cost of $2.00 per gallon.

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EXECUTIVE SUMMARY

This study on the sodium nitrite wastewater treatment process contained 12 simulation runs and six test runs using the boiler hydroblasting wastewater from the Long Beach Naval Shipyard (LBNSY). Reproducible results were obtained showing the total destruction of sodium nitrite by sulfamic acid in Navy boiler hydroblasting wastewater. The removal of heavy metal ions by sodium hydroxide precipitation was equally successful, an approach which resulted in reducing nearly all the ions to the discharge limits by EPA standards. The sludge, filtered out by cartridge contained 30 percent solids by weight, passed the Toxicity Characteristic Leaching Procedure (TCLP) Test required for disposal.

A small temperature rise of 2°F in the 100-gallon batch reactor tank was theoretically predicted and experimentally observed (Appendixes A and B).

It was not necessary to use excessive amounts of sulfamic acid above the stoichiometric ratio to achieve the total conversion of sodium nitrite to nitrogen. However, the addition of sulfamic acid to the reactor and the subsequent mixing rate must be slow enough to avoid the possible formation of NO_x. Since the generation of pure nitrogen is by no means a speedy reaction, as evident from free energy calculations, a batch-wise process is therefore recommended for full-scale production. The estimated cost of treatment remains under $0.30 per gallon compared with the 1990 contract haul cost of $2.00 per gallon at LBNSY.

Contents

INTRODUCTION

Navy shipyards generate sodium nitrite wastewater from three sources: (1) boiler hydroblast cleaning, (2) boiler lay-up, and (3) boiler hydroleak testing. Nitrite wastewater is considered hazardous by the National Pollutant Discharge Elimination System (NPDES) because it contributes to the eutrophication of the surface water streams. Simple oxidation of nitrite to nitrate, as by air blowing, is not acceptable since the formed nitrate is also eutrophic. Although the Environmental Protection Agency (EPA) has not set up a sewer discharge limit for nitrite in wastewater, many local governments have adopted the EPA's intermediate drinking water standard to limit nitrogen content in wastewater discharge, which is 10 mg/L (ppm); which is equivalent to 33 ppm of nitrite. However, Navy shipyard boiler wastewater usually contains around 800 ppm of nitrite (or 1,200 ppm of sodium nitrite). At the present time, the contractors and the public-owned treatment works (POTWs) have no effective treatment method for the total conversion of nitrite in the boiler wastewaters to nitrogen gas.

In addition to sodium nitrite, the waste stream also includes various heavy metals in ionic form. The heavy metal ions, namely, cadmium, copper, nickel, chromium, lead, and zinc, are regulated by the EPA and several states as toxic wastes.

When boiler nitrite wastewater is allowed to mix with other wastes in the ship's bilges, the contractor disposal charge is about $3.25/gallon. If the sodium nitrite streams are segregated from other wastes in the ship's bilges, the disposal cost is about $2.00/gallon.

The Naval Facilities Engineering Command (NAVFAC) tasked the Naval Civil Engineering Laboratory (NCEL) to investigate sodium nitrite wastewater treatment technologies. NCEL laboratory studies (Ref 1) conducted in 1990 showed that sulfamic acid administered at a stoichiometric dosage is capable of completely eliminating nitrite through denitrification (conversion to nitrogen gas). Based on the positive and reproducible results of the laboratory studies, a 100-gallon bench process was successfully tested during 1991 at California State Polytechnic University, Pomona, California. The test results showed that the chemical process can completely convert the nitrite ion, successfully remove heavy metals, and reduce sludge. The treated wastewater meets the NPDES requirements for discharging to the sewer.

Basically, this process involves a three-step procedure: (1) the reduction of both nitrite and nitrate (if any) content in the wastewater by sulfamic acid, (2) the precipitation of heavy metal ions by sodium hydroxide, and (3) the separation of suspended solids and sludge which are reduced by settling and filtering. The chemical equations are shown below:

$$NO_2^- + NH_2SO_2OH \text{ ------> } N_2 \uparrow + HSO_4^- + H_2O \qquad (1)$$

$$(Me)^{++} + 2X(OH)^- \text{ ------> } Me(OH)_2 + 2X \qquad (2)$$

Since sulfamic acid is a strong reducing agent, any nitrate ion present is first reduced to nitrite before being further reduced to inert nitrogen gas (Ref 2). That is:

$$NO_3^- + 2H^+ + 2e^- \text{----->} NO_2^- + H_2O \qquad (3)$$

According to a Russian study (Ref 3), there is an unstable intermediary compound existing between nitrite ion and sulfamic acid before the release of nitrogen gas; that is:

$$NaNO_2 + NH_2SO_2OH \text{<---->} HO\text{-}SO_2\text{-}N_2\text{-}OH + Na^+ + OH^- \qquad (4)$$

This phenomenon (which was not noticeable in the previous laboratory study) probably offers the most plausible explanation of a slower formation of nitrogen gas in a larger reactor tank.

Based on the reproducible data obtained in the laboratory study, the objectives of this bench scale study were as follows:

1. Determine the operating characteristics of treating boiler nitrite wastewater and heavy metal ions in a 100-gallon reactor tank.

2. Evaluate the parametric effects, particularly the mixing speed and sulfamic acid reaction rate, in a 100-gallon reactor tank.

3. Observe any temperature increase other than that theoretically predicted.

4. Confirm if the stoichiometric ratio of sulfamic acid to nitrite concentration behaves identically in the large reactor tank as in a smaller 500-mL flask. Verify absence of nitrate ion.

5. Determine the quantity of sludge and metal oxide precipitate from the treated liquor to meet State and County landfill requirements.

6. Design a pilot-plant process capable of treating up to 500 gallons of Navy boiler sodium nitrite wastewater per day.

BENCH SCALE PROCESS DESCRIPTION

A block flow diagram of the bench scale process is presented in Figure 1. The process is divided into the following four unit operations:

1. Denitrification

2. Metal Precipitation

3. Sludge Dewatering

4. Neutralization

For each unit operation, temperature and pH were measured manually using thermometers and pH papers, respectively. Each of the unit operations is discussed below.

Denitrification

A 175-gallon conical bottom polyethylene tank with 1-inch discharge polypropylene valves served as the denitrification reactor. A slow-speed (220-rpm) mixer with custom-made, large propellers was inserted through the open-mouth top into the tank at an angle to ensure the wastewater was well mixed with no dead spots or short circuiting. Figure 2 shows the reactor tank with the mixer in place. Once 100 gallons of wastewater was transferred to the reactor tank, the pH and the nitrite ion reading (using a Hach colorwheel kit) was determined. Then the sulfamic acid solution was metered into the tank in a stoichiometric ratio with the nitrite concentration. In order to avoid the evolution of N_2O_3 indicated by the brown color, the acid solution was added slowly to the wastewater. The reduction of the nitrite in the reactor was monitored by measuring the temperature, pH, and nitrite concentration throughout the process.

The denitrification process is complete when the nitrite is totally reduced to nitrogen gas.

Metal Precipitation

After the reduction of nitrite was complete, the same conical bottom tank was used for metal precipitation. Fifty percent sodium hydroxide was added to the tank until the pH increased to between 9 and 11. The pH of the wastewater was maintained within this range for the metal oxides to precipitate without using any flocculants. After the mixer was turned off, approximately 2 hours were needed to allow the flocs to settle.

Sludge Dewatering

Metal oxide precipitate and solid particles in the raw wastewater were filtered out and collected by using a single or successive multiple filter cartridges of a 5- to 10-micron pore size. The filter cartridges were placed in a vacuum oven at a moderate temperature for drying and determining the weight percentage of sludge. Metal ion determinations, as well as solid toxicity tests, were all performed by an EPA-approved laboratory in Chino, California.

Neutralization

After completing the transfer of the sludge, the treated wastewater remaining in the reactor tank was transferred to a 100-gallon tank for neutralization purposes. The pH of the water was lowered to between 6 and 9 by adding 20 percent sulfuric acid in order to meet discharge standards. The pH and temperature were monitored before releasing the treated wastewater to the test site industrial sewer system.

Table 1 illustrates a sample data sheet for the denitrification and demetalization process.

TEST RESULTS

There were two groups of tests in the bench study. The first group of tests included 12 simulation runs using synthetic solutions. The second group of tests included six runs using Long Beach Naval Shipyard (LBNSY) boiler nitrite wastewater. Tables 2 and 3 contain the tabulations of these test results, respectively. Table 4 illustrates the experimental matrix and Figure 3 shows the sulfamic acid requirements for total reduction of sodium nitrite for the 12 simulation runs. Figure 4 shows the sulfamic acid requirements for total reduction of sodium nitrite for actual boiler nitrite wastewater.

Total Sodium Nitrite Destruction

The bench scale study further confirmed the results obtained from earlier laboratory studies, which consistently demonstrated that the sulfamic acid sodium nitrite reaction is stoichiometric. The stoichiometric sulfamic acid requirement for nitrite ion reduction to nitrogen based on a 100-gallon solution volume is shown in Figure 5, which can be used as a conversion plot for the convenience of the operator. The detailed calculations and examples are shown in Appendix A. Additional amounts of sulfamic acid, between 5 to 10 percent, were required for the complete reduction of sodium nitrite to nitrogen due to the competition of nitrate ion (in the actual boiler wastewater) and the interference of metal ions (in the case of simulation runs (Ref 3)).

In evaluating the overall study, one may note the pretreatment and post-treatment results of Navy No. 4 Run. As shown in Table 3 and Appendix C, a sodium nitrite concentration of 1,680 ppm was successfully reduced to zero ppm.

Heavy Metal Ion Removal

The results in Tables 2 and 3 show that nearly all the metal ions were removed using sodium hydroxide (NaOH) solution to adjust the pH to 11. The requirement of NaOH is linearly proportional to the metal ion concentration in the wastewater. During the course of the experiment, it was also learned that the time allowed to precipitate played an important role. At least 2 hours were needed for the precipitate to settle. The discharge limit for zinc ion in Los Angeles County sewers is 1.49 ppm. Therefore, the results do not present any liquid disposal problem. The most encouraging run was Navy No. 4, which contained thick waste liquor pumped out of the bottom of several barrels. Not only was the metal ion content in the wastewater extremely high, but it also contained minute metal particles, sludge, oil film, and it was quite odorous (see Appendix C). The post-treatment results revealed that cadium, chromium (total), lead, and nickel (initially 34.0 ppm) were all reduced to nondetectable limits, whereas copper (from 28.8 ppm to 0.17 ppm) and zinc (from 28.0 ppm to 0.04 ppm), as shown in Table 3, were all within the discharge limits.

Sludge Disposal

One of the bench study objectives was to test the possibility of landfilling the sludge resulting from precipitation of metal hydroxides and the solids from the original wastewater.

A Toxicity Characteristic Leaching Procedure (known as a TCLP Test - EPA Standard) was performed. The results indicated that from the eight constituents considered to be toxic (namely, arsenic, barium, cadmium, chromium (total), lead, mercury, selenium, and silver), all were nondetectable except arsenic at 0.009 ppm (detection limit 0.002 ppm), and selenium at 0.03 ppm (detection limit 0.002 ppm). Neither of these two ion concentrations exceeded the most stringent discharge limits.

CONCLUSIONS

In view of the foregoing technical results and the summary data presented herein, it can be concluded that:

1. The stoichiometric ratio between sodium nitrite and sulfamic acid is the same in a 100-gallon reactor tank as in a 500-mL flask. In the presence of nitrate and reducible metal ions, additional sulfamic acid demand occurs but this does not disturb the stoichiometry of the nitrite/sulfamic reaction.

2. The addition of sulfamic acid solution and the subsequent mixing rate must be slow enough to accommodate the generation and bubbling of nitrogen gas in the 100-gallon reactor tank.

3. The sodium hydroxide requirement for precipitation of metal ions is linearly proportional to the metal ion concentration in the wastewater, particularly *in the regions of high* and low metal ion concentration.

4. The time for metal oxide precipitation should be at least 2 hours in a 100-gallon capacity reactor.

5. The chemistry involved in both sodium nitrite and heavy metal ion removal is identical regardless of the reactor size.

RECOMMENDATIONS

Because of the intermittent production of nitrite wastewater and relatively slow chemical reaction, it is recommended that a batch-wise process of multiple reactors would be more suitable for a full-scale production facility as shown in Figure 6.

However, before a full-scale operational facility can be successfully designed, a subscale pilot plant with a capacity of 500 gallons per day for the denitrification of sodium nitrite wastewater followed by the precipitation of heavy metals and the collection of the resulting metal hydroxide sludge should be first tested. A proposed pilot-scale nitrite reduction process is shown in Figure 7. The design basis for the pilot system is outlined in Table 5.

After successfully implementing the NCEL hydroblast recycling process, NCEL has estimated the total volume of sodium nitrite wastewater generated by all Naval shipyards to still be about 3 million gallons each year, and by Navy-wide boiler maintenance operations to be 10 million gallons per year. The proposed chemical denitrification process has the potential of reducing the disposal cost by at least 85 percent (reduced from $2.00/gallon to $0.30/gallon operating cost) or $5M savings per year for Naval shipyards and $17M savings per year for the Navy-wide boiler maintenance operations.

The proposed chemical process will not produce hazardous waste and the effluent produced can be safely discharged to the sanitary sewer.

REFERENCES

1. Naval Civil Engineering Laboratory. Memorandum to files on the initial feasibility study on treatment of sodium nitrite wastewater from Naval Shipyards, by B.Y.K. Pan and Andy Law. Port Hueneme, CA, May 1990.

2. V.P. Razygraev and M.V. Lebedeva. "The influence of some secondary reactions of the oxidizing-reconstructing potential and corrosion processes in nitric-acid environment," Academic Acta of USSR, vol 8, no. 8, 1982.

3. Yu Kostrikin and O.V. Teterina. "Rate of reaction of nitrites with sulfamic acid," Energetik, vol 10, no. 22, 1987.

Table 1. Denitrification and Demetalization Data Sheet

Run No. ________ Date____/____/199__
Volume Treated ______ gallon Type______________

I. Wastewater

1. NO_2^- = _______ppm

2. Cd ______ ppm, Cr ______ ppm, Cu ______ ppm, Fe ______ ppm
Ni ______ ppm, Pb ______ ppm, Zn ______ ppm

3. (a) Temp = _____°F or (______°C) (b) pH = ______

II. Wastewater Treatment

5. Sulfamic acid added = _______g = _______g-mol (per tank)

6. Rate of addition = _______g/min. Acid/NaNO2 (Mole) ratio _____

7. (a) Temp = _____°F or (_____°C) (b) pH = ______

III. Unreacted Nitrite:

8. NO_2^- = _______ppm

IV. Solution Treatment (for demetalization)

9. NaOH (______%); ______mL (per tank)

10. Time (to bring to 9 <pH <11) = _____ min

V. Solution: pH = 9 to 11

11. (a) Temp = _______°F or (_______°C) (b) pH = ______

12. Cd ______ ppm, Cr ______ ppm, Cu ______ ppm, Fe ______ ppm
Ni ______ ppm, Pb ______ ppm, Zn ______ ppm

VI. Solution Treatment (for neutralization)

13. H_2SO_4 (_____%); _____mL (per tank)

14. Time (to bring to 6 <pH <8) = _____ min

VII. Solution: pH = 6 to 8

15. Cartridge (after) = ______g 16. (a) Temp = ____°F or (____°C)
Cartridge (before) = ______g (b) pH = ____
Residue = ______g

Remarks____

Recorded by___________________________________

Table 2. Simulation Run Data

Denitrification and Demetalization Data Sheet

Simulation No.	$NaNO_2$	Cd 213*	Cr 218*	Cu 220*	Fe 236*	Pb 239*	NI 249*	Zn 289.1*	Sulfamic Acid Added (gram)	pH	Nitrite Post Treatment	NaOH Added (50%) mL	pH	Sulfuric Acid (95%) mL	pH	Total Suspended Solids (ppm)
	Wastewater Characteristics								Wastewater Treatment Data							
1	650	0	0	0	0	0	0	0	694	2	ND	271			7	
2	1600	0	0	0	0	0	0	0	1836	2	ND	1200		160	7.5	
3	920	0	0	0	0	0	0	0	736	2.5	ND	700			8	
4A	1020	0.05	0.03	29.9	24.6	0.75	1.66	1.82	848	2	ND	900	12	800	7	
4B		0.01	ND	0.04	ND	ND	ND	0.07								220
5A	1600	0.04	0.14	17.2	6	0.78	1.23	0.92	1266	2	ND	1200	12	500	6	
5B		0.01	ND	0.02	ND	0.08	0.05	0.03								151
6A	900	0.04	0.37	26.7	10.7	2.68	1.35	0.05	717	2.5	ND	500	10	155	7.5	
6B		ND	ND	ND	ND	ND	ND	ND								236
7A	600	0.03	0.38	18.3	8.14	1.68	0.87	1.06	478	2.8	ND	375	11	45	7.8	
7B		ND	ND	ND	ND	ND	ND	ND								203
8A	1200	0.05	1.38	37	17.5	3.43	2.19	2.36	956	2.5	ND	800	12	153	7	
8B		ND	ND	ND	ND	0.35	ND	0.1								
9A	600	0.07	0.13	22.1	11.1	2.32	1.19	1.66	508	2	ND	350	11	75	5	
9B		ND	ND	ND	ND	ND	ND	0.02								262
10A	1200	0.02	0.12	19.4	11.7	26.4	1.31	1.86	1050	2	ND	650	11	120	6	
10B		ND	ND	ND	ND	ND	ND	ND								236
11A	900	0.1	0.02	12.2	7.77	1.69	0.9	1.26	788	2	ND	540	11	70	6.5	
11B		0.01	ND	ND	ND	ND	ND	0.02								198
12A	900	0.04	0.2	24.5	11.9	3.32	1.73	1.76	792	1.5	ND	600	12	100	7	
12B		ND	ND	ND	ND	ND	ND	0.2								

*EPA procedure.

Note: The unit of figures shown above are ppm (mg/L).
Run number with "A" stands for the pre-treatment concentration.
Run number with "B" stands for the post-treatment concentration.
"ND" stands for nondetectable.

Table 3. Navy Actual Wastewater Treatment Data

Denitrification and Demetalization Data Sheet

Navy Sample No.	Wastewater Characteristics							Wastewater Treatment Data							
	$NaNO_2$	Cd 213.1*	Cr 218.1*	Cu 220.1*	Pb 239.1*	NI 249.1*	Zn 289.1*	Sulfamic Acid Added (gram)	pH	Nitrite Post Treatment	NaOH Added (50%) mL	pH	Sulfuric Acid (96.7%) mL	pH	Total Suspended Solids (ppm)
1A	0.164	ND	ND	0.26	0.97	ND	0.21	0.1307	4	ND	80		53.5	5	
1B	ND	ND	ND	0.09	ND	ND	0.12								
2A	250	0.04	ND	1.18	ND	ND	0.75	199	4	ND	108	10.5	55	7.6	13
2B	ND	0.04	ND	0.39	ND	ND	0.22								8
3A	1062	0.22	1.7	17.4	3.4	15.9	15.9	850		ND	490		50	6.5	
3B	ND	ND	ND	0.1	ND	ND	0.04								236
4A	1680	0.42	3.3	28.8	5.4	34	28	800	2	ND	500	11	50	7	
4B	ND	ND	ND	0.17	ND	ND	0.04								203
5A	600	0.01	0.01	3.09	0.04	0.11	0.63	450	1.5	ND	300	11	40	7	
5B	ND	ND	ND	0.05	ND	ND	ND								
6A	520	0.01	0.01	2.03	0.01	0.09	0.69	410	2	ND	320	10.4	52	7	
6B	ND	ND	ND	ND	ND	ND	0.04								

*EPA procedure.

Note: The unit of figures shown above are ppm (mg/L).
Run number with "A" stands for the pre-treatment concentration.
Run number with "B" stands for the post-treatment concentration.
"ND" stands for non-detectable.

Table 4. Experimental Simulation Run Matrix
Using 100-Gallon Solution

$NaNO_2$ Concentration	Sulfamic Acid Added (g)	Run No. for a Metal Ion Concentration of -			
		Low	Medium	High	None
340 g at 600 ppm	478	7	9	4	1
510 g at 900 ppm	717	11	6	12	3
680 g at 1,200 ppm	956	5	10	8	2

Table 5. Design Basis for Nitrite Reduction/Metal Precipitation Pilot Plant

Process	Basis
General	
Flow rate	500 gal/day
Operation	Batch
Nitrite Reduction	
Retention time (minimum)	8 hr
Sulfamic acid dosage	12.7 kg/batch
Final pH	2.0 units
Metal Precipitation	
Retention time	8 hr
Operating pH	9.0 - 11.0 units
Polymer type	Anionic polyacrylamide
Polymer dosage	2 - 5 mg/L
Underflow sludge concentration	0.5 - 1.0% solids by weight
Sludge Dewatering	
Thicker retention time	8 hr
Thickened sludge concentration	1.0% solids by weight
Filter press operating pressure	100 psi
Filter press cake concentration	30% solids by weight
Neutralization	
Retention time	30 min
Operating pH	6.0 - 9.0 units

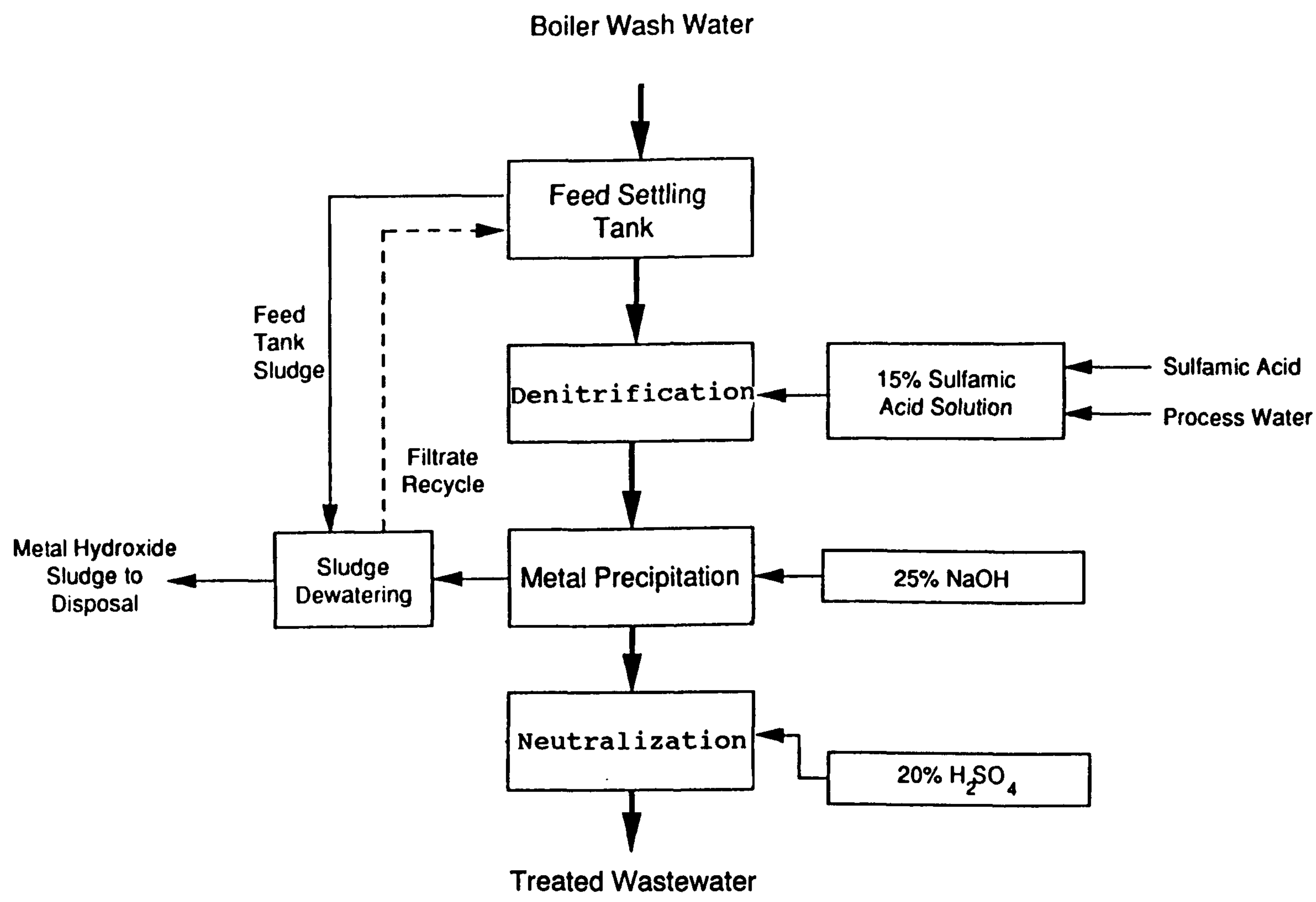

Figure 1
Block flow diagram for the nitrite reduction/metal precipitation system.

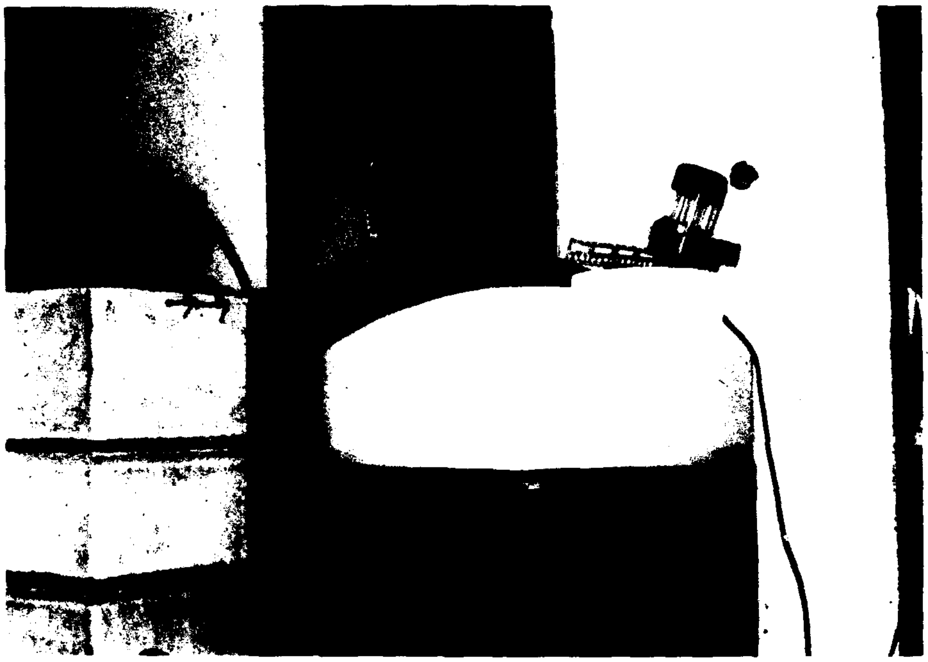

Figure 2
Reactor tank with mixer in place.

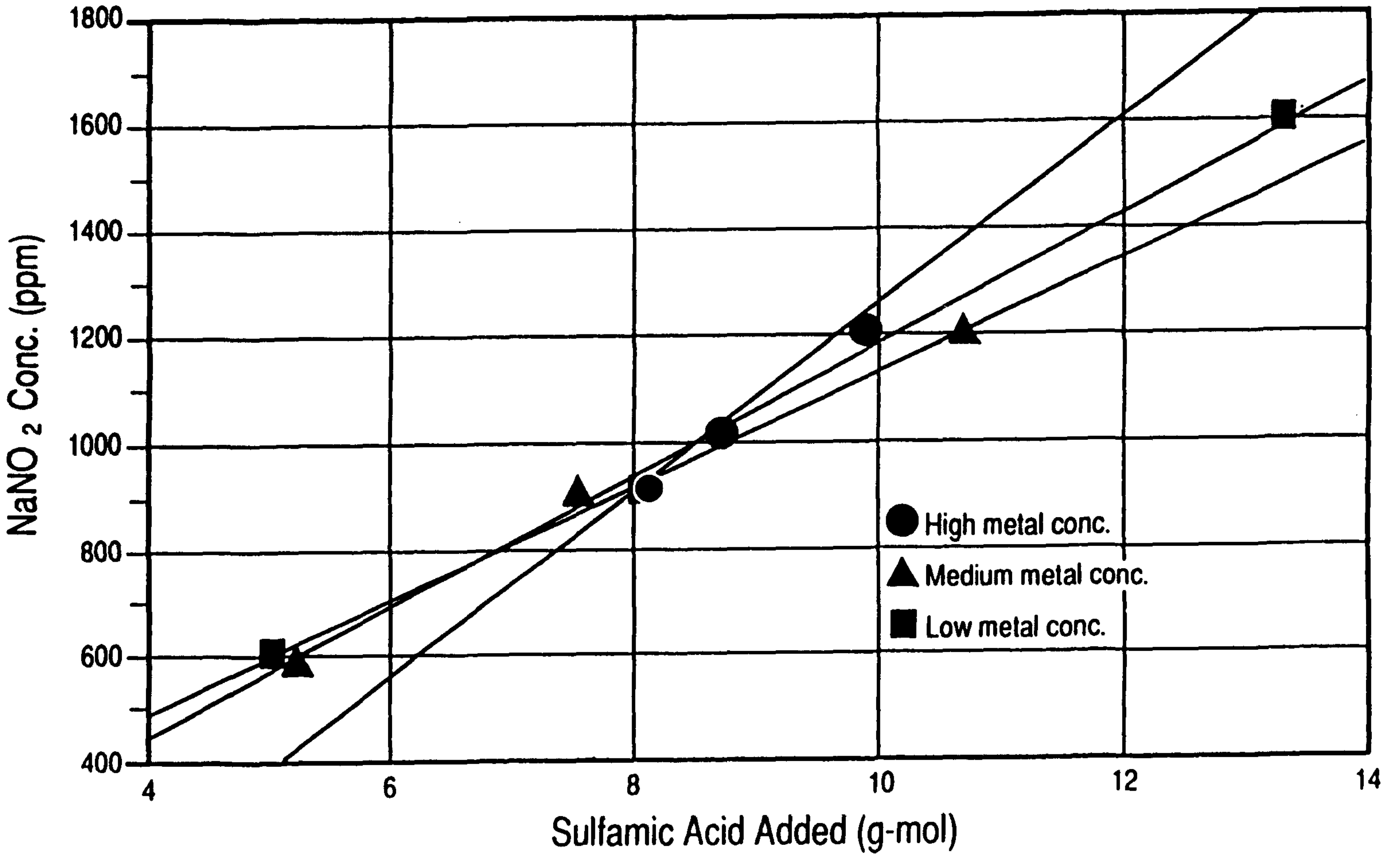

Figure 3
Sulfamic acid requirement for total reduction of sodium nitrite content (100-gallon tank) in synthetic nitrite wastewater.

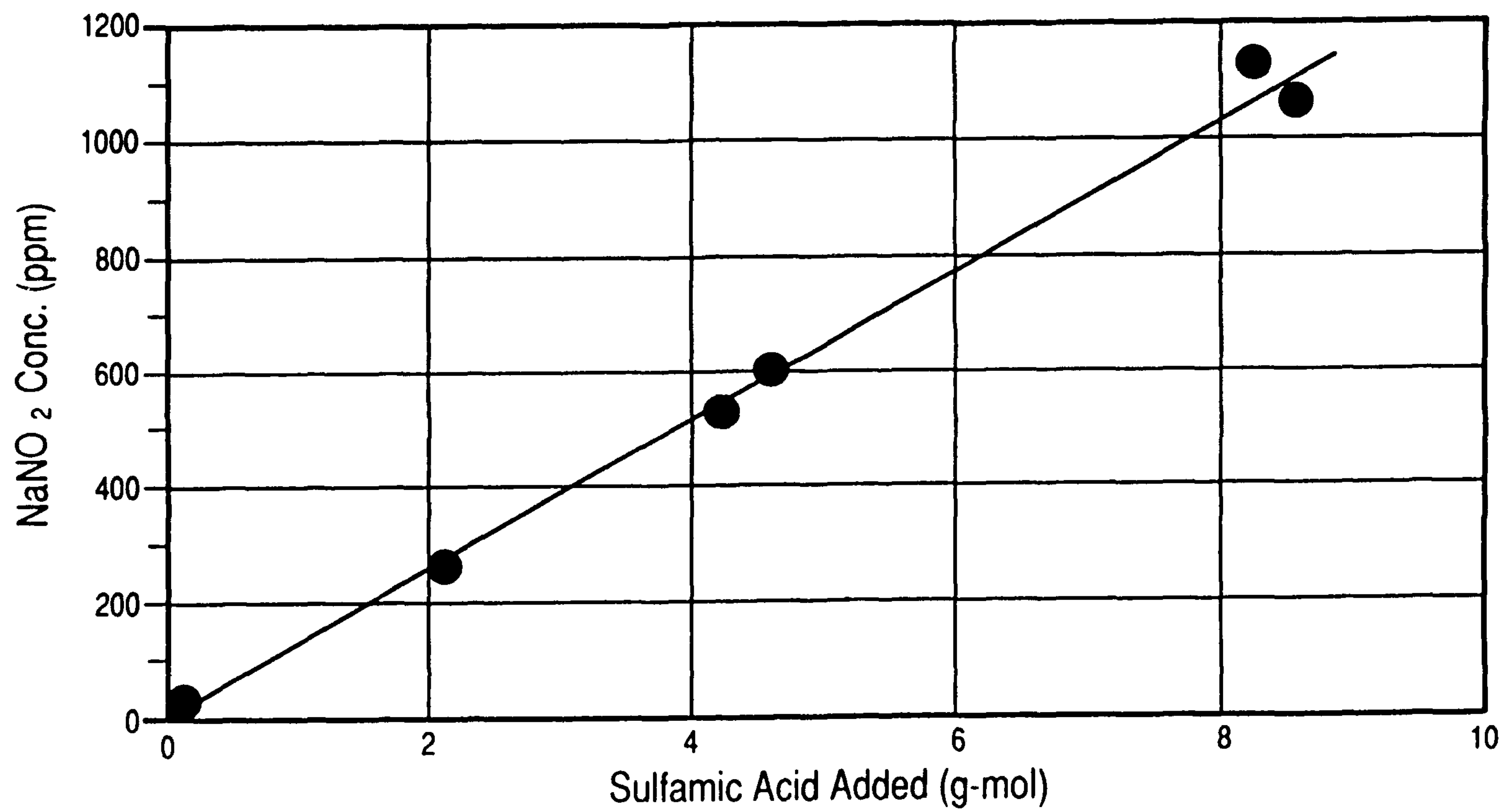

Figure 4
Sulfamic acid requirement for total reduction of sodium nitrite content (100-gallon tank) in actual nitrite wastewater.

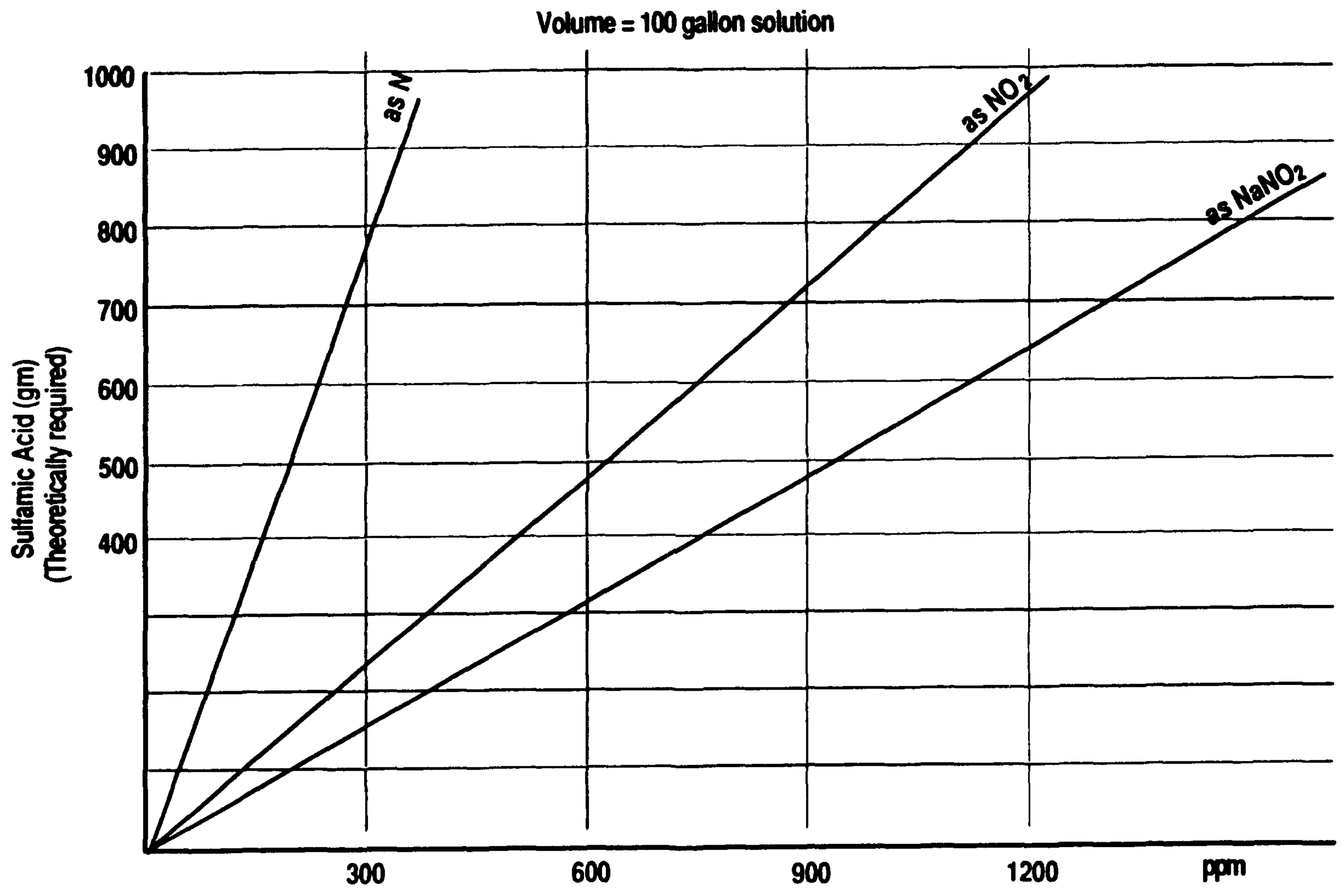

Figure 5
Sulfamic acid requirement for nitrite ion reduction to nitrogen.

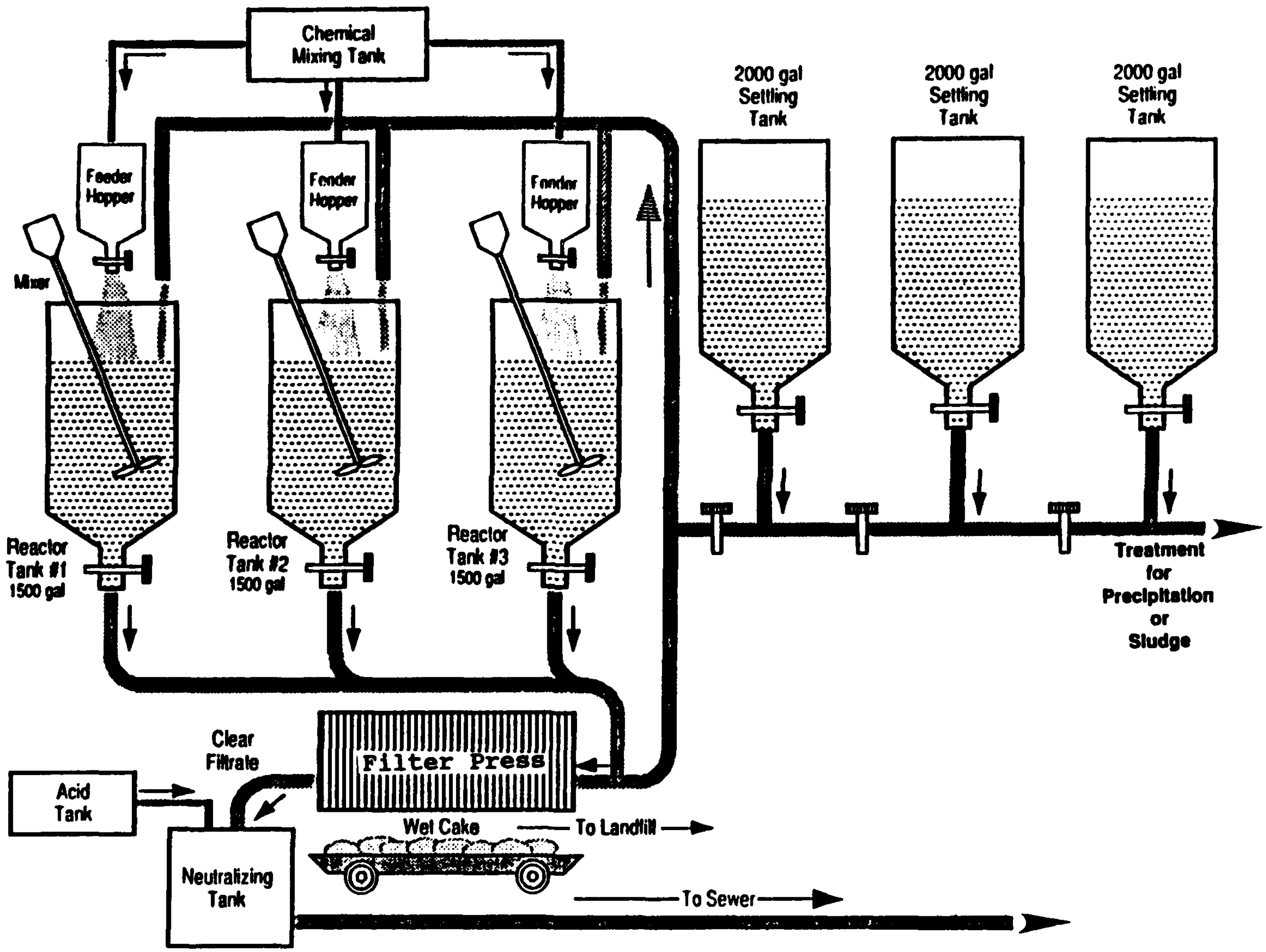

Figure 6
Proposed full-scale sodium nitrite wastewater minimization process.

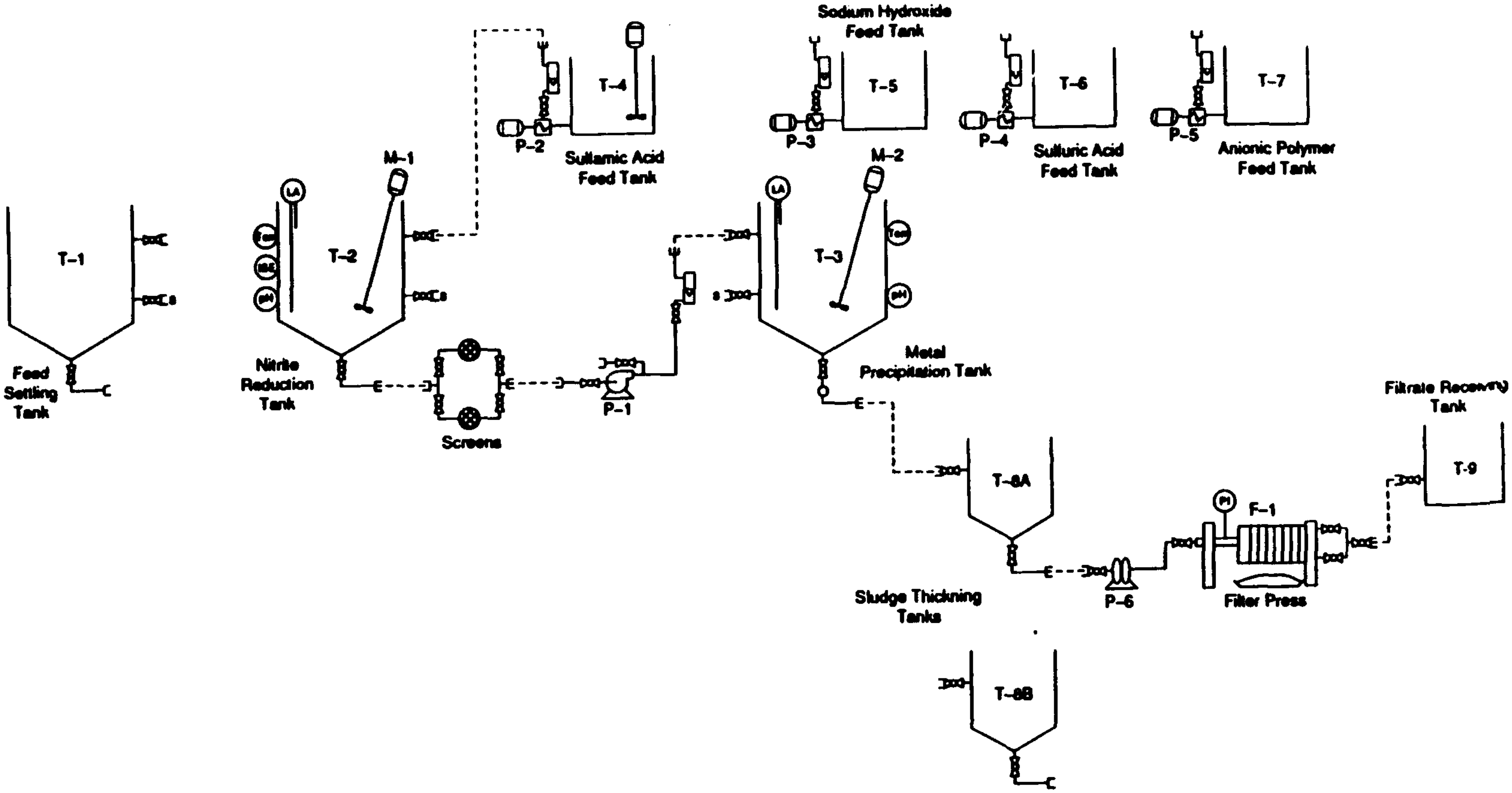

Figure 7
Proposed pilot-scale nitrite reduction system.

Appendix A

BASIC SAMPLE CALCULATIONS

1. Sulfamic Acid Requirement

Basis: 100-gallon Solution

1 lb of $NaNO_2$ in 100 gallons water = (454 g)/(100 gal x 3.78 L/gal)

= 1.20 g/L = 1200 mg/L

Stoichiometric Sulfamic Acid Requirement (M.W. = 97)

$$NaNO_2 + NH_2SO_2OH \text{ <------> } N_2 \uparrow + NaHSO_4 + H_2O$$

1.0 lb $NaNO_2$ (M.W.=69) = 0.0145 lb-mol equivalent x 97

= 1.41 lb NH_2SO_2OH

Expressed as:

N (ppm)	NO_2 (ppm)	$NaNO_2$ (ppm)	NH_2SO_2OH Required
243	800	1200	640 g (1.41 lb)
182	600	900	480 g (1.05 lb)
122	400	600	320 g (0.70 lb)

A conversion plot for the convenience of the operator is shown in the Test Results section in the main text of this report (Figure 4).

Example:

If the solution contains 250 ppm as NO_2^-, it is 375 ppm (as $NaNO_2$)

= 375 mg/L = 0.375 g/L = 0.375 g x 3.78 L/gal x 100

= 141.5 g/100 gal. The conversion factor is therefore 250/141.5

$= 1.767$ (see Data Sheet Line 1)

2. Theoretical Temperature Increase

Using the thermodynamic data shown in Appendix B, the theoretical temperature increase in the 100-gallon reactor tank can be calculated as follows. Using heats of formation of both reactants and products,

$$HOSO_2NH_2 + NaNO_2 \dashrightarrow NaHSO_4 + H_2O + N_2 \uparrow$$

$$\Delta H^o_{reaction} = \Delta H^o_{NaHSO_4} + \Delta H^o_{H_2O} - \Delta H^o_{NaNO_2} - \Delta H^o_{NH_2SO_2OH}$$

$$= 269.0 - 68.3 - (85.7 - 161.3)$$

$$= 90.3 \text{ kcal/mol of reactant}$$

$$90.3 \text{ kcal/mol } NaNO_2 \text{ reacted} = 90.3/69 = 1.3 \text{ kcal/g x } 340 \text{ g/100 gal}$$

$$= 4.43 \text{ kcal/gal x 1 gal/3.78 L x 1 L/1000 mL}$$

$$\text{x } 1000 \text{ cal/1 kcal} = 1.173 \text{ cal/mL}$$

Since the heat capacity of this dilute solution can be assumed to be the same as water; i.e., $C_p = 1.0$ cal/mL-°C, hence:

$$\Delta T = 1.173 \text{ cal/mL x } 1/C_p = 1.17°\text{C or } 2°\text{F}$$

3. Free Energy Change for the Reaction

$$\Delta G = \Delta H - T\Delta S \quad \text{(T in absolute scale)}$$

At equilibrium, $\Delta S_{reaction} = 0$

$$\Delta S^o_{NH_2SO_2OH} = \Delta S_{NaHSO_4} + \Delta S_{H_2O} - \Delta S_{NaNO_2}$$

$$= 27.0 + 16.7 - 24.8 = 18.9 \text{ cal}$$

$$\Delta G^o_{NH_2SO_2OH} = \Delta H^o_{NH_2SO_2OH} - T\,\Delta S^o$$

At room temperature 25°C (298°K),

$$= -161.3 - 298\ (18.9/1000)$$

$$= -166.9 \text{ kcal/mol}$$

$$\Delta pG^{o}_{reaction} = -273.3 - 56.7 - (-68.0 - 166.9)$$

$$= -95.1 \text{ kcal/mol}$$

Appendix B

THERMODYNAMIC DATA

Thermodynamic Values of Reaction Compounds

Compounds	Molecular Weight	ΔH_f^o (kcal/mol)	ΔG_f^o (kcal/mol)	ΔS^o (cal/^{o}K-m)
$NaNO_2$ (c)	69	-85.7	-68.0	24.8
H_2NSO_3H (c)	97	-161.3	-166.9*	18.9*
$NaHSO_4$	120	-269.0	-237.3	27.0
H_2O (1)	18	-68.3	-56.7	16.7
H_2O (g)	18	-57.8	-54.6	45.7

*Calculated. Not available in literature.

Appendix C

SAMPLE ANALYTICAL DATA
FOR NAVY WASTEWATER RUN NO. 4

WESTERN ANALYTICAL LABORATORIES, INC.

13744 MONTE VISTA AVENUE
CHINO, CALIFORNIA 91710
TELEPHONE: (714) 627-3628

DATE RECEIVED: 12/17/90 WAL NO.: 90120409
DATE REPORTED: 01/04/91
CUSTOMER: DR. HENRY SHENG MIS5
ADDRESS: 3316 Woodbend Dr., Claremont, CA 91711 S255
ATTENTION: Dr. Sheng
SAMPLE I.D.: Industrial Wastewater - Grab Sample
SAMPLE POINT: Navy 4B
SAMPLED BY: Customer
DATE & TIME SAMPLED: 12/14/90

PARAMETER	VALUE (Post-treated)	UNIT	DETECTION LIMIT	METHOD	Pre-treatment VALUE
Cadmium	ND	mg/l	0.005	EPA 213.1	0.42 mg/L
Chromium(total)	ND	mg/l	0.02	EPA 218.1	3.30 mg/L
Copper	0.17	mg/l	0.01	EPA 220.1	28.80 mg/L
Lead	ND	mg/l	0.05	EPA 239.1	5.40 mg/L
Nickel	ND	mg/l	0.01	EPA 249.1	34.0 mg/L
Zinc	0.04	mg/l	0.005	EPA 289.1	28.0 mg/L

Sodium nitrite 1680 mg/L (pre-treated)

Sodium nitrite 0.0 mg/L (post-treated)

Note: This sample was pumped out from the bottom of several barrels. It contained sludge, metal precipitates, oil film and bad odor. The worst we have treated.

= Not Detected

Joseph P. Zimmer
Laboratory Director

WESTERN ANALYTICAL LABORATORIES, INC.

13744 MONTE VISTA AVENUE
CHINO, CALIFORNIA 91710
TELEPHONE: (714) 627-3628

DATE RECEIVED: 01/02/91 WAL NO.: 91010007
DATE REPORTED: 01/09/91
CUSTOMER: DR. HENRY SHENG MIS20
ADDRESS: 3316 Woodbend Dr., Claremont, CA 91711 S255
ATTENTION: Dr. Sheng
SAMPLE I.D.: Industrial Wastewater - Grab Sample
SAMPLE POINT: #5 Unfiltered
SAMPLED BY: Customer
DATE & TIME SAMPLED: 01/02/91

PARAMETER	VALUE	UNIT	DETECTION LIMIT	METHOD
ANALYSIS OF TCLP EXTRACT:				
Arsenic	0.009	mg/l	0.002	EPA 206.3
Barium	ND	mg/l	0.1	EPA 208.1
Cadmium	ND	mg/l	0.005	EPA 213.1
Chromium(total)	ND	mg/l	0.02	EPA 218.1
Lead	ND	mg/l	0.05	EPA 239.1
Mercury	ND	mg/l	0.0002	EPA 245.1
Selenium	0.03	mg/l	0.002	EPA 270.3
Silver	ND	mg/l	0.01	EPA 272.1

NOTE: Analysis of TCLP Extract is to determine whether sludge is able to be landfilled. None of the metals are above regulatory limits.

ND = Not Detected

Joseph P. Zimmer
Laboratory Director

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4B Hazardous/toxic materials management
4C Waterwaste management and sanitary engineering
4D Oil pollution removal and recovery
4E Air pollution
4F Noise abatement
5 OCEAN ENGINEERING
5A Seafloor soils and foundations
5B Seafloor construction systems and operations (including diver and manipulator tools)
5C Undersea structures and materials
5D Anchors and moorings
5E Undersea power systems, electromechanical cables, and connectors
5F Pressure vessel facilities
5G Physical environment (including site surveying)
5H Ocean-based concrete structures
5J Hyperbaric chambers
5K Undersea cable dynamics
ARMY FEAP
BDG Shore Facilities
NRG Energy
ENV Environmental/Natural Responses
MGT Management
PRR Pavements/Railroads

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