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Sponsored By Naval Facilities
Engineering Command

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.
13. ABSTRACT (Maximum 200 words)

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.
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 NOₓ. 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.
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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:

\[
\text{NO}_2^- + \text{NH}_2\text{SO}_2\text{OH} \rightarrow \text{N}_2 \uparrow + \text{HSO}_4^- + \text{H}_2\text{O} \quad (1)
\]

\[
\text{(Me)}^{++} + 2\text{X(OH)}^- \rightarrow \text{Me(OH)}_2 + 2\text{X} \quad (2)
\]
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:

\[
\text{NO}_3^- + 2\text{H}^+ + 2e^- \rightarrow \text{NO}_2^- + \text{H}_2\text{O} \tag{3}
\]

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:

\[
\text{NaNO}_2 + \text{NH}_2\text{SO}_2\text{OH} \rightleftharpoons \text{HO-SO}_2\text{-N}_2\text{-OH} + \text{Na}^+ + \text{OH}^- \tag{4}
\]

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 cadmium, 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


Table 1. Denitrification and Demetalization Data Sheet

<table>
<thead>
<tr>
<th>Run No.</th>
<th>Date</th>
<th>Volume Treated</th>
<th>Type</th>
</tr>
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</table>

I. Wastewater

1. \( \text{NO}_2^- = \) ppm
2. Cd \( \text{ppm} \), Cr \( \text{ppm} \), Cu \( \text{ppm} \), Fe \( \text{ppm} \)
   Ni \( \text{ppm} \), Pb \( \text{ppm} \), Zn \( \text{ppm} \)
3. (a) Temp = \( ^\circ\text{F} \) or \( ^\circ\text{C} \) (b) pH = _____

II. Wastewater Treatment

5. Sulfamic acid added = \( \_ \text{g} = \_ \text{g-mol (per tank)} \)
6. Rate of addition = \( \_ \text{g/min} \). Acid/NaNO2 (Mole) ratio ______
7. (a) Temp = \( ^\circ\text{F} \) or \( ^\circ\text{C} \) (b) pH = _____

III. Unreacted Nitrite:

8. \( \text{NO}_2^- = \) ppm

IV. Solution Treatment (for demetalization)

9. \( \text{NaOH (}\_ \%) ; \_	ext{mL (per tank)} \)
10. Time (to bring to \( 9 < \text{pH} < 11 \)) = \_ min

V. Solution: \( \text{pH} = 9 \text{ to } 11 \)

11. (a) Temp = \( ^\circ\text{F} \) or \( ^\circ\text{C} \) (b) pH = _____
12. Cd \( \text{ppm} \), Cr \( \text{ppm} \), Cu \( \text{ppm} \), Fe \( \text{ppm} \)
   Ni \( \text{ppm} \), Pb \( \text{ppm} \), Zn \( \text{ppm} \)

VI. Solution Treatment (for neutralization)

13. \( \text{H}_2\text{SO}_4 (\_ \%) ; \_	ext{mL (per tank)} \)
14. Time (to bring to \( 6 < \text{pH} < 8 \)) = \_ min

VII. Solution: \( \text{pH} = 6 \text{ to } 8 \)

15. Cartridge (after) = \_ g 16. (a) Temp = \( ^\circ\text{F} \) or \( ^\circ\text{C} \)
   Cartridge (before) = \_ g (b) pH = _____
   Residue = \_ g

Remarks

______________________________________________________________

______________________________________________________________

Recorded by ________________________________

7
Table 2. Simulation Run Data

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*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

<table>
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<th>Navy Sample No.</th>
<th>Wastewater Characteristics</th>
<th>Wastewater Treatment Data</th>
<th>Total Suspended Solids (ppm)</th>
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<td></td>
<td>NaN2 213.1* Cd 218.1* Cr 210.1* Cu 220.1* Pb 239.1* Ni 249.1* Zn 289.1*</td>
<td>Sulfamic Acid Added (gram) pH Nitrite Post Treatment</td>
<td>NaOH Added (50%) mL pH</td>
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<td>1A</td>
<td>0.164 ND ND 0.26 0.97 ND ND 0.21</td>
<td>0.1307 4 ND</td>
<td>80 53.5</td>
</tr>
<tr>
<td>1B</td>
<td>ND ND ND 0.09 ND ND ND 0.12</td>
<td>199 4 ND</td>
<td>108 55</td>
</tr>
<tr>
<td>2A</td>
<td>250 0.04 ND ND 1.18 ND ND 0.75</td>
<td>850 ND</td>
<td>490 50</td>
</tr>
<tr>
<td>2B</td>
<td>ND ND ND 0.39 ND ND ND 0.22</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3A</td>
<td>1062 0.22 1.7 17.4 3.4 15.9 15.9</td>
<td>280 2 ND</td>
<td>500 11</td>
</tr>
<tr>
<td>3B</td>
<td>ND ND ND 0.1 ND ND ND 0.04</td>
<td>450 1.5 ND</td>
<td>400 11</td>
</tr>
<tr>
<td>4A</td>
<td>1680 0.42 3.3 28.8 5.4 ND 34 28</td>
<td>236</td>
<td>203</td>
</tr>
<tr>
<td>4B</td>
<td>ND ND ND 0.17 ND ND ND 0.04</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5A</td>
<td>600 0.01 0.01 3.09 0.04 0.11 0.63</td>
<td>410 2 ND</td>
<td>320 10.4</td>
</tr>
<tr>
<td>5B</td>
<td>ND ND ND 0.05 ND ND ND ND</td>
<td></td>
<td></td>
</tr>
<tr>
<td>6A</td>
<td>520 0.01 0.01 2.03 0.01 0.09 0.69</td>
<td></td>
<td></td>
</tr>
<tr>
<td>6B</td>
<td>ND ND ND ND ND ND ND ND</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*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

<table>
<thead>
<tr>
<th>NaNO₂ Concentration</th>
<th>Sulfamic Acid Added (g)</th>
<th>Run No. for a Metal Ion Concentration of -</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Low</td>
</tr>
<tr>
<td>340 g at 600 ppm</td>
<td>478</td>
<td>7</td>
</tr>
<tr>
<td>510 g at 900 ppm</td>
<td>717</td>
<td>11</td>
</tr>
<tr>
<td>680 g at 1,200 ppm</td>
<td>956</td>
<td>5</td>
</tr>
</tbody>
</table>
Table 5. Design Basis for Nitrite Reduction/Metal Precipitation Pilot Plant

<table>
<thead>
<tr>
<th>Process</th>
<th>Basis</th>
</tr>
</thead>
<tbody>
<tr>
<td>General</td>
<td></td>
</tr>
<tr>
<td>Flow rate</td>
<td>500 gal/day</td>
</tr>
<tr>
<td>Operation</td>
<td>Batch</td>
</tr>
<tr>
<td>Nitrite Reduction</td>
<td></td>
</tr>
<tr>
<td>Retention time (minimum)</td>
<td>8 hr</td>
</tr>
<tr>
<td>Sulfamic acid dosage</td>
<td>12.7 kg/batch</td>
</tr>
<tr>
<td>Final pH</td>
<td>2.0 units</td>
</tr>
<tr>
<td>Metal Precipitation</td>
<td></td>
</tr>
<tr>
<td>Retention time</td>
<td>8 hr</td>
</tr>
<tr>
<td>Operating pH</td>
<td>9.0 - 11.0 units</td>
</tr>
<tr>
<td>Polymer type</td>
<td>Anionic polyacrylamide</td>
</tr>
<tr>
<td>Polymer dosage</td>
<td>2 - 5 mg/L</td>
</tr>
<tr>
<td>Underflow sludge concentration</td>
<td>0.5 - 1.0% solids by weight</td>
</tr>
<tr>
<td>Sludge Dewatering</td>
<td></td>
</tr>
<tr>
<td>Thicker retention time</td>
<td>8 hr</td>
</tr>
<tr>
<td>Thickened sludge concentration</td>
<td>1.0% solids by weight</td>
</tr>
<tr>
<td>Filter press operating pressure</td>
<td>100 psi</td>
</tr>
<tr>
<td>Filter press cake concentration</td>
<td>30% solids by weight</td>
</tr>
<tr>
<td>Neutralization</td>
<td></td>
</tr>
<tr>
<td>Retention time</td>
<td>30 min</td>
</tr>
<tr>
<td>Operating pH</td>
<td>6.0 - 9.0 units</td>
</tr>
</tbody>
</table>
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 7
Proposed pilot-scale nitrite reduction system.
Appendix A

BASIC SAMPLE CALCULATIONS

1. Sulfamic Acid Requirement

Basis: 100-gallon Solution

1 lb of NaNO₂ 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)

\[
\text{NaNO}_2 + \text{NH}_2\text{SO}_2\text{OH} \rightarrow \text{N}_2 \uparrow + \text{NaHSO}_4 + \text{H}_2\text{O}
\]

1.0 lb NaNO₂ (M.W. = 69)  = 0.0145 lb-mol equivalent x 97
= 1.41 lb NH₂SO₂OH

Expressed as:

<table>
<thead>
<tr>
<th>N (ppm)</th>
<th>NO₂ (ppm)</th>
<th>NaNO₂ (ppm)</th>
<th>NH₂SO₂OH Required</th>
</tr>
</thead>
<tbody>
<tr>
<td>243</td>
<td>800</td>
<td>1200</td>
<td>640 g (1.41 lb)</td>
</tr>
<tr>
<td>182</td>
<td>600</td>
<td>900</td>
<td>480 g (1.05 lb)</td>
</tr>
<tr>
<td>122</td>
<td>400</td>
<td>600</td>
<td>320 g (0.70 lb)</td>
</tr>
</tbody>
</table>

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 NO₂⁻, it is 375 ppm (as NaNO₂)

\[
= 375 \text{ mg/L} = 0.375 \text{ g/L} = 0.375 \text{ g} \times 3.78 \text{ L/gal} \times 100
\]

\[
= 141.5 \text{ g/100 gal}. \text{ The conversion factor is therefore } 250/141.5
\]
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,

\[
\text{HOSO}_2\text{NH}_2 + \text{NaNO}_2 \rightarrow \text{NaHSO}_4 + \text{H}_2\text{O} + \text{N}_2 \uparrow
\]

\[
\Delta H_{\text{reaction}}^o = \Delta H_{\text{NaHSO}_4}^o + \Delta H_{\text{H}_2\text{O}}^o - \Delta H_{\text{NaNO}_2}^o - \Delta H_{\text{NH}_2\text{SO}_2\text{OH}}^o
\]

\[
= 269.0 - 68.3 - (85.7 - 161.3)
\]

\[
= 90.3 \text{ kcal/mol of reactant}
\]

90.3 kcal/mol NaNO2 reacted = \(\frac{90.3}{69}\) = 1.3 kcal/g x 340 g/100 gal

\[
= 4.43 \text{ kcal/gal} \times 1 \text{ gal/3.78 L} \times 1 \text{ L/1000 mL}
\]

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

Since the heat capacity of this dilute solution can be assumed to be the same as water; i.e., \(C_p = 1.0 \text{ cal/mL} \cdot ^\circ \text{C}\), hence:

\[
\Delta T = 1.173 \text{ cal/mL} \times \frac{1}{C_p} = 1.17^\circ \text{C or 2^\circ F}
\]

3. Free Energy Change for the Reaction

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

At equilibrium, \(\Delta S_{\text{reaction}} = 0\)

\[
\Delta S_{\text{NH}_2\text{SO}_2\text{OH}}^o = \Delta S_{\text{NaHSO}_4}^o + \Delta S_{\text{H}_2\text{O}}^o - \Delta S_{\text{NaNO}_2}^o
\]

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

\[
\Delta G_{\text{NH}_2\text{SO}_2\text{OH}}^o = \Delta H_{\text{NH}_2\text{SO}_2\text{OH}}^o - T \Delta S^o
\]

At room temperature 25°C (298 K),

\[
= -161.3 - 298 (18.9/1000)
\]

\[
= -166.9 \text{ kcal/mol}
\]
\[ \Delta pG^o_{\text{reaction}} = -273.3 - 56.7 - (-68.0 - 166.9) \]

\[ = -95.1 \text{ kcal/mol} \]
# Appendix B

## THERMODYNAMIC DATA

Thermodynamic Values of Reaction Compounds

<table>
<thead>
<tr>
<th>Compounds</th>
<th>Molecular Weight</th>
<th>$\Delta H_f^o$ (kcal/mol)</th>
<th>$\Delta G_f^o$ (kcal/mol)</th>
<th>$\Delta S^o$ (cal/°K-m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NaNO₂ (c)</td>
<td>69</td>
<td>-85.7</td>
<td>-68.0</td>
<td>24.8</td>
</tr>
<tr>
<td>H₂NSO₃H (c)</td>
<td>97</td>
<td>-161.3</td>
<td>-166.9*</td>
<td>18.9*</td>
</tr>
<tr>
<td>NaHSO₄</td>
<td>120</td>
<td>-269.0</td>
<td>-237.3</td>
<td>27.0</td>
</tr>
<tr>
<td>H₂O (l)</td>
<td>18</td>
<td>-58.3</td>
<td>-56.7</td>
<td>16.7</td>
</tr>
<tr>
<td>H₂O (g)</td>
<td>18</td>
<td>-57.8</td>
<td>-54.6</td>
<td>45.7</td>
</tr>
</tbody>
</table>

*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.:** 20120409

**Date Reported:** 01/04/91  
**Customer:** Dr. Henry Sheng

**Address:** 3316 Woodbone Dr., Claremont, CA 91711

**Attention:** Dr. Sheng

**Sample I.D.:** Industrial Wastewater - Grab Sample

**Sample Point:** Navy 4B

**Sampled By:** Customer

**Date & Time Sampled:** 12/14/90

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value (Post-treated)</th>
<th>Unit</th>
<th>Detection Limit</th>
<th>Method</th>
<th>Pre-treatment Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cadmium</td>
<td>ND</td>
<td>mg/L</td>
<td>0.005</td>
<td>EPA 213.1</td>
<td>0.42 mg/L</td>
</tr>
<tr>
<td>Chromium (total)</td>
<td>ND</td>
<td>mg/L</td>
<td>0.02</td>
<td>EPA 218.1</td>
<td>3.30 mg/L</td>
</tr>
<tr>
<td>Copper</td>
<td>0.17</td>
<td>mg/L</td>
<td>0.01</td>
<td>EPA 220.1</td>
<td>28.80 mg/L</td>
</tr>
<tr>
<td>Lead</td>
<td>ND</td>
<td>mg/L</td>
<td>0.05</td>
<td>EPA 239.1</td>
<td>5.40 mg/L</td>
</tr>
<tr>
<td>Nickel</td>
<td>ND</td>
<td>mg/L</td>
<td>0.01</td>
<td>EPA 249.1</td>
<td>34.0 mg/L</td>
</tr>
<tr>
<td>Zinc</td>
<td>0.04</td>
<td>mg/L</td>
<td>0.005</td>
<td>EPA 289.1</td>
<td>28.0 mg/L</td>
</tr>
</tbody>
</table>

| 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.

---

签字：Joseph P. Zimmer  
Laboratory Director

---

**State Certified Laboratory**  
**Domestic Water - Industrial Waste Water - Hazardous Waste**  
**Metal Finishing Solution Analysis and Process Control**
DATE RECEIVED: 01/02/91
DATE REPORTED: 01/09/91
CUSTOMER: DR. HENRY SHENG
ADDRESS: 3316 Woodbend Dr., Claremont, CA 91711
ATTENTION: Dr. Sheng
SAMPLE I.D.: Industrial Wastewater - Grab Sample
SAMPLE POINT: #5 Unfiltered
SAMPLED BY: Customer
DATE & TIME SAMPLED: 01/02/91

<table>
<thead>
<tr>
<th>PARAMETER</th>
<th>VALUE</th>
<th>UNIT</th>
<th>DETECTION LIMIT</th>
<th>METHOD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arsenic</td>
<td>0.009</td>
<td>mg/l</td>
<td>0.002</td>
<td>EPA 206.3</td>
</tr>
<tr>
<td>Barium</td>
<td>ND</td>
<td>mg/l</td>
<td>0.1</td>
<td>EPA 208.1</td>
</tr>
<tr>
<td>Cadmium</td>
<td>ND</td>
<td>mg/l</td>
<td>0.005</td>
<td>EPA 213.1</td>
</tr>
<tr>
<td>Chromium (total)</td>
<td>ND</td>
<td>mg/l</td>
<td>0.02</td>
<td>EPA 218.1</td>
</tr>
<tr>
<td>Lead</td>
<td>ND</td>
<td>mg/l</td>
<td>0.05</td>
<td>EPA 239.1</td>
</tr>
<tr>
<td>Mercury</td>
<td>ND</td>
<td>mg/l</td>
<td>0.0002</td>
<td>IPA 245.1</td>
</tr>
<tr>
<td>Selenium</td>
<td>0.03</td>
<td>mg/l</td>
<td>0.002</td>
<td>IPA 270.3</td>
</tr>
<tr>
<td>Silver</td>
<td>ND</td>
<td>mg/l</td>
<td>0.01</td>
<td>IPA 272.1</td>
</tr>
</tbody>
</table>

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

STATE CERTIFIED LABORATORY
DOMESTIC WATER - INDUSTRIAL WASTE WATER - HAZARDOUS WASTE
METAL FINISHING SOLUTION ANALYSIS AND PROCESS CONTROL
DISTRIBUTION LIST

AF / 92D CES/DEMC, FAIRCHILD AFB, WA; AFSC/DEE, WASHINGTON, DC;
   SM-ALC/DEEN (J PESTILLO), McCLELLAN AFB, CA
AF HQ / LETT (CARGO), WASHINGTON, DC; LEYSF, WASHINGTON, DC; HQ
   PACAF/DEE (EMCS MGR), HICKAM AFB, HI
AFESC / DEB, TYNDALL AFB, FL
AFIT / DEV, WRIGHT-PATTERSON AFB, OH
ANTARCTIC / STAFFO, ALEXANDRIA, VA
ARMY / CH OF ENGRS, DAEN-CWE-M, WASHINGTON, DC; CH OF ENGRS, DAEN-HPU,
   WASHINGTON, DC; CH OF ENGRS, DAEN-FMZ, WASHINGTON, DC
ARMY / ENGR CEN, ATSE-DAC-LC, FORT LEONARD WOOD, MO; HQDA (DAEN-ZCM),
   WASHINGTON, DC; HQDA DANA-CSC, WASHINGTON, DC; SEC OFFR, WASHINGTON,
   DC; KWAJALEIN ATOLL, CSSD-LA-LT, APO AP
ARMY CERL / LIB, CHAMPAIGN, IL
ARMY ENGRG DIV / EUDED-TN (O'MALLY), FRANCE, APO AE
ARMY LMC / FORT LEE, VA
AU REG HOSP/SGPB / GARG, MAXWELL AFB, AL
BATTLENE NEW ENGLAND MARINE RSCH LAB / LIB, DUXBURY, MA
BRITISH EMBASSY / SCI & TECH DEPT (WILKINS), WASHINGTON, DC
CBE / CODE 15, PORT HUENEME, CA; CODE 155, PORT HUENEME, CA; CODE 156,
   PORT HUENEME, CA; FPO (CODE 400), GULFPORT, MS
CECOS / CODE C35, PORT HUENEME, CA
CINC PACFLT / CODE 442, PEARL HARBOR, HI; CODE 443, PEARL HARBOR, HI; SO,
   PEARL HARBOR, HI
CNA / TECH LIB, ALEXANDRIA, VA
CNO / DCNO, LOGS, OP-452, WASHINGTON, DC
COM GEN FMF / PAC, SCIAD (G5), CAMP HM SMITH, HI
COMASWINGPAC / CODE 421, SAN DIEGO, CA; CODE N-316, SAN DIEGO, CA
COMCBPAC / CODE CB22, PEARL HARBOR, HI
COMDT COGUARD / LIB, WASHINGTON, DC
COMFLEACT / CODE 200, FPO AP,
COMFLEACT / FPO, FPO AP; SCE, FPO AP; SO, FPO AP
COMNAVCAT / FPO, LONDON, UK, FPO AE
COMNAV AIRSYSCOM / CODE 41712A, WASHINGTON, DC; CODE 422, WASHINGTON, DC;
   CODE 56W23, WASHINGTON, DC
COMNAVDIST / CODE 313, WASHINGTON, DC; CODE 412, WASHINGTON, DC
COMNAVILOGPAC / CODE 4318, PEARL HARBOR, HI; CODE N41B4, PEARL HARBOR, HI
COMNAVRASISCOM / CODE N4, FPO AP
COMOCEANSYS / PAC, SCE, PEARL HARBOR, HI
COMOPTEVFOR / CO, NORFOLK, VA
COMSUBDEVGRU ONE / CO, SAN DIEGO, CA
COMSUBPAC / CODE 541, SCE, PEARL HARBOR, HI; CODE 542, SCE, PEARL
   HARBOR, HI
COMUSNAV / JAPAN, CODE J42E, APO AP
DEPCOMOPTEVFORPAC / CODE 701A, SAN DIEGO, CA
DOE / WIND/OCEAN TECH DIV, PORT TOBACCO, MD
DOODS / PAC, FAC, FPO AP
NAVFACENGCOM SOUTHWESTDIV / CODE 101.1, SAN DIEGO, CA; CODE 144C, SAN DIEGO, CA

NAVFACENGCOM WESTDIV / SAN BRUNO, CA; CODE 04, SAN BRUNO, CA; CODE 04A2.2 LIB, SAN BRUNO, CA; CODE 05, SAN BRUNO, CA; CODE 09B, SAN BRUNO, CA; CODE 11, SAN BRUNO, CA; CODE 162, SAN BRUNO, CA; CODE 1833, SAN BRUNO, CA; CODE 402, SAN BRUNO, CA; CODE 402, (KELLY), SAN BRUNO, CA; CODE 405, SAN BRUNO, CA; CODE 406.2 (SMITH), SAN BRUNO, CA; CODE 408.2 (JEUNG), SAN BRUNO, CA; CODE 408.2 (SHULL), SAN BRUNO, CA; CODE 411, SAN BRUNO, CA; CODE 4023 (PICQUET), CHARLESTON, SC; CODE 4023 (RDL), CHARLESTON, SC; CODE 403 (GADDY), CHARLESTON, SC; CODE 403 (S HULL), CHARLESTON, SC; CODE 405, CHARLESTON, SC; CODE 405 REL, CHARLESTON, SC; CODE 405 LEA, CHARLESTON, SC; CODE 405, CHARLESTON, SC; CODE 406, CHARLESTON, SC; PWO, CHARLESTON, SC

NAVFACENGCOM SOUTHDIV / CODE 101.1, SAN DIEGO, CA; CODE 144C, SAN DIEGO, CA

NAVFACENGCOM WESTDIV / SAN BRUNO, CA; CODE 04, SAN BRUNO, CA; CODE 04A2.2 LIB, SAN BRUNO, CA; CODE 05, SAN BRUNO, CA; CODE 09B, SAN BRUNO, CA; CODE 11, SAN BRUNO, CA; CODE 162, SAN BRUNO, CA; CODE 1833, SAN BRUNO, CA; CODE 402, SAN BRUNO, CA; CODE 402, (KELLY), SAN BRUNO, CA; CODE 405, SAN BRUNO, CA; CODE 406.2 (SMITH), SAN BRUNO, CA; CODE 408.2 (JEUNG), SAN BRUNO, CA; CODE 408.2 (SHULL), SAN BRUNO, CA; CODE 411, SAN BRUNO, CA; CODE 4023 (PICQUET), CHARLESTON, SC; CODE 4023 (RDL), CHARLESTON, SC; CODE 403 (GADDY), CHARLESTON, SC; CODE 403 (S HULL), CHARLESTON, SC; CODE 405, CHARLESTON, SC; CODE 405 REL, CHARLESTON, SC; CODE 405 LEA, CHARLESTON, SC; CODE 405, CHARLESTON, SC; CODE 406, CHARLESTON, SC; PWO, CHARLESTON, SC

NAVFULE DET / OIC, FPO AP,

NAVINVSERV / SW REG, SO, SAN DIEGO, CA

NAVMEDCOM / SWREG, SCE, SAN DIEGO, CA

NAVOCEANO / LIB, NSTL, MS

NAVORDSTA / CODE 0922B1, INDIAN HEAD, MD; SCSI3, INDIAN HEAD, MD

NAVPETRES / DIR, WASHINGTON, DC

NAVPGSCOL / E. THORNTON, MONTEREY, CA; PWO, MONTEREY, CA

NAVSEACENPAC / CODE 420, SAN DIEGO, CA

NAVSEASYSCOM / CODE 51412, WASHINGTON, DC

NAVSEASYSCOM / CODE 56Z4, WASHINGTON, DC; SEA-6631, WASHINGTON, DC

NAVSECRUACT / PWO, CHESAPEAKE, VA

NAVSHIPREFAC / LIB, FPO AP; SCE, FPO AP

NAVSHIPYARD / CODE 383.4, PORTSMOUTH, VA; CODE 450-HD, PORTSMOUTH, VA; CARR INLET ACOUSTIC RANGE, BREMERTON, WA; CO (PEARL HARBOR), PEARL HARBOR, HI; CO (PHILADELPHIA), PHILADELPHIA, PA; CODE 106.4, STARYNSKI, PHILADELPHIA, PA; CODE 106.4, PEARL HARBOR, HI; CODE 1710, PHILADELPHIA, PA; CODE 1720.04, LONG BEACH, CA; CODE 202.5 (ENGRG LIB), BREMERTON, WA; CODE 244.13, LONG BEACH, CA; CODE 308.05, PEARL HARBOR, HI; CODE 308.3, PEARL HARBOR, HI; CODE 380, PORTSMOUTH, VA; CODE 382.3, PEARL HARBOR, HI; CODE 402.4, PHILADELPHIA, PA; CODE 406, PORTSMOUTH, NH; CODE 443, BREMERTON, WA; CODE 453, CHARLESTON, SC; CODE 453P, PORTSMOUTH, VA; CODE 830 (SEC DIV), BREMERTON, WA; CODE 830.1, PEARL HARBOR, HI; MARE IS, CODE 106.4, VALLEJO, CA; MARE IS, CODE 202.13, VALLEJO, CA; MARE IS, CODE 208.08, VALLEJO, CA; MARE IS, CODE 280, VALLEJO, CA; MARE IS, CODE 280.28, VALLEJO, CA; MARE IS, CODE 401, VALLEJO, CA; MARE IS, CODE 457, VALLEJO, CA; MARE IS, CODE 833, VALLEJO, CA; MARE IS, PWO, VALLEJO, CA; MARE IS, SEC OFFR, VALLEJO, CA; PWO, BREMERTON, WA; PWO, CHARLESTON, SC; PWO, CODE 400, LONG BEACH, CA; SEC OFFR, PORTSMOUTH, NH; SHOP 71, BREMERTON, WA

NAVSTA / CO, LONG BEACH, CA; CODE 0D3, SAN DIEGO, CA; CODE 80B, PEARL HARBOR, HI; PUGET SOUND CODE 413, SEATTLE, WA; PUGET SOUND CODE 922, EVERETT, WA; SCE, PEARL HARBOR, HI; SO, FPO AP
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