

# Memorandum

To: Rick Allen  
From: Terry W. Pepper  
Date: August 1, 1997  
  
Re: **GIANT ARSENIC**  
Testwork Status Report

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The testwork performed to date on the Hot Water Process has not established a process for recovering low antimony arsenic trioxide ( $\text{As}_2\text{O}_3$ ). Some of the results indicate that selective removal of antimony from arsenic might be possible, but additional testwork is required to determine the technical and economic feasibility of the antimony removal.

The Methanol Process has been shown to selectively dissolve arsenic, leaving antimony and iron in the residue. Insufficient testwork has been performed to fully define the process and to anticipate potential problems.

Antimony solubility testwork has been less than satisfactory. The limited solubility data provided in the literature is the best information we have.

## RECOMMENDATION

If:

1. An arsenic product containing less than 0.20% Sb is required and
2. A process must be designated this summer.

It is recommended that either the Fuming Process or Methanol Process be proposed.

If a higher antimony material is acceptable, the Water Leach Process should be proposed and additional testwork should be performed to evaluate recycle streams.

42.480 kg

## HOT WATER LEACH

Testwork has been performed at Lakefield Research to evaluate arsenic, antimony, and iron dissolution during the leaching stage. Hazen Research performed solubility tests to establish the solubility of arsenic and antimony as a function of temperature. Additional work was performed at both locations to attempt to remove antimony from the saturated arsenic solution prior to crystallization.

### Solubility Tests

#### As<sub>2</sub>O<sub>3</sub> Solubility Over 90 g As<sub>2</sub>O<sub>3</sub>/l at 88°C

The attached graph shows solubility data produced by Hazen shows the effect of temperature on As<sub>2</sub>O<sub>3</sub> solubility. The solubilities were from solutions produced from reagent grade As<sub>2</sub>O<sub>3</sub> and Sb<sub>2</sub>O<sub>3</sub> maintained at temperature for at least 24 hours.

#### Sb Solubility Information Flawed

Antimony analyses from the same solutions used above produced solubility trends which did not fit data in the literature or results from leaching tests on Giant arsenic. The available information is presented in an attached table. The Hazen data, plotted near the bottom of the chart contrasts with the information from Seidell (Schulze, Gayer) and from the test data.

### Arsenic Extraction

#### Water Leach Extracts 95.9% of As<sub>2</sub>O<sub>3</sub>

Water Leach Test No. 2, performed June 9 at Lakefield extracted about 42% in Stage 1 and about 89% of the remainder in Stage 2. Total extraction was 95.9%. The test was performed at 95C.

### Antimony Removal

#### Little or Slow Chloride Removal of Antimony

Tests performed at Lakefield showed no antimony removal from arsenic solution in two hours. Cooling to ambient removed some antimony after 48 hours. Arsenic loss resulted from the cooling in one test.

Chloride Addition (% stoichiometric)	Antimony Elimination (%)	Arsenic Loss (%)
41	78	26
83	42	0

### **Neutralization Removes Some Antimony**

Sodium hydroxide, calcium oxide, and ammonia were used to raise solution pH and attempt to precipitate antimony. Solutions were analyzed after 2 hours (Initial Sb Removal) and after cooling and standing for 48 hours (Final Sb Removal). Results of the tests are summarized below:

Neutralizing Agent	Initial Sb Removal (%)	Final Sb Removal (%)	Arsenic Loss (%)
NaOH	0	38	8
CaO	100	100	100
NH <sub>4</sub> OH	42	42	0

### **Tartaric Acid and Activated Carbon Removes Sb and As**

A test was performed at Hazen to evaluate the possibility of complexing antimony with tartarate ion and then recovering the complex on activated carbon. Because of the problems encountered at Hazen in dissolving antimony, the solution concentration was low, ~2.2 ppm Sb. This was entirely removed from solution, but a significant amount of the arsenic (~20%) also condensed on the carbon.

### **Future Testwork**

#### **Lakefield Research Proposal**

Lakefield Research has recommended three test series to further evaluate the Hot Water Process, the Methanol Process, and the solid-liquid separation of the leach pulps from the above tests. Work should be performed on only the process being considered.

The Hot Water Process testwork would be a program of locked cycle tests performed in two phases:

- A six-cycle series to evaluate impurity build-up, and
- A two-cycle series, using the saturated solution from the first series, to evaluate the effect of a dispersant.

The Methanol Process testwork would be a similar six-cycle, locked cycle test series to evaluate impurity build-up. The work would include vacuum distillation of the methanol.

The solid-liquid separation testwork would evaluate the settling and filtration characteristics of both water leached and methanol leached arsenic material.

## **Hazen Research Proposal for Marketing Sample**

Hazen Research has provided a quotation for producing a 200 kg sample of Water Leach Process product. They plan to use a continuous process, with recycles, similar to the locked cycle testwork proposed by Lakefield. This could be a valuable confirmation of the Lakefield work, while producing a marketing sample.

### **Possible Antimony Removal Testwork**

While the testwork has not established an economic method of removing antimony from arsenic solution, it has indicated some possible areas of work. The table below lists potential methods of removing antimony from solution, in estimated order of probability of success:

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|---|---|
| 1. Complexing with tartaric acid and removing the complex on activated carbon | Evaluate reduced carbon addition and carbon recycle.<br><br>Evaluate tartarate complexing, arsenic crystallization, and then carbon removal of complex. |
| 2. Chloride precipitation of antimony   | Determine cause of antimony precipitation with time and cooling. If it is a time effect, seeding with Sb crystals could aid removal.                    |
| 3. Neutralization with ammonia  | Determine ammonia requirement for antimony removal. Evaluate possibility of ammonia recovery and recycle.   |
| 4. Neutralization with lime (CaO)   | Evaluate reduced additions to selectively remove antimony.  |

The first two options would be very important if they allow elimination of antimony from solutions containing ammonia. This would allow use of alternate processes which would use the higher arsenic solubility in ammonia solutions to reduce heat requirements.