

GIANT YELLOWKNIFE MINES LIMITED

PURIFICATION AND RECOVERY OF
ARSENIOUS OXIDE FROM ROASTER DUSTS

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PURIFICATION AND RECOVERY OF

ARSENIOUS OXIDE FROM ROASTER DUSTS

SUMMARY

PURIFICATION AND RECOVERY OF
ARSENIOUS OXIDE FROM ROASTER DUSTS

SUMMARY

The recovery of arsenious oxide (As_2O_3) from Giant roaster dusts has been studied in both laboratory and pilot scale investigations.

Conclusions

General conclusions are as follows:

1. A purified As_2O_3 product, averaging 99.43% As_2O_3 , 0.021% Fe and 0.237% Sb can be recovered from the current production of Giant baghouse dust at a total As_2O_3 extraction efficiency of 99.02% in a two-stage hot water leach process. The marketing standard for maximum impurity levels of 0.02 wt % Fe, 0.20 wt % Sb cannot be consistently achieved. A product specification of 0.05 wt % Fe, 0.40 wt % Sb is required for flexibility in plant operation.
2. A two stage hot water countercurrent leach is required to obtain recoveries of refined arsenious oxide adequate for the process to be technically and environmentally viable.
3. Con arsenic pond sludge and Giant baghouse dust cannot be treated simultaneously in a single leach circuit. The content of water soluble salts, other than arsenic, in the Con material fouls leach solutions and retards the dissolution of arsenious oxide from baghouse dust.
4. An As_2O_3 product averaging 99.70% As_2O_3 , 0.02 wt % Fe and 0.147% Sb can be recovered from the Con arsenic pond sludge at a total As_2O_3 extraction

efficiency of 81.55% when treated separately. The marketing standard for maximum levels of As_2O_5 of 0.125 wt % cannot be met in treating Con material. Concentration of As_2O_5 in the crystal product averaged 0.60 wt %.

No final conclusion should be drawn on treating the Con arsenic sludge until pilot scale testing is complete.

5. Final mill effluent levels of soluble As_2O_3 can be reduced by the hot water washing of Giant cottrell dust. A total of 98.94% of the soluble arsenic contained in cottrell dust was extracted and recovered as a calcium arsenate precipitate in a two stage hot water wash. Inexperience in dealing with this material combined with a short pilot plant campaign leave a number of questions still to be answered concerning the optimum operating conditions. Nevertheless the viability of the process has been proven as a technique for the reduction of total arsenic in the Giant mill effluent.

6. Recovery of gold by cyanidation from leach circuit residues will be 85+ %. The washed cottrell dust-leach circuit residue would be treated in the existing Giant carbon plant. The action of cyanide will solubilize some As_2O_3 however the magnitude is expected to be 25% lower than presently experienced. The major environmental gains are in eliminating the soluble As presently reporting in the carbon plant wash thickener overflow solutions.

Recommendations

1) The conventional hot water leach process is both energy cost intensive and inefficient when compared to the pressurized hot water leach process patent claims. It is recommended that the pressurized hot water leach process be studied, in pilot scale if necessary, before proceeding with the conventional process.

2) Heat dissipated during crystallization of As_2O_3 from saturated leach solution should be recovered and recycled to off-set the high energy requirements of the conventional hot water leach process. Several options are discussed in Section 4.

Process Options

Three viable options are known for the purification of arsenious oxide dusts:

1. Conventional Hot Water Leach

The hot water leach process is based on the difference in the solubility of As_2O_3 in hot water (70 g/l at 98 to 100° C) and in cold water (28 g/l at 25° C). The arsenious oxide dust is water leached at 98 to 100° C to produce a saturated solution of As_2O_3 . This saturated solution is separated from the undissolved solids, clarified and cooled to 25° C. As the saturated solution cools, supersaturation is achieved and As_2O_3 begins to crystallize. These crystals are withdrawn and the stripped mother liquor is recycled.

2. Aqueous Ammonia Extraction

The dissolution of arsenious oxide is more efficient in aqueous ammonia solution than in hot water. One pound of arsenic trioxide will dissolve in 1.81 pounds of 9% ammonia solution at 160° F as compared to one pound of arsenic

trioxide in 22.2 pounds of water at 212° F. The energy saving benefits are substantial.

Unfortunately antimony also readily dissolves in aqueous ammonia. Antimony levels in purified As_2O_3 product were two to three times the market standard.

3. Pressurized Hot Water Leach

A Japanese patent issued in 1979 describes a pressure leach process for the purification of As_2O_3 . The patent claims a solubility of 250 g/l As_2O_3 at 160° C and 2 kg/cm² pressure as compared to 70 g/l As_2O_3 at 100° C and atmospheric pressure. If valid the pressurized hot water leach is more efficient and less energy cost intensive than the conventional hot water leach process.

Pilot Scale Studies

1. Giant Baghouse Dust

A two stage countercurrent hot water leach process for the recovery and purification of arsenious oxide (As_2O_3) from current production of Giant baghouse dust has been demonstrated in pilot scale. A crystalline arsenious oxide product assaying 99.43% As_2O_3 , 0.021% Fe and 0.237% Sb was recovered from baghouse dust at a total As_2O_3 recovery of 99.02 ± 0.40%. The final plant residue contained less than 1% of the total As_2O_3 contained in the baghouse dust feed. No soluble arsenic was detected by chemical titration.

A one month 125 locked cycle, pilot plant campaign showed no evidence of impurity accumulation in either recycled leach solutions or crystalline product. No solution purge or bleed was required. Make-up water

(estimated at 4.6%/cycle) was only added to compensate for solution lost by evaporation, sampling and entrainment in plant residue and crystal product.

Product impurities were dependent upon process conditions:

Iron

The Fe standard of 0.02 wt% was exceeded 28.4% of the time. Filtration techniques are available to meet the 0.02 wt% standard however a product specification of 0.02 to 0.05 wt% would allow more flexibility. The concentration of Fe in product is related to:

- 1) Efficient clarification of leach solutions
- 2) Solution pH should be maintained above 4.0 to inhibit the dissolution of iron.

Antimony

The Sb standard of 0.20 wt% was exceeded 46% of the time. Control of solution pH above 4.0 is a necessity to attain the 0.20 wt%. The crystallization of Sb appears to be pH dependant. Antimony does not appear to be a major concern of manufacturer's of arsenic based wood preservative products. A product specification of 0.20 to 0.50 wt% Sb would be more realistic.

As₂O₃ Specification

The As₂O₃ standard of 98% was consistantly attained. Product quality exceeded 99% As₂O₃ 85% of the time.

A two stage hot water leach is required to attain environmentally acceptable residues. Theoretical solubilities were not attained in the pilot plant due to loss of temperature in the thickening-filtration phase of the flowsheet. A total of 33.6 kg of solution must be treated to recover one k.g. of purified arsenious oxide.

Heat removed during crystallization must be recovered to defray the energy costs of the hot water leach process.

An extrapolated flowsheet showing the recovery of As_2O_3 from Giant baghouse dust based on pilot plant results is presented. Figure S1.

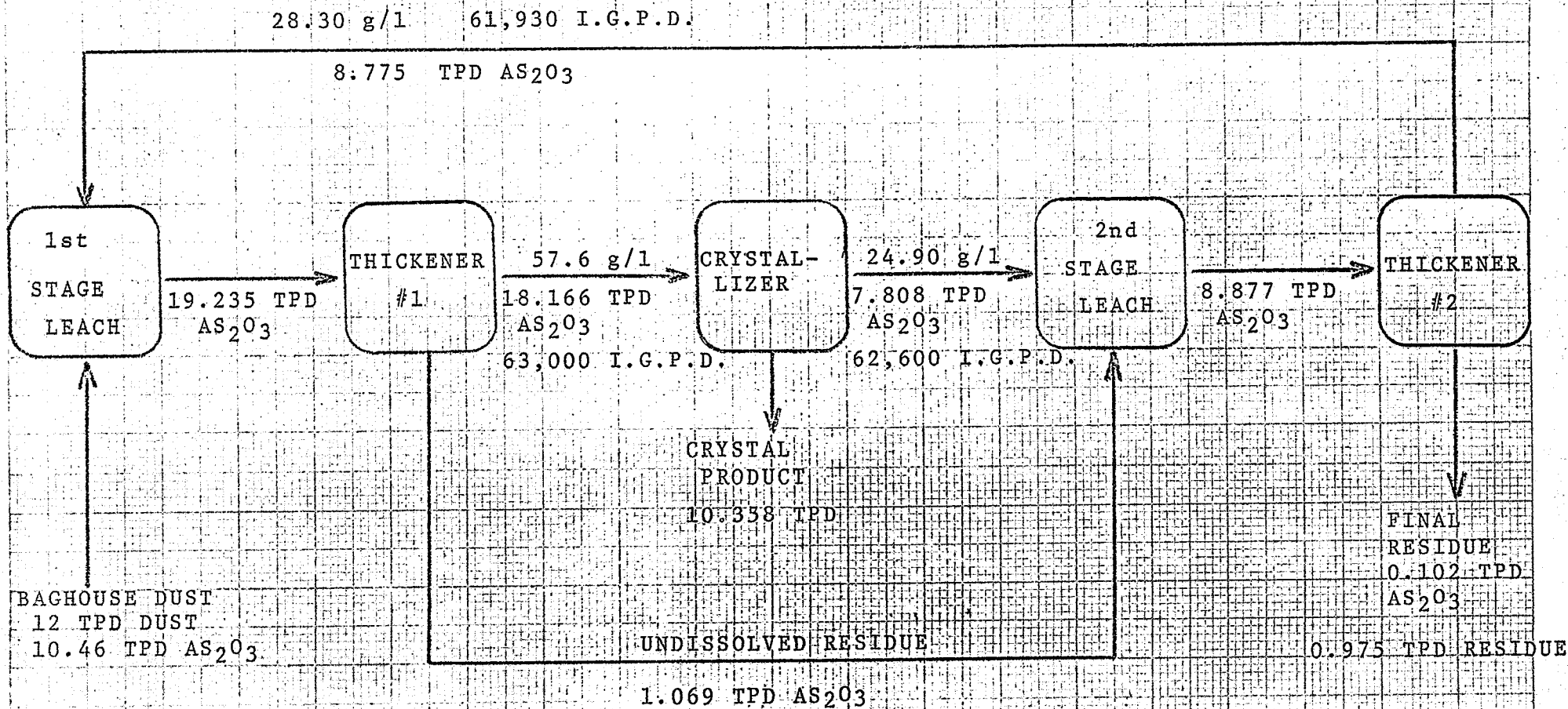
2. Giant Cottrell Dust

Cottrell dust is low in arsenic (2.40% As) and yields a low arsenic content in leach solutions. Cottrell dust has an extremely large surface area, and in the process of introducing this material into the arsenic leaching circuit containing saturated arsenic solutions, it is possible it will absorb arsenic in excess of its original content. Due to the large surface area of the material, it will be difficult or impossible to again remove it by dewatering and washing. For this reason, it has been suggested the cottrell dust be left out of the leach feed and then combined with the leach residue for washing treatment prior to the gold extraction process.

Under optimum conditions 98.94% of the soluble As_2O_3 contained in cottrell dust can be extracted in a two stage hot water wash and recovered as a calcium arsenate precipitate. The calcium arsenate precipitate can be stored underground or chemically fixed and disposed of on surface.

The efficiency of removal is related to: 1) wash temperature;
2) slurry density.

FIGURE S1. Recovery of As_2O_3 from Baghouse Dust



3. Con Arsenic Pond Sludge

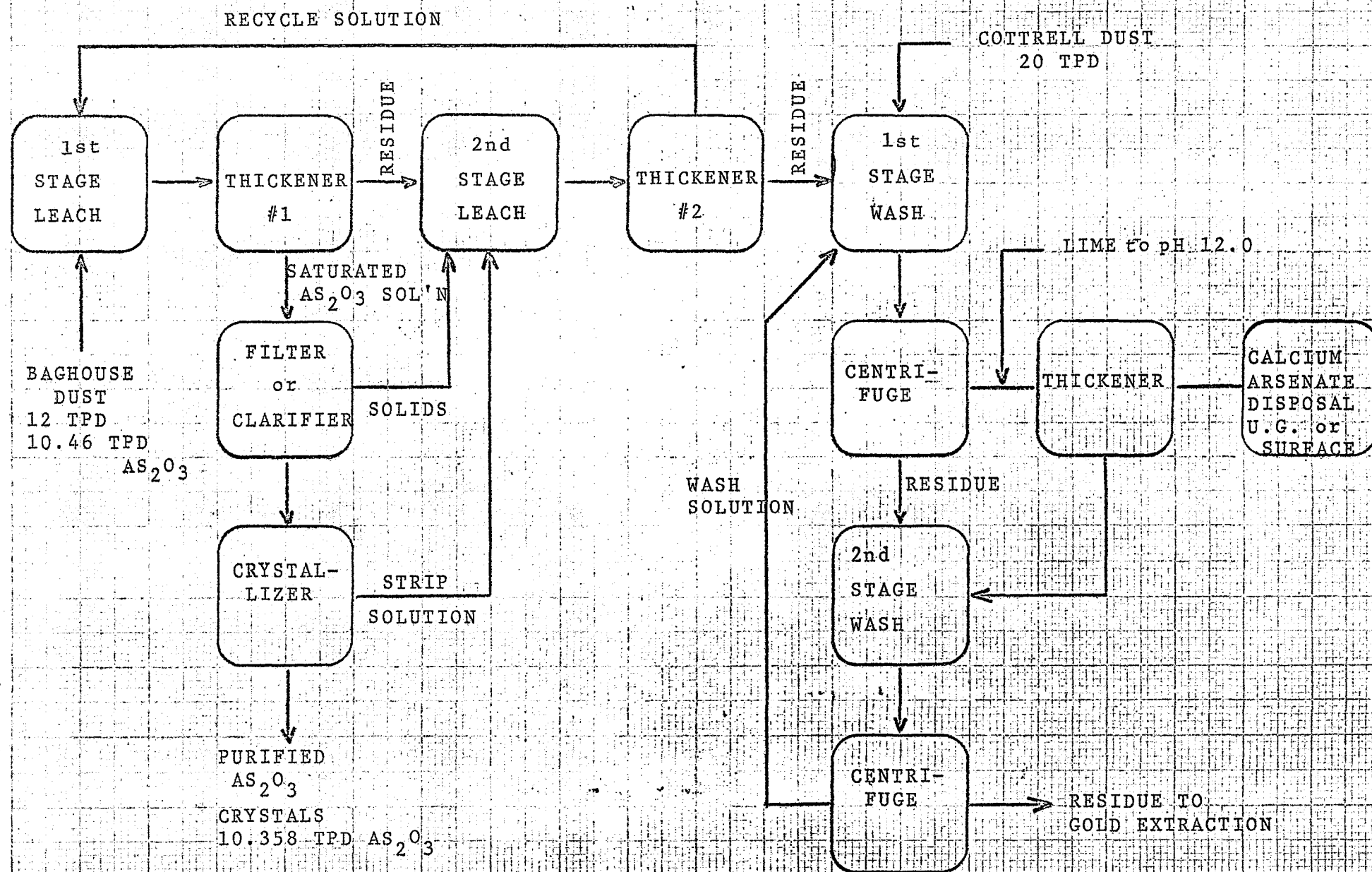
A two stage countercurrent leach process for the recovery and purification of As_2O_3 from Con arsenic pond sludge is presently being piloted. Results are not yet available. Early indications suggest a separate circuit will be required to treat this material.

Flowsheet

A flowsheet (Figure 5.3) was derived from the pilot plant studies, including: 1) Recovery of As_2O_3 from current production of baghouse dust and the production of a purified As_2O_3 product.

2) Extraction of soluble As_2O_3 from cottrell dust prior to gold cyanidation and its subsequent recovery as calcium arsenate.

FIGURE S.3 Schematic Flowsheet: AS_2O_3 Purification and Recovery



PURIFICATION AND RECOVERY OF
ARSENIOUS OXIDE FROM ROASTER DUSTS

SECTION 1

PROJECT HISTORY AND BACKGROUND

GIANT YELLOWKNIFE MINES LIMITED

PURIFICATION AND RECOVERY OF ARSENIOUS OXIDE FROM ROASTER DUSTS

SECTION 1: PROJECT HISTORY AND BACKGROUND

1.1 Background

Low grade arsenic trioxide is a major by-product of the Giant Yellowknife gold milling process. The gold at Giant occurs in complex association with a mixture of arsenopyrite, pyrite, stibnite and a wide variety of antimony sulphosalts. Most of the gold is believed to occur either as submicroscopic inclusions or in solid solution in arsenopyrite. Economic recovery of this gold is thus dependent upon the removal of arsenic, antimony and sulphur prior to cyanidation.

This elimination is achieved by the roasting of a sulphide concentrate, fuming off the arsenic as gaseous arsenic trioxide. This arsenic trioxide is condensed and recovered from the roaster off-gas stream in a baghouse type dust collector. The arsenic trioxide dust is then pneumatically conveyed from the baghouse to specially prepared underground storage vaults located in the permafrost.

1.2 Environmental Restrictions

The level of arsenic trioxide in the Yellowknife environment has become an emotional issue in the past several years and is thus receiving priority attention from government and environmental institutions.

In May of 1978 the N.W.T. Water Board issued Giant Yellowknife Mines with a water use license. Among other requirements, this license dictates that by 1981 total arsenic in the Giant mill tailings pond effluent average 0.5 ppm. Present effluent levels average greater than ten times this standard.

Arsenic is present in the Giant mill effluent streams in many different forms ranging from the relatively innocuous stable arsenopyrite to the soluble As^{+3} and As^{+5} states. The total arsenic contained in the mill effluent can be broken down as follows:

<u>Mill Tailings Stream</u>	<u>As Disposal Rate</u>	<u>% of Total As In Final Tailings</u>
Carbon Plant Thickener O/F	166 lbs/day	17.7%
Carbon Plant Barren	468 lbs/day	71.3%
Calcine Wash Thickener O/F	64 lbs/day	9.8%
Calcine Barren Bleed Solution	6 lbs/day	0.9%
Flotation Tails & Calcine Residue	2 lbs/day	0.3%
	656 lbs/day	
Minewater	30 lbs/day	

It is quickly apparent that 89% of the total arsenic in the mill tailings is associated with the treatment of roaster cottrell dust. A significant percentage of this arsenic is in the soluble As^{+3} and As^{+5} states hence by eliminating or reducing this release of arsenic, significant improvements in effluent quality would be made.

Technically, the options available to meet the Water Board arsenic standard prove to be costly and limited in effectiveness. The major options considered at Giant include:

- A) Ferric Sulphate Treatment
- B) Underground Storage of Cottrell Dusts
- C) Separate Ponding of Cottrell Dust Treatment Wastes.

See Appendix A.

The removal of soluble arsenic from cottrell dust prior to treatment for gold recovery is favoured over any of the above options.

In October of 1979 the Department of Environment announced in the Canada Gazette their intention to set a gold roasting plant stack emission of 20 mg/scm. Giant Yellowknife will continue to meet these standards by recovering greater than 99.9% of the arsenic fumed off during roasting. The

recovered arsenic trioxide dust has to be stored in costly underground storage vaults selectively located in the permafrost. The long term presence of this stored material is a continued source of friction between the Environmental Protection Service and Giant Yellowknife Mines.

1.3 Project History

In January of 1978 Giant Yellowknife Mines was approached by the forest products division of Koppers Co. Inc.. Koppers expressed an interest in purchasing unrefined arsenic trioxide from Giant for use in producing arsenic acid for wood preservatives and other agricultural applications at their Conley, Georgia plant. A similar request was received from the F.M.C. Corporation of Chicago in April 1978. Small samples of Giant baghouse dust were shipped to both companies.

F.M.C. reported the unrefined baghouse dust to be unsuitable for use in their process. Discussions held with Koppers over the summer of 1978 led to an exchange of plant visits. During this period arrangements were made to ship approximately 14,000 pounds of Giant baghouse dust to the Kopper's Georgia plant for pilot scale studies. Results of the trial run indicated that the Fe content of the Giant baghouse dust made the material unsuitable for use in the Kopper's process. Apparently the Fe arsenates contaminate the final product.

In the interim a market study conducted by the Falconbridge marketing department indicated an increasing demand for arsenic trioxide in the U.S. for use in the manufacture of wood preservatives. A steady domestic production level coupled with an increased market demand had resulted in an arsenic deficit in the U.S. in 1973 with corresponding increases in the price paid for arsenic.

Asarco's Tacoma copper smelter is experiencing difficulty in complying with new environmental restrictions which will affect their ability to manufacture arsenic products. In addition the upcoming Philippine Lepanto

smelter will consume its own high arsenic copper concentrates which are currently treated by Asarco at Tacoma, thereby removing a source of arsenic to the U.S. market. These two factors could combine in the coming years to further increase the deficit of arsenic in the U.S. market. (1) The marketing department subsequently indicated a potential market for 6,000 tons/year of refined As_2O_3 .

The favourable market conditions have combined with tightening environmental restrictions to point out the need for a feasibility study of the potential for recovering, refining and marketing arsenic trioxide from Giant roaster dusts. This report deals with the evolution and subsequent testing of a process to refine arsenic trioxide.

1.4 Sources of Arsenic Trioxide

Supplies of low grade arsenic trioxide are readily available to Giant from three major sources:

Current Giant Baghouse Dust Production

The Giant gold milling process produces approximately 10 to 12 tons per day of a low grade arsenic trioxide dust, averaging 70% As (92% As_2O_3 equivalent), 2% Fe, 0.4% Sb and 0.10 oz/ton Au. This stream is estimated as a potential source of 3,100 tons/year of purified As_2O_3 .

Underground Storage at Giant Yellowknife

Low grade arsenic trioxide has been recovered as a mill by-product since the installation of the cold baghouse in late 1958. Total tonnage stored since 1958 is estimated at 154,000 tons at an average grade of 64% As (84% As_2O_3 equivalent). The gold content is variable ranging from 0.09 to 0.94 oz/ton. The average is estimated to be 0.40 oz/ton Au.

Recovery of this material is not being considered at present due to the potential environmental hazards in mining or disturbing these underground storage vaults.

Con Arsenic Ponds

The Con Mine in Yellowknife also recovered arsenic trioxide from their now defunct arsenopyrite roaster operation, however, the arsenic trioxide recovered is in the form of a water slurry. These high density sludges are presently stored on surface in containment ponds with approximately 40,000 tons of material available at an average grade of 54% As (70% As_2O_3 equivalent), 10.6% Fe, 0.8% Sb and 0.37 oz/ton Au. Slightly greater than 90% of the contained As_2O_3 is soluble.

Giant Cottrell Dust

Approximately 15 tons/day of cottrell dust is recovered from the Giant roaster off-gas stream. This material is predominantly fine roaster calcine containing some unroasted sulphide. On average the cottrell dust also contains 2% arsenic, 50% of which is water soluble. Although not significant on a market basis the removal of this arsenic will greatly improve effluent levels.

1.5 Market Standard for Purified As_2O_3

The market standard for refined arsenic trioxide varies dependent upon the desired end product. In studying these standards, the Falconbridge marketing department has suggested the following: (2)

<u>Element</u>	<u>Maximum Impurity Level</u>
As_2O_3	Greater than 98.0 wt%
As_2O_5	0.125 wt%
Fe	0.02 wt%
Sb	0.20 wt%
Pb	0.02 - 0.08 wt%
Zn	0.005 wt%
Bi	0.1 - 0.25 wt%
S (Sulfides)	0.05 - 0.1 wt%

No screen analysis was specified however a particle size greater than 10 microns was preferred to minimize dusting during handling.

Colour: Off-white to light grey.

PURIFICATION AND RECOVERY OF
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SECTION 2

LABORATORY METALLURGICAL INVESTIGATIONS

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LABORATORY METALLURGICAL INVESTIGATIONS

2.1 Preliminary Studies

The Falconbridge Metallurgical Laboratory initiated on Giant's behalf a literature search and lab scale study on the purification of arsenious oxide from Giant roaster dusts. (3)

2.1.1. Literature Survey

A survey of the literature and other information sources provides three viable options for the purification of arsenious oxide dusts:

2.1.1.1. Hot Water Leach Process

The hot water leach process was used by Beattie Mines (Quebec) and Deloro Mines (Ontario) to refine arsenious oxide, however very little information can be found on either process. The hot water leach process is based on the difference in the solubility of As_2O_3 in hot water (70 g/l at 98 - 100° C) and in cold water (28 g/l at 25° C). Basically the arsenious oxide dust is water leached at 98 to 100° C to produce a saturated solution of As_2O_3 . This saturated solution is separated from the undissolved solids, clarified and cooled to 25° C. As the saturated solution is cooled, supersaturation is achieved and As_2O_3 begins to crystallize. These crystals are withdrawn and the stripped mother liquor is recycled.

The South African National Institute of Metallurgy has published a report on the feasibility of using the hot water leach process to refine arsenious oxide in flue dusts from the Fairview Mine. (4)

The investigation showed that arsenious oxide of sufficient purity to meet market specifications could be produced if the concentration of impurities in the leach liquors did not exceed certain values. It was found that a two-stage counter current leach was required to obtain recoveries of refined arsenious oxide adequate for the process to be technically viable.

Ammonia Leach

In March of 1979, Koppers Company provided Giant Yellowknife Mines with a report on an aqueous ammonia leach process for the purification of As_2O_3 . (4) This process differs from the hot water leach process in that a 9 to 10% aqueous ammonia solution is used in place of water. One pound of arsenic trioxide will dissolve in 1.81 pounds of 9% ammonia solution at 160°F as compared to one pound of arsenic trioxide in 22.2 pounds of water at 212°F . The energy saving benefits are substantial.

Following dissolution of the As_2O_3 the leach solution is filtered to remove the insolubles. The saturated aqueous ammonia solution is then cooled under vacuum to precipitate a pure arsenic trioxide. The advantages of this process over the hot water leach include:

- 1) Lower energy requirements
- 2) Lower solution volume to be handled
- 3) Lower retention times during leaching. Dissolution is claimed to be almost instantaneous.

Koppers reported arsenic trioxide products containing between 98.0 and 99.5% As_2O_3 and 0.019% Fe obtained from Giant baghouse dust.

Subsequent testing at Thornhill and Giant found that Sb was readily soluble in the aqueous ammonia leach process. The dissolved Sb was recovered in the purified As_2O_3 product resulting in Sb levels consistently above the 0.20 wt% standard.

Hot Water Pressure Process

A Japanese patent issued in 1979 describes a pressure leach process for the purification of As_2O_3 . The patent claims a solubility of 250 g/l As_2O_3 at 160°C and 2 kg/cm^2 pressure as compared to 70 g/l As_2O_3 at 100°C and atmospheric pressure. The patent also claims increased leaching rates at the increased pressure. The major advantages of the pressure leach over the

conventional hot water leach process include:

- 1) Lower energy requirements
- 2) Higher leach densities or lower solution volumes to be handled
- 3) Lower retention times during leaching

2.1.2 Preliminary Laboratory Studies - F.M.L.

The Falconbridge Metallurgical Laboratory initiated, on Giant's behalf, a preliminary laboratory investigation into the purification of arsenious oxide (As_2O_3) produced by the roasting of arsenopyrite gold ores. (3) The testwork was carried out on samples of Giant baghouse dust, recovered from underground storage vaults, and on samples of Con arsenic pond sludge.

The study establishes:

- A) The chemistry of the hot water leach process as it applies to the purification of arsenious oxide from both Giant and Con roaster dust products.
- B) A preliminary flowsheet was developed upon which all future testwork was based. Mr. Hatch's report has been appended, (see Appendix B) as an important source of reference information.

Some of the areas studied are summarized under the following headings:

2.1.2.1 Hot Water Leaching of Arsenious Oxide Bearing Dusts

Solubility of As_2O_3 in Aqueous Solutions

There is a considerable amount of conflicting information regarding the solubility of As_2O_3 . The Handbook of Chemistry and Physics lists solubility values for amorphous As_2O_3 and cubic crystalline As_2O_3 as 101.4 and 114.6 g/l respectively. Mellor lists solubility values for the cubic crystalline form ranging from 60 to 114 g/l. Values between 70 and 80 g/l have been obtained in the treatment of flue dusts from roasting operations.

(3)

The solubility of Giant and Con arsenious oxide bearing dusts was determined by water leaching samples of both material in an excess of water.

	As_2O_3 (Total)	As_2O_3 (Soluble)	Fe	Sb
Giant baghouse dust (from UG storage)	70.7	64	10.6	0.82
Con arsenic pond sludge	66.7	60	2.5	2.5

Effect of Leaching Conditions (Redox - pH^+) on As_2O_3 Dissolution

Leaching tests were carried out on Con material under various conditions, adjusting the pH^+ and lowering the redox of the solution to determine whether greater solubility could be achieved by solution adjustment. Tests were carried out on 94 g of sample (dry basis) and 800 ml of solution, leaching at 95°C for one hour. Residues were washed with 100 ml of hot water to give a total filtrate volume of 900 ml. The conditions and results of these tests are given in Table 2.1.2.1.1

Table 2.1.2.1.1 Solubility of As_2O_3 Under Various Leaching Conditions

Test	Solution	pH^+	Redox (mV SCE)	R E S I D U E		Solution As_2O_3 (g/L) *
				Wt(g)	As_2O_3 % (Total)	
1	Water	2.4	+ 300	29.0	28.6	72
2	Water-Hydrazine	3.0	+ 150	33.2	43.4	67
3	Water - NaOH	5.4	+ 150	33.3	41.5	67

* calculated

All arsenic in the residue is expressed as As_2O_3 . Thus, in Test #1, the residue contains 2.6 g As_2O_3 and 26.5 g of residue containing arsenic in various forms equivalent to 17% As (21.7% expressed as As_2O_3).

The results of these tests indicate that there is no improvement in leaching under reducing conditions or at higher pH^+ . This is in agreement with literature references which indicate the stability of various arsenates under mild reducing conditions. The arsenates which have formed (presumably during roasting) are associated with iron, calcium, etc., and are inert to these leaching conditions. Only the free As_2O_3 is solubilized.

Rate of Dissolution and Solubility of As_2O_3 at Various Temperatures

Tests were carried out to determine the solubility and rate of dissolution of As_2O_3 from Con material. An excess of feed (150 g) was added to 800 ml of hot water in a stirred reaction kettle. Approximately ten minutes were required for the slurry to reach the desired temperature. Samples were withdrawn at various time intervals and rapidly filtered for solution analysis.

The results of these tests carried out at 85°C and 95°C were as follows:

Dissolution Time (h)	Solution As_2O_3 (g/l)	
	85°C	95°C
0.5	47	58
1.0	52	57
2.0	49	61
7.0	53	60

The final solutions at 85°C and 95°C had pH^+ - redox values of 2.05, +325 mV and 2.05, +295 mV respectively. The results of these tests show that dissolution of As_2O_3 is rapid with greater than 90% of the maximum solubility being attained in a 0.5 hour leaching period. The final solubilities of 53 g/l (85°C) and 60 g/l (95°C) are probably low since some cooling of the slurry, occurred during sampling.

Crystallization of As_2O_3

Arsenious oxide is recovered from leach solutions by crystallization. The crystallization of As_2O_3 occurs as the solution is cooled, eventually reaching an equilibrium value of 21 g/l As_2O_3 at 25°C . The precipitation rate is determined by the rate of cooling of solution and also the following which determines the rate at which equilibrium is attained:

- 1) Liquid-crystalline As_2O_3 ratio
- 2) Solution agitation (stirring)

It has been found that seeding the pregnant leach solution causes more rapid crystallization and a closer approximation to the equilibrium solubility line. Mild agitation also brings fresh solution in contact with As_2O_3 crystals.

Using recycle solution in the leaching stage, an 18 hour crystallization period was necessary to reach 28 g/l As_2O_3 . The solution was only subjected to room air temperatures during the cooling-crystallization process. In larger scale operations, the cooling rate can be increased by evaporation of water from the crystallizer.

2.2 Laboratory Studies - Giant

While the second phase pilot plant was being constructed, Mr. E. Foster directed a laboratory study into the hot water leaching of arsenious oxide from varying combinations of Giant roaster dusts and Con arsenic pond sludge. The remainder of Section 2 covers this work.

The solubility of As_2O_3 in each of the arsenic bearing materials under study was determined by leaching each in an excess of water at 100°C .

Solubility of As_2O_3 in Materials Tested

<u>Sample</u>	<u>% Weight Loss</u>	<u>% As_2O_3 Solubility</u>
Con Arsenic Sludge	59.8	89.70
Giant Baghouse Dust	86.2	94.35
Giant Cottrell Dust	9.0	55.21
Three Product Mixture	47.0	94.09
Con Sludge/Baghouse Mixture	57.0	89.70

At the project outset it was planned to combine the Con arsenic pond sludge, Giant baghouse dust and Giant cottrell dust in a single leach circuit. Pilot locked batch tests were simulated in the laboratory using 1500 ml beakers, heated externally, and agitated by mechanical stirring. Solid-liquid separation of the pulp was affected by vacuum filtering with a Buechner funnel and filter flask. Crystallization of the saturated liquor was difficult in the laboratory, but appeared to be resolved by transferring the liquor to a winchester type bottle and agitating it on the rolling machine while it cooled to room temperature.

The product streams tested included:

1) A three product mixture of Con arsenic pond sludge, Giant baghouse dust, and Giant cottrell dust proportioned in the ratio 1:1:1.7.

2) A mixture of Con arsenic pond sludge and Giant baghouse dust proportioned in the ratio of 1:1.

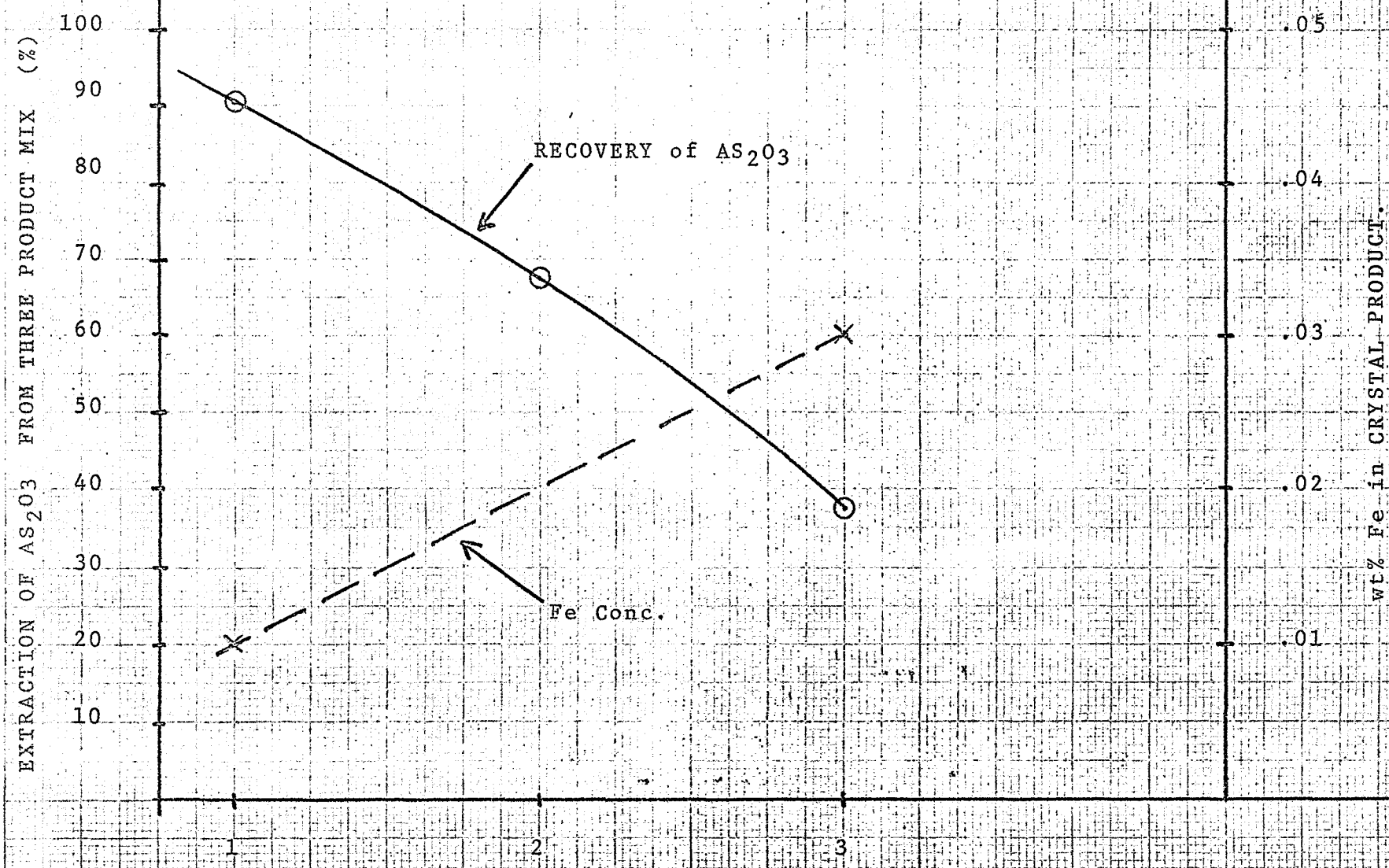
2.2.1 Three Product Mixture

In this locked batch test, a 150 gm sample was pulped with 1,000 ml water and agitated for two hours at 200° F. Pulp was filtered and the saturated solution crystallized. The residue was repulped in the stripped solution for re-leaching. Pulp filtered. The solution was recycled with another 150 gm sample for the second cycle.

Results of the first cycle were encouraging. Arsenic crystals assaying 99.96% As_2O_3 and 0.01% Fe were produced. The final residue weighed 82.9 gm and assayed 4.75% As_2O_3 . Unfortunately, recycling solutions had an adverse effect, both on arsenic solubility and crystal production. At the end of the second cycle the residue weight increased to 100.2 gm and assayed 21.74% As_2O_3 . After the third cycle the residue increased to 120 gm, and assayed 34.59% As_2O_3 . The quantity of arsenic crystals was drastically reduced for the second cycle; the grade remained high, 99.95% As_2O_3 but the iron content increased to 0.03% Fe_2O_3 . Figure 2.2.1

FIGURE 2.2.1.

Recovery of As_2O_3 from mixture of Con Arsenic Pond Sludge, Giant Baghouse dust and Cottrell dust with closed recycle of solutions.



Water leaching cottrell dust demonstrated large amounts of iron and alumina are readily soluble. It is suggested the adverse results after the initial cycle are attributed to the presence of cottrell dust in the mixture. Iron and alumina are dissolved in preference to arsenic.

Three cycles of hot water leaching on the Three Product Mixture were required to produce a low arsenic residue. Solubility was 47.6%. The leach residue assayed:

<u>Au</u>	<u>Fe</u>	<u>As</u>	<u>Sb</u>
1.83 oz/t	26.3%	2.79%	0.64%

The final leach solution contained 8.81 gpl As_2O_3 . As solubility of As_2O_3 is quite rapid (within one hour), this solution assay indicates the dissolved values retained in the pulp are difficult to remove by washing, due to the large surface area of the cottrell dust.

2.2.2 Con Arsenic Sludge & Baghouse Dust

Cottrell dust is normally relatively low in arsenic (2.40% As) and yields a low arsenic content in leach solutions. Cottrell dust has an extremely large surface area, and in the process of introducing this material into the arsenic leaching circuit containing saturated arsenic solutions, it is possible it will absorb arsenic in excess of its original content. Due to the large surface area of the material it will be difficult or impossible to again remove it by dewatering and washing. For this reason, it has been suggested the cottrell dust be left out of the leach feed and then combined with the leach residue for washing treatment prior to the gold extraction process.

In two series of laboratory locked batch tests treating a mixture of Giant baghouse dust and Con arsenic material, the concentration of impurities resulting from recycling leach solution increased to the degree the solubility of arsenious oxide and production of crystals were drastically reduced. After nine cycles, the final residue assay for As_2O_3 was the same as the feed sample.

An attempt to clean the solutions by adjusting the pH to 9.0 and filtering off the precipitate, removed the impurities but also precipitated half the arsenic content in the solution as ferrous and aluminium arsenates.

The bulk of the contaminants, iron, alumina, and magnesia originate in the Con arsenic pond material. A rather complete analysis of this material reported here refers to a sample collected in July 1977 and reported by T.L. Charlton, Cominco Ltd.

<u>Au</u>	<u>As₂O₃</u>	<u>Sb</u>	<u>Fe</u>	<u>SO₂</u>	<u>Al</u>	<u>Zn</u>	<u>CaO</u>	<u>SO₄/S</u>
0.42 oz/t	69.45%	0.40%	3.6%	4.4%	0.74%	0.16%	0.89%	1.0%

The trend of increasing contamination by recycling solutions is indicated in the following tables. Although the dissolution of arsenious oxide was reduced, the purity of the arsenic crystals was not materially affected.

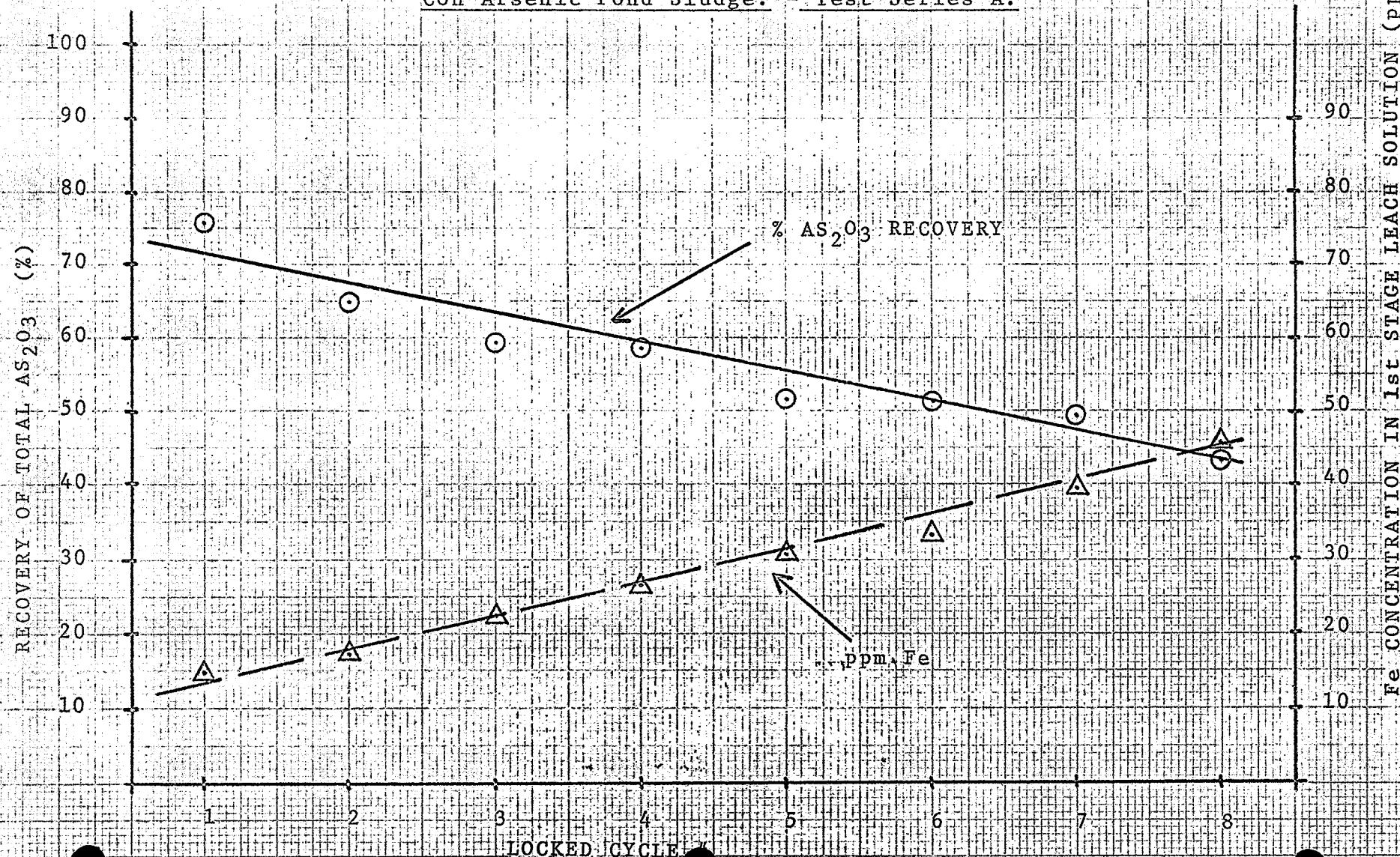
Series A

A 100 gm mixture in 1000 ml water for the first cycle. Subsequent batches were leached in recycle solution. Figure 2.2.2.A.

<u>Cycle</u>	<u>1st Stage Fe (ppm)</u>	<u>Sol'n Sb (ppm)</u>	<u>2nd Stage Fe (ppm)</u>	<u>Sol'n Sb (ppm)</u>	<u>As₂O₃ As₂O₃%</u>	<u>Crystals Fe %</u>	<u>As₂O₃ Solu- bility Percent</u>
1	14.8	27	3.2	48	99.7	.03	75.9
2	17.2	33	9.2	50	99.6	.01	65.5
3	22.4	39	11.6	55	98.3	.02	59.3
4	26.7	48	12.0	67	100.0	.04	58.4
5	30.9	59	13.2	76	99.7	.02	51.6
6	33.3	75	16.9	89	99.1	.02	51.2
7	39.6	89	20.7	92	99.2	.03	49.3
8	45.9	97	27.3	99	99.7	.02	43.2

In test series 'B', baghouse dust and Con material were leached at 10% solids using synthetic arsenic liquors (40 gpl As₂O₃) for the first cycle leach and 20 gpl As₂O₃ for the second cycle leach. In all subsequent cycles, the stripped solution after crystallization and second stage liquor were recycled.

FIGURE 2.2.2.A. Recovery of As_2O_3 from a mixture of Giant Baghouse dust plus Con Arsenic Pond Sludge. - Test Series A.



The natural pH of the pulp varied between 3.1 and 3.5. After seven cycles were completed it was noted the final residue weight increased from 115 gm (1st cycle) to 200 gm (7th cycle). There was a corresponding decrease in the solubility of arsenious oxide, from 67.0% to 45.5%. Chemical analysis of leach solutions indicate a gradual increase of iron and antimony. However qualitative tests indicate the bulk of the contaminants to be Group II metals including alumina and silica.

Series B

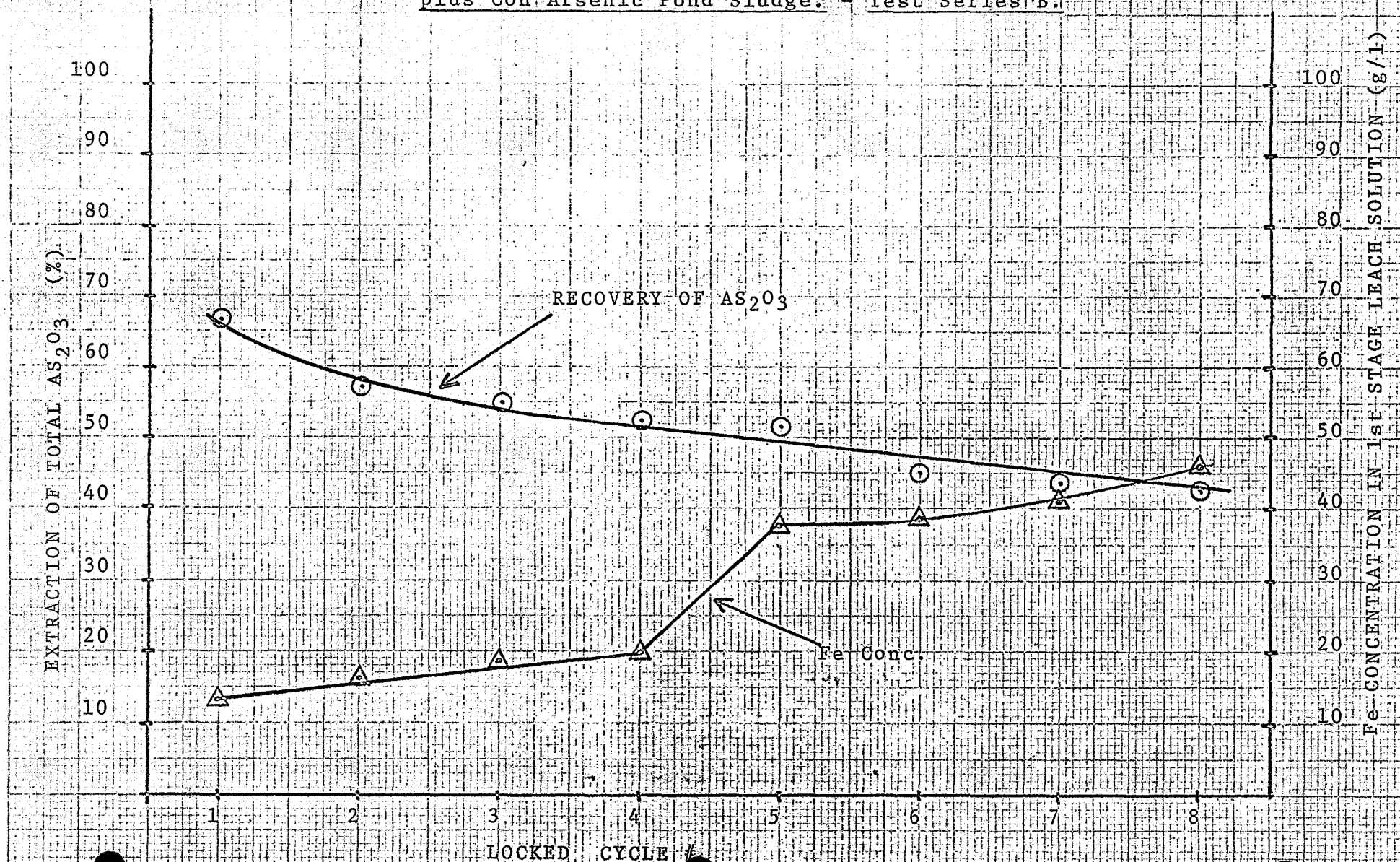
A 300 gm mixture in 3000 ml synthetic arsenic solution for the first cycle. Subsequent batches were leached with recycle solution. Figure 2.2.2.B.

Cycle	1st Stage Fe (ppm)	Sol'n Sb (ppm)	2nd Stage Fe (ppm)	Sol'n Sb (ppm)	As ₂ O ₃ Crystals		As ₂ O ₃ Solu- bility Percent
					As ₂ O ₃ %	Fe %	
1	13.4	28	2.8	54	99.95	.02	67.0
2	16.2	35	8.4	57	98.95	.02	57.8
3	18.8	41	16.8	58	99.92	.02	55.3
4	19.6	58	17.6	62	99.88	.02	52.7
5	37.6	69	18.4	73	99.95	.02	52.1
6	38.6	72	21.9	86	99.90	.02	45.5
7	41.2	89	23.3	92	99.35	.02	44.0
8	46.0	91	27.0	97	99.02	.02	43.3

In test series 'A' where water was used to start the leach cycle, the same decrease in solubility of As₂O₃ was experienced as the test progressed. After three cycles, due to loss of solution by evaporation and samples for analysis, it was necessary to replenish the volume with water for the fourth cycle. Diluting the impurities restored the solubility of As₂O₃ to 75.3%.

The high content of water soluble salts, other than arsenic, in both cottrell dust and Con material, results in a high concentration of impurities when leach solutions are recycled. This retards the dissolution of arsenious oxide and produces a residue with a high arsenic content.

FIGURE 2.2.2.B. Recovery of As_2O_3 from a mixture of Giant Baghouse dust plus Con Arsenic Pond Sludge. - Test Series B.



Leach tests conducted at the Cominco research facilities in Trail, B.C. on Con arsenic pond sludge and Giant baghouse dust suggest that:

1) Increased As_2O_5 concentrations in Giant baghouse dust leach solutions improves the rate of solubility of As_2O_3 . See Table 2.2.2.1

2) Increased As_2O_5 concentrations in Con arsenic pond sludge has no effect on the rate of As_2O_3 solubilization. See Table 2.2.2.2.

No locked cycle tests were conducted. These results do not indicate what would happen during crystallization.

2.2.3 Giant Baghouse Dust

A series of lab scale locked batch leach tests were commenced using only Giant baghouse dust as the feed sample. Control of several variables in lab scale tests was difficult due to an inadequate heat supply to achieve optimum leaching temperature and the loss of solution by evaporation. The natural pH of baghouse slurry is 6.1. While adjusting pH to the desired range of 4.5 to 5.0, the acidity of the pulp was inadvertently allowed to drop to pH 2.6 for the second and third cycle. This increased the solubility of iron drastically. The initial cycle on baghouse dust extracted 94.5% of the As_2O_3 from the feed sample. The residue assayed 55.05% As_2O_3 and contained only 7.18% weight of the original feed sample.

A total of 18 cycles were completed however the results from the first six were discarded due to the operational problem mentioned above. The results from the other 12 cycles are tabulated in Table 2.2.3.

On average, 99.59 percent of the As_2O_3 contained in Giant baghouse dust is soluble. Control of pH was shown to be very important; readings lower than 4.0 increase the solubility of iron. Figure 2.2.3.1. The average grade of crystals produced was 99.54% As_2O_3 ; 0.028 % Fe, 0.14 % Sb.

TABLE 2.2.2.1

Giant Yellowknife BHD Leach

-All leaches contained 60 g/l BHD
 -All results in g/l

0.0 g/l As₂O₅ Added

<u>Time</u>	<u>As₂O₃</u>	<u>As₂O₅</u>
0	18.0	0.0
10	55.4	0.5
20	56.1	0.6
30	56.2	0.4
60	57.3	0.4

Residue - 17.1 g/l

28.0 g/l As₂O₅ Added

<u>Time</u>	<u>As₂O₃</u>	<u>As₂O₅</u>
0	18.0	28.0
10	62.2	27.7
20	63.1	27.8
30	63.7	28.4
60	64.4	27.8

Residue - 11.6 g/l

60.0 g/l As₂O₅ Added

<u>Time</u>	<u>As₂O₃</u>	<u>As₂O₅</u>
0	18.0	60.0
10	65.7	57.8
20	66.9	57.0
30	69.1	57.8
60	70.7	58.6

Residue - 5.2 g/l

10.0 g/l As₂O₅ Added

<u>Time</u>	<u>As₂O₃</u>	<u>As₂O₅</u>
0	18.0	10.0
10	60.8	9.8
20	61.8	10.0
30	62.6	9.8
60	64.4	10.0

Residue - 12.0 g/l

46.0 g/l As₂O₅ Added

<u>Time</u>	<u>As₂O₃</u>	<u>As₂O₅</u>
0	18.0	46
10	67.0	45.9
20	68.1	46.0
30	68.9	46.0
60	69.4	46.0

Residue 4.3 g/l

* 28.0 g/l As₂O₅ Added

<u>Time</u>	<u>As₂O₃</u>	<u>As₂O₅</u>
0	20.0	-
10	62.2	-
20	61.5	-
30	61.5	-
60	66.6	28.0 g/l

Residue - 9.2 g/l

* In this case the temperature of the leach solution at 20 & 30 minutes was 86° C.

Difference Between As₂O₃ Leached By Assay From Solids Leached in HBD

<u>As₂O₅ Added</u>	<u>As₂O₃ By Assay</u>	<u>Solids Leached</u>	<u>Difference</u>
0 g/l	39.3 g/l	42.9 g/l	3.6 g/l
10	46.4	48.0	1.6
28	46.6	50.8	4.2
46	51.4	55.7	4.3
60	52.7	54.8	2.1

TABLE 2:2.2.2

CAPS Leach

- All leach tests contained 60 g/l CAPS
- All results in g/l

<u>0.0 g/l As₂O₅ Added</u>		
<u>Time</u>	<u>As₂O₃</u>	<u>As₂O₅</u>
0	21.0	0.0
10	40.9	2.0
20	43.2	2.3
30	45.1	2.7
60	45.7	3.4

Residue - 29.5 g/l

<u>20.0 g/l As₂O₅ Added</u>		
<u>Time</u>	<u>As₂O₃</u>	<u>As₂O₅</u>
0	21.0	21.5
10	47.6	21.8
20	48.0	21.5
30	48.9	21.5
60	49.5	20.4

Residue - 24.0 g/l

<u>50.0 g/l As₂O₅ Added</u>		
<u>Time</u>	<u>As₂O₃</u>	<u>As₂O₅</u>
0	20.6	50.5
10	51.5	50.5
20	47.0	50.4
30	52.5	50.7
60	53.2	51.1

Residue - 21.4 g/l

<u>10.0 g/l As₂O₅ Added</u>		
<u>Time</u>	<u>As₂O₃</u>	<u>As₂O₅</u>
0	20.5	10.6
10	48.4	10.9
20	50.2	11.1
30	51.5	11.1
60	52.1	10.9

Residue - 22.6 g/l

<u>28.0 g/l As₂O₅ Added</u>		
<u>Time</u>	<u>As₂O₃</u>	<u>As₂O₅</u>
0	21.0	27.8
10	49.0	43.0
20	50.5	27.8
30	51.0	27.8
60	52.5	27.8

Residue - 23.4 g/l

Difference Between As₂O₃ Leached By Assays From Solids Leached in CAPS

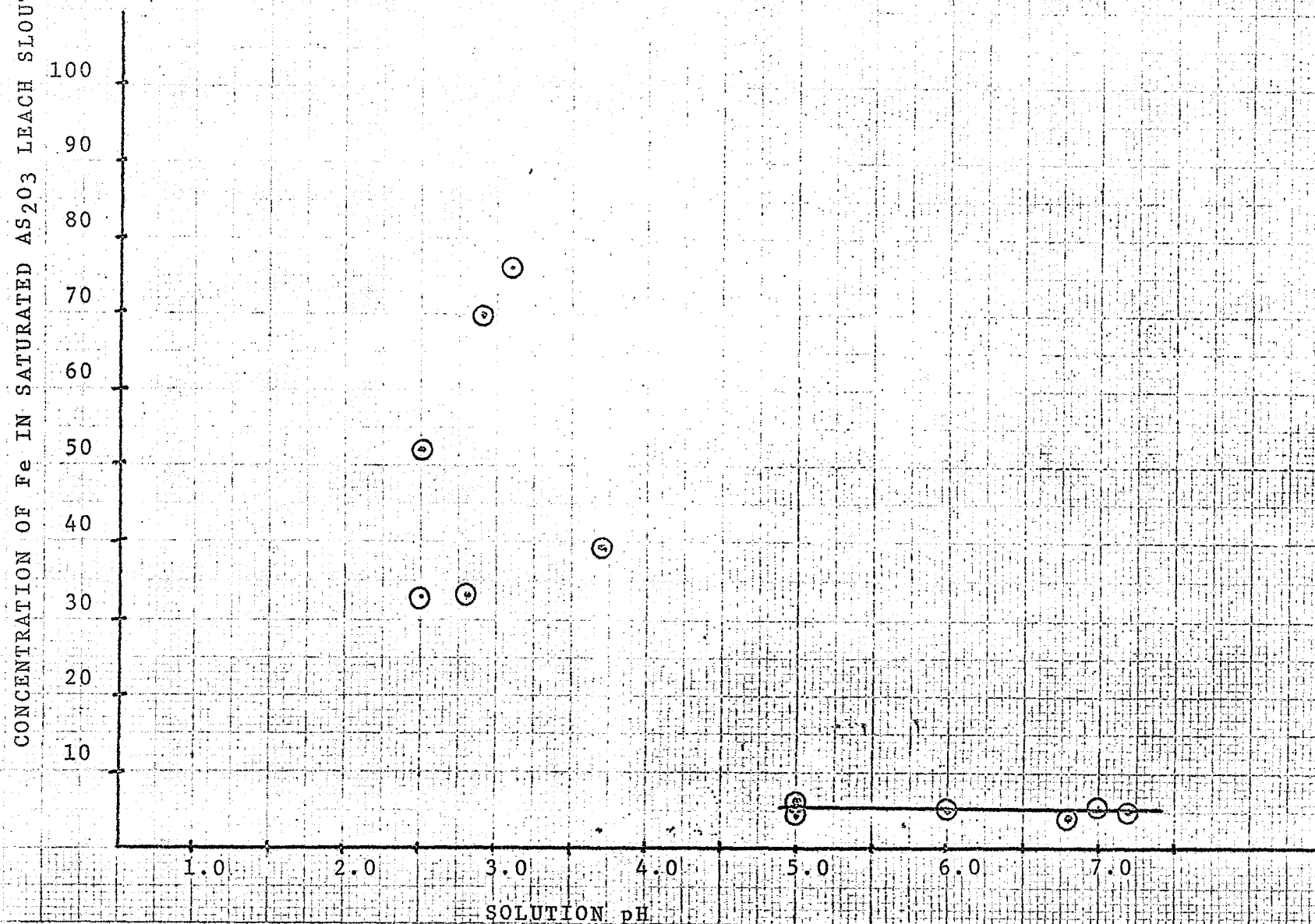
<u>As₂O₅ Added</u>	<u>As₂O₃ By Assay</u>	<u>Solids By Weight</u>	<u>Difference</u>
0 g/l	24.7 g/l	30.5 g/l	5.8 g/l
10	31.1	37.6	6.3
20	28.5	36.0	7.5
28	31.5	36.6	5.1
50	32.2	38.6	6.4

Table 2.2.3

Leaching and Crystallizing Tests on Giant Baghouse Dust - 96.30% of As_2O_3

Cycle	Feed Wt (gm)	Crystallizer Solution			pH	Crystal Product			Stripped Solution			2nd Leach Solution			Final Residue		Recovery Total As_2O_3
		As_2O_3	Fe	Sb		As_2O_3	Fe	Sb	As_2O_3	Fe	Sb	As_2O_3	Fe	Sb	Wt (gm)	As_2O_3	
7	85	57.46	6.4	42.0	4.5	99.70	.02	.14	16.58	2.4	23.0	27.54	47.0	62.0	3.13	15.37	99.41
8	85	59.34	39.0	35.0	3.2	99.20	.03	.13	16.30	35.0	38.0	21.12	2.2	84.0	3.71	15.76	99.29
9	85	49.18	4.2	73.0	6.3	98.95	.02	.14	17.10	1.8	34.0	17.38	21.0	94.0	3.08	10.28	99.61
10	85	42.50	4.2	56.0	4.5	99.95	.03	.12	15.96	2.2	28.0	17.54	2.8	84.0	3.02	17.14	99.37
11	85	44.94	5.0	90.0	6.7	100.28	.03	.15	16.76	1.6	31.0	17.54	1.6	53.0	3.11	9.82	99.63
12	85	45.49	5.2	95.5	5.5	99.28	.02	.15	15.96	1.6	24.0	20.20	2.4	105.0	3.03	7.69	99.72
13	85	46.00	5.4	101.0	6.5	99.55	.06	.18	18.08	2.2	18.0	18.50	2.0	79.70	2.74	8.38	99.72
14	85	44.66	32.8	35.0	2.0	99.55	.04	.13	15.96	30.2	13.0	23.14	10.6	78.0	2.84	12.96	99.55
15	85	48.12	52.0	43.0	2.0	99.88	.02	.13	15.42	37.6	24.0	21.00	35.4	121.0	2.16	3.58	99.91
16	85	54.50	84.0	33.0	2.3	99.56	.02	.15	14.40	67.0	32.0	21.72	38.8	80.0	2.95	12.19	99.56
17	85	46.62	70.0	64.0	2.4	99.22	.02	.12	16.40	76.0	14.4	17.48	51.0	82.0	2.82	7.63	99.74
18	85	42.92	76.0	21.0	2.6	99.39	.02	.14	15.20	72.2	23.3	23.86	77.0	54.0	3.07	12.99	99.51

FIGURE 2.2.3.1. Effect of pH⁺ on Dissolution of Fe from Giant Baghouse dust.



2.2.4 Con Arsenic Pond Sludge

Previous lab scale testing indicates treatment of Con arsenic pond material could involve a potential problem with a build-up of impurities (Group 2 Metals) resulting from recycling leach solutions. A short test program to further investigate the treatment of Con material alone showed that 91.0% of the arsenic (As_2O_3) content was extracted leaving a residue assaying 25.39% As_2O_3 , having 32.2% weight of the original feed. Unlike Giant arsenic, the Con material contains 2.87% As_2O_5 in the leach feed, and 1.49% As_2O_5 in the final residue, an indicated extraction of 83.27% of the contained As_2O_5 . Arsenic pentoxide is an undesirable material in arsenic crystals designated for use as wood preservative.

The problem of decreasing extraction previously encountered by recycling solutions when treating a mixture of Giant baghouse dust and Con arsenic material was not experienced in the series when Con material was treated separately. Extraction of arsenious oxide was 81.55 percent of the arsenic content in the feed. Grade of crystals produced averaged 99.70% As_2O_3 ; .02% Fe; .147% Sb.

The Con arsenic pond sample contained 2.87% As_2O_5 . Concentration of As_2O_5 in the leach liquor was 0.92% and in the crystal product 0.60%.

The locked cycle results are tabulated in Table 2.2.4. The solubility or recovery of As_2O_3 from Con arsenic pond sludge varies over the nine cycles however there is no steady decrease. The concentration of Fe in recycled solution did increase over the nine cycles, however, there appeared to be no effect on crystal product. Figure 2.2.4.1. The natural pH^+ of the Con arsenic pond material lies between 2 and 3. The dissolution of Fe can be raised by raising the pH^+ .

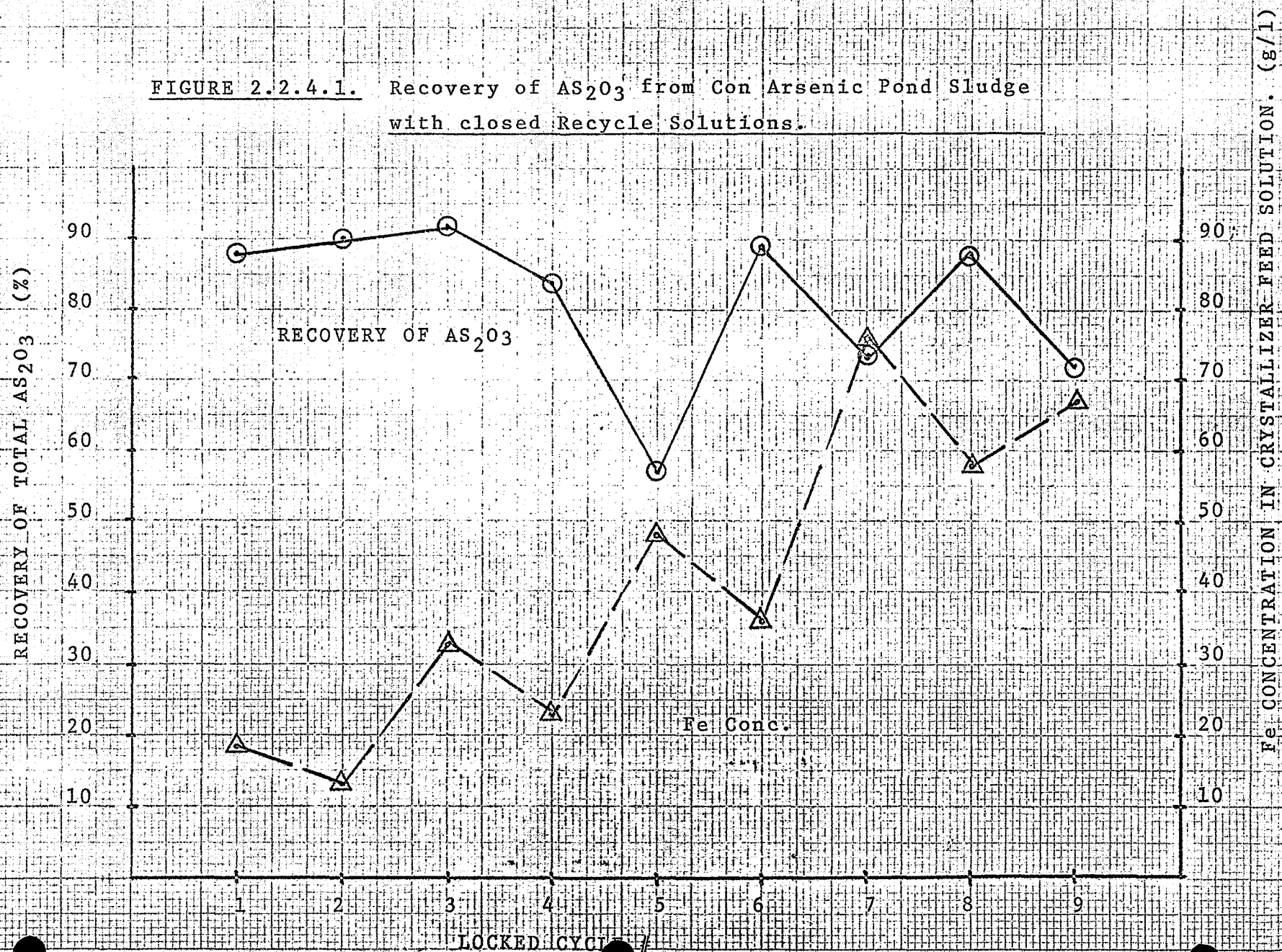
Results suggest the need to treat the Con material on a circuit separate from the Giant baghouse dust.

TABLE 2.2.4

Leaching & Crystallizing Tests on Con Arsenic Pond Samples - 66.80% As₂O₃

Cycle	Feed Wt(gm)	Crystallizer Solution			pH	Crystal Product			Stripped Solution			2nd Leach Solution			Final Residue	
		As ₂ O ₃	Fe	Sb		As ₂ O ₃	Fe	Sb	As ₂ O ₃	Fe	Sb	As ₂ O ₃	Fe	Sb	Wt(gm)	As ₂ O ₃
1	200	36.44	18.4	108.0	3.3	99.59	.02	.11	19.78	15.6	45.0	26.2	4.2	2.18	64.43	25.39
2	200	37.68	13.2	111.0	3.25	99.99	.02	.15	15.50	8.6	19.0	28.06	16.0	2.51	54.30	24.19
3	200	36.70	33.0	97.0	2.85	99.61	.02	.13	26.58	12.0	82.0	27.88	22.4	5.75	48.00	22.18
4	200	41.22	23.0	91.0	3.3	99.94	.02	.17	17.82	22.0	53.0	27.66	21.8	2.70	60.00	36.12
5	200	41.74	48.0	105.0	2.19	99.88	.02	.15	18.62	47.0	38.0	28.72	35.2	2.33	102.5	54.99
6	200	39.08	30.6	154.0	2.35	99.56	.02	.15	18.54	32.2	68.0	26.32	29.6	7.0	54.0	27.11
7	200	46.36	76.0	126.0	2.40	99.22	.02	.14	18.22	59.0	50.0	29.94	55.0	7.25	81.5	43.78
8	200	59.62	58.0	131.0	2.60	99.72	.02	.16	19.12	45.5	55.2	38.88	57.7	4.25	71.0	23.03
9	200	53.08	67.0	147.0	2.80	99.81	.02	.17	18.87	53.3	57.7	40.42	63.3	5.75	81.8	45.87

FIGURE 2.2.4.1. Recovery of As_2O_3 from Con Arsenic Pond Sludge with closed Recycle Solutions.



2.2.5 Gold Extraction From Leached Residues

2.2.5.1 Three Product Mix

Samples of Con arsenic pond sludge, Giant baghouse dust and Cottrell dust were proportioned in the ratio 1:1:1.7 and the mix treated by hot water leaching to provide a supply of low arsenic residue for precious metal extraction tests. A partial analysis of the products follow:

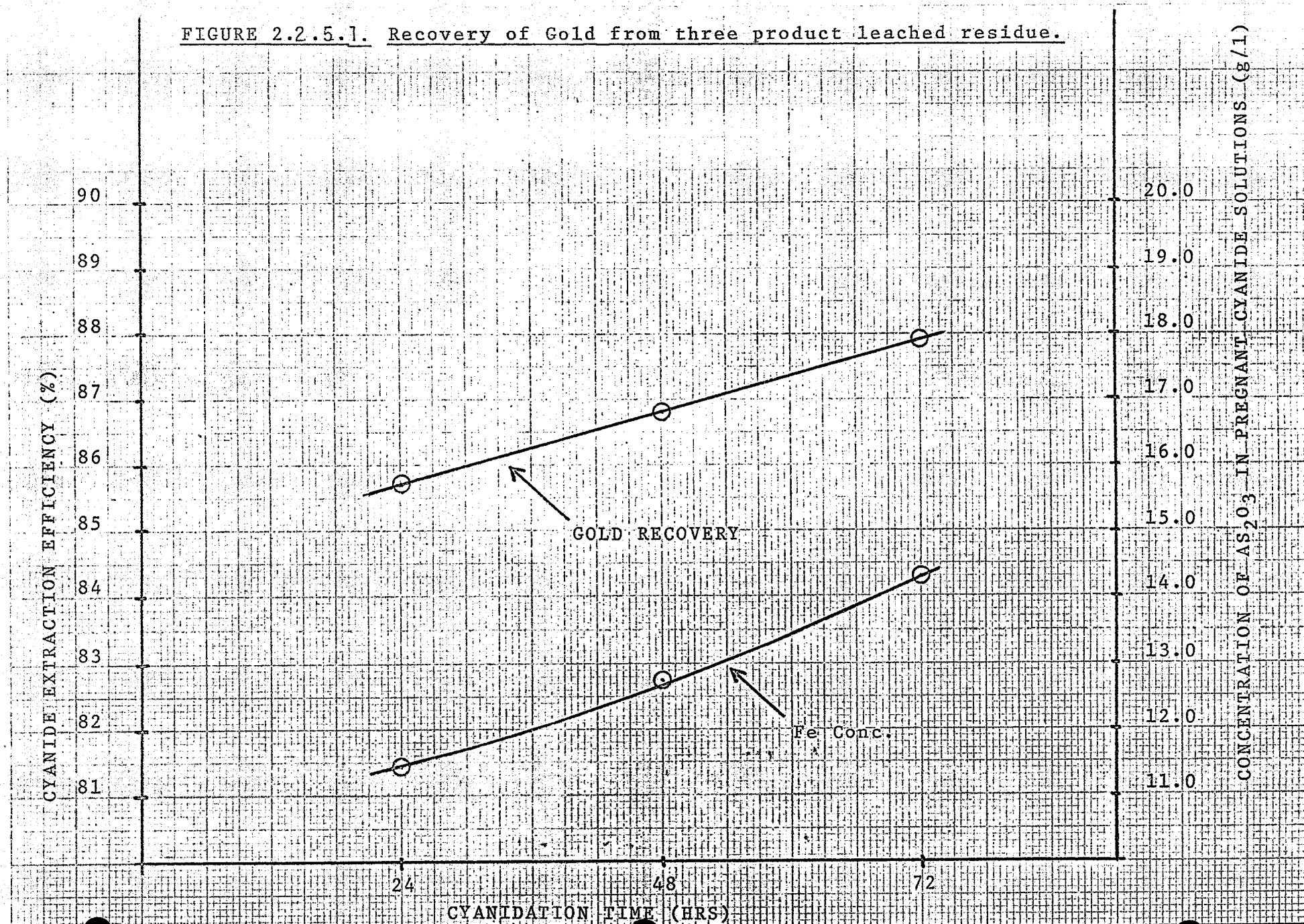
	<u>Au</u> <u>Oz/t</u>	<u>Ag</u> <u>Oz/t</u>	<u>Fe</u>	<u>S</u>	<u>As</u>	<u>Sb</u>	<u>Pb</u>	<u>Zn</u>	<u>Cu</u>	<u>Insol</u>
Con Mine Dust	1.12	3.31	9.25	2.64	50.55	1.19	0.32	0.21	0.07	-
Giant Baghouse Dust	0.06	-	-	-	-	-	-	-	-	-
Giant Cottrell Dust	0.93	-	-	-	-	-	-	-	-	-
3 Product Mix	0.90	0.42	10.8	1.71	33.37	5.58	0.31	0.16	0.08	18.63

Hot water leaching (3 cycles) at a natural pH 6.7 produced a residue for gold and silver extraction tests assaying:

	<u>Au</u>	<u>Ag</u>	<u>Fe</u>	<u>S</u>	<u>As</u>	<u>Sb</u>	<u>Cu</u>
3 Product Mix Residue	1.82 oz /ton	3.14 oz /ton	16.0%	1.29%	17.43%	1.71%	0.23%

Gold extraction tests demonstrated the gold content in the residue is amenable to extraction by either direct cyanidation or by the carbon cyanide process. Gold extraction was 85.7% during a 24-hour period. The pregnant gold solution contained 11.44 gpl As_2O_3 (cyanidation) and 12.76 gpl As_2O_3 (carbon/cyanide) respectively. Extending the treatment period increased gold extraction to 86.8% after 48 hours and 87.9% after 72 hours. Dissolution of arsenic also increased 14.30 gpl As_2O_3 (48 hours) and 17.75 gpl (72 hours). Figure 2.3.1.

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Wash Treatment for Leach Residue & Cottrell Dust Prior to Cyanidation

A 110 gm sample of leach residue (baghouse dust & Con material) was mixed with 340 gm cottrell dust.

<u>Au</u>	<u>As₂O₃</u>	<u>Sb</u>
1.02 oz/t	18.62%	0.55%

The mixture was washed by agitating with water at 100° F for 15 minutes.

Input:	450 gm solids 18.62% As ₂ O ₃ , 0.55% Sb	<u>As₂O₃ (gm)</u>	<u>Sb (gm)</u>
	water (1.0 L nil As ₂ O ₃ , Sb)	83.79	2.47
		0	0
		<u>83.79</u>	<u>2.47</u>

Output:	312 gm residue 13.99% As ₂ O ₃ , 0.60% Sb		
	wash sol'n (1.0 L 40.14 gpl As ₂ O ₃ , 0.6 gpl Sb)	43.65	1.87
		40.14	0.60
		<u>83.79</u>	<u>2.47</u>

The washed residue was treated for gold extraction by the carbon-cyanide process:

	<u>Wt (gm)</u>	<u>Au Oz/T</u>	<u>As₂O₃</u>	<u>Sb</u>
Leach Residue	225	1.14	13.99%	0.60%
Pregnant Sol'n	500	0.32	12.44 gpl	0.38 gpl
Cyanide	215	0.45	11.75%	0.54%

Extraction of gold from the leach residue 62.5 percent. Approximately 19.75% of arsenic and 14.0% of antimony content were taken into solution by the alkaline cyanide solution.

The leach residue from a mixture of baghouse dust and Con arsenic pond material is not sufficiently low in arsenic content to yield a favourable extraction of gold without excessive pollution in the plant discharge effluent.

2.2.5.2 Water Leaching Cottrell Dust For Gold Extraction Treatment

A 250 gm sample of Giant cottrell dust was pulped with 500 ml water at 70° F, agitated 15 minutes and filtered. Procedure repeated twice to provide three cycles of washing:

	<u>Wt</u>	<u>A s s a y s</u>				<u>% Distribution</u>		
		<u>Au</u>	<u>Fe</u>	<u>As</u>	<u>Sb</u>	<u>Fe</u>	<u>As</u>	<u>Sb</u>
Feed	250 gm	-	17.0%	2.29%	0.21%	100.00	100.00	100.00
Filtrate	1400 ml	-	14.01	3.975	0.294 gpl	46.14	9.79	7.85
Residue	236 gm	1.30	2.19%	2.19%	0.205%	53.86	90.21	92.15

A large quantity of gelatinous ($\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$) precipitate was produced by making the filtrate alkaline with ammonia.

Cyanidation of the washed residue using the carbon extraction process reduced the gold content in the residue to 0.16 oz/ton Au, an indicated gold extraction of 87.69%. The pregnant solution assayed 0.762 oz/ton Au, 1000 ppm As_2O_3 and 70 ppm Fe. Currently the solution from the carbon process contains 1300 to 1400 ppm As_2O_3 . It appears the carbon treatment for gold extraction from cottrell dust will invariably produce foul solutions.

2.3 Pulp Settling Rates

Filtration of residues from early pilot plant campaigns had proven to be very difficult. The need for a thickener was quickly realized. Settling rates are available for several of the streams to be treated.

A) Cottrell Dust: 1.46×10^{-3} ft/minute at 20% solids.

B) Residue from Leaching a Mixture of Con Arsenic Pond Sludge, Giant Baghouse Dust and Cottrell Dust

Pulp % Solids	2	4	6	8	10
Settling Rate (Ft/min)	0.206	0.206	0.220	0.464	0.688

The same tests were repeated with the addition of 3 ppm of an anionic flocculant Dearborn, Aquafloc 467.

Pulp % Solids	2	4	6	8	10
Settling Rate (Ft/min)	3.628	3.300	2.681	2.553	2.371

2.4 Centrifuge Testing

The separation of saturated solution from undissolved residue has proven to be extremely difficult in pilot plant scale, especially in the case of Giant cottrell dust. The fine grained solids (100% -400 m, 80% -2 microns) have extremely slow settling rates and tend to quickly blind filter cloth or other filtering media.

The settling rate of undissolved residues from the hot water leaching of Giant baghouse dust is increased by a factor of 10 upon the addition of an anionic flocculant. Cottrell dust settling rates, however, appear to be completely impervious to the addition of flocculating agents. It is theorized

that the high surface area of the wetted cottrell dust quickly absorbs any flocculant. In addition, thus necessitating the use of high flocculant dosages before increased settling rates are observed. In past studies the addition of large dosages of flocculant to cottrell dust appear to impart phixotropic properties to cottrell dust slurry resulting in even more difficult handling problems.

Two pilot scale centrifuges were rented:

- A) A horizontal solid bowl centrifuge with scroll discharge from Bird Machine Co.
- B) A vertical solid bowl centrifuge from Western States Machinery.

Cottrell dust was water quenched and fed to both centrifuges. The results are as follows:

2.4.1 Vertical Bowl Centrifuge

Test Procedures

- 1) Speed control was calibrated to actual bowl speed in R.P.M.
- 2) At a given speed the feed rate was optimized against cake moisture content and centrate % solids.
- 3) At both a high and low feed rate, % moisture in the cake and % solids in the centrate were evaluated against machine speed.

Results:

<u>Vari-Drive Setting</u>	<u>R.P.M.</u>
0 (minimum)	550
1	750
2	975
3	1250 ... severe vibration in this range,
4	1600 ... unsuitable for test work.
5	2075
6	2675
7 (maximum)	3375
8	-
9	-
10	-

2.4.1

Continued

<u>Feedrate</u>	<u>Speed</u>	<u>Feed (% Solids)</u>	<u>Centrate (% Solids)</u>	<u>Cake (% H₂O)</u>
0.5 igpm	2075 RPM (5)	41.5%	0.3 g/l (0.03%)	36.6
1.0 igpm	2075 RPM (5)	41.5%	2.5 g/l (0.25%)	35.9
1.5 igpm	2075 RPM (5)	41.5%	64.7 g/l (6.5 %)	35.3
2.0 igpm	2075 RPM (5)	40.0%	61.8 g/l (6.2 %)	34.2

<u>Speed</u>	<u>Feedrate</u>	<u>Feed (% Solids)</u>	<u>Centrate (Solids)</u>	<u>Cake (% H₂O)</u>
7 (3375 RPM)	0.5 IGPM	40.5%	1.0 g/l	32.4
	2.0	40.5	6.4	34.6
2 (974 RPM)	0.5	42.0	1.6	37.4
	2.0	42.0	33.5	36.5
6 (2675 RPM)	0.5	42.5	0.2	33.7
	2.0	42.5	9.5	32.7

The cake moisture content varies little with machine speed or feed rate, all results falling between 32.7 and 36.6% moisture. Low feed rates and high machine speed gave the cleanest centrate. The best results obtained were 0.2 and 0.3 g/l solids at the 0.5 IGPM feedrate and machine speeds of 2075 and 2675 RPM respectively. The addition of flocculant should readily clear the remaining suspended solid from the centrate even at low dosages.

2.4.2

Horizontal Bowl Centrifuge

The Bird Centrifuge gave a sludge having similar moisture content to that obtained from the Western States machine. It would appear that 32% moisture is the minimum cake moisture obtainable by the use of these centrifuges. The Western States machine was superior in giving the cleaner centrate. Feeding the thickened HCD slurry to the Bird Machine resulted in centrate containing 11% solids.

2.4.3 Washing Entrained Arsenic Solution From Centrifuge Cake (8)

The vertical bowl centrifuge was chosen to determine the washing efficiency of the centrifuge for the removal of residual As_2O_3 solution from the cake.

Procedure

Arsenic pilot plant final residue was leached at 7% solids for one hour, then centrifuged at the optimum speed of 2700 RPM and feedrate of 1/2 IGPM. Once the centrifuge became filled with solids, a cold water wash was introduced at 1/2 IGPM for four minutes. The entire wash discharge was collected at one minute intervals to determine the effectiveness of each subsequent wash. A duplicate test was made using cottrell dust, leached at 15% solids. The low leach densities were chosen to enable maintenance of a slow, constant feedrate.

Data

<u>Arsenic Residue Test</u>	<u>Weight/Volume</u>	<u>As_2O_3 Conc.</u>	<u>% of Total As_2O_3</u>
Cake	7.35 kg	5.11%	46.06
Centrate	97.7 l	4.84 g/l	55.52
Wash #1	2.3 l	1.44 g/l)	0.42
#2	2.3 l	0.07)	
#3	2.3 l	0.02)	
#4	2.3 l	0.01)	
<u>Cottrell Dust Test</u>	<u>Weight/Volume</u>	<u>As_2O_3 Conc.</u>	<u>% of Total As_2O_3</u>
Cake	11.9 kg	2.21%	78.71
Centrate	68.2 l	1.04 g/l	21.27
Wash #1	2.3 l	0.03 g/l)	0.02
#2	2.3 l	N.D.)	
#3	2.3 l	N.D.)	
#4	2.3 l	N.D.)	

Washing of a leached centrifuge cake appears to have an insignificant effect on arsenic removal, the majority of the elimination being in the centrate. The relatively minute quantities removed by washing, 0.42 and 0.02%, were likely dissolved in the water contained within the cake. Washing merely replaced this solution with arsenic-free water.

PURIFICATION AND RECOVERY OF
ARSENIOUS OXIDE FROM ROASTER DUSTS

SECTION 3

PILOT PLANT INVESTIGATIONS

SECTION 3

PILOT PLANT INVESTIGATIONS

The hot water leach process flowsheet found to be technically viable in the lab was chosen as the basis for the pilot scale program. In March of 1979 the first pilot plant was constructed at the Giant Mill. Over the first weeks of operation the pilot plant flowsheet underwent several changes as process difficulties were encountered. A chronological description of each flowsheet and the problems encountered follows:

3.1 Flowsheet #1

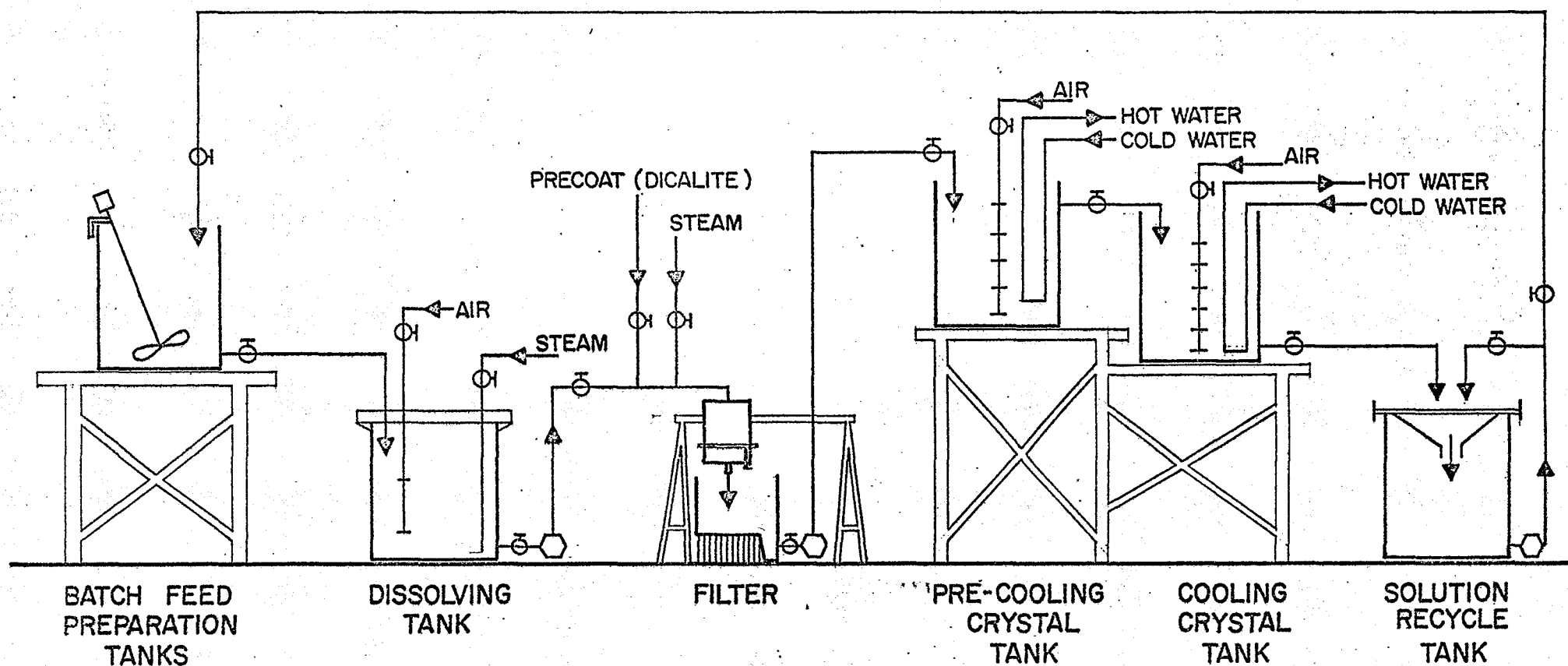
The initial pilot plant flowsheet was an extrapolation of the process scheme evolved in the lab (Fig. 3.1).

Giant baghouse dust was mixed and heated in a 45 gallon polyethylene drum using live steam as the heating media. The slurry was transferred by gravity to a second polyethylene drum to allow sufficient retention time for dissolution of arsenic trioxide. The first tank was mechanically agitated while an air sparge was used in the second tank. The second tank was also heated using live steam.

Following a two-hour leach period, the slurry was pumped through a filter bed of diatomaceous earth (dicalite) separating the undissolved residue from the saturated As_2O_3 solution. The clarified saturated solution was transferred to a third polyethylene tank serving as the first of two crystallizers. Each of the two crystallizer tanks was equipped with a stainless steel cooling coil through which cold water was circulated. Agitation was provided by means of air sparges. The saturated solution overflowed the first crystallizer into the second tank which in turn drained across a 100 mesh screen. The crystallized arsenic trioxide was to be collected on the screen while the stripped mother liquor was collected in a tank for recycle to the batch feed tank.

Figure 3.1

HOT WATER LEACH PROCESS - FLOWSHEET No. 1



Leach conditions employed were as follows:

Leach Tank:	Slurry Density:	4% solids by weight
	Temperature:	95 to 100° C.
	pH:	Natural pH of 7.6 to 8.0
	Retention Time:	2 hours

Unfortunately several process problems were encountered which brought the first pilot plant run to a quick halt. These difficulties included:

- A) The use of live steam in the batch feed and leach tanks upset the water balance by dilution with condensate water. The moisture carried with the steam over the two hour leach time accounted for approximately 25% of the solution volume processed. This continual addition of fresh water interfered with the recycle of solutions from the crystallizer. The problem was corrected by replacing the open steam coils with closed loops which exhausted the condensate into a nearby sump.
- B) The filterability of the undissolved residue proved to be extremely poor. The fine particle size of the residue solids resulted in filter blinding despite experiments with various pore sized filter cloths, different thickness of dicalite and bulking of the filter bed with granular activated carbon. Blinding occurred after passing as little as two to five gallons of leach slurry across a filter surface area of 0.39 square feet. The problem was overcome by placing a heated thickening tank ahead of the filter.
- C) Recycle stripped solutions of plus 120 g/l As_2O_3 were being obtained at 12 - 15° C, indicating a problem in the crystallizing tanks. The rapid cooling of saturated solutions resulted in the production of numerous small crystal nuclei which had insufficient retention time for growth and consequently passed through the screen. The suspension of numerous fine grained As_2O_3 crystals in the recycled solutions gave the appearance of supersaturation. The only crystals recovered in this short run were those that formed on the external surfaces of the cooling coils and crystallizer tanks. The problem was tackled by allowing natural slow cooling of the saturated As_2O_3 solutions in air agitated tanks.

3.2 Flowsheet #2

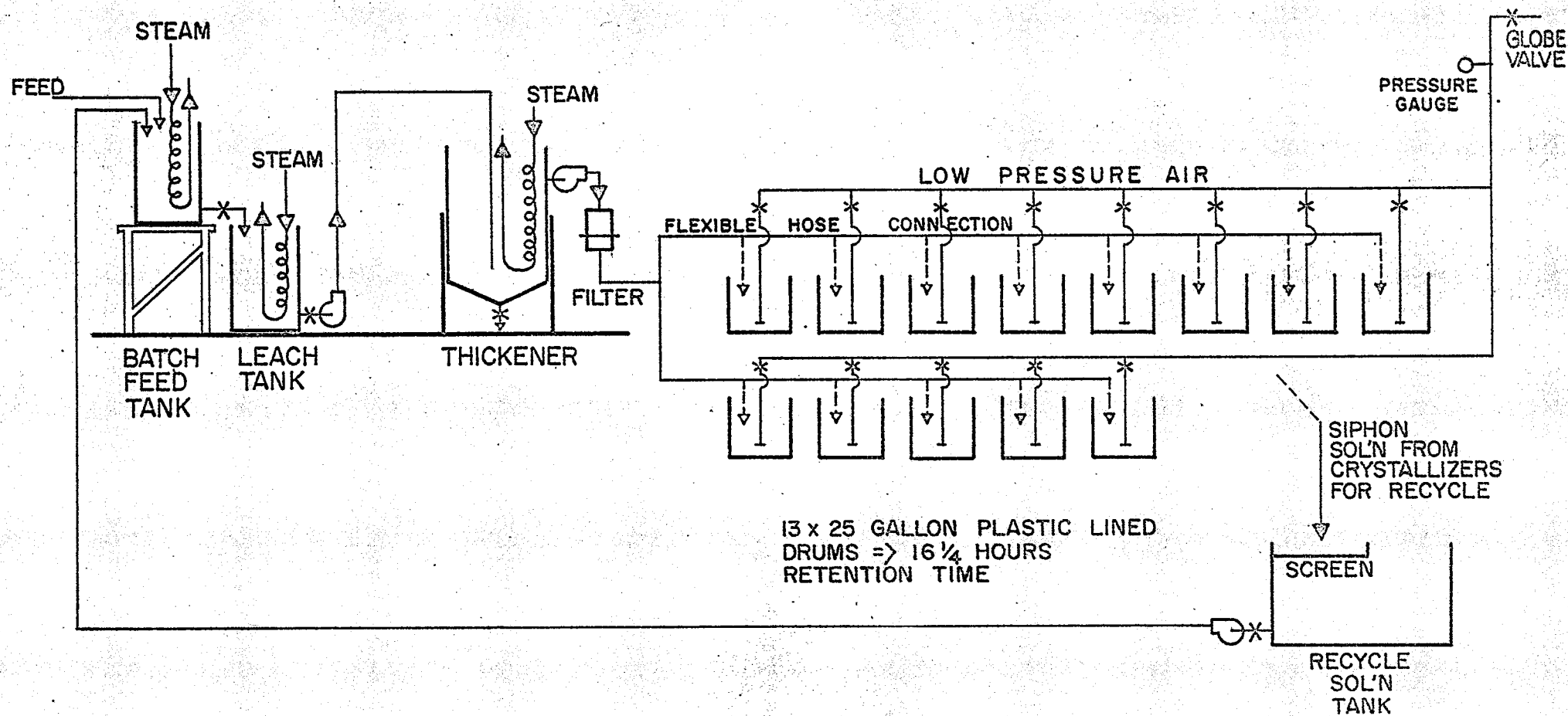
Following a two-week shutdown to facilitate the necessary changes in the pilot plant flowsheet, a second campaign was begun. This new flowsheet is diagramed in Figure 3.2.

As in the first run Giant baghouse dust was mixed and heated in two polyethylene tanks using steam passed through closed copper coils as the heating media. Following a two hour retention period, the leach tank slurry was transferred by pump to a thickener. The thickener temperature was maintained at 85 to 90° C using steam passed through a closed copper coil. At a later date the thickener was insulated and closed to minimize heat and evaporation losses.

Natural settling rates of the undissolved leach residue were found to be too slow for the pilot plant thickener (0.20 ft/min). The addition of 3 ppm of Dearborn Aquafloc 467 (an anionic polymer) dramatically increased these settling rates allowing a clear overflow to be drawn off the thickener. Flocculant consumption was in the order of 0.10 lbs/ton of material leached giving settling rates of 3.3 ft/min. Flocculant consumption was later reduced to 0.014 lbs/ton. The thickener underflow was stored for future testwork while the overflow solutions were pumped through a filter bed of diatomaceous earth to remove residual solids that could prove to contaminate crystal product. It was estimated that 2 lbs. of suspended solid per ton of material treated were retained on the polishing filter.

The clarified saturated arsenic trioxide solution was drained from the polishing filter to a series of 25 gallon tanks lined with plastic. Each tank was agitated with low pressure air spargers and allowed to naturally cool to room temperature. The As_2O_3 crystals were allowed to settle with the stripped solutions being recycled to the leach tank.

Figure 3.2 **HOT WATER LEACH PROCESS - FLOWSHEET No.2**



Leach conditions employed were as follows:

Slurry Density:	4% solids by weight
Temperature:	95 to 100° C
pH:	4.0 adjusted using 1:1 H ₂ SO ₄ . The lower pH was found to inhibit the dissolution and crystallization of Sb.
Retention Time:	2 hours

Under natural cooling conditions a retention time of 32 hours was required to reduce the saturated solution from a concentration of 68 g/l to 30 g/l As₂O₃. The relation between recovery of As₂O₃ from saturated solution and cooling time is plotted in Figure 3.2.A.

The addition of seed crystal only slightly improved the rate of crystallization. It was felt that the air sparges provided insufficient agitation for crystal growth as evidenced by the build-up of crystalline As₂O₃ on the plastic drum liners.

It was theorized that a more effective means of agitation would maintain the As₂O₃ crystal load in suspension and thus provide growth sites for the release of supersaturated As₂O₃ from solution as the temperature drops. At the same time increased agitation would increase the solution cooling rate. A second test was conducted replacing the air sparge with a mechanical mixer. As shown in Figure 3.2.B., a retention time of only 5 hours was required to move the saturated solution from a concentration of 80 g/l to 30 g/l As₂O₃. The rate of crystallization was observed to increase as the stripped solution was recycled to dissolve more baghouse dust. Steady state was achieved at the third cycle. Based on these results, strong mechanical agitation was seen as a necessity in increasing crystallization rates.

The average grade of the arsenic trioxide crystal produced in the second pilot plant run was as follows:

99.67 wt% As ₂ O ₃
0.032 wt% Fe
0.10 wt% Sb

FIGURE 3.2.A. Relation between cooling time and the Recovery of As_2O_3 from Saturated Solution - Natural Cooling.

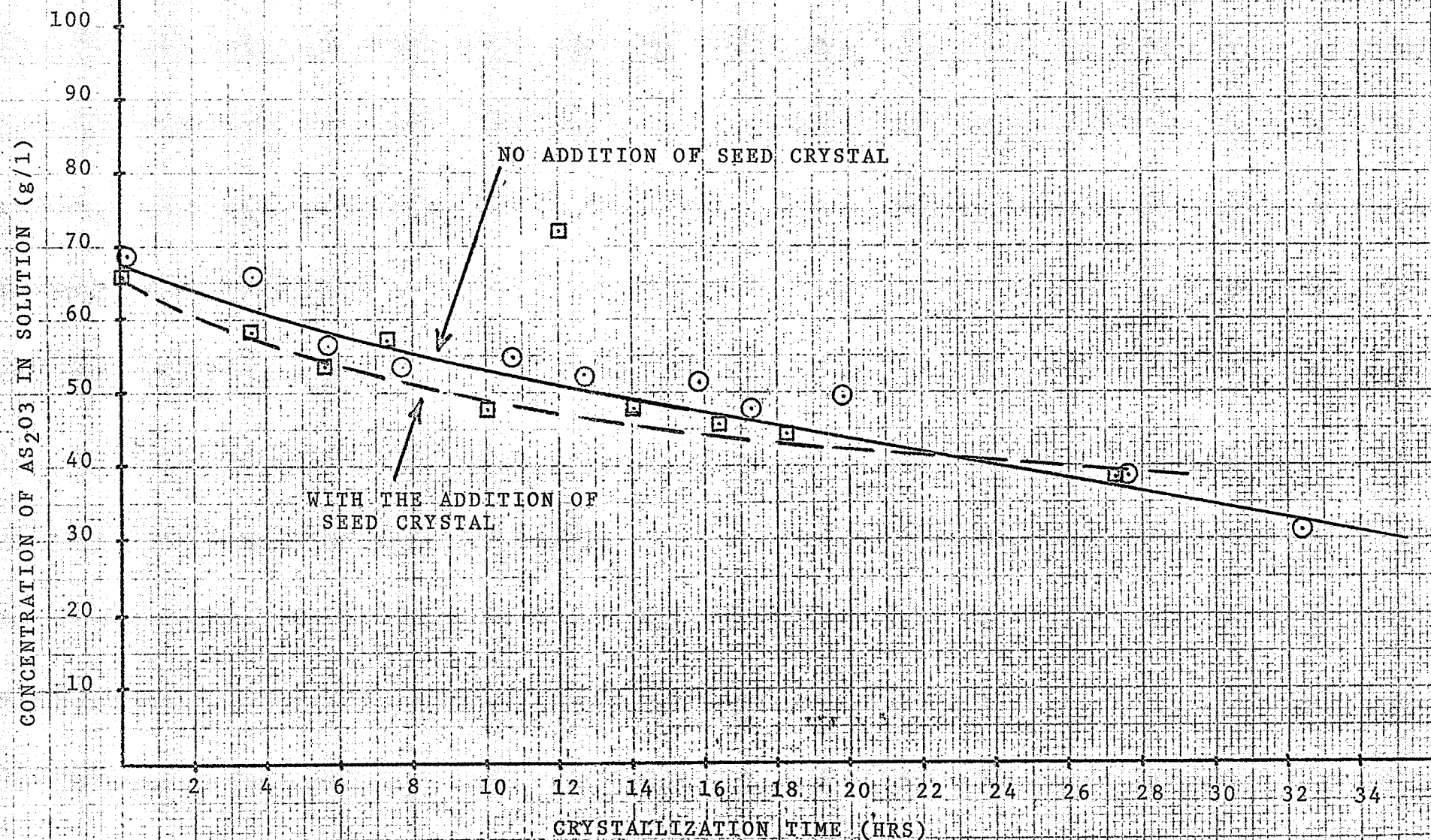
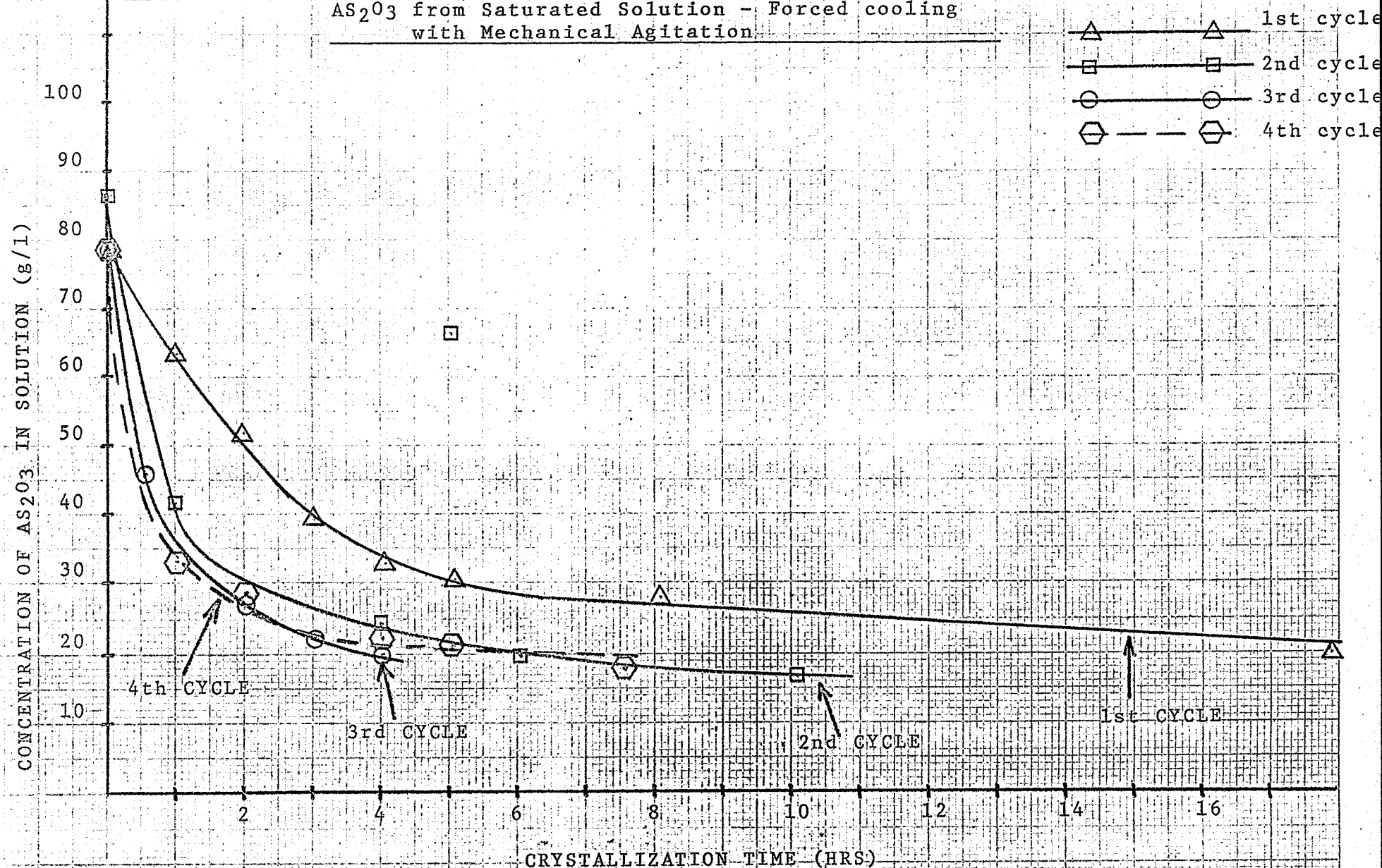


FIGURE 3.2.B. Relation between cooling time and the Recovery of As_2O_3 from Saturated Solution - Forced cooling with Mechanical Agitation



3.3 Flowsheet #3

The second pilot plant campaign was brought to a halt once the benefit of mechanical agitation on crystallization rates had been verified. Lab studies indicate that forced cooling of the crystallizer vessel in the presence of strong mechanical agitation further improves crystallization rates. Based on these indications new agitators were ordered and the crystallizer tanks were placed in external water cooling jackets.

By mid-June the alterations to the pilot plant flowsheet were completed and a third campaign was begun. Figure 3.3. The plant was run from June 12 to July 6th on a 5-day per week, 3 shift per day basis, with the following objectives:

- 1) To define the level and rate of impurity accumulation in the crystallizer products under continuous recycle of stripped As_2O_3 solutions. This information is essential to determine if a solution bleed is necessary.
- 2) To define operational difficulties that only become evident over a period of continuous operation.
- 3) To obtain crystal product for market surveys.
- 4) To obtain leach residues for laboratory gold recovery tests.

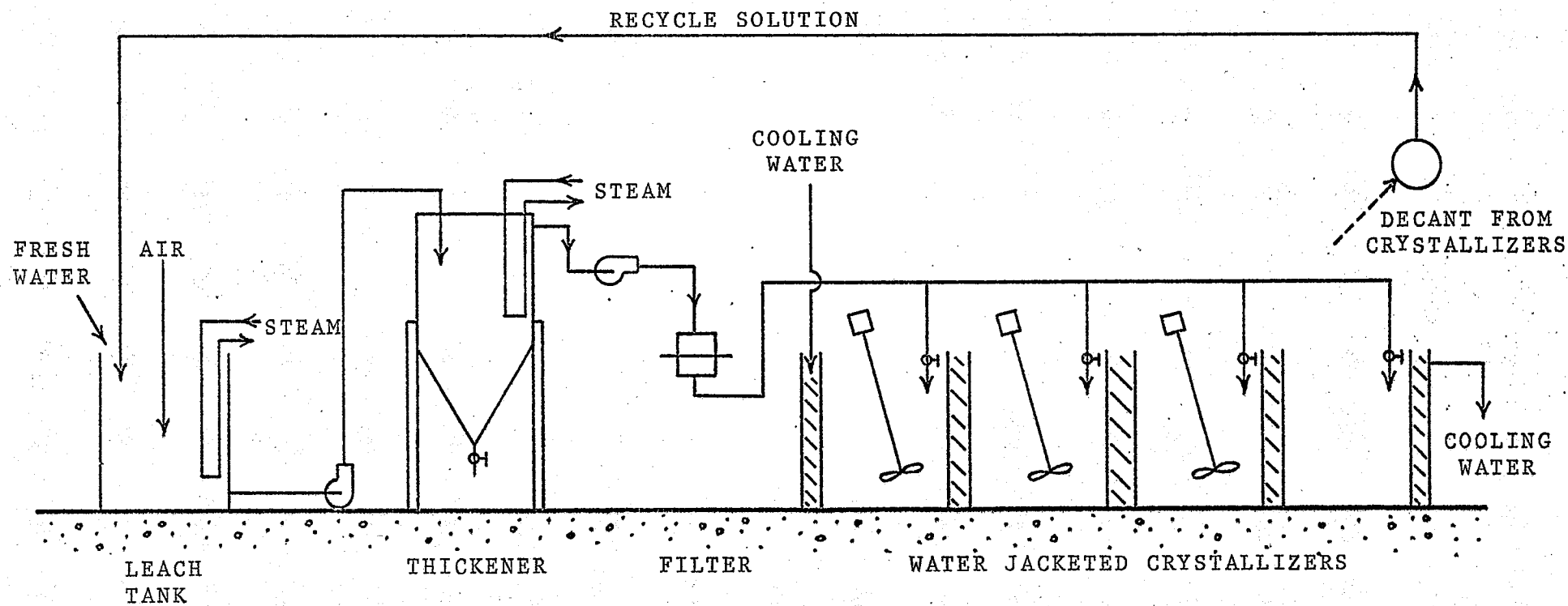
Operation

The plant operation was similar to that described in the second pilot plant campaign. Over the one month run a total of 109 closed cycles were completed. Current production of Giant baghouse dust was the only material tested. Water was only added to compensate for solution lost by evaporation or as solution entrained in crystal product and final residue. Equipment design resulted in heavy loss of solution when sampling final residue (Thickener U/F). For this reason final residue samples were not taken on a regular basis.

Observations

The data obtained from the campaign is tabulated in Table 3.3.1. (6)

Figure 3.3 Hot Water Leach Process - Flowsheet #3



51	RNS	22	62.16	95.52	68.08
52	RNS	22	63.80	95.52	54.12
53	RNS	22	-	96.54	53.08
54	Recycle NS	22	57.88	-	-
55	33.10	22	69.16	-	-
56	31.36	22	63.24	95.21	63.24
57	Recycle NS	22	67.00	-	67.52
58	RNS	22	63.24	-	65.92
59	RNS	22	68.08	93.21	62.72
60	Fresh	22	56.80	92.86	69.68
61	RNS	22	60.04	93.87	66.48
62	RNS	22	65.40	90.87	68.08
63	RNS	22	68.08	89.52	-
64	RNS	17	-	-	-
65	RNS	17	-	-	-
66	30.15	17	59.48	90.20	64.84
67	RNS	17	57.52	89.52	65.92
68	RNS	17	64.84	89.52	-
69	Fresh	28	-	-	-
70	27.34	17	64.84	85.52	-
71	23.85	17	60.56	83.51	-
72	25.99	17	60.56	77.84	-
73	28.27	17	57.36	89.09	-
74	35.09	17	69.16	89.77	-
75	32.70	17	57.88	90.79	-
76	31.89	17	-	-	-
77	24.89	17	77.8	89.77	-
78	26.52	17	64.2	90.79	-
79	20.81	17	-	-	-
80	21.08	17	64.72	92.16	-
81	22.44	17	61.48	92.84	-
82	Fresh	28	55.48	92.16	-
83	24.75	17	68.0	93.18	-
84	19.45	17	-	-	-
85		17	-	-	-
86	26.93	17	51.68	84.99	-
87	Fresh	28	49.52	84.52	-
88	Fresh	28	62.56	83.96	62.56
89	RNS	17	66.36	No assay	64.20
90	31.03	17	73.44	92.16	60.4
91	33.32	17	-	80.55	-
92	29.65	17	-	94.21	-
93	RNS	17	-	93.52	-
94	RNS	17	62.0	89.43	-
95	32.10	17	50.6	91.47	-
96	28.15	17	57.12	88.4	-
97	RNS	17	76.16	92.16	-
98	29.38	17	68.0	76.46	65.28
99	23.12	17	-	87.04	-
100	No assay	17	-	-	-
101	-	17	-	77.25	-
102	20.26	17	-	77.58	-
103	36.99	17	-	86.12	-
104	49.37	17	68.56	91.05	-
105	31.14	17	56.56	92.37	-
106	36.04	17	72.6	92.04	-
107	-	17	76.44	81.52	-
108	32.22	17	-	88.75	-
109	21.30	17	Pump broke down		

A total of 2,303 lbs. of baghouse dust was fed into the pilot plant, approximately 910 lbs. of crystal was removed.

NOTE: RNS - Recycle Solution - No sample Analysis.

Thickener Underflow

<u>Date</u>	<u>% As₂O₃</u>	<u>Au Oz/T</u>	<u>% Fe</u>	<u>% S</u>	<u>Sb</u>	<u>Pb</u>	<u>Zn</u>	<u>Cu</u>
June 21	93.19							
June 22	90.87	0.12	0.93	0.11	0.9	.06	.02	.06
Water soluble As ₂ O ₃ - 90.2%								
Acid insoluble As ₂ O ₃ - 0.2%								
June 23	91.19							

As₂O₃ Crystal Purity

A trend of increasing Fe contamination in the As₂O₃ crystal product was observed however it was found difficult to differentiate whether the Fe was originating from the material treated or from the corrosion of exposed metal surfaces in the pilot plant thickener and piping. This increase is demonstrated in Figure 3.3.1. Daily samples of both As₂O₃ crystal product and recycle solution were collected and sent to Thornhill for analysis. (5)

Table 3.3.2. Chemical Analysis of As₂O₃ Products

<u>Sample</u>	<u>Day/Month</u>	<u>As₂O₃</u>	<u>Fe</u>	<u>Sb</u>
A7773	13/06	99.9	0.024	0.06
7726	14/06	99.9	0.018	0.10
7727	20/06	99.9	0.020	0.15
7728	21/06	99.9	0.018	0.19
A7774	21/06	99.9	-	-
A7729	22/06	99.9	0.020	0.14
30	25/06	99.9	0.016	0.13
31	26/06	99.2	0.030	0.18
32	27/06	99.7	0.040	0.21
33	28/06	99.7	0.040	0.21
A7888	29/06	99.3	0.040	0.16
89	03/07	99.3	0.05	0.15
90	04/07	99.7	0.05	0.17
91	05/07	99.9	0.04	0.14
92	05/07	99.6	0.05	0.28
93	06/07	99.6	0.04	0.25

All crystal samples contain 99+ percent As₂O₃ and are above the specification concentration (98%) for this product. All impurity levels are low and with the exception of Fe are well below specification levels. An increase in solution pH to 4.5 (\pm 0.5) was recommended to reduce both Fe and Sb levels.

Other impurities detectable by spectrographic analysis are extremely low and show no increase with repeated cycles. The concentration of

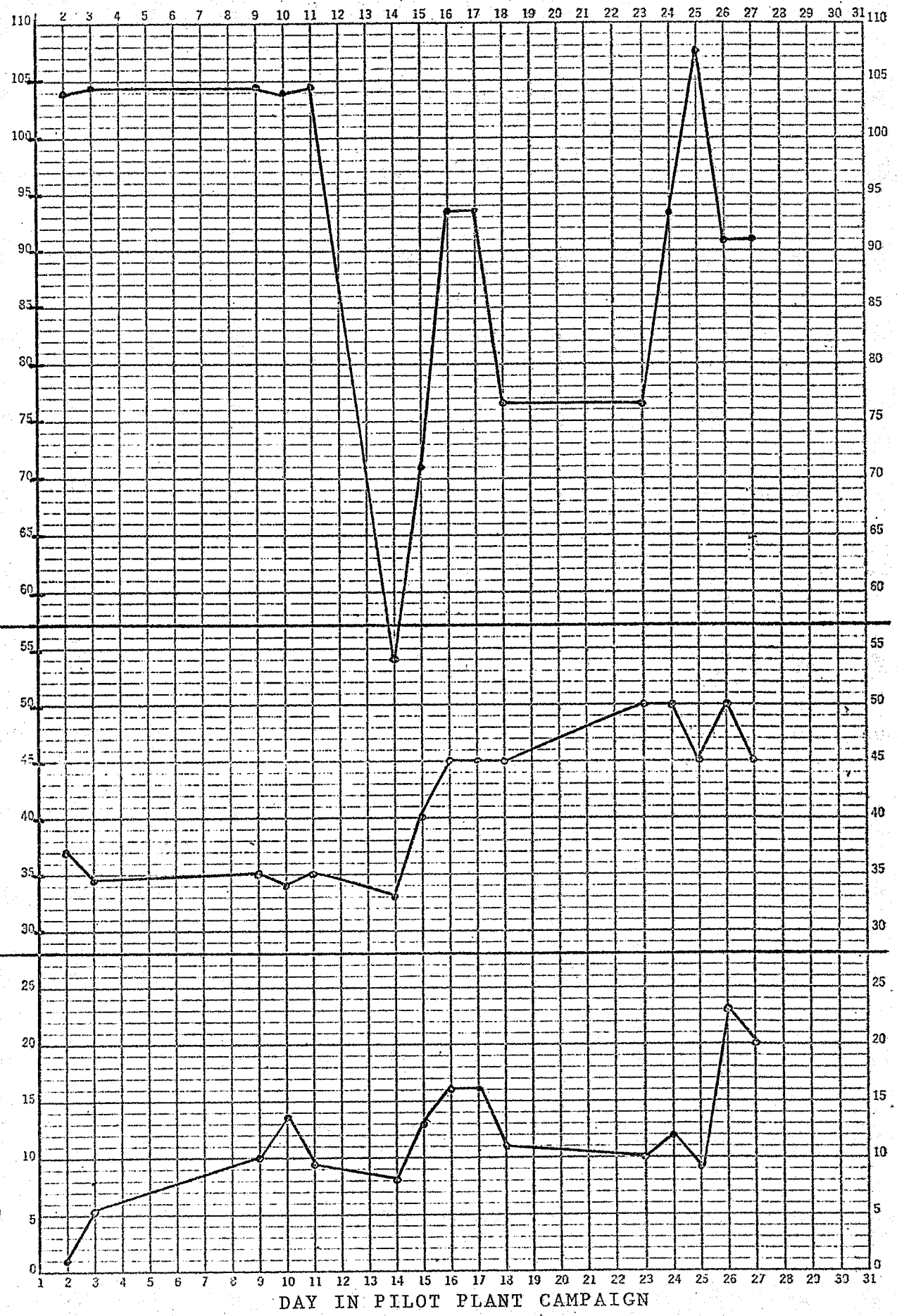
AS₂O₃ Crystal Impurity
 IMPURITY DATA
 DIVISION
 HUFFEL & ESSER CO.

Crystals

wt% AS₂O₃

wt% Sb

wt% Fe



DAY IN PILOT PLANT CAMPAIGN

Month _____ 19__

Figure 3.3.1: . Impurity Accumulation in As₂O₃ Crystal.

impurities in the recycle solutions are generally very low with the exception of sulphate and calcium (1.5 g/l and 0.37 g/l respectively). Both elements appear to have levelled off at these concentrations and are not contaminating the product.

Table 3.3.3.

Chemical Analysis of Crystallizer Recycle Solutions

FML #	Date (1979)	Batch	As ₂ O ₃ FML	As ₂ O ₃ (Giant)	Sb	Fe	SiO ₂	Ca	Mg	SO ₄	pH+	Redox (mv)
A7734	25/06	55	28.7	33.1	0.009	0.018	0.006	0.15	0.072	0.94		
35	25/06	66	27.9	30.1	0.011	0.029	0.006	0.15	0.075	0.19		
36	26/06	69	27.2	-	0.007	0.023	0.004	0.10	0.48	0.63		
37	26/06	71	24.7	23.9	0.013	0.039	0.007	0.16	0.080	0.30		
38	27/06	74	35.8	35.1	0.012	0.041	0.007	0.17	0.074	1.0		
39	29/06	83	25.5	24.8	0.019	0.046	0.011	0.23	0.11	1.5		
A7780	03/07	86	26.0	26.9	0.014	0.040	0.007	0.34	0.11	1.3	2.9	+280
81	03/07	91	32.2	33.3	0.012	0.026	0.008	0.36	0.12	1.5	2.9	+280
82	04/07	95	28.8	32.1	0.016	0.028	0.008	0.37	0.12	1.5	2.9	+280
83	04/07	98	28.6	29.4	0.015	0.020	0.008	0.40	0.13	1.6	2.9	+280
84	05/07	102	77.2	20.3	0.047	0.010	0.008	0.09	0.04	1.5	2.9	+280
85	05/07	104	49.6	49.4	0.016	0.004	0.008	0.03	0.01	1.4	2.9	+280
86	06/07	105	37.2	31.1	0.015	0.028	0.007	0.37	0.12	1.5	2.9	+280
87	06/07	108	34.4	32.2	0.017	0.008	0.008	0.33	0.08	1.6	2.9	+280

NOTE: pH⁺ of A7784, A7785 and A7787 adjusted to 9.4, 9.7 and 8.2 respectively before analysis thereby precipitating Ca, Mg and Fe.

Recovery of As₂O₃

Recovery of soluble As₂O₃ was not calculated for every cycle due to inadequate sampling. In a single stage leach approximately 70% of the soluble As₂O₃ is solubilized leaving a residue unacceptably high in soluble As₂O₃. This run points out the necessity of abandoning a single stage leach in favour of multi-stage countercurrent leaching.

In general, sampling during this third campaign proved to be woefully inadequate. The equipment did not lend itself to representative sampling. The campaign was plagued with operational difficulties as evidenced by the lack of data for the first 45 batches.

3.4 Phase II Pilot Plant Program

Difficulties encountered during the third pilot plant campaign suggested that the equipment available in the Phase I pilot plant was inadequate to provide accurate data for metallurgical evaluation and scale up. In specific:

- 1) It was not possible to determine whether Fe contaminants of the As_2O_3 crystal product originated from the material treated or resulted from the corrosion of the exposed metal surfaces in pipelines, valves and in particular the thickener.
- 2) A recovery of only 70 to 80% of the soluble arsenic in Giant baghouse dust was being obtained in a single stage leach.
- 3) Accurate sampling was proving difficult due to equipment design.

In mid July it was decided to construct a second pilot plant to overcome these short-comings and to allow more flexibility in operation. New tanks, thickeners and agitators were ordered. The new plant was arranged as a two stage countercurrent leach using all polyethylene tanks and C.P.V.C. piping. Figure 3.4.

Four new employees were hired and trained as pilot plant operators to allow for continuous operation on a seven day per week basis. The new plant was commissioned on August 30, 1979.

The second phase of the pilot plant program was broken down into three campaigns, each expected to require a one-month run:

- A) Giant Baghouse Dust
- B) Giant Cottrell Dust
- C) Con Arsenic Pond Sludge

FLOW SCHEMATIC: ARSENIC UPGRADING PILOT PLANT

3.4.1 Giant Baghouse Dust Campaign

Objectives

The major objective in treating Giant baghouse dust is to recover a marketable arsenic trioxide product. However, in order to meet environmental restrictions, greater than 99% of the contained As_2O_3 must be recovered from the baghouse dust to leave a "clean" residue. In addition the water balance must be closed, i.e., no bleed or purge of plant solutions is acceptable. The pilot plant campaign was planned to meet these objectives as well as the following:

- 1) Provide sufficient data to define the plant flowsheet as well as material and solution balances.
- 2) To determine the concentration and rate of impurity accumulation in both the crystalline arsenious oxide product and leach solutions under a closed recycle of all solution.
- 3) To define operational difficulties that may only become evident over an extended period of steady operation.
- 4) To obtain crystal product for market surveys.
- 5) To obtain leach residue samples for gold recovery tests.

Operational Procedure

A predetermined weight of baghouse dust was slurried with a recycled solution in a 45 gallon batching tank. Once mixed the slurry was drained by gravity into a closed leach tank (leach tank #1) and heated by a closed steam coil to 95° to 100° C. After a predetermined leach time, an anionic flocculant was added and the leach slurry was pumped into a heated thickener tank. (Thickener #1)

The thickener overflow (a saturated solution of As_2O_3) was pumped through a filter bed of dictamaceous earth and then drained into an agitated crystallizer tank. Each crystallizer tank was sitting in a water cooling bath to allow for controlled cooling. As the saturated solution cools, supersaturation is achieved and the As_2O_3 in solution begins to crystallize on seed crystal kept in suspension by the agitator. After four hours of cooling the agitator was shut off allowing the arsenic crystals to settle.

The undissolved residue from Thickener #1 was drained by gravity into a second leach tank. This residue was slurried with the stripped mother liquor decanted from above the settled crystal bed in the crystallizer tank. The crystals were sampled and left as seed for the next cycle.

The second leach tank was then heated by closed steam coil to a temperature between 95° and 100° C. This second leach was intended to recover any soluble As_2O_3 not recovered in the first stage leach. The solutions obtained were not saturated. After a predetermined leach period, flocculant was again added and the leach slurry was pumped into a second heated thickener. The overflow solution from the second thickener (not saturated) was recycled to slurry up the next batch or cycle of baghouse dust. The undissolved residue from the second leach was drained by gravity from the second thickener, filtered, dried and weighed. The cycle was then repeated.

Operating Conditions

Leach #1

Slurry Density:	4% solids - campaign began at a density of 8% solids (cycle 1-10) until recycle solution was available. Cycles 11-31 were run at a density of 5% solids. This was reduced to 4% solids in cycles 31-125 due to poorer recoveries at the higher density
Retention Time:	2 hours (not including heat-up time)
Temperature:	$95 - 100^\circ$ C

pH⁺: 4.5 \pm 0.5 adjusted using 1:1 H₂SO₄
pH⁺ was on occasion outside this range due to operator error.

Acid Consumption: 253 ppm (13.51 lbs/ton of baghouse dust)
of 95.5% H₂SO₄

Flocculant Consumption: 3 ppm (0.16 lbs/ton of baghouse dust) of
Dearborn Aquafloc 467 added in a 0.1%
solution.

Thickener #1

Temperature: 90 - 95⁰ C

Leach #2

Slurry Density: Estimated at 1.5% solids - this density obviously
varies with the efficiency of the first leach. No
record of this variance was collected.

Retention Time: 2 hours (not including heat-up time)

Temperature: 95 - 100⁰ C

Flocculant Consumption: 1.5 ppm (0.08 lbs/ton of baghouse dust) of
Dearborn Aquafloc 467 added in a 0.1% solution

Thickener #2

Temperature: 90 to 95⁰ C.

Crystallizer Tanks

Temperature: 5 - 15⁰ C

Retention Time: 4 to 12 hours (4 hours with agitation)

Campaign Results

During the one month campaign a total of 125 closed cycles were
completed. Fresh water additions were restricted to only that needed to
compensate for solution lost through evaporation, sampling and solution
entrained with plant residue and crystal product. These losses have been

calculated as follows:

Evaporation:	1.60 Imp. gal/cycle	4.00%
Sampling:	0.07 " " "	0.18%
Solution in Residue:	0.16 " " "	0.40%
<u>Sol'n in Crystal Samples:</u>	<u>0.02 " " "</u>	<u>0.05%</u>
Total Solution Loss:	1.85 Imp. gal/cycle	4.63%
Total Solution Volume:	40.00 Imp. gal/cycle	100.00%

Samples were collected and analyzed on a batch basis. The resulting chemical analysis are tabulated in Table 3.4.1.

Cycles #1 - 10

No recycle solutions were available for batches #1 to 10. In effect the first 10 batches were used to charge the pilot plant with arsenic bearing solution.

As fresh water was being used for each batch a higher weight of baghouse dust (30 lbs) was charged to give a theoretical leach solution concentration of 71.8 g/l As_2O_3 at 100° C.

Operations during the first week (Batch #1 - 30) were involved primarily in training new employees in operating techniques and improving the mechanical operation of the pilot plant wherever possible. Temperature and pH control were poor during the period.

The water supply available for making up batches 1 to 10 contained 2.1 ppm Fe (rust stained) and contributed to the high iron content in the first lot of crystals produced. The Fe problem resolved itself once sufficient water had run through the inactive water lines feeding the pilot plant area.

Treatment of Giant Baghouse Dust

Table 3.4.1. Pilot Plant Campaign

Batch	lb	BATCH FEED			CRYSTALIZER FEED			CRYSTALS			STRIP SOL'N			RECYCLE SOL'N			wt lb	Au oz/Tn	RESIDUE		
		As ₂ O ₃ %	Fe %	Sb %	As ₂ O ₃ g/l	Fe ppm	Sb ppm	As ₂ O ₃ %	Fe %	Sb %	As ₂ O ₃ g/l	Fe ppm	Sb ppm	As ₂ O ₃ g/l	Fe ppm	Sb ppm			As ₂ O ₃ %	Sb %	As ₂ O ₃ % Rem ⁺
1	30	96.40	0.71	0.49																	
2	30	96.40	0.58	0.55																	
3	30	96.40	0.71	0.65																	
4	30	96.73	0.68	0.66	54.12	14.4	-	N.S.	Allowed to		55.66	8.8									
5	30	-	-	-	54.64	10.4	-	N.S.	build up as		26.14	2.3									
6	30	96.40	0.71	0.59	54.64	10.4	-	N.S.	seed		55.89	6.4									
7	30	96.40	0.65	0.54	-	-	-	99.85	.04	.20	39.81	3.9	-	58.88	14.0	-					
8	30	96.40	0.80	0.61	53.04	15.2	-	99.86	.02	.24	27.59	2.7	-	55.72	11.6	-	4.91	.27	79.87	.20	86.44
9	30	95.40	0.75	0.58	-	-	-	99.89	.02	.26	28.64	4.4	-	-	-	-	10.43	.23	82.02	1.24	70.11
10	30	95.06	0.76	0.54	54.12	7.2	-	99.15	.03	1.15	14.19	2.0	-	55.16	6.8	-	5.17	.21	84.35	1.19	84.71
11	20	-	-	-	-	-	-	99.86	.02	.24	23.99	2.0	-	56.24	4.8	-	3.90	.22	81.67	1.08	-
12	20	96.40	0.78	0.60	54.64	2.2	-	99.89	.02	.26	32.84	2.9	-	52.52	10.4	-	12.93	.14	88.36	1.18	40.74
13	20	96.40	0.76	0.63	-	-	-	99.36	.02	1.49	29.22	2.6	-	51.92	6.4	20.2	4.41	.17	84.68	.74	80.63
14	20	97.08	0.91	0.69	58.36	8.4	-	99.89	.02	.56	31.90	2.5	-	50.32	6.4	84.8	3.98	.20	82.02	-	83.19
15	20	96.73	0.78	0.74	61.56	11.2	-	-	-	-	22.00	3.2	-	58.80	12.8	84.8	6.97	.09	91.52	-	66.93
16	20	94.73	0.76	0.73	53.04	15.2	-	99.89	.02	.19	26.81	2.2	-	50.84	10.8	81.6	4.52	.15	87.32	-	79.17
17	20	96.06	0.76	0.66	64.20	9.2	-	99.90	.02	.69	34.57	3.0	8.2	118.12	10.0	31.6	2.20	.22	82.70	-	90.53
18	20	96.40	0.76	0.71	60.48	8.4	-	98.99	.02	.27	47.02	4.6	10.3	51.92	7.6	98.0	5.25	.16	87.32	-	76.22
19	20	96.06	0.69	0.66	64.72	8.4	-	98.99	.02	.17	16.29	1.7	7.5	51.92	8.0	96.8	5.45	.19	84.02	-	76.17
20	20	96.40	0.70	0.64	60.92	10.0	62.8	99.66	.02	.04	22.51	1.9	7.8	130.32	12.8	38.0	1.06	.24	78.91	-	95.66
21	20	95.45	0.41	0.39	59.32	9.6	55.6	98.90	.02	.36	30.86	2.9	8.8	49.80	7.2	92.4	4.21	.22	80.27	-	82.30
22	20	95.12	0.76	0.56	64.12	8.8	55.2	98.90	.02	.49	35.76	3.4	9.4	57.20	6.4	86.4	3.31	.23	79.88	-	86.17
23	20	95.12	0.45	0.35	66.24	8.4	52.4	98.90	.02	.22	16.82	1.6	8.6	53.52	11.2	85.6	1.39	.26	77.36	-	94.35
24	20	95.93	-	0.66	125.04	12.0	36.8	99.56	.02	.13	34.57	3.0	10.8	52.12	7.2	83.6	3.74	.19	82.80	-	83.86
25	20	94.92	0.70	-	64.12	7.6	68.8	99.56	.02	.12	37.22	3.1	11.8	53.64	6.8	83.2	5.32	.15	85.13	-	76.17
26	20	95.25	0.71	-	63.56	9.2	64.8	99.89	.02	.14	27.22	2.5	8.9	52.88	8.4	87.6	-	-	-	-	-
27	20	95.25	0.68	0.55	126.08	12.4	41.6	99.56	.01	.12	16.16	1.6	9.6	55.20	10.0	52.4	-	-	-	-	-
28	20	95.25	0.68	0.60	43.44	6.4	38.8	99.56	.02	.16	30.72	3.2	13.6	47.16	8.4	85.2	-	-	-	-	-
29	20	95.25	0.68	0.60	62.52	8.4	61.2	99.56	.02	.04	26.63	3.1	9.6	46.08	6.0	81.2	7.50	.26	77.55	-	69.47
30	20	94.92	0.61	0.42	60.4	15.2	49.6	99.23	.01	.09	36.55	5.8	18.3	47.16	10.0	79.2	8.42	.32	73.67	-	67.37
31	20	95.58	0.59	0.41	63.76	9.6	50.0	99.23	.01	.11	16.14	1.7	9.6	51.4	6.4	81.6	5.57	.32	69.97	-	79.57
32	15	94.92	0.63	0.41	59.88	9.2	58.8	99.23	.01	.12	25.56	2.9	9.4	47.16	5.2	81.6	3.07	.34	71.14	-	84.67
33	15	94.92	0.66	0.41	59.88	9.6	62.4	99.56	.01	.10	23.31	2.5	10.7	45.60	10.4	83.6	2.09	.40	64.73	-	90.50
34	15	94.92	0.65	0.41	59.12	11.2	58.0	99.23	.01	.12	18.28	2.1	10.7	43.56	11.2	94.8	1.11	.58	47.05	-	96.37
35	15	95.25	0.63	0.42	63.56	7.2	59.6	99.12	.12	.13	15.76	1.6	10.7	40.44	7.6	71.2	2.64	-	-	-	-

Treatment of Giant Baghouse Dust

Table 3.4.1. Pilot Plant Campaign

Batch	lb	BATCH FEED			CRYSTALIZER FEED			CRYSTALS			STRIP SOL'N			RECYCLE SOL'N			wt lb	Au oz/Tn	RESIDUE		
		As ₂ O ₃ %	Fe %	Sb %	As ₂ O ₃ g/l	Fe ppm	Sb ppm	As ₂ O ₃ %	Fe %	Sb %	As ₂ O ₃ g/l	Fe ppm	Sb ppm	As ₂ O ₃ g/l	Fe ppm	Sb ppm			As ₂ O ₃ %	Sb %	As ₂ O ₃ %
36	15	95.25	0.63	0.40	60.4	6.8	63.6	99.12	.02	.25	24.90	2.4	10.6	41.2	8.4	67.6	2.64	.58	51.51	-	90.4
37	15	94.92	0.63	0.40	61.44	12.4	42.4	99.61	.02	.15	27.61	2.8	13.4	34.20	6.0	52.8	3.15	.49	55.59	-	87.70
38	15	95.25	0.62	0.40	62.0	6.8	60.0	99.61	.02	.11	22.16	2.1	13.2	26.44	8.4	56.8	1.81	.63	44.11	-	94.4
39	15	93.88	0.74	0.66	61.44	7.6	54.4	99.61	.02	.13	15.56	2.4	14.8	24.88	9.6	43.6	1.52	.70	34.07	1.21	96.3
40	15	84.94	2.31	0.66	57.76	8.0	60.4	-	-	-	25.47	2.7	10.8	17.12	9.2	40.4	.76	.67	34.07	.93	97.9
41	15	85.72	2.16	0.56	62.20	13.6	78.8	99.12	.02	.12	30.72	2.9	11.9	19.72	10.0	41.6	.61	.85	15.40	.73	99.2
42	15	88.44	1.79	0.37	60.64	9.2	62.4	94.26	.02	.08	21.19	2.6	10.3	25.44	8.0	32.0	1.81	.74	26.06	.86	96.4
43	15	84.89	2.75	0.44	55.2	8.4	65.6	95.24	.02	.12	15.75	2.9	11.5	27.0	6.0	37.2	2.75	.55	42.89	1.08	90.7
44	15	85.91	3.51	0.66	56.0	7.6	67.2	95.73	.02	.12	22.55	2.3	10.7	22.16	12.0	42.0	2.12	.80	22.16	.80	96.3
45	15	81.44	3.14	0.53	49.76	13.2	53.6	100.10	.02	.32	29.14	3.0	10.8	27.92	5.2	33.2	2.74	.67	36.73	1.11	91.7
46	15	85.13	2.99	0.65	49.0	6.8	72.0	99.61	.14	.31	27.91	3.2	11.7	26.28	6.8	30.0	1.93	.46	60.53	1.13	90.8
47	15	82.41	2.44	0.70	53.64	9.2	93.6	100.10	.02	.31	16.62	1.9	10.9	24.64	4.8	24.8	1.34	.44	66.07	1.35	92.8
48	15	81.44	2.39	0.46	52.56	14.0	48.8	99.61	.02	.33	26.07	2.8	12.2	21.36	7.6	32.8	-	-	-	-	-
49	15	88.52	2.28	0.51	46.8	10.8	76.8	100.59	.02	.36	23.81	2.6	10.4	28.72	7.6	34.0	3.04	.86	13.14	-	96.9
50	15	86.01	2.33	0.56	55.0	12.8	80.8	100.59	.02	.14	21.96	1.6	10.7	25.44	6.0	37.6	2.57	.83	11.70	-	97.6
51	15	86.01	2.34	0.35	52.56	9.6	74.8	99.61	.05	.21	15.80	2.8	12.6	25.44	12.8	35.6	3.12	.86	10.88	-	97.3
52	15	86.01	2.20	0.50	50.92	14.0	75.2	99.61	.02	.34	32.84	2.2	13.5	37.76	8.4	53.6	2.97	.85	11.08	-	97.4
53	15	85.03	2.30	0.46	52.56	9.2	77.2	99.61	.02	.24	17.45	2.4	11.7	-	-	-	-	-	-	-	-
54	15	85.52	2.34	0.31	53.36	13.6	34.4	99.00	.02	.12	22.17	2.7	13.4	25.44	9.2	34.0	0.58	.86	10.06	-	99.5
55	15	86.01	2.33	0.40	52.56	12.4	79.2	-	-	-	-	-	-	29.56	7.2	30.0	1.16	.87	14.56	-	98.6
56	15	85.52	2.36	0.50	50.08	15.6	85.2	99.52	.02	.20	23.40	2.4	13.2	27.92	38.4	38.8	0.40	.80	17.64	1.06	99.4
57	15	85.52	2.31	0.43	47.60	12.0	83.2	99.52	.02	.15	20.73	2.6	13.5	37.76	9.2	38.8	-	-	-	-	-
58	15	84.53	2.30	0.46	56.64	10.8	86.4	99.52	.02	.24	29.76	3.2	12.7	32.00	6.4	29.6	-	-	-	-	-
59	15	84.12	2.99	0.60	69.80	11.6	100.4	99.00	.04	.17	24.83	2.9	12.6	27.92	34.8	37.2	1.63	.83	9.76	.53	98.7
60	15	81.88	3.14	0.53	55.84	11.6	83.0	99.52	.03	.36	37.97	4.8	14.5	25.44	4.4	30.0	1.96	.76	8.77	.38	98.6
61	15	84.33	2.39	0.50	55.84	11.2	87.2	99.52	.03	.20	17.24	2.1	10.4	37.44	11.6	40.4	1.63	.88	9.36	.43	98.7
62	15	85.77	2.34	0.56	45.16	8.4	68.4	99.52	.03	.24	18.06	1.6	12.4	26.28	8.0	41.6	1.46	.87	9.76	.46	98.8
63	15	84.53	2.39	0.44	57.48	9.6	78.8	99.00	.03	.18	16.01	2.1	12.6	29.48	22.8	33.6	1.65	.87	9.76	.49	98.7
64	15	84.95	2.68	0.49	53.36	12.4	76.8	99.52	.03	.48	43.92	4.4	16.2	27.88	7.6	32.4	1.72	.87	9.96	.50	98.6
65	15	84.95	2.55	0.49	59.12	9.6	67.2	99.49	.03	.19	17.12	2.1	10.5	33.44	9.2	34.4	1.44	.91	10.35	.33	98.8
66	15	87.82	2.28	0.40	61.56	25.6	65.6	98.49	.03	.20	17.52	2.4	11.7	34.24	11.2	41.6	1.29	.95	9.36	.64	99.0
67	15	87.82	1.96	0.41	38.12	7.6	41.2	99.52	.03	.18	18.12	2.1	12.4	25.48	8.8	36.8	-	-	-	-	-
68	15	87.82	1.96	0.26	54.96	9.2	65.2	99.52	.03	.22	35.05	4.2	10.9	27.88	11.2	36.8	0.40	.79	23.49	.73	99.2
69	15	89.26	2.10	0.46	47.80	10.4	52.8	99.06	.02	.21	18.92	1.9	12.2	44.60	12.0	36.8	1.76	.83	11.55	.63	98.4

Treatment of Giant Baghouse Dust

Table 3.4.1. Pilot Plant Campaign

Batch	lb	BATCH FEED			CRYSTALIZER FEED			CRYSTALS			STRIP SOL'N			RECYCLE SOL'N			RESIDUE				
		As ₂ O ₃ %	Fe %	Sb %	As ₂ O ₃ g/l	Fe ppm	Sb ppm	As ₂ O ₃ %	Fe %	Sb %	As ₂ O ₃ g/l	Fe ppm	Sb ppm	As ₂ O ₃ g/l	Fe ppm	Sb ppm	wt lb	Au oz/Tn	As ₂ O ₃ %	Sb %	As ₂ O ₃ % Rem
70	15	89.26	2.38	0.49	55.76	16.8	77.6	99.06	.02	.22	18.92	2.3	13.5	31.84	15.6	41.6	0.87	.79	9.36	.63	99.39
71	15	89.26	2.29	0.54	53.36	13.6	63.2	98.57	.02	.22	18.52	2.1	12.4	24.68	8.0	28.0	0.78	.99	11.94	.61	99.30
72	15	88.74	2.28	0.13	55.76	12.8	69.2	98.57	.03	.51	34.85	4.3	14.4	24.68	14.8	31.2	2.22	1.10	9.96	.53	98.34
73	15	88.23	2.09	0.43	56.56	8.4	84.0	-	-	-	24.69	4.1	12.5	31.84	52.4	58.4	1.36	1.08	9.36	.51	99.04
74	15	88.11	1.90	0.53	54.16	10.4	67.6	96.06	.02	.21	18.92	2.3	12.1	23.08	12.4	26.8	0.91	1.11	12.15	.55	99.16
75	15	88.11	1.93	0.56	44.60	18.4	67.6	99.56	.01	.19	17.32	2.1	11.9	25.48	19.6	51.2	1.72	1.05	9.76	.46	98.73
76	15	89.77	2.03	0.48	50.96	10.0	76.4	99.06	.02	.22	25.89	2.7	12.3	28.68	3.2	47.2	0.59	1.07	12.74	.51	99.44
77	15	89.26	2.40	0.43	53.36	18.0	71.2	100.06	.03	.17	22.70	2.4	15.5	24.68	7.2	40.8	-	-	-	-	-
78	15	88.11	1.90	0.58	54.16	10.0	78.8	100.55	.02	.19	18.72	2.3	14.6	22.32	8.8	39.2	-	-	-	-	-
79	15	89.10	1.89	0.30	54.96	10.4	72.4	100.55	.03	.19	16.93	5.4	16.9	24.68	6.8	40.8	1.31	0.98	14.54	.53	98.57
80	15	88.11	1.89	0.34	54.16	26.4	79.2	99.06	.02	.14	25.89	9.5	13.4	22.32	5.6	27.6	1.10	0.98	14.74	.36	98.77
81	15	89.10	2.04	0.41	54.96	10.0	86.4	99.56	.02	.53	20.71	2.2	14.8	22.32	12.4	24.8	0.69	0.88	10.15	.29	99.48
82	15	89.60	1.83	0.43	49.40	9.2	90.8	98.57	.02	.24	26.48	2.2	14.9	18.32	4.0	27.2	0.71	1.08	10.95	.43	99.42
83	15	90.10	1.96	0.46	47.80	7.2	81.2	98.57	.02	.22	18.52	2.1	14.6	15.12	2.4	14.4	0.53	1.10	11.75	.49	99.54
84	15	87.61	2.18	0.48	56.56	10.0	90.8	99.06	.02	.24	20.71	1.5	12.4	24.68	3.2	13.6	-	-	-	-	-
85	15	88.11	2.08	0.53	52.56	24.8	84.8	98.57	.04	.53	16.53	2.4	10.0	22.32	2.8	16.4	1.16	1.10	13.75	.47	98.79
86	15	88.11	2.10	0.50	55.76	10.0	96.4	99.56	.01	.24	15.73	1.6	14.2	21.52	6.0	16.8	1.09	1.11	9.76	.33	99.20
87	15	87.61	2.00	0.49	55.76	10.0	104.4	99.06	.02	.39	21.50	1.2	9.7	24.68	4.0	21.6	1.17	0.96	10.75	.41	99.04
88	15	87.61	2.06	0.40	58.92	22.0	92.4	100.06	.02	.65	31.06	1.7	9.7	23.08	3.2	18.8	0.88	0.95	12.74	.35	99.15
89	15	88.61	2.09	0.41	54.16	8.0	89.6	99.56	.05	.22	20.11	1.3	9.7	26.28	4.8	16.8	1.10	1.00	11.14	.41	99.08
90	15	88.61	2.19	0.39	53.36	6.0	60.4	100.06	.03	.22	20.11	1.5	9.2	35.84	12.8	19.6	1.77	1.07	5.20	.26	99.31
91	15	88.61	2.10	0.54	50.96	5.6	58.0	99.56	.02	.27	19.51	1.5	8.9	24.68	4.4	26.8	1.00	0.99	5.32	.26	99.60
92	15	88.11	2.05	0.55	54.16	7.6	53.6	100.06	.02	.39	39.23	2.3	9.3	22.52	2.4	19.6	0.77	1.11	5.64	.27	99.67
93	15	87.61	2.00	0.34	48.60	12.0	45.6	100.06	.04	.31	22.10	1.7	7.4	37.80	5.6	24.8	1.12	0.94	10.15	.35	99.13
94	15	87.12	2.24	0.43	-	-	-	100.06	.04	.31	37.24	2.7	12.1	24.12	2.4	15.6	0.80	0.95	8.90	.38	99.46
95	15	87.12	2.06	0.39	58.16	6.8	54.0	100.06	.02	.22	11.55	1.0	6.4	24.12	7.2	17.2	-	-	-	-	-
96	15	87.61	1.96	0.38	60.52	8.8	62.4	-	-	-	39.61	2.3	11.9	22.04	5.2	18.8	2.08	0.97	7.84	.23	98.76
97	15	88.11	2.15	0.40	57.36	6.8	38.4	99.93	.02	.09	21.71	1.4	10.4	33.08	6.0	25.6	2.31	0.90	7.84	.20	98.63
98	15	85.61	2.40	0.40	58.92	8.8	56.8	99.93	.01	.06	28.35	1.8	10.2	29.08	6.4	22.4	0.91	0.88	8.85	.21	99.37
99	15	86.62	2.11	0.40	54.68	9.2	61.6	99.93	.01	.17	23.81	1.8	9.6	27.08	4.0	18.8	-	-	-	-	-
100	15	87.40	1.53	0.26	80.44	18.4	84.0	99.93	.03	.65	46.49	3.1	12.3	35.60	8.0	26.8	-	-	-	-	-
101	15	84.58	1.60	0.26	49.88	5.2	53.6	99.31	.02	.11	27.19	1.6	9.4	39.08	10.4	34.8	2.33	0.94	8.44	.35	98.45
102	15	84.89	1.70	0.24	53.08	9.6	54.4	99.93	.02	.15	26.44	1.6	9.6	43.12	6.8	30.4	2.33	0.97	9.44	.30	98.29
103	15	85.52	1.65	0.26	50.64	9.2	58.4	99.93	.01	.20	46.12	3.3	10.3	35.08	8.8	26.4	-	-	-	-	-

Treatment of Giant Baghouse Dust

Table 3.4.1. Pilot Plant Campaign

Batch	lb	BATCH FEED			CRYSTALIZER FEED			CRYSTALS			STRIP SOL'N			RECYCLE SOL'N			RESIDUE				
		As ₂ O ₃ %	Fe %	Sb %	As ₂ O ₃ g/l	Fe ppm	Sb ppm	As ₂ O ₃ %	Fe %	Sb %	As ₂ O ₃ g/l	Fe ppm	Sb ppm	As ₂ O ₃ g/l	Fe ppm	Sb ppm	wt lb	Au oz/Tn	As ₂ O ₃ %	Sb %	As ₂ O ₃ % Rem*
104	15	86.78	1.95	0.31	53.12	6.4	49.6	98.48	.02	.72	34.84	2.5	9.6	34.08	6.4	24.4	1.55	0.93	9.04	.28	98.92
105	15	86.78	2.15	0.39	55.16	7.2	61.6	99.98	.02	.33	21.30	2.0	5.9	41.0	5.6	27.2	2.62	0.93	9.44	.27	98.10
106	15	85.52	2.38	0.41	56.16	7.2	61.6	99.48	.01	.13	18.42	1.1	8.8	33.76	8.8	24.8	1.61	0.98	10.05	.34	98.74
107	15	86.14	2.05	0.39	52.64	6.8	60.0	99.98	.02	.13	23.69	1.3	9.3	26.52	4.8	5.6	1.08	0.92	9.44	.30	99.21
108	15	85.84	1.61	0.30	58.64	8.8	61.2	98.98	.02	.82	50.00	2.9	9.9	28.96	5.2	4.8	0.99	0.96	9.04	.30	99.30
109	15	87.09	1.54	0.33	57.64	9.2	45.2	98.98	.01	.10	27.55	1.4	8.9	31.36	5.6	4.0	1.07	0.98	8.03	.21	99.34
110	15	85.92	1.65	0.26	58.64	9.6	58.4	99.48	.01	.07	19.10	1.1	9.2	30.56	2.4	20.0	0.40	0.98	9.44	.29	99.71
111	15	88.43	1.34	0.26	60.64	10.8	60.4	99.98	.01	.15	23.12	1.2	9.2	25.72	4.4	18.4					
112	15	88.43	1.34	0.28	58.72	7.2	57.6	99.98	.02	.03	34.18	1.7	9.1	water	0.7						
113	15	89.93	1.68	0.28	60.32	10.0	51.6	99.48	.01	.07	23.73	1.3	10.1	water							
114	15	89.93	1.89	0.51	60.32	14.8	12.2	99.48	.01	.12	23.52	1.4	9.8	24.92	2.4	21.6					
115	15	89.43	1.99	0.54	57.92	7.2	13.8	99.89	.01	.12	19.70	1.1	10.0	32.96	4.0	20.0					
116	15	88.93	1.80	0.44	56.28	7.6	14.4	99.48	.01	.07	26.94	1.4	9.7	16.88	1.6	13.2					
117	15	89.93	1.98	0.39	71.56	9.6	60.0	99.48	.01	.08	16.69	0.9	9.3	19.32	4.4	14.4					
118	15	88.19	1.20	0.26	54.68	6.0	52.4	99.98	.01	.12	18.70	1.6	10.1								
119	15	86.42	1.19	0.49	46.64	6.0	60.8	99.48	.01	.18	22.92	1.2	5.3								
120	15	87.42	1.24	0.50	46.72	7.6	49.6	99.98	.01	.11	21.71	1.1	7.5								
121	15	87.42	1.23	0.48	49.04	5.2	46.4	99.48	.01	.11	23.32	1.6	8.6								
122	15	86.92	1.23	0.49	48.24	6.4	54.8	98.98	.01	.15	27.55	1.6	9.2								
123	15	85.42	1.51	0.48	49.04	4.8	56.4	99.48	.02	.26	35.19	1.9	9.1								
124	15	83.90	0.94	0.48																	
125	15	83.40	1.66	0.46																	

*Rem. Recovery % AS₂O₃

Cycles 11 - 31

Once recycle solution was available, the charge of baghouse dust to each batch was reduced to 20 lbs to give a theoretical leach solution of 70 gm per litre As_2O_3 (assuming a recycle solution of 22 gm/l As_2O_3) at 100°C . Actual solubility averaged only 52.6 gm/l As_2O_3 in the first stage of leaching. The lower value left more arsenious oxide to be recovered in the second stage leach, increasing the concentration of soluble As_2O_3 in the recycled solutions. The net effect was poor overall recoveries of arsenious oxide (78.14%).

Operator inexperience in sample dilution and preparation techniques coupled with fluctuating leach temperatures further complicate the relevance of the data obtained from the first 31 cycles.

Crystal product purity remained fairly constant despite plant upsets, with the exception of antimony. Antimony levels fluctuated quite erratically over the first 22 cycles. Antimony was observed to readily crystallize from solution whenever the pH^+ dropped below 4.0. During these periods of low solution pH antimony appeared to act as a cement, agglomerating the entire crystallizer circulating load. This condition would have to be avoided in a full scale plant. On the pilot scale, the effected crystallizer tank had to be manually cleaned before being placed back in service.

Cycles 32-53

The charge of baghouse dust to each cycle was further reduced to 15 lbs. commencing with batch #32. The solubility of As_2O_3 in the first stage leach did not appreciably improve, however the net recovery of arsenious oxide increased by 15.86% to average 94.0%.

Investigation of the poor recovery indicate that although the thickeners were constructed with heating elements, saturated leach solutions were being allowed to cool to 80 to 90°C . The reduced temperatures were

allowing As_2O_3 to crystallize prematurely in the first stage thickener. This loss of solubility in the first stage placed a heavy burden on the second stage leach, eventually causing high As_2O_3 concentrations in the recycled solutions and poor overall recoveries.

Pilot Plant Cycle 54 Through 125

Steady state conditions were maintained on a fairly reasonable basis in cycles 54 through 125. Rheostat controls on the thickener heaters were adjusted to maintain a steady 90 to 95° C on both thickeners. Some variation in thickener temperatures was still experienced but not of the magnitude observed in cycles 1 through 53.

The test results for cycles 54 through 125 were used to derive a balance for total As_2O_3 (Figure 3.4.1). In summary:

1st Stage Leach

Recovery of total As_2O_3 from baghouse dust: 88.45% with a standard deviation of 26.41%.

Pick-up of As_2O_3 by recycled solution: 26.3 gm/l with a standard deviation of 7.91 gm/l.

2nd Stage Leach

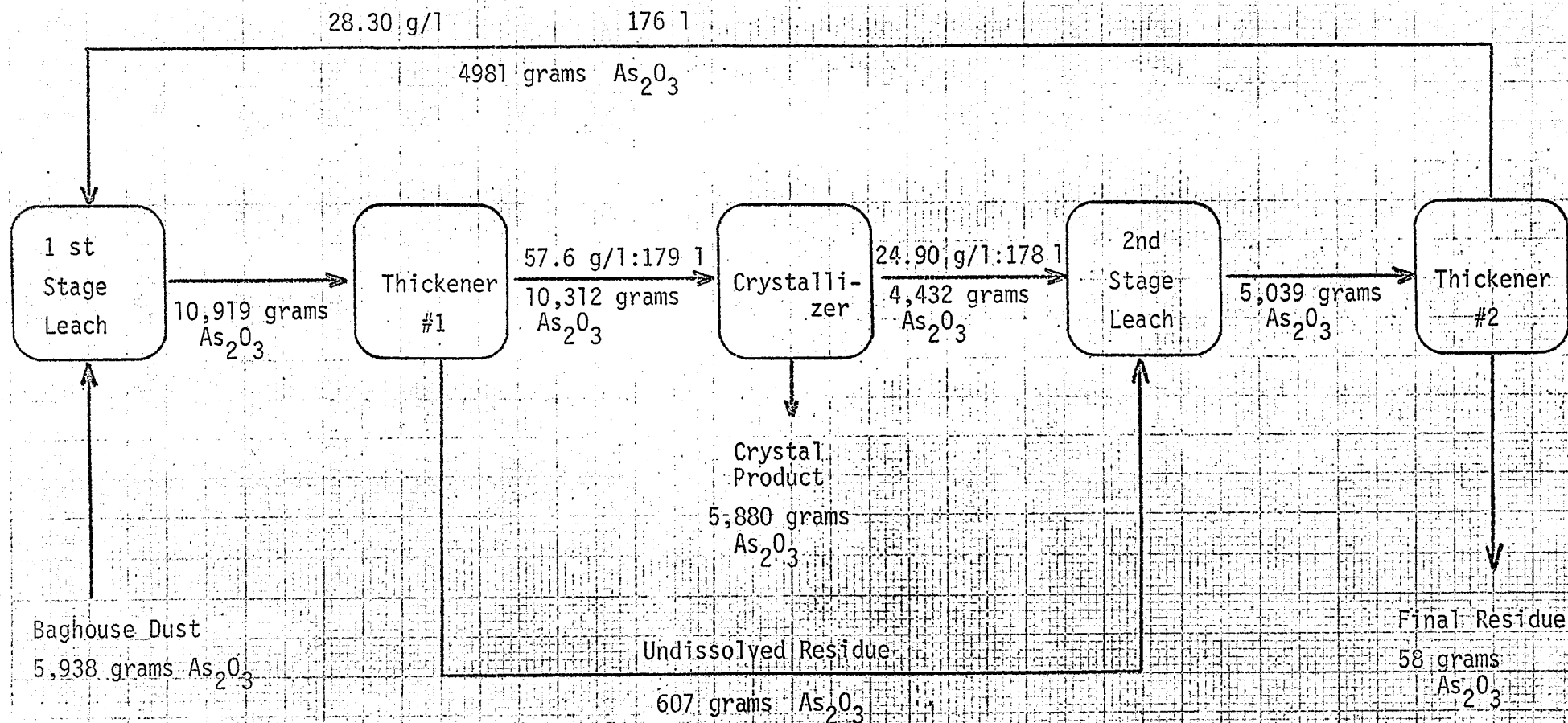
Recovery of total As_2O_3 from 1st Stage Leach Residue: 91.52%.

Incremental recovery of total As_2O_3 : 10.57%

Pick-up of As_2O_3 by crystallizer strip solution: 3.40 gm/l with a standard deviation of 10.38 gm/l.

Figure 3.4.1.

Pilot Plant Balance For Total As_2O_3 - Cycle 54 - 125



Overall Recovery

Recovery of total As_2O_3 : 99.02% with a standard deviation of 0.40%

Solubility of As_2O_3

The solubility of As_2O_3 contained in baghouse was observed to vary over a very wide range. This variance is partially due to inconsistent operating conditions. On occasion thickener temperatures were inadvertently allowed to drop, crystallizing As_2O_3 from solution to the thickener residue. In a full scale plant these operating deviations could be avoided by the use of instrumented temperature control devices.

Theoretical solubilities of 70 gm/l As_2O_3 were not attained due to loss of temperature in both the thickener, polishing filter and interconnected piping (solubility of As_2O_3 in leach solution was measured in crystallizer feed solutions). As a result the pick-up of As_2O_3 from baghouse dust by recycled solutions averaged only 26.3 gm/l at 100° C as compared to the 40 to 45 gm/l obtained in the lab. The pick-up of As_2O_3 per unit of solution would improve if temperatures could be maintained in the thickening and filtering stages of the process.

The principle of a two-stage countercurrent leach proved to be necessary as evidenced by the overall recovery of As_2O_3 obtained in the pilot plant. The wide variance of solubility of arsenious oxide in the first stage leach was counter-balanced by the recovery of an undersaturated solution from the second stage leach. This is further reinforced by the wide variance observed in the second stage leach pickup of As_2O_3 , 3.40 gm/l with a standard deviation of 10.38 gm/l. To meet the environmental restrictions imposed on this process a two stage counter-current leach will be required. Unfortunately the two stage leach will require greater energy input per pound of arsenic recovered. Recovery of the heat removed during crystallization will be needed to temper this increased energy requirement.

A total of 33.6 kg of solution must be heated to recover each kg of purified arsenic trioxide. Ideally a heat energy input of 3,360 kilocalories are required for each kg of purified As_2O_3 produced.

Assuming that 60% of the heat removed during crystallization can be recovered, it is estimated that actual energy requirements will be closer to 6,720 k. cal./kg of purified As_2O_3 produced.

Crystallization of Arsenious Oxide

The crystallized arsenious oxide product averaged 99.43% As_2O_3 , 0.021% Fe and 0.237% Sb. Impurity accumulation in the crystallized product from closed recycle of solutions does not appear to be a problem. The recovery of As_2O_3 from saturated leach solution averaged 32.9 grams/litre.

Fe Contamination

An Fe standard of 0.02 wt % cannot be consistently achieved. A product specification of 0.02 to 0.05 would allow more operational flexibility. The presence of Fe in saturated crystallizer feed solutions is related to two factors:

- 1) Efficiency of leach solution clarification;
- 2) The pH of leach solutions. pH must be controlled above 4.0 to inhibit the dissolution of iron.

The Fe standard of 0.02 wt % was exceeded in approximately 28.4% of the leach cycles.

Sb Contamination

An Sb standard of 0.20 wt % was exceeded in approximately 46% of the leach cycles. The crystallization of antimony is pH dependent with increased recovery in the crystal product with decreasing pH levels below 4.0. Reliable pH control would have reduced many of these higher Sb levels to meet the 0.20 wt % standard.

Antimony does not appear to be a major concern of manufacturer's of wood preservative products. A product specification of 0.20 to 0.50 wt % would allow more operational flexibility.

As₂O₃ Specification

The As₂O₃ standard of 98% was exceeded consistently and appears to present no problem. Average product quality should exceed 99% As₂O₃ approximately 85% of the time.

Spectrographic analysis indicate no other potential impurity problems.

Final Residue

The undissolved residue remaining after two stage leaching of baghouse dust represents 8.5% of the total weight treated. On average the residue assayed 10.46% As₂O₃. Less than 1% of the total arsenious oxide contained in the baghouse dust reported to the final residue. No soluble arsenic was detected by chemical titration. To be consistent all total arsenic assays were converted by a factor of 1.3204 to be reported as As₂O₃. Due to the insoluble nature of the residual arsenic it probably is in the form of ferro and ferric arsenate.

Objectives

Cottrell dust treatment for gold recovery generates 89% of the arsenic going to Giant's tailings pond. The present lime treatment method effectively precipitates soluble arsenic and retains it from solution while the pH is maintained high. In the tailings pond a neutral decant is desired and at such a lowered pH the calcium arsenate slowly resolubilizes. It is suspected that other soluble arsenic compounds in the tailings are not completely dissolved when lime treated and hence unaffected. The prime objective now is to come up with economically viable technology to eliminate this major arsenic water pollution source.

Subjecting the cottrell dust to a hot leach should dissolve the arsenic which would ever do so under the cold conditions existing in the tailings area. The initial intention to treat the cottrell dust with the baghouse dust to recover saleable arsenic trioxide was abandoned for two reasons. Arsenic content of baghouse dust and solids load from the cottrell dust would combine to result in leach residue cake, requiring complicated and expensive liquid-solid separation techniques to wash the moisture from that cake. Conceivably at 30% moisture assaying the same as recycle solution, the residue would be higher in arsenic content than the original cottrell dust. Secondly the high soluble iron content of the cottrell would contaminate crystal product.

Ruling out the combined treatment brought out the idea of separate circuits, but hot leaching of cottrell dust would not result in saturated solutions amenable to arsenic trioxide crystallization. However a hot leach to remove the soluble arsenic prior to gold recovery may be beneficial, clarified leach liquor then treated with lime generates a calcium arsenate-lime sludge which can be gravity collected.

The existing (Fig. 3.4.) pilot plant was put to task to test this process. Goals were to determine:

1. Could a soluble arsenic free residue be obtained and

if so could lower temperatures be used.

2. The efficiency of lime-arsenic precipitation and settleability
3. Liquid solid separation difficulties.
4. Recycle problems if any with specific buildups.
5. Retention times.

Also to provide residue for cyanidation testing and precipitate for arsenic recovery tests.

Operational Procedure

Basically the flowsheet remains the same as with the preceeding baghouse dust pilot test except for the crystallizer tanks. These were used as stirred lime reactors for approximately 30 minutes after the pH of the filtered #1 thickener overflow was brought to the 11.5 - 12.0 range by quicklime additions. The agitators were then shut down and these same vessels served as settling tanks for the resulting precipitate. Cooling water was not circulated about these tanks as was the practice when they were used for crystallization. The clear supernatant resulting after a settling period was used for re-slurrying the first stage thickener underflow in the second stage leach tank.

Operating Conditions

Leach # 1

Slurry Density: 12.5 % Solids - Batch 1 thru 23.

18.75 % Solids - Batch 24 thru 32.

25.0 % Solids - Batch 33 thru 81.

Retention Time: 2 hours (not including heat-up time)

Temperature: 95° - 100° C Batch 1 thru 44

90° C Batch 45 thru 49

80° C Batch 50 thru 60

60° C Batch 61 thru 69

40° C Batch 70 thru 81

Flocculant Consumption: 3 p.p.m./batch of Dearborn Aquafloc
467 added in a 0.1 % solution.

Thickener # 1

Temperature: 90° - 95° C

Leach # 2

Slurry Density: Density measurement and weighed bucket addition of # 1 Thickener underflow to approximate matching the #1 leach slurry density.

Temperature: 95°-100° C

Flocculant Consumption: 3ppm/batch of Dearborn Aquafloc
467 added in a 0.1 % solution.

Thickener # 2

Temperature : 90° - 95° C

Lime Reactor / Settling Tanks

Retention Time: 30 minutes agitation.

Slow Thickener settling times dictated entire process
slowdown resulting in approximately 32 hours settling
in these vessels.

Campaign Results

In general the campaign results were disappointing. Improper sampling, lack of data, and operational difficulties combined to complicate the data analysis. In retrospect this is not surprising considering the early difficulties experienced in treating baghouse dust and the neglect of conducting laboratory locked cycle tests prior to piloting.

The inability to separate wash solution from the undissolved residue overshadowed the entire run. Leach residence time was dictated by the cottrell dust settling rate in the small pilot plant thickeners. The natural settling rate of cottrell dust was measured at 1.46×10^{-3} ft/min. at 20% solids. The addition of neither low (3 ppm) nor high (9 ppm) flocculant dosages appears to have any effect on settling rates. The high surface area of the cottrell dust quickly absorbs flocculant, necessitating the addition of exorbitant levels before any increase in settling rate is attained. Centrifuging has proven to be the only successful means of liquid-solid separation.

Effect of Wash Temperature

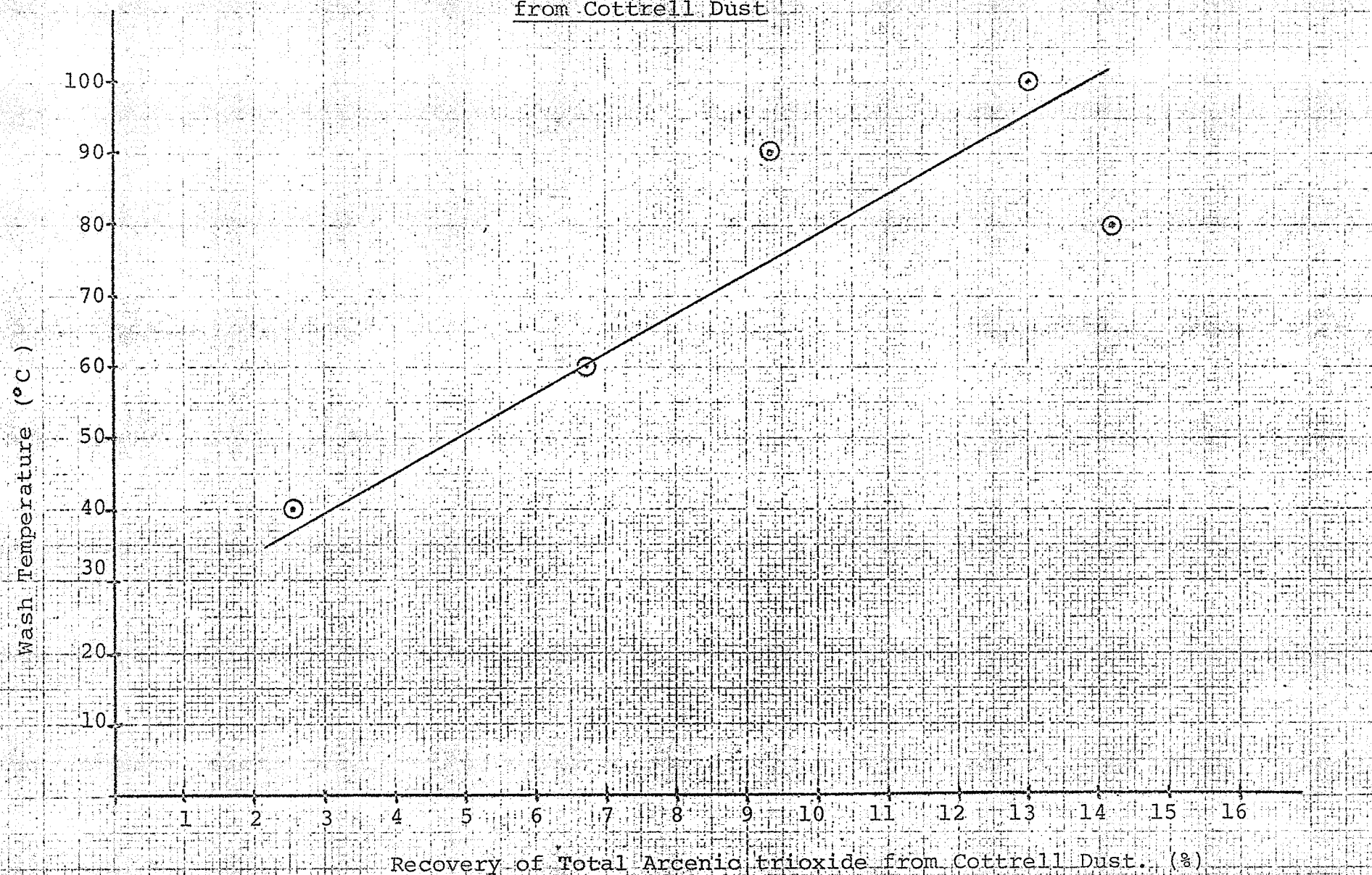
A 25% solids by weight slurry of cottrell dust was washed at varying wash temperatures (Figure 3.4.2.A.). The recovery of total As_2O_3 was low in all cases however as expected recovery increases with temperature. At the higher temperatures, slurry viscosity is lower, allowing better "wetting" of Cottrell dust and subsequent dissolution of As_2O_3 .

The variance in test results suggests that washing efficiency does not improve at incremental temperatures above 80 C.

Wash Temperature:	100 C	90 C	80 C	60 C	40 C
Recovery of Total As_2O_3 in %:	13.00	9.34	14.19	6.77	2.56

The low recoveries were later found to be a function of slurry density as well as temperature.

FIGURE 3.4.2.A. Effect of Wash Temperature on Total As_2O_3 Extraction
from Cottrell Dust



Effect of Slurry Density

Varying density slurries of cottrell dust were washed at 100 C (Figure 3.4.2.B.). The best washing efficiency was attained at the lowest density.

Slurry Density in % Solids:	12.5	18.75	25.0
Recovery of Total As_2O_3 in %:	50.71	13.82	13.00

As would be expected, the lower slurry viscosity lends itself to more efficient washing and hence removal of As_2O_3 . Too little data is available for more detailed analysis.

Washing Efficiency

The cottrell dust pilot plant campaign results are presented in detail in Table 3.4.2.A. and summarized to some extent in Table 3.4.2.B. .In summary, the best attainable wash efficiency was the extraction of 98.94% of the soluble As_2O_3 contained in cottrell dust. The percentage of total As_2O_3 in cottrell dust that is water soluble was seen to vary from 16% to 56% with evidence that a great deal of the dust treated in the pilot plant campaign falling at the lower end of the range.

Soluble As_2O_3 in Washed Cottrell Dust

Every fourth pilot plant cottrell wash circuit residue was analyzed for water soluble As_2O_3 . No soluble As_2O_3 was detectable by chemical titration analysis. More sensitive analytical techniques indicate the presence of 0.017 ± 0.016 % soluble As_2O_3 . On average only 0.651 ± 0.590 % of the total As_2O_3 in washed cottrell dust was water soluble. A wide variance was observed on all results. A calculated balance (Figure 3.4.2.C.) suggests that the residue passing on to the carbon plant for gold recovery will contain only 6 lbs./day of water soluble As_2O_3 , down from the present calculated value of 164.65 to 566.0 lbs./day (16 to 55 % soluble).

Lime Treatment of Wash Solutions

The addition of lime to the As_2O_3 bearing wash solutions was found to be 99.53 ± 1.5 % efficient at a pH of 11.5 to 12.0, i.e. greater than 99% of the soluble As_2O_3 in the wash solutions was recovered in the resulting calcium arsenate-lime precipitate. In full scale the lime required for this process would not be an additional milling cost as this lime is presently being consumed to treat the carbon plant wash thickener overflow and barren streams.

The As_2O_3 content of the calcium arsenate-lime precipitate was highly variable, averaging 2.30 %. The anticipated dry tonnage of calcium arsenate-lime precipitate produced each day is calculated to be 2.48 tons. This material could be pumped at 50% solids to underground storage or possibly chemically fixed with silicates and disposed of on surface. Future work may even find a use for calcium arsenate.

FIGURE 3.4.2.B. Recovery of Total As_2O_3 vs Slurry Density

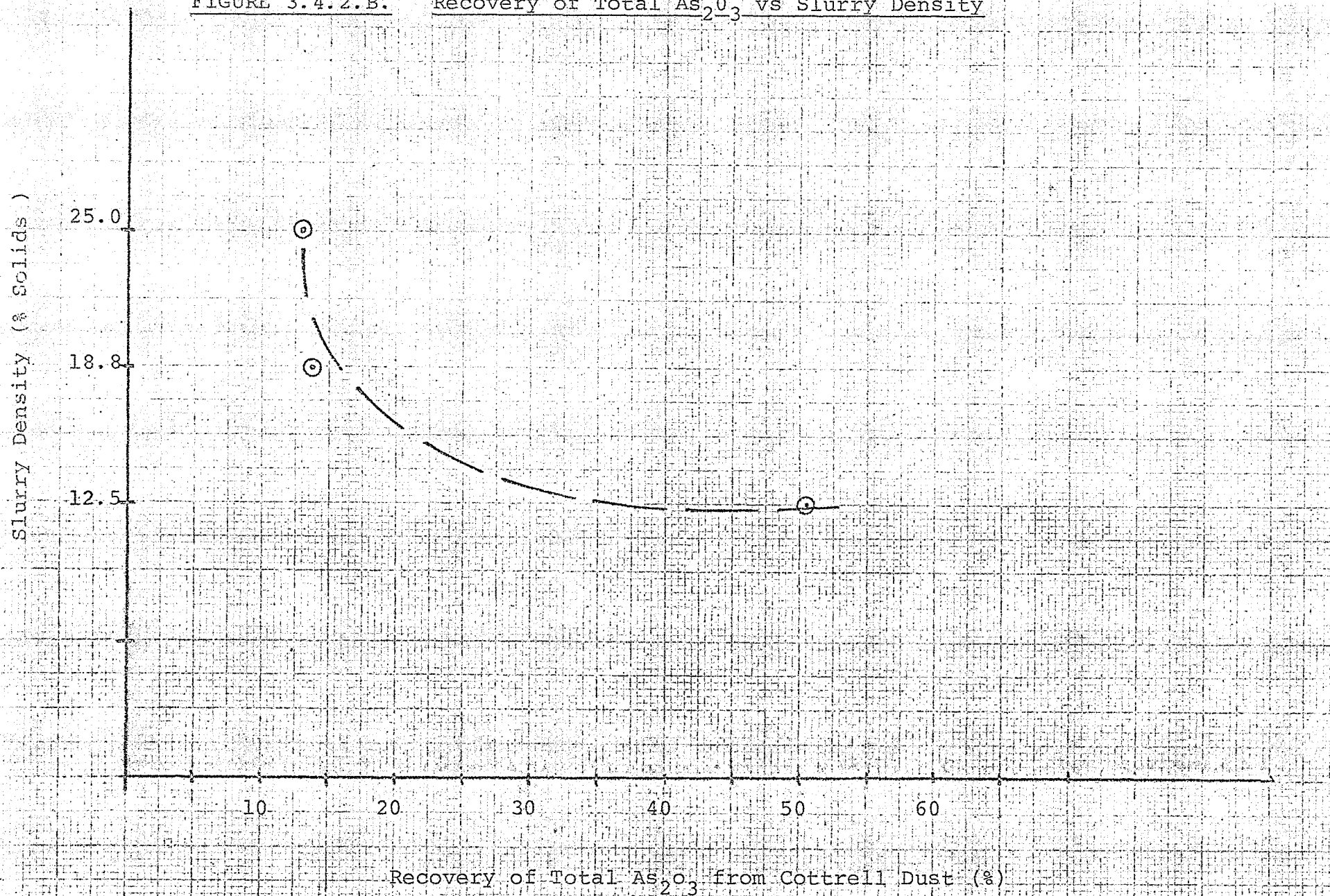


TABLE 3.4.2a TREATMENT OF GIANT COTTRELL DUST

Pilot Plant Campaign

Batch	lb	BATCH FEED			CRYSTALIZER FEED			SLUDGE			STRIP SOL'N			RECYCLE SOL'N			RESIDUE			Insol As ₂ O ₃
		As ₂ O ₃ %	Fe %	Sb %	As ₂ O ₃ g/l	Fe ppm	Sb ppm	As ₂ O ₃ %	Fe %	Sb %	As ₂ O ₃ g/l	Fe ppm	Sb ppm	As ₂ O ₃ g/l	Fe ppm	Sb ppm	As ₂ O ₃ %	Sb %	Fe %	
1	50	---			---			---			---			---			---			
2	50	2.40	18.0	0.32	---			---			---			---			14.04			
3	50	2.40	18.0	0.35	---			---			---			0.50	2.0	15.0	2.60			
4	50	2.40	18.0	0.37	---			---			---			0.46	0.2	16.4	2.31	0.27	18.5	
5	50	2.60	18.0	0.36	1.42	2.0	24.8	1.51	.04	0.10	0.01	nd	6.6	0.30	0.8	14.2	2.60	0.28	16.25	
6	50	2.40	18.0	0.30	1.06	6.2	21.6	27.81	.04	0.30	0.04	nd	8.4	0.26	3.6	15.0	2.21	0.26	17.5	2.18
7	50	2.40	18.0	0.28	1.26	1.2	22.6	36.63	.09	0.36	0.04	nd	3.3	0.58	0.8	12.0	2.40			
8	50	3.60	18.0	0.49	1.54	1.0	24.6	3.91	.04	0.13	0.01	nd	2.4	0.74	nd	10.2	3.00	0.33	17.5	
9	50	4.21	18.0	0.49	1.80	1.2	23.2	13.63	.08	0.26	0.01	nd	2.3	0.50	1.2	10.0	2.90	0.25	17.25	
10	50	2.40	18.0	0.30	1.36	4.0	19.2	10.72	.04	0.20	0.01	nd	2.6	0.38	1.0	9.0	3.00	0.25	16.75	3.02
11	50	2.60	18.0	0.39	8.72	1.8	25.8	12.68	.02	0.13	nd	nd	2.2	0.18	0.6	8.0	3.10	0.29	16.75	
12	50	2.40			4.38	0.4	23.2	1.85	.02	0.10	nd	nd	2.6	0.48	1.0	8.8	5.61	0.26	16.75	
13	50	3.01			2.60	0.6	23.4	12.42	.05	0.20	nd	nd	1.7	0.48	0.8	7.6	5.02	0.25	17.5	
14	50	3.01			2.42	2.2	27.8	0.61	.05	0.11	nd	nd	1.5	0.12	0.4	5.4	2.69	0.21	18.0	
15	50	3.01	17.75		6.36	1.6	27.8	5.18	.03	0.08	nd	nd	1.6	0.02	1.2	3.2	2.71			
16	50	14.43			0.20	2.6	3.6	23.87	.08	0.12	nd	0.01	1.5	0.14	0.8	6.6	2.79	0.24	19.0	
17	50	3.01			1.20	nd	9.2	0.73	.03	0.07	0.02	nd	3.5	0.14	4.2	8.0	1.88	0.22	18.25	
18	"	1.70			0.68	0.8	7.4	1.03	.03	0.07	0.02	nd	1.5	0.18	3.4	7.0	2.38	0.21	19.0	2.28
19	"	2.10			0.14	0.2	4.0	0.41	.02	0.07	0.01	nd	3.3	0.20	1.0	9.8	2.59	0.22	18.75	
20	"	2.10			0.50	0.6	10.0				0.02	0.10	3.2	0.16	1.0	8.4	3.00	0.27	18.25	
21	"	2.21			0.34	1.8	10.2	1.54	.03	0.09	nd	0.10	3.3	0.30	1.2	13.6				
22	"	2.21	18.25		0.04	1.2	5.8	0.30	.06	0.07	nd	nd	3.5	0.16	2.4	10.2	2.38	0.22	18.0	2.71
23	"	2.60			1.24	1.2	19.4	1.35	.03	0.05	0.02			0.18	0.4	8.4	2.38	0.21	19.25	
24	75	5.22			2.12	1.6	23.0	3.41	.03	0.07	nd	0.10	3.5	nd	nd	3.6	2.38	0.20	17.5	
25	75	3.30			2.44	0.2	9.0	1.86	.03	0.05	nd	0.10	3.5				2.38	0.25	19.25	
26	75	2.21			1.26	2.0	11.2	1.86	.10	0.05	nd	nd	3.6	0.10	1.0	6.4	2.69	0.26	19.25	2.71
27	"	2.21			0.64	0.6	10.6	3.21	.06	0.07	nd	0.50	2.6	0.70	1.8	13.2				
28	"	2.81			nd	nd	5.6	5.59	.04	0.07	nd	0.10	3.2	0.18	0.8	8.4	3.00	0.24	19.00	
29	"	2.31	18.25		1.1	1.0	11.0	5.18	.04	0.15				0.28	1.0	9.6	2.89	0.22	19.25	
30	"	2.40			6.12	4.2	23.6	8.17	.19	0.15	nd	nd	2.6	0.34	1.8	8.0	2.69	0.20	19.0	
31	"	2.71			nd	1.4	5.8	2.59	.09	0.14	nd	0.10	2.3	0.36	0.8	12.0	2.59	0.21	19.0	
32	"	2.31			0.66	1.2	11.2	1.04	.04	0.13	nd	0.10	3.1	0.18	1.0	10.6	2.79	0.22	19.0	
33	100	2.38			0.78	1.2	10.0	3.10	1.50	0.17	nd	0.10	2.6	0.20	1.0	7.8	2.69	0.24	19.25	
34	100	2.38			1.16	1.6	12.2	1.54	1.50	0.13	0.01	0.30	2.5	0.02	0.2	4.0	2.50	0.21	18.25	2.60

TABLE 3.4. 2a. TREATMENT OF GIANT COTTRELL DUST

Pilot Plant Campaign

Batch	BATCH FEED				CRYSTALLIZER FEED			SLUDGE			STRIP SOL'N			RECYCLE SOL'N			RESIDUE			
	lb	As ₂ O ₃ %	Fe %	Sb %	As ₂ O ₃ g/l	Fe ppm	Sb ppm	As ₂ O ₃ %	Fe %	Sb %	As ₂ O ₃ g/l	Fe ppm	Sb ppm	As ₂ O ₃ g/l	Fe ppm	Sb ppm	As ₂ O ₃ %	Sb %	Fe %	Insol As ₂ O ₃
35	100	2.38			0.68	1.2	11.4	0.51	0.05	0.13	nd	0.10	2.8	0.68	1.2	12.6	2.71			
36	100	2.38	18.25		1.00	1.8	15.0	0.41	0.10	0.14	0.02	0.10	2.9	0.46	1.8	11.6	2.71	0.28	18.00	
37	100	2.59			1.05	1.8	16.2	1.15	0.11	0.08	nd	0.10	3.6	1.56	0.8	18.8	2.50	0.25	17.00	
38	100	2.59			2.96	4.8	22.4	1.77	1.50	0.08	nd	0.20	4.2	1.90	1.8	20.4	2.60	0.29	18.00	
39	100	3.00			4.22	2.4	21.2	12.29	0.14	0.11	nd	nd	3.1	nd	1.0	6.0	2.92			2.60
40	100	2.48			1.70	2.8	15.8	4.07	0.10	0.09	nd	0.10	2.9	0.36	2.4	8.6	2.71	0.29	17.00	
41	"	2.27			nd	0.6	7.0	1.24	0.14	0.11	nd	0.30	2.8	0.38	1.6	11.8	2.30	0.21	16.00	
42	"	2.50			1.86	2.0	18.2	1.96	0.50	15.5	0.32	1.8	11.8	0.32	1.8	11.8	2.30	0.26	16.25	
43	"	2.40			1.24	2.6	13.4	1.15	0.03	0.09	nd	nd	3.8	1.16	1.6	18.8	2.40	0.25	17.75	
44	"	2.60	16.75		nd	0.6	6.4	1.56	0.05	0.09	nd	nd	3.3	0.26	5.8	6.8	2.39	0.20	12.00	
45	"	2.50			1.16	2.6	17.0	2.30	0.08	0.09	0.03	0.40	1.8	nd	1.0	5.2				
46	"	2.35			1.10	2.6	14.2	2.71	0.12	0.09	nd	0.10	4.4	nd	1.0	5.6	2.92	0.29	16.25	
47	"	2.19			1.14	1.2	12.8	5.11	0.11	0.11	nd	0.30	4.2	0.02	2.6	7.6	3.02	0.33	16.50	
48	"	2.19			0.94	0.6	14.2	1.56	1.38	0.07	nd	nd	2.2	0.16	1.6	10.6	2.71	0.26	17.00	
49	"	---			0.84	3.2	15.6	1.66	0.75	0.09	nd	nd	2.6	nd	nd	4.0	2.19	0.25	16.75	
50	"	2.39	16.75		0.50	0.8	11.6	1.25	6.63	0.13	nd	nd	2.4	0.48	0.4	12.4	2.30	0.26	15.50	
51	"	2.39			1.16	1.2	17.6	2.92	1.63	0.10	nd	0.80	2.7	0.38	1.0	23.8	2.50	0.34	17.00	
52	"	2.60			1.32	1.2	32.0	0.73	0.75	0.10	nd	0.20	2.1	0.86	1.0	30.8	2.30	0.34	17.00	
53	"	2.50			1.64	2.6	34.0	0.94	0.50	0.08	nd	0.10	3.6	0.98	1.6	19.2	2.30	0.38	17.50	
54	"	2.71			1.80	2.0	38.4	2.30	6.63	0.15	nd	0.10	2.4	0.06	1.4	5.8	2.50	0.31	17.00	2.60
55	"	3.13			1.36	1.6	24.2	3.33	1.38	0.10	nd	0.10	2.8	0.04	3.0	6.0	2.60	0.25	16.75	
56	"	3.13						1.36	1.25	0.09	nd	0.30	3.1	0.20	2.4	13.2	2.50	0.25	16.75	
57	"	3.13	16.75		1.20	3.4	25.6	6.98	1.25	0.17	nd	0.40	3.5	0.16	1.6	13.4	2.50	0.49	16.75	
58	"	2.92			1.14	3.2	29.4	1.45	6.50	0.13	nd	nd	2.7	0.16	1.6	17.0	2.60			2.54
59	"	2.71			1.12	1.8	28.8	0.42	1.38	0.07	nd	nd	3.3	0.16	0.16	21.0	3.43			
60	"	2.92			0.78	1.8	27.6	3.64	1.25	0.09	nd	nd	1.6	0.10	1.2	12.2	3.02	0.33	17.50	
61	"	3.13			1.00	1.8	31.6	4.05	0.75	0.10	nd	0.20	1.7	0.64	3.8	19.6	3.02	0.28	16.75	
62	"	3.54			0.64	1.4	21.6	1.56	4.88	0.11	nd	0.20	1.3	0.02	1.0	9.0	2.60	0.48	16.75	
63	"	3.33			0.62	3.0	9.2	0.94	1.95	0.07	nd	nd	3.1	0.06	1.8	7.4	2.39			
64	"	2.19	17.00		0.98	3.8	19.6	1.77	1.50	0.08	nd	nd	2.0	0.14	2.2	16.2	2.92	0.54	17.75	
65	"	2.09			0.78	2.2	18.0	5.10	1.00	0.12	nd	nd	2.4	nd	nd	4.4	2.60	0.33	16.25	
66	"	2.19			0.42	4.2	13.4	2.50	2.00	0.09	nd	nd	2.4	0.04	1.2	2.4	2.60			2.28

TABLE 3.4.2a TREATMENT OF GIANT COTTRELL DUST

Pilot Plant Campaign

Batch	lb	BATCH FEED			CRYSTALIZER FEED			SLUDGE			STRIP SOL'N			RECYCLE SOL'N			RESIDUE			
		As ₂ O ₃ %	Fe %	Sb %	As ₂ O ₃ g/l	Fe ppm	Sb ppm	As ₂ O ₃ %	Fe %	Sb %	As ₂ O ₃ g/l	Fe ppm	Sb ppm	As ₂ O ₃ g/l	Fe ppm	Sb ppm	As ₂ O ₃ %	Sb %	Fe %	Insol As ₂ O ₃
67	100	2.30			0.34	1.4	8.2	2.92	0.63	0.10	0.28	0.3	5.4	0.28	0.6	9.0	2.39			
68	"	2.19			0.28	1.0	6.4	1.25	0.75	0.10	nd	nd	2.5	nd	nd	3.6	2.28	0.29	17.25	
69	"	2.39			0.50	1.4	7.6	0.83	0.12	0.08	nd	nd	2.6	0.24	1.2	8.6	2.60	0.32	17.25	
70	"	2.30			0.34	1.0	7.4	0.83	1.88	0.09	nd	nd	2.4	nd	nd	4.6	2.60	0.33	16.75	
71	"	2.30	17.25		0.18	1.2	6.8	0.11	0.50	0.08	nd	nd	2.5	nd	nd	4.6	2.81			
72	"	2.19			0.04	0.4	7.2	0.32	0.38	0.08	nd	nd	2.6	nd	nd	5.2	2.92			
73	"	2.18			0.26	nd	8.4	0.21	0.06	0.09	nd	nd	2.5	0.02	0.6	8.6	2.50			
74	"	2.39			0.22	1.2	9.4	1.15	2.81	0.10	nd	nd	2.5	0.02	0.8	7.2	2.28	0.27	17.25	
75	"	2.28			0.08	1.2	9.2	0.62	0.13	0.09	nd	nd	2.6	0.04	1.6	9.2	2.09	0.20	18.00	2.18
76	"	2.28			0.26	0.8	7.6	0.73	0.44	0.10	nd	nd	2.5	nd	nd	5.0	2.18	0.22	16.50	2.28
77	"	2.18			0.24	1.6	6.8	2.18	0.16	0.09	nd	nd	2.6							
78	"	2.28	17.25		0.26	1.8	10.2	0.51	1.69	0.08	nd	nd	2.7							
79	"	2.18																		
80	"	2.18																		
81	"	2.18																		

Cycle Temperatures: Batch 1 thru 44 100 C
Batch 45 thru 49 90 C
Batch 50 thru 60 80 C
Batch 61 thru 69 60 C
Batch 70 thru 81 40 C

TABLE 3.4.2b Means and Standard Deviations of Data from Treatment of Giant Cottrell Dust

Batch	lb	BATCH FEED			CRYSTALIZER FEED			SLUDGE			STRIP SOL'N			RECYCLE SOL'N			wt lb	RESIDUE			
		As % ₂ O ₃	Fe %	Sb %	As g/l ₂ O ₃	Fe ppm	Sb ppm	As % ₂ O ₃	Fe %	Sb %	As g/l ₂ O ₃	Fe ppm	Sb ppm	As g/l ₂ O ₃	Fe ppm	Sb ppm		Au oz/Tn	As % ₂ O ₃	Sb %	Fe %
1-23	50	M	2.61	18.0	0.37	1.30	1.61	17.56	8.68	0.04	0.14	0.01	0.01	3.06	0.31	1.33	9.85		2.88	0.25	17.84
T-100°	SD		0.54	0.10	0.07	1.05	1.42	8.36	10.54	0.02	0.09	0.01	0.03	1.75	0.18	1.12	3.34		0.67	0.03	0.88
24-32	75	M	2.83	18.25		1.59	1.36	12.33	3.66	0.07	0.10	nd	0.13	3.05	0.27	1.03	8.98		2.68	0.23	18.91
T-100°	SD		0.91			1.79	1.18	6.21	2.14	0.05	0.04	0.0	0.15	0.46	0.20	0.54	2.89		0.21	0.02	0.54
33-44	100	M	2.50	17.5		1.39	1.95	14.10	1.65	0.47	0.11	0.003	0.15	3.14	0.61	1.75	11.58		2.56	0.25	17.5
T-100°	SD		0.18			1.15	1.10	4.85	1.07	0.63	0.03	0.006	0.14	0.51	0.59	1.34	5.15		0.19	0.03	0.97
45-49	100	M	2.31	16.75		1.04	2.04	14.76	2.67	0.49	0.09	0.006	0.16	3.04	0.04	1.24	6.60		2.71	0.28	16.63
T-90°	SD		0.13			0.12	0.97	1.43	1.29	0.51	0.01	0.012	0.16	1.06	0.06	0.85	2.31		0.32	0.03	0.28
50-60	100	M	2.78	16.75		1.20	1.96	26.92	2.30	2.65	0.11	nd	0.18	2.75	0.33	1.40	15.89		2.60	0.33	16.86
T-80°	SD		0.27			0.36	0.82	7.41	1.80	2.43	0.03	0.0	0.23	0.58	0.31	0.77	7.15		0.33	0.07	0.55
61-69	100	M	2.59			0.62	2.24	15.07	2.32	1.51	0.09	0.03	0.08	2.60	0.16	1.31	8.91		2.60	0.37	17.00
T-60°	SD		0.54			0.25	1.09	7.89	1.38	1.33	0.01	0.09	0.11	1.11	0.20	1.12	5.38		0.23	0.09	0.48
70-81	100	M	2.24			0.21	1.02	8.11	0.74	0.89	0.09	nd	nd	2.54	0.01	0.43	6.34		2.48	0.26	17.13
T-40°	SD		0.07			0.09	0.53	1.17	0.59		0.01	0.0	0.0	0.08	0.01	0.57	1.82		0.29	0.05	0.57

Figure 3.4.2.C. Extraction of As_2O_3 from Cottrell Dust

Baghouse dust leach residue.

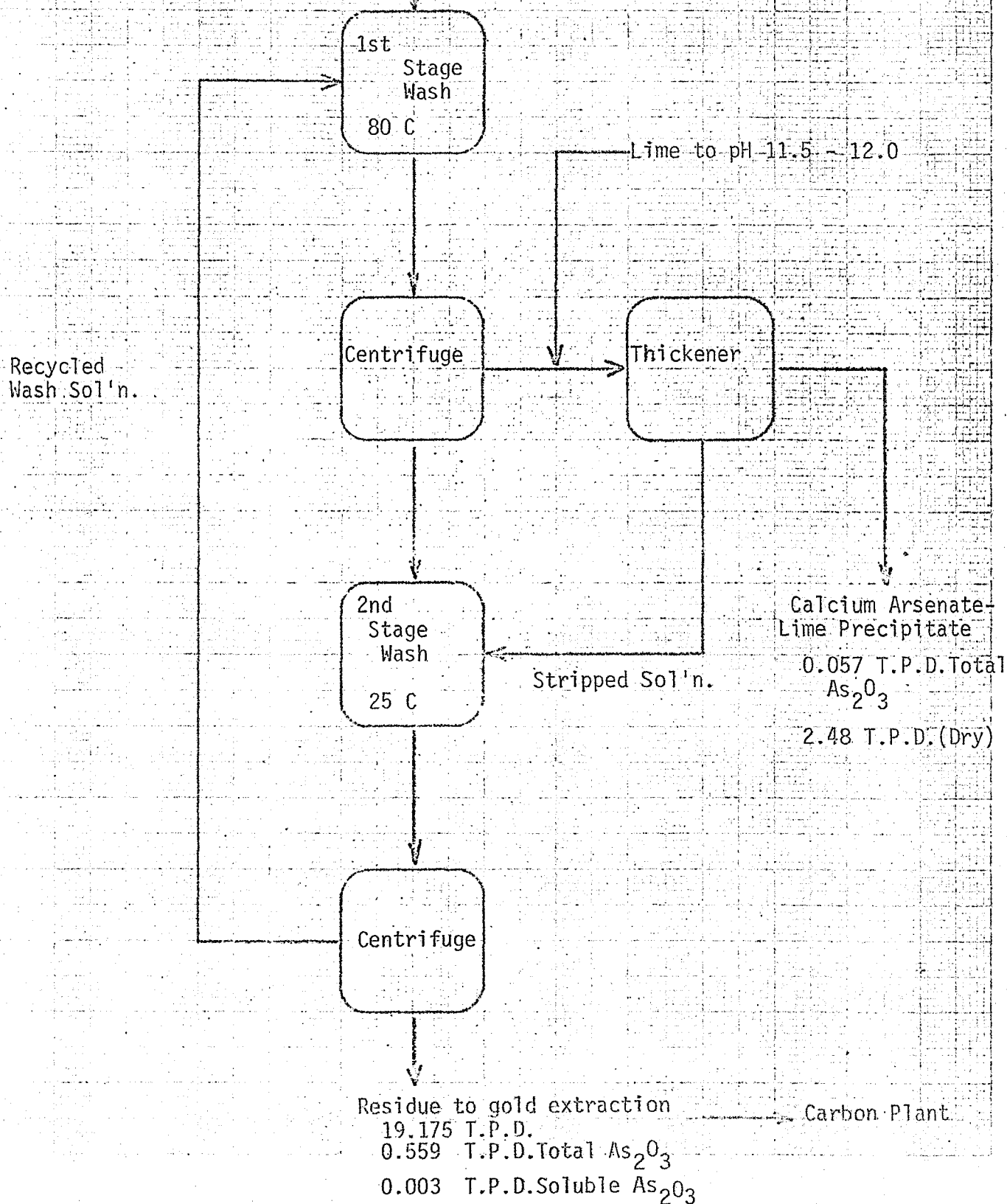
0.975 T.P.D.

0.102 T.P.D. Total As_2O_3

Cottrell Dust

20 T.P.D. (0.514 T.P.D. Total As_2O_3)

0.283 T.P.D. Soluble As_2O_3



Gold Extraction from Washed Cottrell Dust

Approximately 25% of the pilot plant cottrell wash circuit residues were cyanided in the mill lab, simulating the carbon plant process. Recoveries obtained after 60 hours retention time averaged 89.15 ± 3.53 %. Carbon plant recoveries presently average 80 to 84 %. The 1.6 to 5.6 % improvement in recovery translates to an additional 8 to 28 ounces of gold per month which should help offset the operating costs of a two stage cottrell dust washing circuit.

Although only speculation a 90 + % decrease in the soluble arsenic presently reporting with the carbon plant wash thickener overflow stream should reduce final tailings pond decant levels of total arsenic by 60 to 70 %. This claim is based on calculated reductions only and does not take into account presently unknown factors or reaction that may occur in the tailings pond. Arsenic levels in the final decant can be further reduced by recovering calcium arsenate-lime sludge presently precipitated from roaster calcine wash thickener overflow solutions.

3.4.3

Con Arsenic Pond Sludges

A two stage countercurrent leach process for the recovery and purification of As_2O_3 from Con arsenic pond sludge is presently being piloted. Results are not yet available. Early indications suggest a separate circuit will be required to treat this material.

PURIFICATION AND RECOVERY OF
ARSENIOUS OXIDE FROM ROASTER DUSTS

SECTION 4

CRYSTALLIZATION OF As_2O_3 FROM SATURATED SOLUTION

Crystallization from solution is a common method either for purification and separation or for achieving a desirable particle size. Successful design of crystallization equipment depends largely on detailed experiments conducted with the particular material under consideration. The design must also take into account standard crystallization practices.

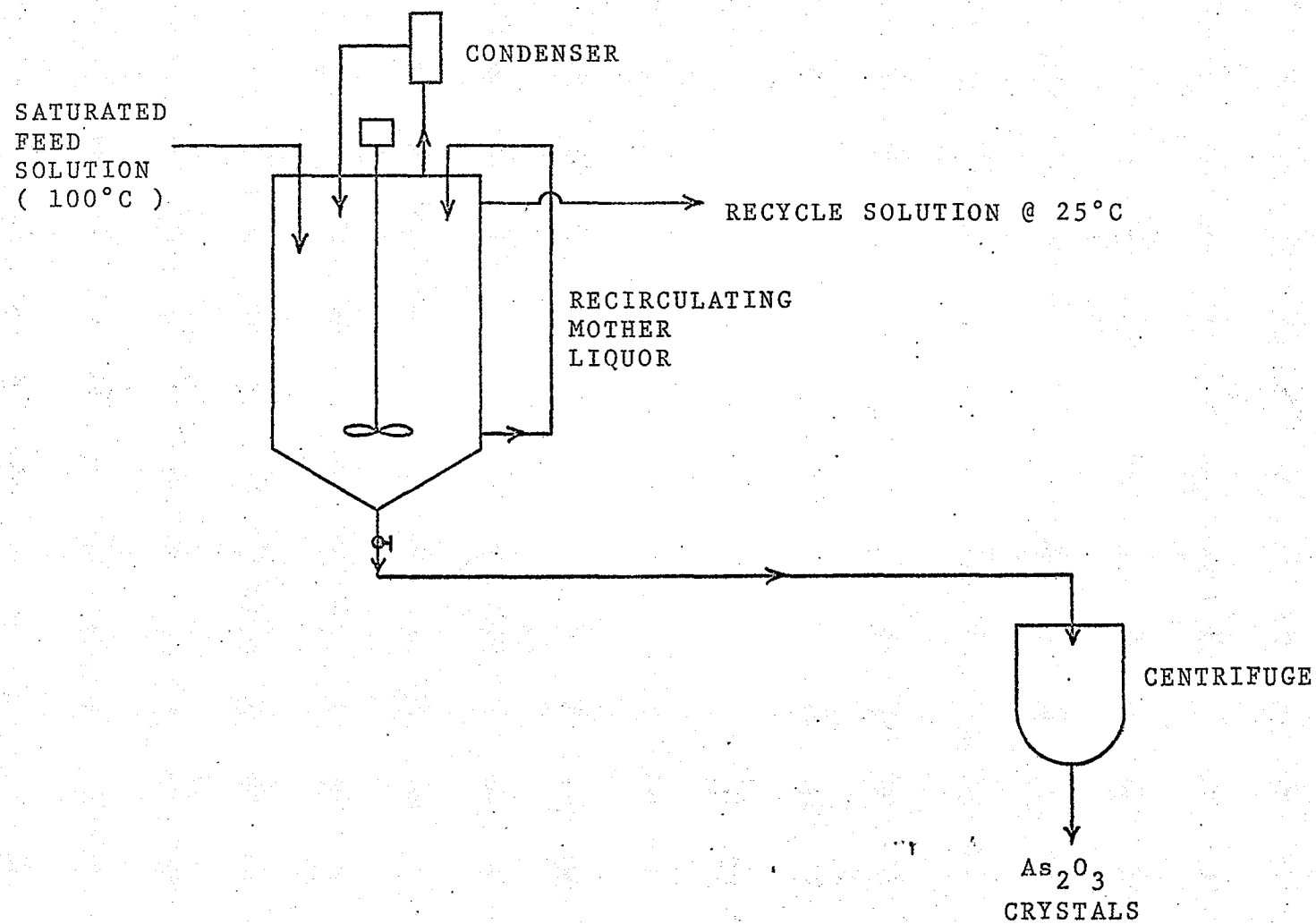
In surveying the literature on crystallization equipment, there appears to be four methods available to Giant for the crystallization of arsenic trioxide. The pros and cons of each method are as follows:

4.1 Option 1 - Slow Cooling

Common practice in plants of very small capacity dealing with concentrated solutions and materials of normal solubility is to pump the hot feed solution into an unagitated tank and allow cooling to occur either by natural convection and radiation or by surface cooling through coils in the tank or a jacket on the outside of the tank. Figure 4.1.1.

While the equipment required for such a system is extremely inexpensive and simple, there is nothing simple about its operation. Nucleation is difficult to control or predict and the cooling rate varies considerably in an open tank depending on the humidity and air velocity. The addition of a propellor or turbine type agitator and a cooling system (jacket or coils) will greatly increase the capacity of a tank crystallizer but introduces other problems.

Figure 4.1.1 Slow Cooling Crystallizer



Agitation sufficient to suspend the crystals introduces the possibility of increased nucleation as the solution reaches its initial crystallization point and during the cooling process from mechanical stimulation of the super saturated solution and contact nucleation. Generally the build-up of crystallized material on cooling coils and surfaces limits more widespread application of this tendency, it usually does not eliminate it. For maximum production capacity, the coolant should be tempered by recirculation so that the temperature difference across the coils is as small as feasible. For materials which adhere readily to the crystallizer walls and cooling surfaces, the temperature difference should be in the range of 5° to 10° F. This imposes a severe limitation on the capacity of the equipment.

A clarified saturated solution of arsenic trioxide (100° C) would be fed to a crystallization tank and allowed to slowly cool to room temperature under strong agitation. The crystals that form would settle to the bottom of the tank and be recycled to encourage crystal growth and minimize wall build-up. Retention time has been estimated between 32 to 40 hours to attain a recycle solution of less than 30 gm/l As_2O_3 .

Advantages

- low capital cost
- simple operation

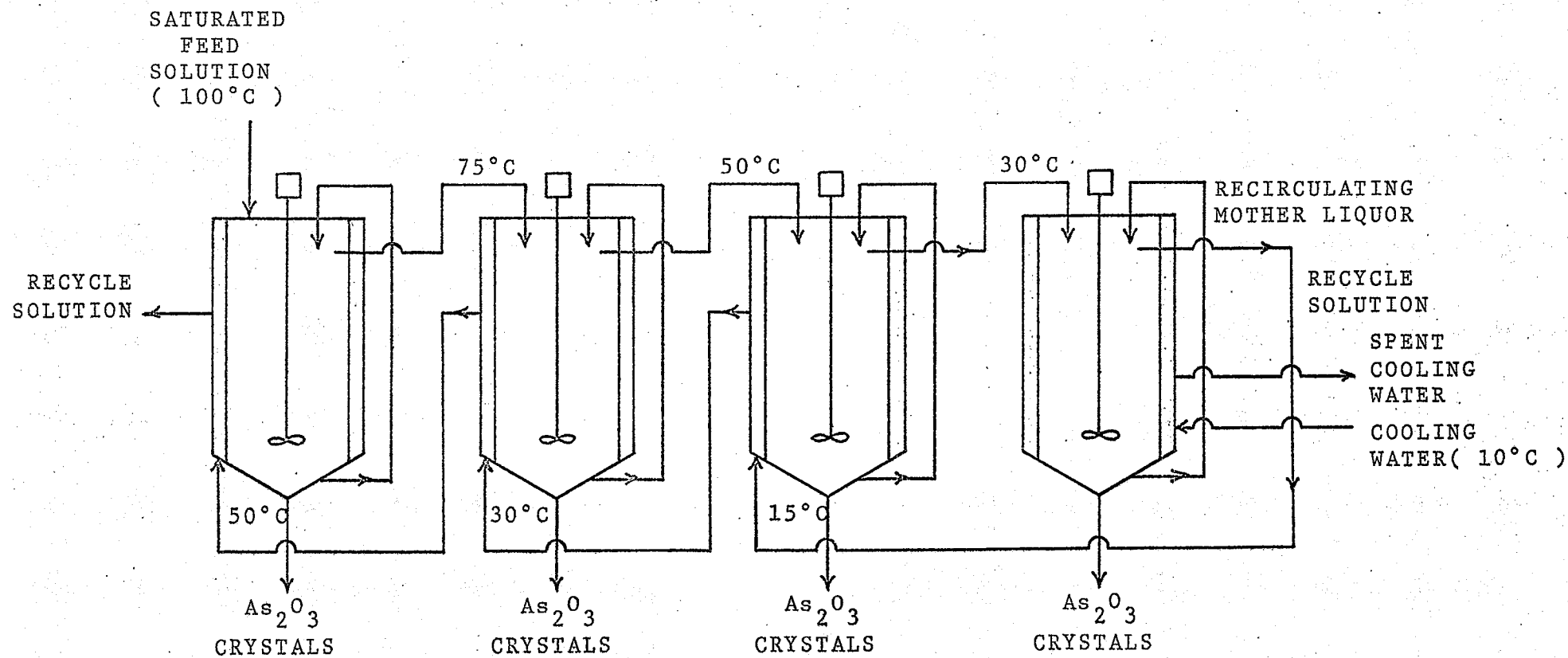
Disadvantages

- no recovery of heat
- tendency for crystals to grow on tank walls
- long retention times

4.2 Option 2 - Forced Cooling Crystallization

A clarified saturated solution of As_2O_3 (100° C) would be passed through a series of double walled agitated crystallizer tanks. Figure 4.2.1. The final tank would be cooled by passing external cooling water through the cavity left between the double walls of the crystallizer. The recycle solution would overflow the final tank and be passed countercurrent through the double wall cavity of the other crystallizing tanks. This would allow for more rapid

Figure 4.2.1 Forced Cooling Crystallizer



cooling of the saturated solution, thus increasing crystallizer capacity while also recovering some of the sensible heat contained in the feed solution. (The temperatures shown in Figure 4.2.1 are solely speculation.) As in the case of slow cooling crystals settling to the bottom of the tanks would be recycled to encourage crystal growth and minimize wall build-up. Retention time has been estimated between 6 to 8 hours to attain a recycle solution of less than 30 gm/l As_2O_3 .

Advantages

- Partial recovery of the sensible heat contained in the saturated feed solution

Disadvantages

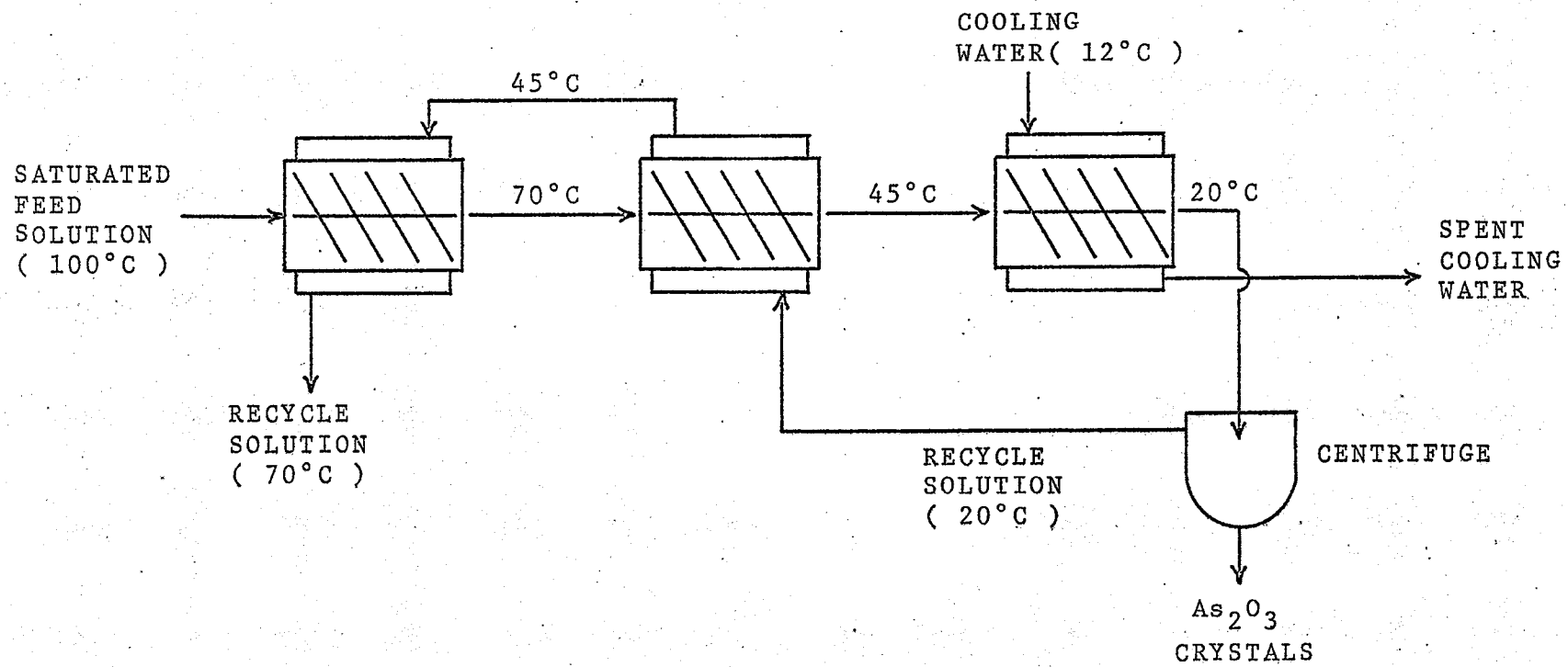
- Tendency for scaling on all cooling surfaces
- High capital cost of double walled tanks

4.3 Option 3 - Scraped Wall Crystallizer

For relatively small scale applications a number of crystallizer designs have been developed employing direct heat exchange between the slurry and a jacket or double wall containing a cooling medium. Figure 4.3.1. The heat transfer surface is scraped or agitated in such a way that crystal deposits do not build up. The scraped surface crystallizer provides an effective and relatively inexpensive method of producing crystals as the equipment does not require expensive installation or supporting structures.

An example is the Swenson-Walker crystallizer consisting of a trough 24 inches wide, with a semi-circular bottom and a cooling jacket welded to the outside. Within the crystallizer is a spiral agitator turning 3 to 10 rpm. The blades suspend the growing crystals and break from the wall any deposits of crystalline material which have attached themselves to the cooled surface. The unit is manufactured in lengths of 10 to 40 feet. Although the Swenson-Walker scraped wall crystallizer can be operated in a batch manner, more often solution is fed into one end continuously and crystal slurry overflows from the opposite end. The units can be cascaded. Generally, the product from such a crystallizer is relatively wide in crystal size distribution.

Figure 4.3.1 Scraped Wall Crystallizer



A clarified saturated solution of As_2O_3 (100°C) would be passed through a series of scraped wall crystallizer tanks. The final tank would be cooled by passing external cooling water through the cooling jacket. The crystal slurry overflowing the final crystallizer tank would be centrifuged to remove the As_2O_3 crystals with the centrate or recycle solution being passed counter-current through the cooling jackets on the preceeding scraped wall crystallizer tanks. Again this would allow for more rapid cooling of the saturated solution, increasing crystallizer capacity while also recovering some of the sensible heat contained in the feed solution. (The temperatures shown in Figure 4.3.1 are estimates only.) Retention time has been estimated between 6 to 8 hours to attain a recycle solution of less than 30 gm/l As_2O_3 :

Advantages

- Partial recovery of the sensible heat contained in the saturated feed solution

Disadvantages

- High degree of mechanical maintenance required
- No control over crystal growth size

4.4 Option 4 - Classified - Suspension Crystallizer

The classified suspension crystallizer is characterized by the production of supersaturation in a circulating stream of liquor. Super-saturation is developed in one part of the system by evaporative cooling or by cooling in a heat exchanger and it is relieved by passing the liquor through a fluidized bed of crystals. The fluidized bed may be contained in a simple tank or in a more sophisticated vessel arranged for a pronounced classification of the crystal sizes. Two examples of this type of crystallizer are:

- A) Oslo surface cooled crystallizer - cooling of the saturated solution is accomplished in a heat exchanger. Figure 4.4.1.
- B) "Krystal" type crystallizer - vacuum cooling of saturated solutions. Figure 4.4.2.

Figure 4.4.2 Vacuum Cooled Classified Suspension Crystallizer

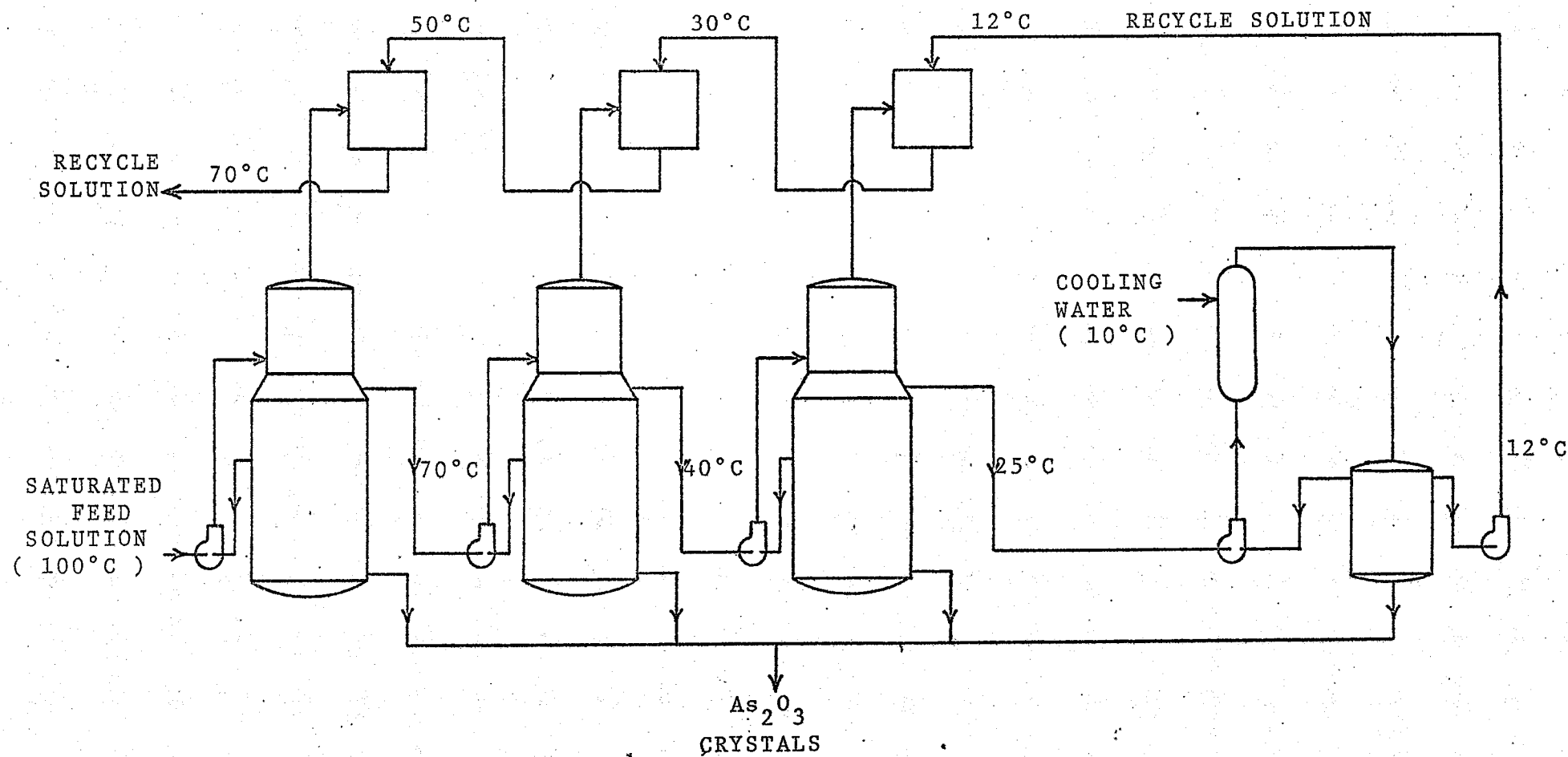
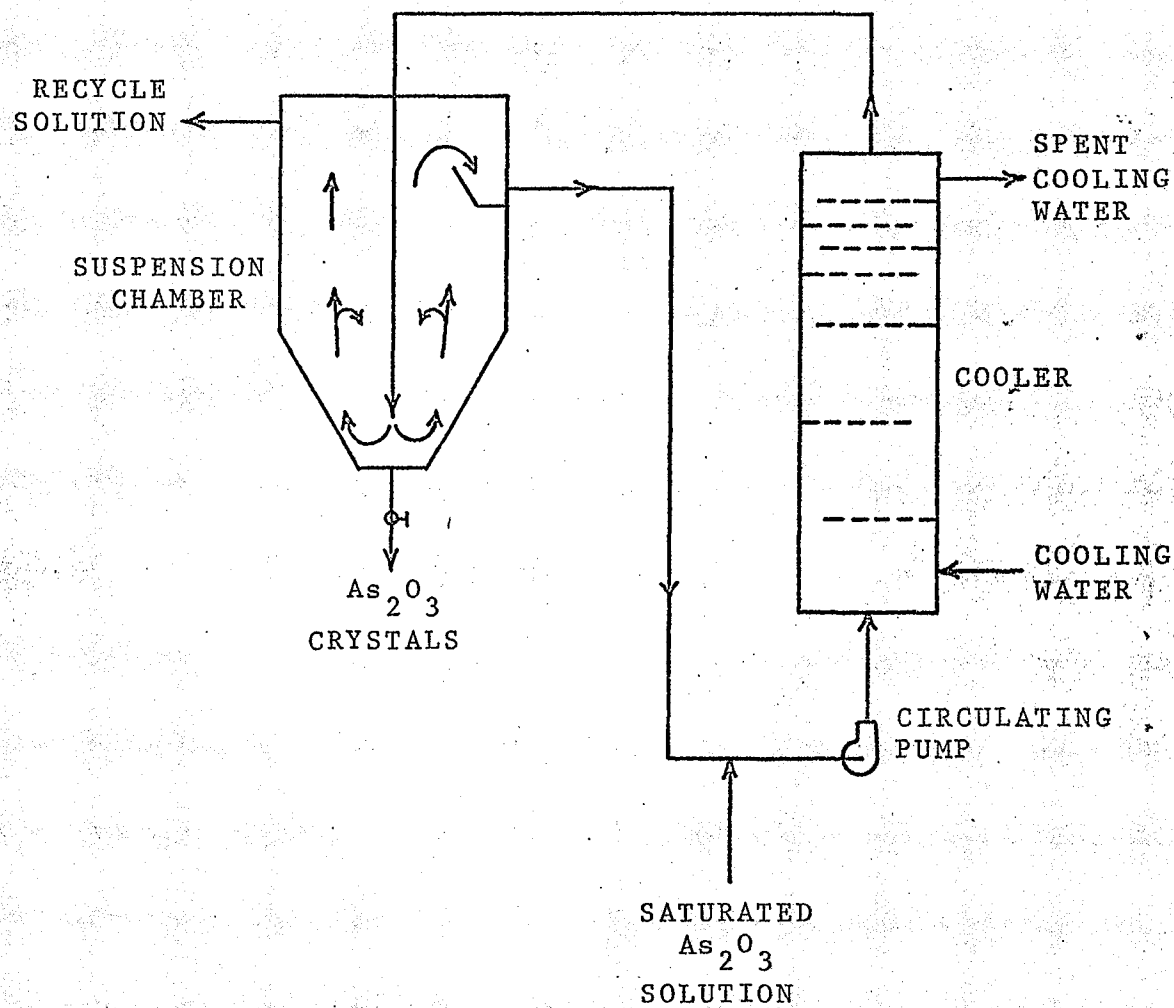


Figure 4.4.1 Oslo Surfaced- Cooled Crystallizer



The main advantage of the classified suspension crystallizer over other types lies in its flexibility in controlling crystal product size.

Advantages

- Control over crystal growth size hence better dewatering and solids handling characteristics.
- Higher yield per unit of solution. The evaporation of water results in a lower hydraulic load being processed (vacuum).

Disadvantages

- High capital cost.

4.5 Vacuum Crystallization of As_2O_3 at Struthers Wells

During the week of May 27 to June 1st, a series of As_2O_3 crystallization tests were conducted at the pilot facilities of Struthers Scientific and International Corporation in Warren, Pennsylvania. Struthers Scientific and International Corporation is a subsidiary of Struthers-Wells established to market the Struthers Wells' "Krystal" line of continuous crystallization equipment. The purpose of these tests were to enable Struthers to formulate a commercial proposal on a full scale As_2O_3 crystallization plant for Giant Yellowknife.

Struthers offers crystallization equipment in three main categories:

- 1) Evaporative crystallizers - used to produce crystals from feed solutions that are not saturated or from which all of the solvent is to be evaporated.
- 2) Vacuum cooling crystallizers which continuously cool hot feed solutions, usually near the saturation point, to produce a crystalline product.
- 3) Contact cooling crystallizers which are used for low temperature operation or with solutions that have no appreciable vapour pressure at the operating temperature.

4.5.1 Test Program

The Struthers engineering group chose to initiate the test program using vacuum cooling crystallization equipment. This choice was based on:

- 1) The steep slope of the theoretical As_2O_3 saturation versus temperature curve, and
- 2) Their previous experience in crystallizing As_2O_3 from solutions provided by ASARCO.

Vacuum crystallizers induce crystallization by continuously cooling a saturated feed solution by vacuum evaporation. Basically a saturated solution of As_2O_3 at 95° to 100° C would be mixed with a large amount of circulating mother liquor. This mixture of saturated feed and mother liquor is pumped into a vessel called a vaporizer, where under the action of vacuum the sensible heat in the feed plus the heat of crystallization are removed by evaporation. Evaporation causes the solution to become slightly supersaturated. This supersaturated mixture flows down the central tube of the crystallizer and up through a fluidized suspension of crystals where the supersaturation is released on the crystals. Fluidization of the crystal suspension is produced only by the circulating liquor. All mechanical mixers are avoided. This method of fluidization is intended to provide the optimum medium for good crystal growth. Each crystal surface, kept in contact with continuously renewed supersaturated solution, is free to grow on all sides. Finished crystals are removed from the suspension container by a variety of means to suit any particular arrangement of equipment in the process. Vacuum crystallizers may be used singly or in multiple effect (See Brochure Appendix A)

4.5.2 Pilot Plant

A pilot scale single effect vacuum crystallizer was employed by Struthers Wells for the Giant test run (See flowsheet in Appendix C). A saturated solution of arsenic trioxide at 90° C was produced by dissolving an excess of baghouse dust which in turn was clarified and fed to a heated holding tank. The saturated solution was combined with circulating mother liquor and pumped to the vaporizer where under vacuum pressure the solution was cooled to 25° C by evaporation.

At first no crystal load appeared to be forming. However, it soon became apparent that the arsenic trioxide was crystallizing on the walls of the separation column effectively plugging the flow through the crystallizer. The unit was cleaned out and the run restarted. The crystals recovered from the cleanout were fed to the unit as seed material. The circulating load of crystal was allowed to slowly climb to 22% by volume at which point crystals were periodically withdrawn through a salt trap.

Over the four-day run crystallizer feed solutions averaged 61.41 g/l As_2O_3 while the crystallizer overflow solutions averaged 30.37 g/l As_2O_3 giving a recovery of 31.04 g/l of As_2O_3 . The heating coils in the feed tank could only maintain solution temperatures between 85° and 90° C resulting in the lower feed concentrations of As_2O_3 . The crystals produced were extremely fine grained averaging 85.3% -325 mesh, but were well within marketable standards. A more detailed analysis of the crystal product is presented in Appendix C.

Struthers Wells suggested the use of a contact cooling crystallizer to further recover arsenic trioxide from the overflow solution from vacuum cooling. The unit would take advantage of the year-round availability of cold water to further cool the overflow solution from the vacuum cooling crystallizer and thus

increase the recovery of As_2O_3 per unit of solution. This was tested in a separate pilot run.

Mr. Hatch of the Falconbridge Metallurgical Laboratories attended this second pilot run and reported the following conclusions: (9)

Arsenious oxide can be continuously crystallized from solution operating the Krystal crystallizer as a contact cooled unit. Satisfactory operation was achieved when baghouse dust was leached in recycle solution with no scaling evident over a 50 hour operating period.

To avoid short circuiting of feed solution through the crystallizer, two or more units would be required in plant design. The contact cooled crystallizer could be operated in conjunction with one or more vacuum crystallizers recycling the overflow through the condensor. Assuming that the previous stage vacuum crystallizer was operated at 25°C , there would appear to be little added advantage cooling to 18°C in an additional unit using cold water as coolant. From a knowledge of the temperature-solubility curve one would expect an additional four g/l As_2O_3 product. A drop of at least 30°C would provide a reasonable product yield in the crystallizer.

Any of the mentioned crystallization methods should work on recovering As_2O_3 from saturated leach solutions. The choice between methods is basically a compromise between high capital cost and savings attained in heat recovery. No cost benefit evaluation for each option has been attempted at Giant at this time.

APPENDIX A

Treatment of Soluble Arsenic in Giant Mill Effluent: Options

The DPAT Project

The joint Giant-Federal Government DPAT project was successful in reducing arsenic levels in the Giant mill effluent to 0.2 mg/l. The DPAT project removed soluble arsenic from mill effluent streams by precipitating both As^{+3} and As^{+5} forms of arsenic with ferric hydroxide ($\text{Fe}(\text{OH})_3$). The exact mechanism of the process is not known, but it very likely involves the incorporation of arsenic ions in the hydroxyl network of ferric hydroxide via chemical and physical adsorption phenomena. Unfortunately the arsenic sludges formed are not permanently stable. As the pH of the sludges drop, as they would be expected with years of contact with groundwaters, the arsenic would resolubilize creating further environmental problems.

In general, the cost to Giant of reducing arsenic and cyanide levels using the DPAT technology would exceed half a million dollars a year. The resulting arsenic sludges are known not to be stable and may result in further environmental damage.

Ferric Sulphate Treatment Process

Following the completion of the DPAT project, Giant conducted an in-plant test to determine the effectiveness and actual cost involved in suppressing arsenic using lime and ferric sulphate. The two calcine wash thickener streams were combined with the carbon plant thickener overflow and treated with ferric sulphate and lime. A summary of the results is as follows:

	Lime pH 8.2	<u>Fe/As Addition Ratio</u>			
		<u>2/1</u>	<u>3/1</u>	<u>4/1</u>	<u>5/1</u>
% Dissolved As Suppressed	22	85	92	92	93
Avg ppm Dissolved As in Final Tailings	12.4	8.1	12.4	7.3	7.1
Extrapolated Cost of Fe Addition to Treat As Streams. Cost of Ferric Sulphate only:		\$2,116 /day	\$3,174 /day	\$4,232 /day	\$5,290 /day

The test was run continuously over a five-day period. As expected, the amount of dissolved arsenic suppressed increased with increased Fe salt addition, however, beyond an addition ratio of 3/1 (Fe/As) the ferric sulphate is believed to be primarily acting to fix the arsenic sludge. While water soluble arsenic can be precipitated using ferric sulphate, the cost to Giant of treating their effluent streams would be enormous.

Sludge Fixation - Canada Waste Technology

The ultimate disposal of any physical-chemical treatment sludges produced from Giant's mill effluent streams through chemical fixation is the goal of work being done by Canada Waste Technology (formerly Krofchak Ltd.) under contract to EPS on Giant mill waste products.

The lime treatment process currently in practice at Giant will lower dissolved arsenic concentrations by a factor of 95% provided the pH is maintained at 12.0. However, as the pH drops towards neutrality (as in the tailings pond) calcium arsenate will resolubilize restoring original dissolved arsenic concentrations.

The equilibrium between free and complex metal cyanides also contributes to maintaining dissolved metal values in future pond decant and leachate. As free cyanide is destroyed by the ultraviolet content of sunlight, complex metal cyanides break down in an effort to maintain equilibrium balance. The net result is an increase in dissolved metals concentration.

The process under investigation by Canada Waste Technology is chemical fixation of Giant mill waste by solidification. This is a method whereby waste materials are complexed as silicate compounds which form rock-hard stable compounds analogous to the forms the elements are naturally found in the earth.

A report was published in June of 1978, although technique performance was poor, the results were justified because of methodology weaknesses. The lessons learned from these mistakes offer encouragement for a comprehensive economic and practical feasibility evaluation resulting from the second attempt underway at this time. The new report should be available in April.

The application of this technology is being tested on the calcine circuit residue and the carbon plant barren slurry. Flotation tailings are being tested as a potential source of silica.

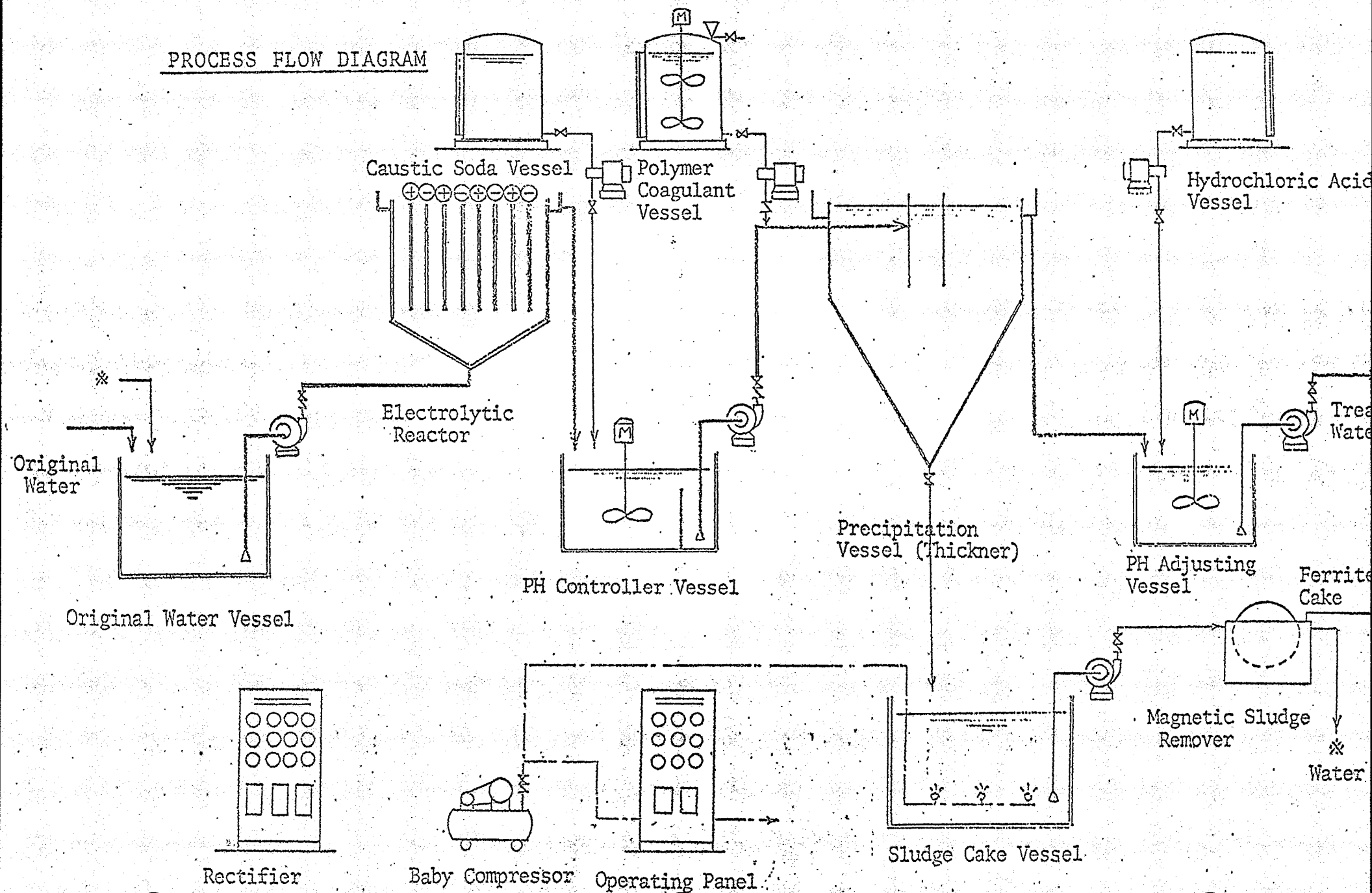
Mitsubishi Electrolytic Ferrite Formation System

In February of 1978 Mitsubishi Petrochemical Engineering Company of Japan was contacted regarding a new process pioneered by them for the removal of heavy metal ions from plating process wastewaters. Briefly the process is as follows: iron dissolved in the waste stream by electrolysis of iron anodes produces hydroxide which in turn coagulates the dispersed pollutants. Alkali for pH control is then added with a flocculant. The suspension moves onto a thickener from which the sludge underflow is removed and aerated to complete the formation of ferrite, a complex salt of the hydroxide and heavy metals. Ferrite is a general term used to describe ferric/ferrous oxides having a spinel structure. The heavy metal ferrite complexes will not redissolve even after many years contact with groundwaters. The process is being used commercially in Japan to remove Cr^{+6} ions from plating solutions and may offer an alternative to the DPAT use of ferric sulphate for complexing arsenic.

Mitsubishi informs us that the process is applicable to all metal ions which form a spinel crystal structure. Arsenic does form an insoluble ferrite compound known as arsenoferrite (FeAs_2) however we do not know whether the Mitsubishi process is applicable to Giant effluent streams. We are maintaining contact with the Canadian arm of Mitsubishi to learn more of the process's applicability in treating Giant's effluent problems.

MITSUBISHI ELECTROLYTIC FERRITE FORMATION SYSTEM

PROCESS FLOW DIAGRAM



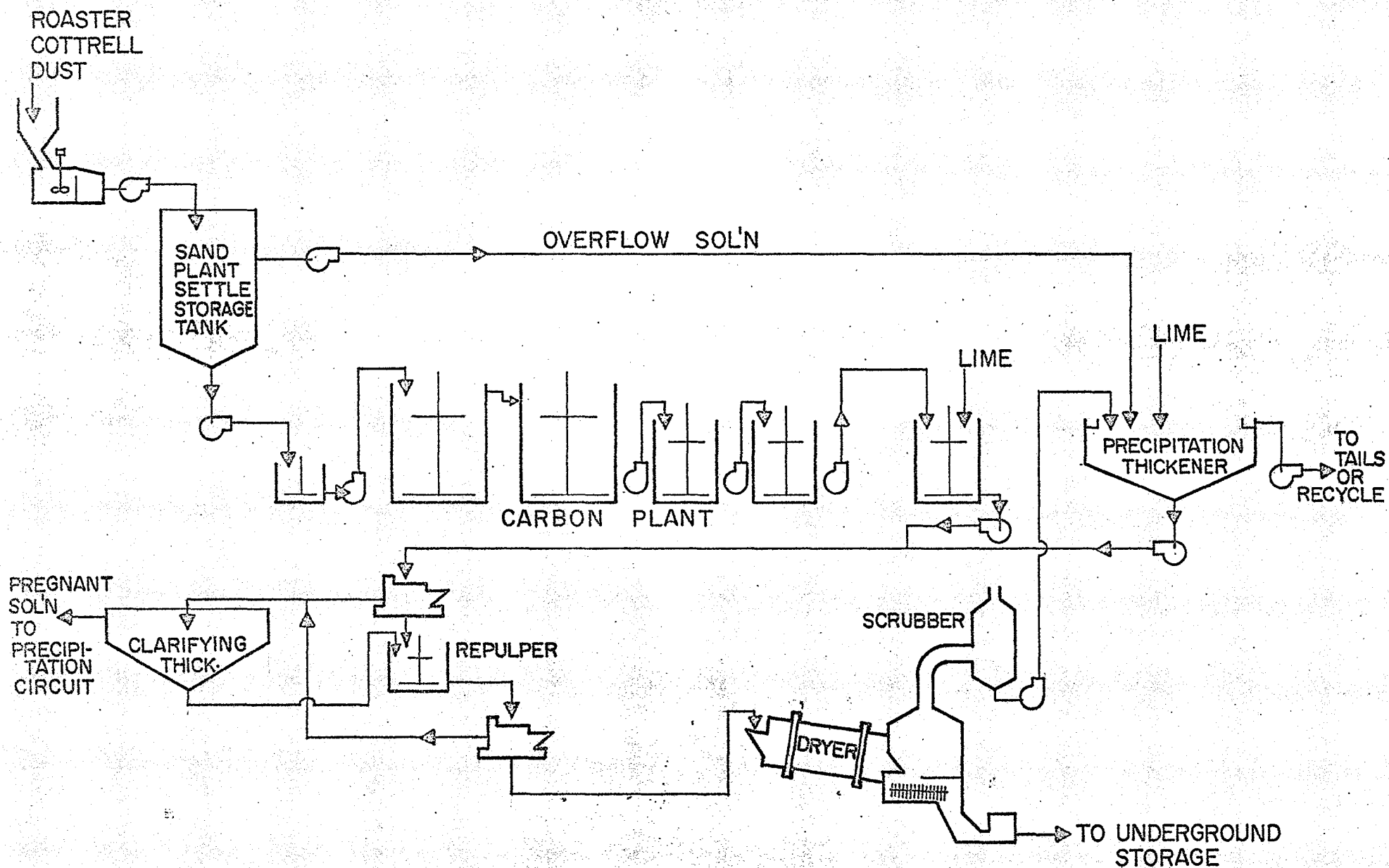
Carbon Plant Expansion

Close to 90% of the total arsenic reporting in the final mill tailing is associated with the treatment of roaster cottrell dust. By eliminating this release of arsenic, significant improvements in effluent quality would be expected.

Based on this premise a flowsheet was developed in the summer of 1978 envisioning underground storage of the total arsenic and solids contained in the cottrell dust, following treatment for gold (see attached flowsheet). Basically the cottrell dust would be water quenched and then thickened in a settle storage tank. The settle storage tank overflow would then be treated with lime to precipitate the contained soluble arsenic as calcium arsenate. The calcium arsenate would be removed in a thickener allowing the overflow solution to be recycled as cottrell dust quench water. The settle storage tank underflow would be treated for gold recovery by cyanidation in an expanded leaching circuit and then treated with lime to again precipitate soluble arsenic as calcium arsenate. The calcium arsenate from the precipitation thickener would be combined with the treated solids and then centrifuged, washed and recentrifuged. The resulting clear liquor would be treated with zinc dust to precipitate the contained gold while the cottrell dust solids and precipitated calcium arsenate is dried and pneumatically transported to underground storage vaults.

As is the case with the DPAT project, the flowsheet as envisioned would have no net economic benefit to Giant and would only be a partial solution at a considerable cost in capital outlay for equipment and operating cost. Nevertheless as an alternative to the DPAT project the flowsheet did warrant some investigation. For this reason the rental of two pilot centrifuges has been arranged for the first half of 1979. They will enable a more detailed study of the viability of this approach to reducing arsenic concentrations in the final mill effluent.

Figure A1 Precipitation and U/G Storage of Roaster Cottrell Dust



Appendix B

Purification of Arsenious Oxide
from Roaster Dusts
by the Hot Water Leach Process

FALCONBRIDGE NICKEL MINES LIMITED
METALLURGICAL LABORATORIES
THORNHILL, ONTARIO

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January 25, 1979

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SUMMARY

A preliminary laboratory investigation has been carried out on the purification of arsenious oxide (As_2O_3) produced from the roasting of arsenopyrite gold ores. Testwork was carried out on samples of Giant Yellowknife Gold Mines underground storage material containing 60% soluble As_2O_3 and Con Mines tailing pond material containing 64% soluble As_2O_3 .

The process involves an initial water leaching step which solubilizes the As_2O_3 at $98^\circ\text{--}100^\circ\text{C}$ to produce a saturated solution containing 70 g/L As_2O_3 . No reagents are required for dissolution of the As_2O_3 which dissolves to form arsenious acid (H_3AsO_3) in solution having a pH^+ of 2.5-4.0. The washed residue contains insoluble gangue components, arsenopyrite, pyrite and ferric arsenate which are not attacked under the pH-redox conditions of the leach. This residue is dried and treated for gold recovery.

The saturated arsenious acid solution is cooled to room temperature to crystallize out the arsenious oxide product which is separated from the mother liquor by filtration. The mother liquor contains approximately 28 g/L As_2O_3 which is recycled to the leach producing a closed loop with no effluent discharge.

The product As_2O_3 is dried to remove surface water. The dried product has a purity of 99.5% As_2O_3 and meets A.E. and C.I. purity specifications.

INTRODUCTION

The roasting of arsenopyrite ores at Giant Yellowknife Mines and the recovery of arsenic as baghouse dust has been described(1). The recovery of arsenic and gold from underground storage areas at Giant as well as from current production is presently under consideration. Substantial quantities of baghouse dust from the Con Mine are also available for recovery from a tailings pond.

Preliminary results of a hot water leach process have been described and a proposal made for a more detailed investigation and flowsheet preparation(2). The objective of Stage I was the preparation of a high purity (min. 98% As_2O_3) product containing less than 0.2% Sb_2O_3 and less than 0.1% Fe. The product should also be white in colour and suitable for the glass industry.

The objective of Stage II was the recovery of gold from the leach residue obtained in the treatment of the impure As_2O_3 baghouse dust. This report covers Stage I investigations with preliminary work on Stage II reported by Lakefield Research.

Testwork has been carried out on Con Mines tailing material (FML #78-346) which consisted of several samples taken at various depths of the disposal area. A 35 lb pail of arsenious oxide baghouse dust from an underground Giant Yellowknife storage area was also evaluated for As_2O_3 purification (FML #78-476).

FLOWSHEET

A preliminary flowsheet is given in Figure 1 based on the experimental work carried out to date. The flowsheet is based on the production of 1.0 t per hour of As_2O_3 on an 8 hour per day operating basis.

FEED

The flowsheet is based on the treatment of Con tailings pond material containing 26% free water. All water in the feed material is saturated with As_2O_3 and if the feed is slurried, recycle solution (30 g/L As_2O_3) should be used to maintain the water balance in the system. The various samples of Con material taken at different depths have averaged approximately 64% soluble As_2O_3 (dry basis). Mineralogical examination of the feed from Con reveals crystalline cubic As_2O_3 to be the major phase with minor amounts of ferric arsenate ($\text{FeAsO}_4 \cdot 2\text{H}_2\text{O}$) quartz, magnetite, pyrite and arsenopyrite(3). It is not known whether the amorphous glassy form of As_2O_3 is present since it is not detectable by X-ray diffraction.

The analysis of Con tailings pond and Giant underground storage material samples is as follows:

FIGURE 1: Treatment of Con Tailings Pond As₂O₃ by Hot Water Leach Process

Feed 2.11 tph (wet)

As ₂ O ₃ (soluble)	- 47.4%	1.0 tph
Gangue (insoluble)	- 26.7%	0.56 tph
Water	- 25.9%	0.55 tph
		2.11 tph

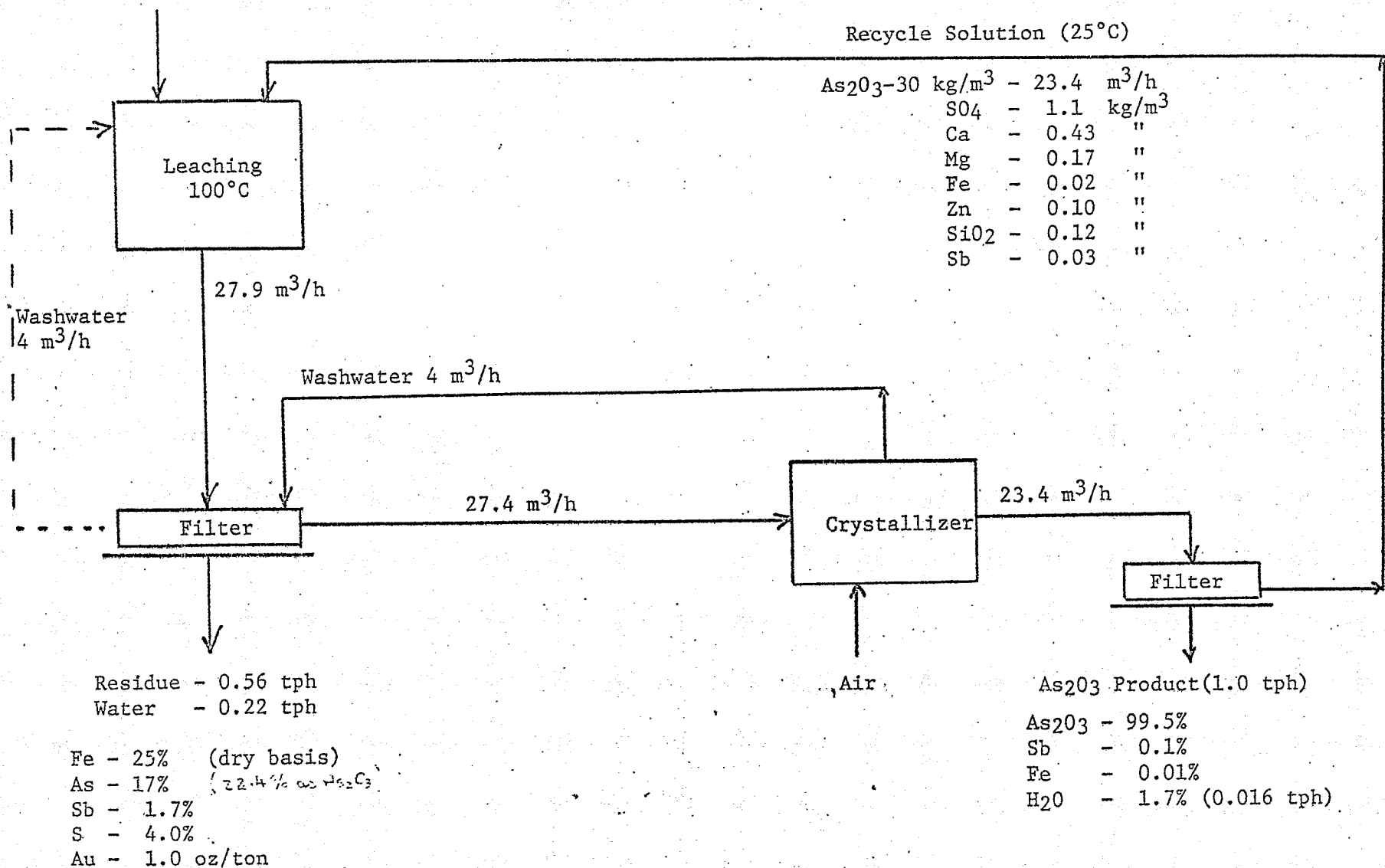


TABLE I: Analysis of Arsenic-bearing Dusts

	Lab #	As ₂ O ₃ (Total)	As ₂ O ₃ (Sol.)	Fe	Sb	Au*	SO ₄ ^x
Con Tailings Pond	78-346	70.7	64	10.6	0.82	0.37	2.0
Giant Underground Storage	78-476	66.7	60	--	2.5	1.3	

* Au in oz/ton, other results expressed as percent

x SO₄ on Con Sample 0-3' = 2.15% , 12 1/2' sample 1.94%
LEACHING

The feed is leached in recycle solution at the boiling temperature (approximately 100°C) for a period of one hour. Testwork carried out at both 85°C and 95°C has shown that the solution has reached saturation after a 0.5 h leaching period. The solubility at various temperatures was found experimentally to be as follows:

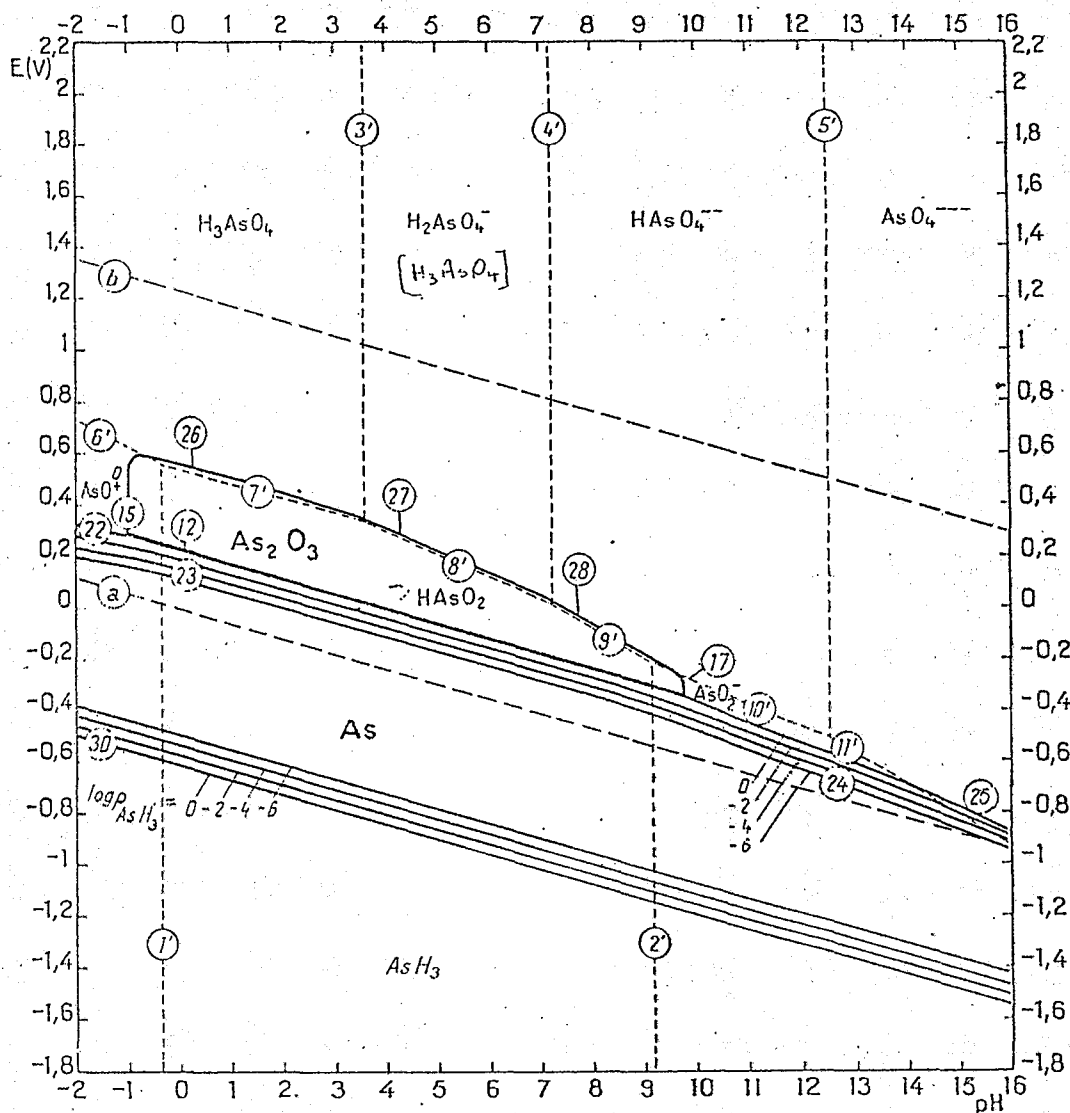
Temp.	As ₂ O ₃ (g/L)	
85°C	53	185°F
95°C	61	203°F
100°C	70	212°F

There is a considerable amount of conflicting information regarding the solubility of As₂O₃. The Handbook of Chemistry and Physics(4) lists solubility values for amorphous As₂O₃ and cubic crystalline As₂O₃ as 101.4 and 114.6 g/L, respectively. Mellor(5) lists solubility values for the cubic crystalline form ranging from 60 to 114 g/L. Values between 70 and 80 g/L have been obtained in the treatment of flue dusts from roasting operations(6,7).

The leaching operation solubilizes the free As₂O₃ in the feed while the arsenates of iron, calcium, etc. report to the residue. The aqueous chemistry of arsenic is described by the Eh-pH⁺ diagram shown in Figure 2. Water leaching of the Con material results in a solution having a pH⁺ of 3.0 ± 0.5 at a potential of about +160 mV (SCE) or +402 mV (vs hydrogen electrode). These conditions correspond to the stability region for arsenious acid (HAsO₂) in Figure 2. The actual species in solution has been shown to be As(OH)₃(8). Tests have been carried out under various pH⁺ and redox conditions to assess influences on As₂O₃ solubility and impurity dissolution. Reducing (hydrazine hydrate) or mild oxidizing conditions (air sparging) have no significant effect on the As₂O₃ solubility due to the fact that the oxidation of arsenious acid [As(OH)₃] to arsenic acid [H₃AsO₄] or vice versa is irreversible requiring very strong oxidizing or reducing conditions. The solubility of arsenious acid is not affected by pH⁺ over the range 1.0 to 8.5. Acid solutions (pH⁺ less than 2.0) are to be avoided due to the increased solubility of the various iron/calcium/magnesium arsenates and hydroxides. In all leaching tests

*new comment
pH 4.5 - 5.0
max. acidity
for leaching*

FIGURE 2: Potential-pH Equilibrium Diagram for the System Arsenic-Water at 25°C



Reference: M. Pourbaix, "Atlas of Electrochemical Equilibria in Aqueous Solutions", Pergamon Press, 1966.

carried out without the addition of reagents to control pH^+ , the solution has been well above $\text{pH}^+ = 2.0$ and iron values in solution have been low.

Antimony is of particular interest to product purity. The solubility of Sb_2O_3 is given as 0.1 g/L at 100°C and 0.0088 at 25°C . Thus it has been found that greater than 90% of the antimony remains in the residue. The small amount which dissolves at 100°C precipitates rapidly as the solution cools. The result is that the initial crystallized product of As_2O_3 is high in Sb_2O_3 . The average antimony values of the crystallized product has been 0.1% Sb.

The leaching tank should be closed with an attached condensor to prevent excessive water loss. Only mild agitation, (stirring) is required to maintain the finely divided solids in suspension.

Both single-stage and two-stage leaching tests have been carried out on the feed materials. A lower residual arsenic value in the residue is obtained in a two-stage leach with all free As_2O_3 being removed. The residue contains approximately 17% As as a ferric arsenate, arsenopyrite, etc. The double L/S separation required in the two-stage leach makes this approach less attractive if a small amount of free As_2O_3 can be tolerated in the residue and if the recycle and feed materials can be accurately proportioned to the leach tank.

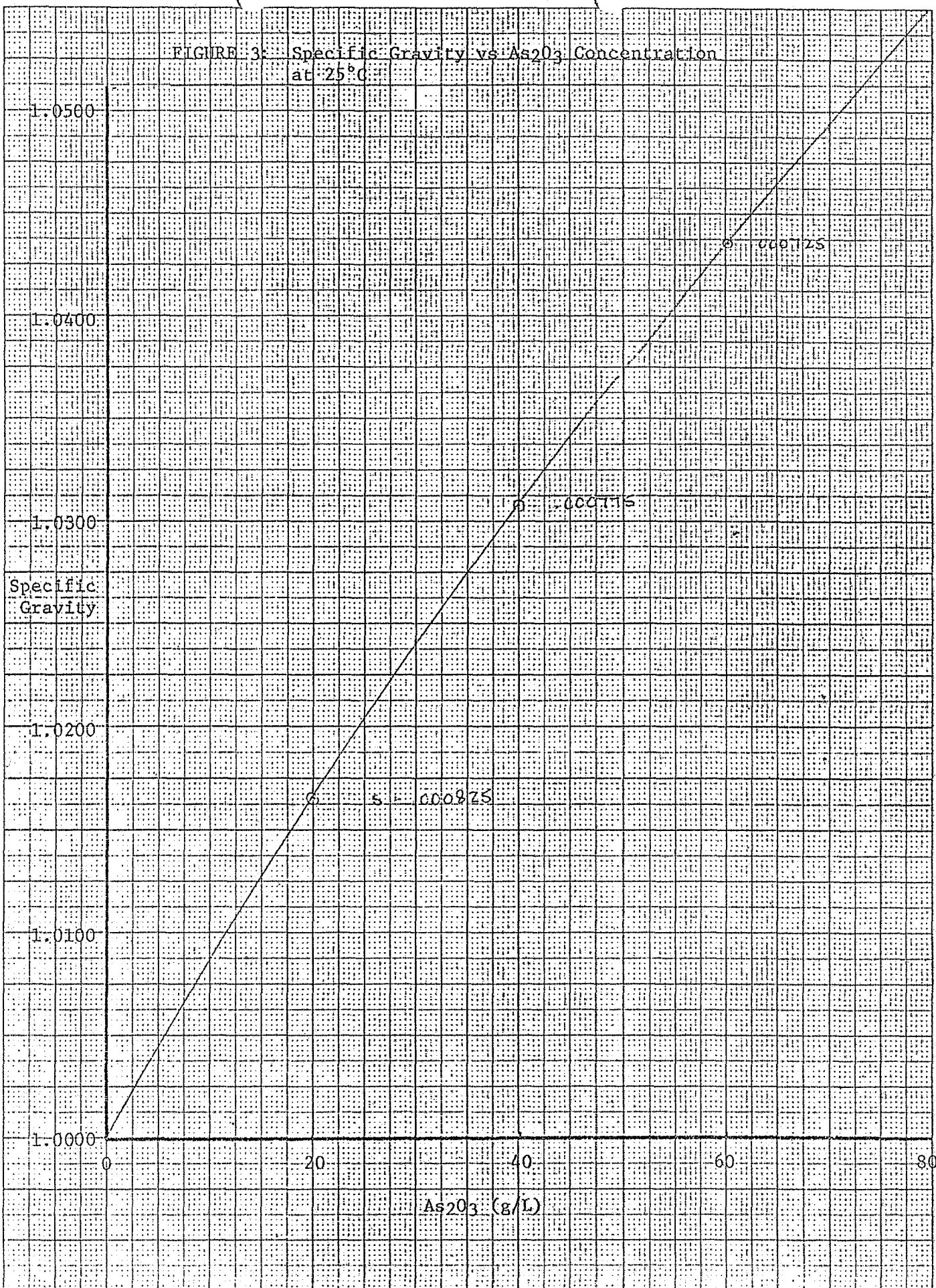
The concentration of various aqueous solutions of As_2O_3 may be determined by measuring the density of the solution. A calibration curve has been prepared covering the range 0-80 g/L As_2O_3 (Figure 3). The aqueous solution is transferred to a tared 100 mL volumetric flask and cooled to 25°C in a water bath. Solution is removed to bring the volume to 100 mL and the contents weighed on an analytical balance. The density ($\text{wt.} \div 100$) is then used to calculate concentration from Figure 3. If the cooling is rapid, little or no crystallization occurs. Even when moderate amounts of As_2O_3 have precipitated, no significant volume change results. This calibration curve has been compared with chemical analysis and found to be accurate within 5% of the amount present and provides a rapid analytical technique for process control.

LIQUID-SOLID SEPARATION

All L/S separations have been carried out on a Buchner funnel with mild suction using a glass filter (Whatman GF/A). It has been found that As_2O_3 crystallizes slowly from clarified solutions but in the presence of slurry containing free As_2O_3 , the precipitation from solution is rapid, following the normal temperature solubility relationship. Thus, the solution must be kept hot (greater than 95°C) during the filtration.

The residue had the following wet screen analysis:

461510

K&E 10 X 10 TO THE CENTIMETER 18 X .75 CM
KEUFFEL & ESSER CO. MADE IN U.S.A.FIGURE 3: Specific Gravity vs As_2O_3 Concentration
at 25°C 

Tyler Mesh Size	Percent
+100	2.9
-100	3.2
-200	1.6
-325	92.3

The wet filter cake had a bulk density of 1.7 g/cc at the 30% moisture level. Filtration rate studies were not carried out and this is suggested as further investigation.

It was found that under the conditions described, a small amount of fine iron hydroxide/arsenate passed through the filter and partially occluded the As_2O_3 crystals. Treating the pregnant solution on a fine polishing filter (Whatman #42) produced a colourless solution and resulted in a low iron (0.01%) white crystalline As_2O_3 product. .

The filtercake is washed with hot recycle solution and/or hot water on the filter to remove pregnant solution as well as some of the remaining free As_2O_3 . Hot water washing of the residue has resulted in wash solutions containing 25-40 g/L As_2O_3 . This wash solution may be discharged to the crystallizer provided the volume is relatively low. Otherwise, the solution should be recycled to the leach.

CRYSTALLIZATION OF As_2O_3

The crystallization of As_2O_3 occurs as the solution is cooled, eventually reaching an equilibrium value of 21 g/L As_2O_3 at 25°C. The precipitation rate is determined by the rate of cooling of solution and also the following which determine the rate at which equilibrium is attained:

- 1) liquid-crystalline As_2O_3 ratio
- 2) solution agitation (stirring)

It has been found that seeding the pregnant leach solution causes more rapid crystallization and a closer approximation to the equilibrium solubility line. Mild agitation also brings fresh solution in contact with As_2O_3 crystals.

Using recycle solution in the leaching stage, an 18 hour crystallization period was necessary to reach 28 g/L As_2O_3 . The solution was only subjected to room air temperatures during the cooling-crystallization process. In larger scale operation, the cooling rate can be increased by evaporation of water from the crystallizer. In stirred open vessels allowed to cool from 98°C to room temperature, a 15% water loss was realized. Further testwork should be carried out either reducing the pressure above the crystallizer or sparging air

through the solution. Water vapour from the crystallizer can be condensed and used to control the water balance in the process. When the solution has cooled to 50-60°C, a cooling coil using minewater (5°C) may be used to bring the solution temperature below 25°C.

In all cases, a crystalline As_2O_3 product was obtained, the actual crystal size depending on the conditions under which the crystallization was carried out. The crystals were easily removed from clean, unetched glassware and teflon stirring impellers. On etched glassware, the crystals were difficult to remove. It is suggested that future work be carried out using crystallizer tanks having an organic material as lining (PVC, polypropylene, etc.).

It was found during the course of investigation that As_2O_3 crystallization was very poor from solutions which had been produced from a direct water leach rather than from a leach using recycle solution. The reason for this is not known and does not appear to be related to the redox or pH^+ of the solution. This same phenomenon has been experienced by others(7). The small amount of As_2O_3 which does precipitate contains essentially all of the antimony which dissolved and samples containing up to 4.2% Sb have been obtained. In normal operation using recycle solution as leachant, no crystallization problems were encountered. Low temperature air drying is required to remove the surface water from the crystals. The product was 99.5% As_2O_3 with Sb and Fe averaging 0.1% and 0.01%, respectively. The product was white meeting A.E. and C.I. specifications.

The combined average chemical and spectrochemical (qualitative) analysis of six product samples is as follows:

<u>Element</u>	<u>%</u>
As_2O_3	99.5
Sb	0.1
Fe	0.01*
Zn	0.003-0.03
Si	0.001-0.01
Pb	0.001-0.01
Mg	0.0003-0.003
Ca	0.0003-0.003

* crystallized product from polishing filter.

Recycle solutions from the crystallizer contain 28-30 g/L As_2O_3 and various concentrations of impurities. A complete study on recycle levels and build-up has not been carried out but the indications with many elements Fe, Zn, Ca, etc., is that their concentrations in the aqueous solution are controlled by the solubility products of their hydroxides or arsenates. Thus, from preliminary indications, it will not be necessary to treat this solution for impurity removal prior to

recycling to the leach. Further testwork and confirmation of this is required in continuous operation.

RECOVERY OF GOLD FROM As_2O_3 LEACH RESIDUES

Gold is contained in various minerals associated with the water insoluble leach residue. A large-scale leaching test produced 320 g of leach residue which was sent to Lakefield Research for direct cyanidization leach tests and for roast cyanidization leach tests. The results show that 59% of the gold is extracted from this residue which analyzed 1.2 oz/ton gold. Roasting prior to cyanide leaching greatly improved the gold recovery(9). Additional roast-leach work is required on these residues.

WRH/bc

W. R. Hatch

W.R. Hatch

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- (2) Purification of Arsenious Oxide from Giant Yellowknife, W.R. Hatch, 201-781019.
- (3) Phases Present in a Sample of Tailings from the Con Mine, J.E.Muir, 201-781124.
- (4) Chemical Rubber Company, 52nd Edition, 1971-72.
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- (6) Improvements In or Relating To the Treatment of Arsenious and/or Sulpho-Arsenious Ores, Br. Pat. 795,793.
- (7) The Hot Water Leaching Process for Refining Arsenious Oxide in Flue Dusts from the Fairview Mine, R.W. Whiate, NIM Report No. 333.
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APPENDIX A

Experimental Testwork

The samples of Con Tailings material taken at various depths were sampled, dried and submitted for qualitative spectrographic analysis to determine whether there was a significant difference in major and minor element distribution. The five samples submitted from one test hole showed all elements within the same concentration range as shown in Table I. Moisture contents ranged from 26% to 30%.

TABLE I: Analysis of Con Tailings Pond Drill Samples
A-6008 - A-6012 (L#78-346)

Element	Concentration Range (%)
As	10-100
Fe	3-30
Zn, Si	1-10
Sb, Mg, Ca, Al	0.1-1.0
Pb, Ti	0.03-0.3
Au, Cu, B	0.003-0.03
Ni, Co	0.001-0.01
Ag, Mn	0.0003-0.003

The Giant underground storage material was sampled and found to contain 15% moisture. The analysis of Con and Giant samples used for experimental work was as follows:

	Lab #	As ₂ O ₃ (Total)	As ₂ O ₃ (Soluble)	Fe	Sb	Au*
Con Tailings Pond	78-346	70.7	64	10.6	0.82	0.37
Giant Underground Storage	78-476	66.7	60	--	2.5	1.3

* Au in oz/ton, other results expressed as percent.

Water Solubility of Various Components of the Feed Samples

Initial leaching testwork was carried out on these samples to determine the amount of water-soluble As₂O₃ present using an excess of water. Ten grams of dry material was leached in 100 mL of boiling water for one hour and the solution was then filtered hot. The residue was dried, weighed and the overall solubility determined. It was found that the Con material contained 64% and the Giant underground material 60% water soluble As₂O₃.

A larger test was carried out on Con material using an excess of water. A 35 g sample (dry basis) was leached in 800 mL of water at 100°C for 2 hours. The residue was filtered, washed with 200 mL of hot water, dried and weighed. The results were as shown in Table II.

(ii)

TABLE II: Solubility of Arsenic and Antimony in Con Material

	Wt. (g)	As ₂ O ₃	Sb	Wt. As ₂ O ₃ (g)	Wt. Sb (g)
Head	35.0	64% (sol.)	0.82%	-22.4 (sol.)	0.287
Residue	14.8	21.7% (tot.)	1.70%	3.2116	0.252
Solution	1000 mL	19.9 g/L	0.024 g/L	19.9	0.024
				<u>23.1116</u>	<u>0.276</u>

58-837
[Handwritten signature]

The results of this test confirmed the solubility of the As₂O₃ in that X-ray diffraction was unable to detect arsenolite (As₂O₃) in the residue. The major phase in the residue was ferric arsenate (FeAsO₄·2H₂O) with minor amounts of quartz, pyrite, arsenopyrite and magnetite. Only a small amount of antimony dissolved with 90% remaining in the residue.

Effect of Leaching Conditions (Redox - pH⁺) on As₂O₃ Dissolution

Leaching tests were carried out on Con material under various conditions, adjusting the pH⁺ and lowering the redox of the solution to determine whether greater solubility could be achieved by solution adjustment. Tests were carried out on 94 g of sample (dry basis) and 800 mL of solution, leaching at 95°C for 1 hour. Residues were washed with 100 mL of hot water to give a total filtrate volume of 900 mL. The conditions and results of these tests are given in Table III.

TABLE III: Solubility of As₂O₃ Under Various Leaching Conditions

Test	Solution	pH ⁺	Redox (mV SCE)	Residue		Solution As ₂ O ₃ (g/L)*
				Wt. (g)	As ₂ O ₃ % (Total)	
1	Water	2.4	+300	29.0	28.6	72
2	Water-Hydrazine	3.0	+150	33.2	43.4	67
3	Water-NaOH	5.4	+150	33.3	41.5	67

* calculated

All arsenic in the residue is expressed as As₂O₃. Thus, in test #1, the residue contains 2.6 g As₂O₃ and 26.5 g of residue containing arsenic in various forms equivalent to 17% As (21.7% expressed as As₂O₃).

The results of these tests indicate that there is no improvement in leaching under reducing conditions or at higher pH⁺. This is in agreement with literature references which indicate the stability of various arsenates under mild reducing conditions. The arsenates which have formed (presumably during roasting), are associated with iron, calcium, etc. and are inert to these leaching conditions. Only the free As₂O₃ is solubilized. ?

(iii)

Rate of Dissolution and Solubility of As₂O₃ at Various Temperatures

Tests were carried out to determine the solubility and rate of dissolution of As₂O₃ from Con material. An excess of feed (150 g) was added to 800 mL of hot water in a stirred reaction kettle. Approximately 10 minutes were required for the slurry to reach the desired temperature. Samples were withdrawn at various time intervals and rapidly filtered for solution analysis.

The results of these tests carried out at 85°C and 95°C were as follows:

<u>Dissolution Time (h)</u>	<u>Solution As₂O₃ (g/L)</u>	
	<u>85°C</u>	<u>95°C</u>
0.5	47	58
1.0	52	57
2.0	49	61
7.0	53	60

The final solutions at 85°C and 95°C had pH⁺-redox values of 2.05, +325 mV and 2.05, +295 mV respectively. The results of these tests show that dissolution of As₂O₃ is rapid with greater than 90% of the maximum solubility being attained in a 0.5 hour leaching period. The final solubilities of 53 g/L (85°C) and 60 g/L (95°C) are probably low since some cooling of the slurry occurred during sampling.

An additional leaching test was carried out on Con material using a recycle solution from a previous leach which analyzed as follows: (g/L)

<u>Sample</u>	<u>As₂O₃</u>	<u>Fe</u>	<u>Ca</u>	<u>Mg</u>	<u>Sb</u>
A-6190	42.2	0.008	0.047	0.027	0.027

The sample (75 g dry basis) was leached in 800 mL of solution at 100°C for 2 hours. The slurry was filtered and washed with 100 mL water at 98°C. The filtrate analyzed as follows: (g/L)

<u>Sample</u>	<u>As₂O₃</u>	<u>Fe</u>	<u>Ca</u>	<u>Mg</u>	<u>Sb</u>
A-6191	68.0	0.027	0.068	0.032	0.028

The filtrate had a yellow colour. It was later confirmed that this was caused by fine iron hydroxide-arsenate which passed through the filter.

The overall balance for total arsenic (as As₂O₃) was as shown in Table IV.

(iv)

TABLE IV: Materials Balance for Recycle Leach

Sample	Wt. (g)	%	Vol. (mL)	As ₂ O ₃ (g/L)	As ₂ O ₃ (g)
Feed, 78-346 <i>2nd</i>	75	71	--	--	53
Recycle Solution, A-6190	--	--	800	42.2	34
					87
Leach Solution, A-6191	--	--	770	68.0	52
Residue, A-6193	48	56	--	--	27
Wash Sol'n., A-6192	--	--	91	44.6	4
					83

Leaching - Crystallization Tests

A number of tests were carried out using various leaching conditions followed by crystallization of the As₂O₃ from solution. Both Con and Giant underground storage material were evaluated in this process. The four tests are described and the conditions summarized in Table V.

Test #1 was carried out on Con material (78-346) using recycle solution, investigating the effect of slightly higher pH⁺ (lime addition) and the precipitation of As₂O₃ at 20°C. The solution was seeded with a small amount of fine As₂O₃ prior to crystallization. A total of 4.4 g Ca(OH)₂ was added to the solution to bring the pH⁺ to 6.8. The final residue was washed with 100 mL of hot water and retained separately. The pregnant solution was cooled in a water bath to 20°C and crystallized 20 hours.

Test #2 was an aqueous leach carried out on 78-346 material without the addition of reagents. Crystals of As₂O₃ (16.5 g) were added to the hot pregnant solution and the product was crystallized over a 20 h period with constant stirring. The solution was allowed to cool to ambient temperature without forced cooling.

The residue (washed) was releached in water to produce a clean (As₂O₃-free) residue.

Test #3 was an aqueous leach carried out on 78-346 material using a large excess of feed and carrying out repeated crystallization and releaching with aqueous solutions under various conditions. The leaching was carried out in a 4.0 L reaction kettle with stirring.

In the initial leach (3-1), water was used and 5 g of granular, activated carbon were added to determine its effect on the redox and on antimony dissolution. The wet residue from 3-1 was releached with recycle solution making the volume to 3.5 L with water. The crystallization was repeated. The procedure was again repeated in leach 3-3. In Test #3-4,

the wet cake was again releached with recycle + make-up water. The pH⁺ of the slurry was lowered to 2.8 by the addition of 10 g H₂SO₄. The As₂O₃ was crystallized from the leach solution as in other tests. In test #3-5, the wet residue was releached with distilled water as in 3-1 and a product As₂O₃ was crystallized as in other tests, allowing the solution to reach room temperature overnight (20 h). No seeding or stirring was employed. The filtercake was washed with 1 L hot water and retained separately. The cake moisture was determined.

In test #3-6, the dry residue was releached with 3-4 recycle solution, the residue separated and washed with 1 L hot water. The residue was very low in free As₂O₃ at this point, but since the material was to be submitted to Lakefield Research for leaching tests, a final recycle leach (3-7) was carried out to ensure a clean residue. Test #4 was carried out on Giant material 78-476 leaching with recycle solution and crystallizing the As₂O₃ overnight without seeding or stirring.

The results of the tests are given in Table V. It is evident from the weight of product obtained that the crystallization from water leaching is very poor compared to solutions that have been prepared by recycle leaching. This is particularly evident from tests 3-1 and 3-4 where a small amount of product was obtained and the crystallizer liquor was relatively high in As₂O₃, indicating a reluctance to crystallize. No explanation appears obvious to the author, but the same phenomenon was observed in similar studies⁽⁷⁾.

Leach solutions containing approximately 70 g/L As₂O₃ were obtained in these tests and there appeared to be little difference in As₂O₃ solubility or crystallization of As₂O₃ under the various conditions. Continuous stirring (test 2) produced a 29 g/L crystallizer liquor, however in test 3-3 and 3-4, As₂O₃ values of 28 g/L and 26 g/L were obtained at 22-24°C.

Antimony crystallized well from solution whether or not there is efficient crystallization of the As₂O₃. Thus, in test 3-5 (water leach), only 2.0 g of product was obtained but this contained a very high concentration of antimony (4.2%).

A solution loss of approximately 13% caused by evaporation was realized in tests using open crystallization vessels (#4) and even higher water losses in stirred open vessels (16% in #2). This can be used to obtain a water balance in the system condensing the water vapour for use in washing or for discharge.

The As₂O₃ products were of satisfactory purity with the exception of the small amount of material obtained from 3-5.

The As₂O₃ balances for three tests are given in Table VI expressing all forms of arsenic as As₂O₃. Test #3 involved numerous samples which were not analyzed, making overall balances impossible.

TABLE V: Leaching - Crystallization Tests on Con and Giant Materials

Test No.	Feed		Leach Solution			Leaching Conditions				Pregnant Solution		Crystallizer Liquor		As ₂ O ₃ Product				Leach Residue		
	No.	Wt. (g)	Type	Vol. (L)	As ₂ O ₃ (g/L)	Time (h)	Temp. (°C)	Redox (mV)	pH ⁺	Vol. (L)	As ₂ O ₃ (g/L)	Vol. (L)	As ₂ O ₃ (g/L)	Wt. (g)	As ₂ O ₃ (%)	Sb (%)	Fe (%)	Wt. (g)	As ₂ O ₃ (%)	Sb (%)
1	78-346	70	Recycle	0.80	39.7	2	100	--	6.8	0.74	71.6	--	--	29.0	--	--	--	36	46.8	--
2	78-346	88	H ₂ O	0.80	--	2	100	250	3.0	0.67	70.0	0.565	29	31.5	99.5	0.026	0.01	43	39.9	--
3-1	78-346	1000	H ₂ O	3.5	--	3	100	+100	3.6	3.2	--	3.2	54	18.0	99.5	0.44	0.024	--	--	--
3-2	3-1 Res.	--	Recycle	3.5	--	3	100	+15	3.6	3.5	--	3.5	36	69	99.5	0.10	0.019	--	--	--
3-3	3-2 Res.	--	Recycle	3.5	--	3	100	+40	4.0	3.5	--	3.2	28	75	99.5	0.04	0.022	--	--	--
3-4	3-3 Res.	--	Recycle	3.5	--	3	100	+185	2.8	3.5	--	3.5	26	91	99.5	0.06	0.014	--	--	--
3-5	3-4 Res.	--	H ₂ O	3.5	--	3	100	+110	3.8	3.5	--	3.5	35	2	94.0	4.2	0.66	455	--	--
3-6	3-5 Res.	455	Recycle	3.5	--	3	100	+125	2.8	3.0	--	3.0	--	30	99.5	0.34	0.012	386	--	--
3-7	3-6 Res.	376	Recycle	3.5	--	2	100	+105	3.2	3.5	42	3.3	35	25	99.5	0.65	0.014	320	--	--
4	78-476	180	Recycle	3.0	34	2	100	+5	4.0	3.0	70	2.6	39	100	99.5	0.12	0.044	88	36.3	5.4

TABLE VI: As₂O₃ Balances for Leaching-Crystallization Tests

Test No.	Sample	Solids		Solution		Feed	Product
		Wt. (g)	As ₂ O ₃ (%)	Vol. (L)	As ₂ O ₃ (g/L)	As ₂ O ₃ (g)	As ₂ O ₃ (g)
1	Feed 78-346	70.0	70.1	--	--	49.0	--
	Leach Solution	--	--	0.8	39.7	31.7	--
	Leach Residue	36.0	46.8	--	--	--	16.8
	Preg. Solution	--	--	0.75	71.6	--	53.7
	Wash Solution	--	--	0.1	26.5	--	2.7
						80.7	73.2
2	Feed 78-346	88.0	70.1	--	--	61.6	--
	As ₂ O ₃ (Seed)	16.5	99.5	--	--	16.4	--
	Leach Residue	43	39.9	--	--	--	17.1
	As ₂ O ₃ Crystals	30.5	99.5	--	--	--	30.3
	Crystal Liquor	--	--	--	--	--	22.0
	Wash	--	--	--	--	--	4.6
						78.0	74.0
4	Feed 78-476	180	66.7	--	--	120.1	--
	Leach Solution	--	--	3.0	34.0	102.0	--
	As ₂ O ₃ Crystals	100	99.5	--	--	--	99.5
	Leach Residue	88	36.3	--	--	--	31.6
	Crystal Liquor	--	--	2.6	39.0	--	101.4
						222.	232.5

Conclusions and Recommendations

The data derived from this testwork has been used to outline a preliminary flowsheet for the purification of As₂O₃ from Con and Giant roaster dusts. Areas requiring further testwork are suggested in the process description. Providing preliminary cost estimates are favourable it is recommended that the process be piloted on a semi-continuous basis to define filtration rates, solution recycle impurity concentrations and to optimize crystallization of As₂O₃. Larger tests are also required to produce residues which can be used to evaluate Stage II. - the recovery of gold from the leach residue.

APPENDIX B

Literature Survey

A literature search of Chemical Abstracts was carried out for the years 1930 to 1977 on Arsenious Oxide - As_2O_3 - Production - Purification. The hot water leach process is known and has been practised by Beattie Mines (Quebec) and Deloro Mines (Ontario)*. Little documentation of the process can be found in the literature with the exception of the research study done by National Institute for Metallurgy(7). This report, issued in 1968 states that from their literature search, "it does not appear that any practical application of the solubility of arsenic trioxide in water has been used to refine this material". This NIM report is the most useful piece of published information turned up in our search. An abstract of their work is included in this survey along with abstracts from patents and Chemical Abstracts relating to the subject area.

A list of abstracts follows.

- (1) National Institute for Metallurgy. Research Report No. 333, 1968.
The Hot-Water Leaching Process for Refining Arsenious Oxide in
Flue Dusts from the Fairview Mine
-

Laboratory testwork was carried out at the National Institute for Metallurgy to investigate the technical feasibility of producing refined arsenious oxide from the arsenical flue dust from the Fairview Mine by hot-water leaching of the dust and the subsequent recovery of the refined arsenious oxide from the cool pregnant solution by crystallization.

The results of this investigation show that arsenious oxide of sufficient purity to meet the specification can be produced if the concentration of impurities in the leach liquors does not exceed certain values. The effect of the concentration of impurities beyond these levels is not known. It was also found that a double leach, that is, leaching of the dust followed by leaching of the residue from this leaching, carried out at the boiling point, produced recoveries of refined arsenious oxide adequate for the process to be technically viable.

If an economic assessment of the process should prove satisfactory, the process should be studied in pilot plants.

- (2) British Patent - 795,793 (1958)
Improvements In or Relating To the Treatment of Arsenious and/or
Sulpho-Arsenious Ores
-

It is an object of the present invention to provide an improved process for the treatment of arsenical and/or sulpho-arsenical

* private communication - F.R. Archibald and H.T. Evans

(viii)

ores with a mixture of sulphuric and nitric acids. On completion of the acid treatment, arsenious oxide is allowed to crystallize in the presence of the suspension, the crystallized arsenious oxide and any insoluble residue from the acid treatment of the ore is removed, the combined arsenious oxide and insoluble residue is washed with cold water to remove soluble metal compounds retained thereon, the arsenious oxide is removed from the insoluble residue by aqueous extraction in the hot and then crystallized and separated from the resulting solution to yield pure (99.5 to 99.8%) arsenious oxide.

(3) CA 78:86597v

86597v Feasibility of producing technical arsenic trioxide from arsenic-containing tin industry dusts. Terebenin, A. N.; Bykov, A. P. (USSR). *Tsvet. Metal.* 1972, (7), 31-2 (Russ). In lab. expts. on As_2O_3 recovery from Sn smelter flue dust, heating the dust in a current of air for 30 min at 500-600° gave a condensate contg. 98.0-9.5% Al_2O_3 with condensation efficiency 97-9%. The residue contained As 7-10 and Sn 3-4%. Raising the temp. >600° did not affect the volatilization and made possible formation of As_2O_3 and nonvolatile compds. E. M. Elkin

(4) CA 60:3753f

Manufacture of high-purity arsenic. III. Behavior of trace amounts of P, Fe, Te, and Cu in the process of purification of arsenic trichloride. Yasuki Osumi, Masanori Nakane, and Yoshizo Miyake (Govt. Chem. Res. Ind. Inst. Osaka, Japan). *Kogyo Kagaku Zasshi* 66(4), 437-42(1963); cf. CA 57, 9456a; 58, 3117c. In order to obtain high-purity As, behaviors of trace amt. of P, Fe, Te, and Cu in the process of purification of As_2O_3 and $AsCl_3$ were investigated by using radioisotopes, ^{32}P , ^{59}Fe , and ^{125}Te while Cu was detd. by coloration method with bathocuproine. By the sublimation of As_2O_3 , Fe and Cu could be eliminated by >99%, while P and Te by only 30-50%. Washing of $AsCl_3$ with aq. HCl resulted in effective sepn. in all cases, though a little loss of $AsCl_3$ occurred as the concn. of aq. HCl decreased. In the hydrolysis of $AsCl_3$, 15-35% of copptn. occurred in the order, Fe > P > Te > Cu. By adsorption method, P was eliminated by >95% with activated Al_2O_3 . Aq. HCl soln. of $AsCl_3$ was distd. with a fractionating column packed with adsorbent such as activated charcoal, which eliminated P, Fe, Te, and Cu by >99.9%. Consequently, P, Fe, Te, and Cu contents could be reduced to the amt. of 1/10,000 of initial content together with Sb, Se, and S by distg. aq. HCl soln. of As_2O_3 purified by sublimation, by using a fractionating column packed with activated charcoal. Yukio Takigawa

(5) CA 54:19249

Method for the preparation of arsenic of high purity. N. A. Goryunova, L. V. Kradinova, V. I. Sokolova, and E. V. Sokolova (Phys.-Tech. Inst. Acad. Sci. U.S.S.R., Leningrad). *Zhur. Priklad. Khim.* 33, 1409-10(1960).— As_2O_3 (50 g.) free of Bi and contg. only small amts. of Sb, Cu, Al, Ca, Fe, Si, and Mn was boiled in 200 ml. 1:1 HCl, filtered, cooled 20-24 hrs., filtered, washed till neutral, and dried to yield practically spectrographically pure As_2O_3 contg. traces of Si. This As_2O_3 was mixed with $1/3$ its wt. of activated C, the mixt. was covered with a layer of C, and heated 4-5 hrs. The remaining As_2O_3 was distd. at 300° and about $2/3$ of the As at 450°. I. Bencowitz

(6) CA 89:8409y

89:8409y Wet-process recovery of arsenious oxide. Aoy Eiichi (Rasa Industries, Ltd.) Japan. Kokai 78 14,691 C01B27/00, 09 Feb 1978, Appl. 76/89,370, 27 Jul 1976; 3. Pure As_2O_3 is obtained safely and in high yields from ash, p. and sludges contg. As in the form of As_2O_3 , by extg. As_2O_3 hot water above atm. pressure, sepg. the insol. fraction by filtration, lowering the pressure, and sepg. the As_2O_3 which

(7) U.S. Pat. 3,923,478

Process and an Apparatus for Recovering Arsenic Trioxide from Gases, Assignee - Boliden.

The invention refers to a process and to an apparatus for recovering arsenic trioxide from a gas by washing out said oxide from the gas with a liquid having the ability of dissolving arsenic trioxide. The invention is characterized thereby that non-desired deposits in the apparatus formed in connection with saturated solution are removed by dissolution with an unsaturated solution of arsenic trioxide.

(8) CA 81:123757g

123757g Purification of arsenic trioxide and apparatus therefor. Iwai, Hideichiro; Omura, Koji (Mitsui Mining Smelting Co., Ltd.) Japan. Kokai 74 26,267 (Cl. 15 B71), Mar 1974, Appl. 72 70,415, 15 Jul 1972; 4 pp. As_2O_3 contg. Pb, Sb, Hg, Se, etc., is mixed with gypsum or silicates to pre-melting, heated in a stream of air to sep. from Pb and filtered at 150-250° into a bag-filter to collect As_2O_3 , and t. at a lower temp. into a 2nd bag filter to collect the impure As_2O_3 . Thus, 97.0% pure As_2O_3 contg. Pb 0.10, Sb 0.12, Hg 0.20, and 0.08% (2 kg) was mixed with 1 kg gypsum, introduced in drum at 500° by a screw-conveyer, heated for 1 hr, and vapor was swept by air to contain 120 g/m³ into the 1st bag f. of glass fiber at 150-250° and into the 2nd of Tetron fiber 60-100°. The yield in the 1st bag filter was 1850 g of >99% pure As_2O_3 contg. Pb 0.002, Sb 0.01, Hg 0.0005, and Se 0.0005 and in the 2nd bag filter the resp. values were 70 g, >85, 0.60, 0.16, and 0.40%, resp. K. Kodan

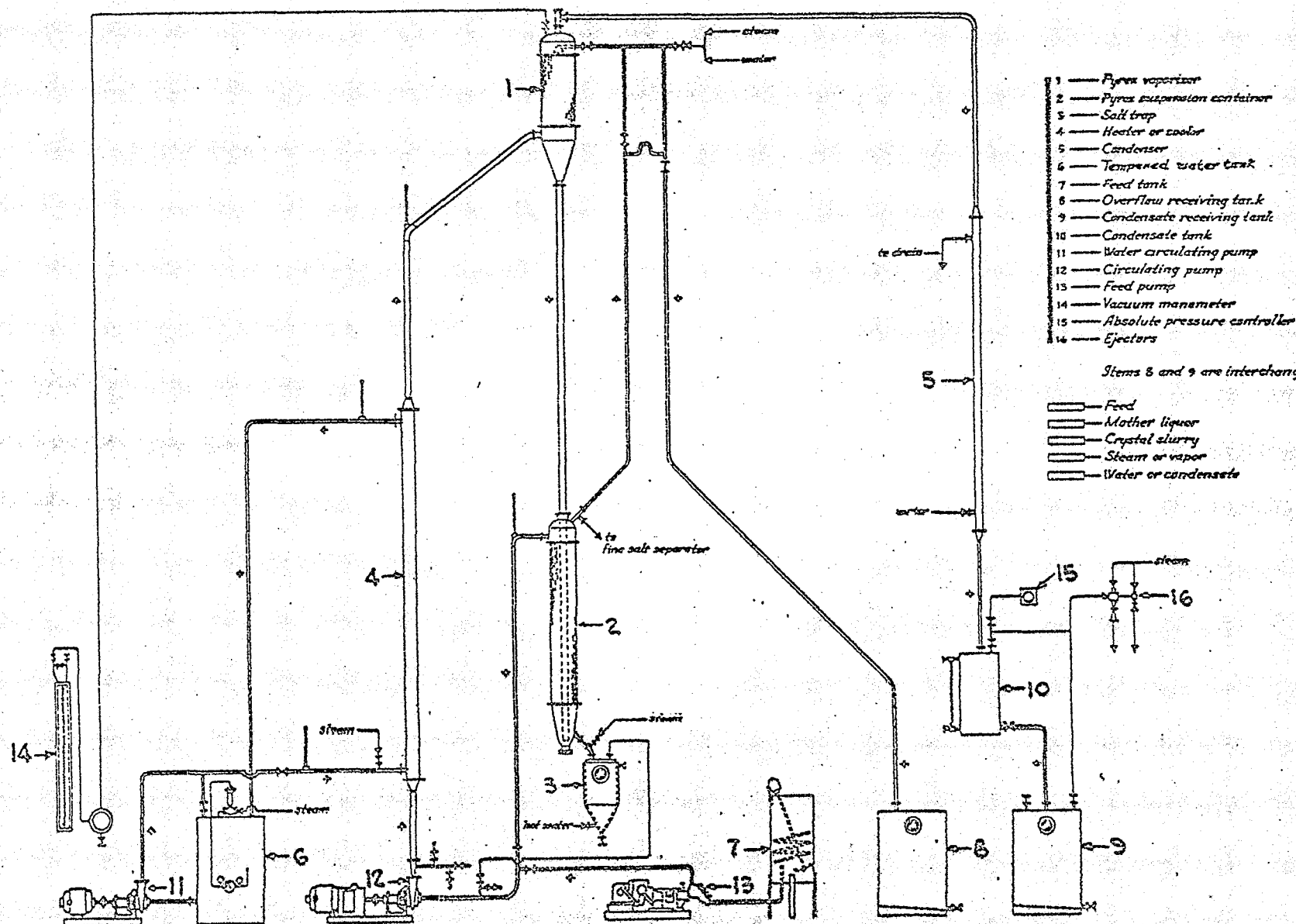
(9) CA 28:3844

Production of arsenic trioxide at Anaconda. Louis V. Bender and Harold H. Goe. *Trans. Am. Inst. Mining Met. Engrs.* 106, 324-8(1933).—The Anaconda As_2O_3 plant, and flow sheet are described. Alden H. Em

PURIFICATION AND RECOVERY OF

ARSENIOUS OXIDE FROM ROASTER DUSTS

APPENDIX "C"



STRUTHERS TECHNICAL SERVICES LABORATORY
 KRYSTAL PILOT PLANT CRYSTALLIZER

STRUTHERS SCIENTIFIC &
 INTERNATIONAL CORP.

KR-010

FALCONBRIDGE NICKEL MINES LIMITED

INTER OFFICE MEMORANDUM

MEMO TO: L.J. Connell

FROM: W.R. Hatch

DATE: June 14, 1979

SUBJECT: Evaluation of As₂O₃ Products from
Struthers Wells Crystallization Tests

KEYWORDS: Giant Yellowknife

COPIES TO: RAB, MC, PJR/LSP, FTP, File

PROJECT No. 201-0-790614

JO#2484

The following is a summary of the evaluation carried out on the samples of As₂O₃ which were produced in crystallization tests at Struthers Wells. The chemical and physical properties measured are given in the following table.

Sample No.	Date	%H ₂ O	Bulk Density (g/cc)		%As ₂ O ₃	%Sb ₂ O ₃	%Fe
			Wet	Dry			
79-353	5-31-79	5.5	1.85	1.95	99.5+	0.11	0.012
-354	6-1-79	4.5	1.78	2.03	99.5+	0.09	0.010
-355	6-2-79	6.8	1.76	2.05	99.3+	0.11	0.012

Dry Screen Analysis

Sample No.	Percentage			
	-48	-100	-200	-325
79-353	-	1.5	8.7	89.8
-354	0.7	3.8	8.9	86.6
-355	-	6.0	14.4	79.6

(Spectrographic qualitative analysis indicated only traces of Si, Mg and Ca as follows:

Si	0.003 - 0.03%
Mg	0.0003 - 0.003%
Ca	0.0001 - 0.001%

The products are of excellent quality as far as chemical composition is concerned. It is of interest to note the higher Sb_2O_3 level and one wonders whether this is due to a higher Sb_2O_3 content in the dust or to higher solubility in the leach. You may have some thoughts on this.

The material is fine and meets size specification. There appears to be a slight increase in particle size on successive days.

A sample has been sent to Jennike and Johanson for product handling evaluation. We froze samples containing 4.5 and 6.8% moisture and found that the crystals formed a fairly solid cake. There was a difference in hardness between the two moisture levels and further tests at 2-3% moisture may be warranted.

WRH/lbm

W. R. Hatch

W.R. Hatch

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