

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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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 As_2O_3 by Hot Water Leach Process

Feed 2.11 tph (wet)

As_2O_3 (soluble)	- 47.4%	1.0	tph
Gangue (insoluble)	- 26.7%	0.56	tph
Water	- 25.9%	0.55	tph
		2.11	tph

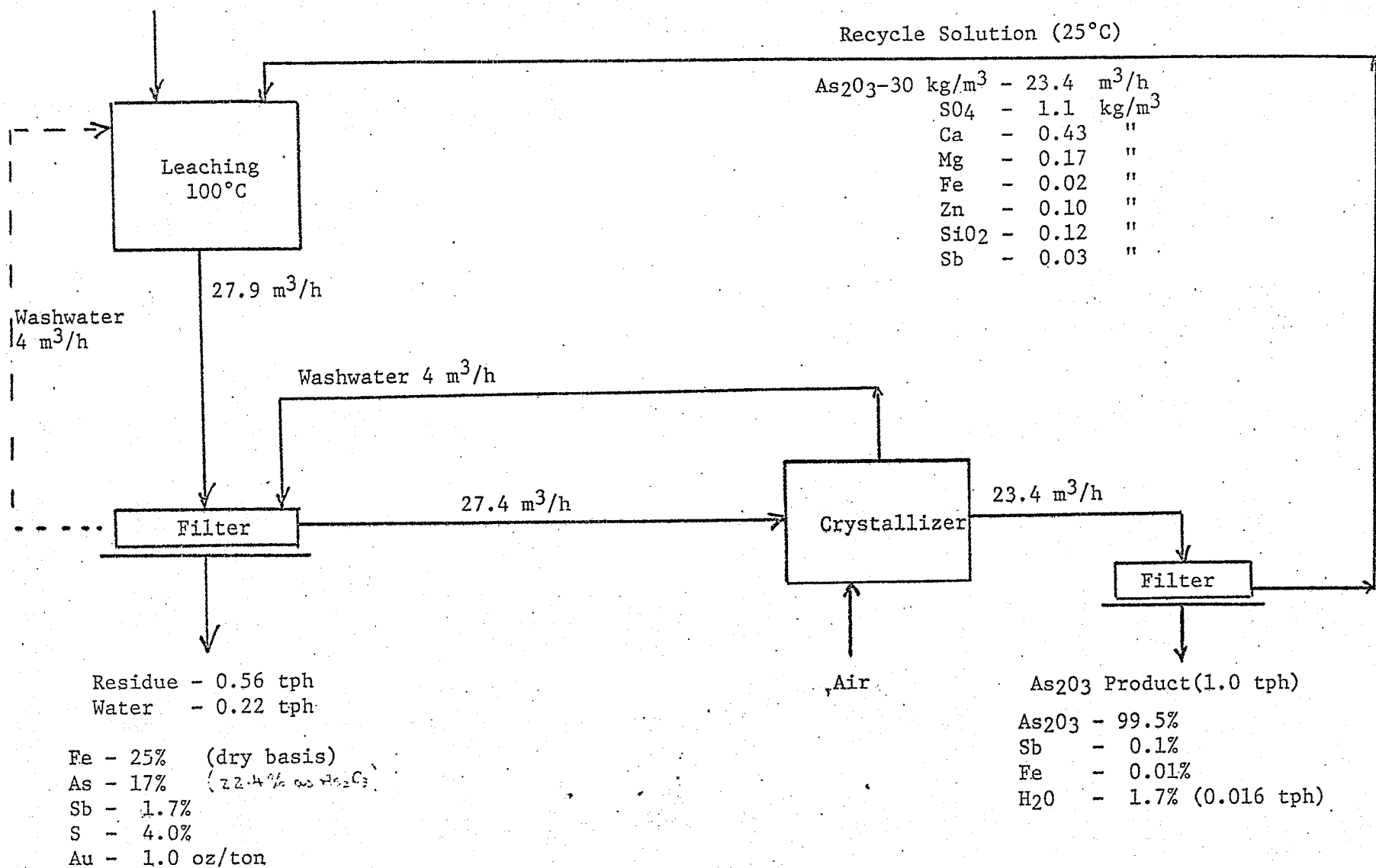


TABLE I: Analysis of Arsenic-bearing Dusts

	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:

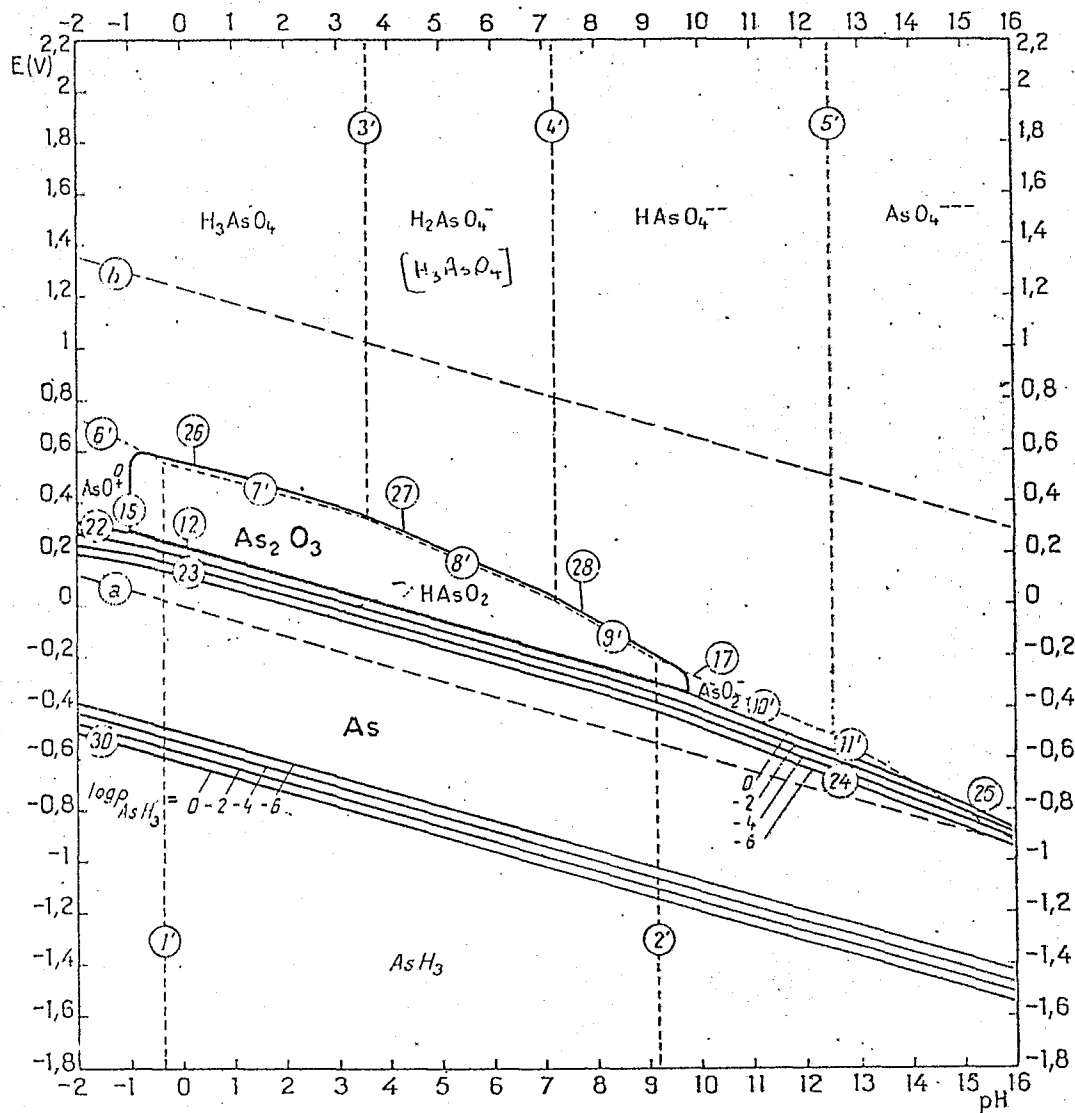
Temp.	As ₂ O ₃ (g/L)	
85°C	53	185°F
95°C	61	203°F
100°C	70	212°F

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 conclusion
pH 4.5 - 5.0
more accuracy
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.

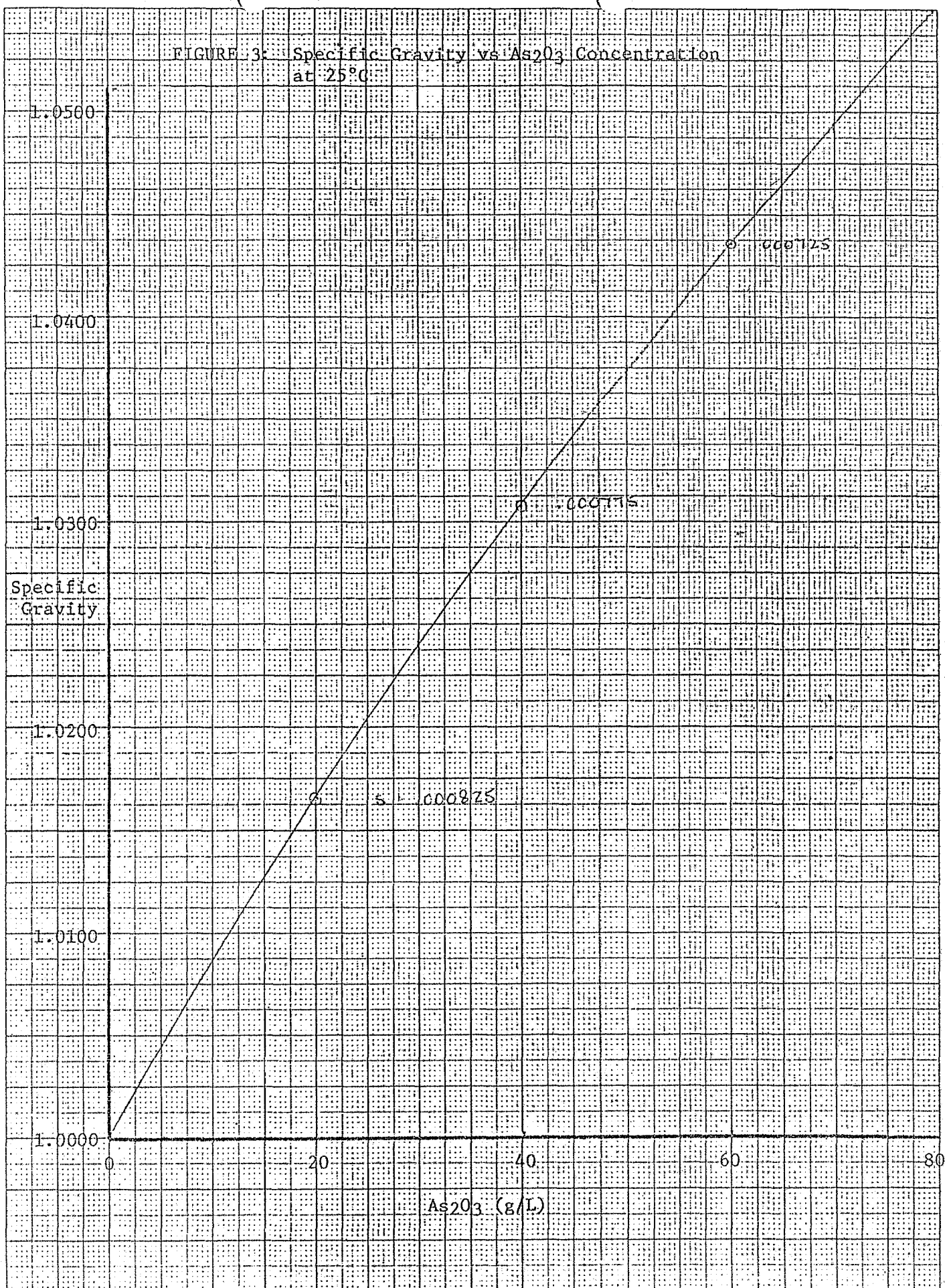
The concentration of various aqueous solutions of As_2O_3 may be determined by measuring the density of the solution. A calibration curve has been prepared covering the range 0-80 g/L As_2O_3 (Figure 3). The aqueous solution is transferred to a tared 100 mL volumetric flask and cooled to 25°C in a water bath. Solution is removed to bring the volume to 100 mL and the contents weighed on an analytical balance. The density (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

10 X 10 TO THE CENTIMETER 18 X 25 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.

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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>

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.

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>346</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,

(v)

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