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FALCONBRIDGE NICKEL MINES LIMITED  
INTER OFFICE MEMORANDUM

GEN. ENGR.

MAY 28 1980

MEMO TO: P.H. Lindon

FROM: R.E. Ranford/M. Sheviak

DATE: May 25, 1980

SUBJECT: Purification of Arsenious Oxide Bearing  
Dust from Giant Yellowknife by Fuming

KEYWORDS: (in title)

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PROJECT No. 201-0-800525  
JO#2682

INTRODUCTION

Fuming of  $As_2O_3$  bearing Giant Yellowknife baghouse dust has been proposed as an alternative to hydrometallurgical treatment for production of a high purity saleable product and concentration of gold in the residue.

Therefore, some testwork has been done to investigate the technical feasibility of the fuming approach. Since preliminary elutriation tests of dust from a fluidized sand bed were quite encouraging, i.e. the measured retention time of dust particles in the bed was in minutes rather than seconds, the experimental work was done feeding preheated nitrogen to a fluid bed reactor where the fuming of the arsenious oxide was carried out. The results of the first 4 preliminary tests are reported in this memo and a recommendation is made.

SUMMARY

1. The fuming of  $As_2O_3$  from Giant Yellowknife Baghouse Dust has been demonstrated in a fluid bed reactor with a sand bed, using preheated nitrogen at 400-800°C.

2. The product purity is quite good at +99%  $As_2O_3$  with antimony at 0.06% and iron at 0.009%. Some contamination of the product occurred due to the apparatus construction.

3. A hot filter consisting of a fiberglass cloth and a Fibrefrax paper felt was satisfactory in preventing the ash from passing into the product  $As_2O_3$ . The operating temperature was 250-300°C.

4. The elutriation tests conducted at room temperature with the original baghouse dust suggest that a fluid bed with a sand bed could be used to lengthen the particle retention time in the "hot fuming zone" and hence minimize the size of the reaction vessel. Knowledge of the Nikkelverk fluidized bed hydrolyzer for nickel chloride could therefore be conveniently used in designing a propane-fired "fuming reactor".

(SUMMARY, Cont'd.)

5. The largest problem in this work was feeding the dust to the fuming reactor, at a low rate, 1-10 g/min. The material agglomerates and sticks to itself and the equipment very readily and has a angle of repose of 90-120°. Therefore any fuming process must address this problem very seriously. Powder dispensers such as those designed by Irsid for lime powder injection into BOF's might be suitable for this process.

6. Some observations made in these tests suggest that the product vapour should not be quenched very severely. The largest crystals of  $As_2O_3$  occurred with the lowest degree of super-cooling of the gas.

7. Testwork should be carried on to develop some indication of the maximum rate of arsenic fuming with a given condition of gas flowrate and temperature in the fluid bed. This requires further work on a batch feeder for the dust.

MATERIALS AND PROCEDURES

The Giant Yellowknife baghouse dust has the particle size and chemical analysis shown in Table I. The particle size was determined at Indusmin Laboratory using the Sedigraph. The liquid used was a 50% water, 50% glycerol solution with 2 drops of Triton surfactant added.

TABLE I: Particle Size and Chemical Analysis of Giant Yellowknife Baghouse Dust (L80-225, A9214)

Size ( $\mu m$ )	-15 + 10	-10 + 7	-7 + 5	-5 + 4	-4 + 3	-3 + 2	-2 + 1	-1 + 0
Wt. %	3.5	8	15.5	15.5	21.5	15.5	7	13.5
Cum. %	3.5	11.5	27	42.5	64	79.5	86.5	100

Wt. %			
<u>As<sub>2</sub>O<sub>3</sub></u>	<u>Fe</u>	<u>SiO<sub>2</sub></u>	<u>Sb</u>
91.5	1.8	3.4	0.14

ELUTRIATION TESTS

The elutriation tests were carried out with a sized sand bed, -14 + 65 m, fluidized with nitrogen at room temperature in a 1" quartz tube. A fritted glass disc was used as a porous hearth plate and the distance from hearth to the transition to 2" diameter was 2.4". The nitrogen flow was measured with a rotameter type flowmeter and then passed through the sand bed. The gas was cleaned by passing it through a cold fabric filter consisting of a polyester fabric overlying a polypropylene felt clamped to a 1 gallon can. The area of the filter was 28 in<sup>2</sup>.

(ELUTRIATION TESTS, Cont'd.)

The tests were carried out by dropping the 100 g dust batchwise through the freeboard onto the bed over a short period, approx. 1 min. The drop length and gas offtake were about 15" above the hearth.

After a given length of time -5, 10, 20 or 40 minutes, the air was turned off and the filter and bed materials were weighed. The weight gain of the filter was checked against the weight loss of the dust from the bed.

## FUMING TESTS

Four fuming tests were carried out using an apparatus similar to that shown schematically in Figure 1. There were a number of modifications after each test and these are tabulated in Table II. In general, nitrogen was preheated indirectly to 600-700°C in a stainless steel heat exchanger and passed through a 1" stainless steel fluid bed reactor with the same dimensions as the quartz fluidizer described earlier. A perforated S.S. sheet was used as a hearth plate. The arsenic bearing dust was fed with a fluidized bed feeder or pocket feeder into the fuming reactor. The off gases from the reactor and entrained dust were passed through a flanged copper cannister 6" diam. x 12" long shown in Figure 1. At the flange there was a fiberglass cloth, FSG 25 or 40, backed by a "Fibrefrax" felt paper filter, 1/8" thick.

A punched S.S. plate was used to support the filters and a 1/2" thick "Fibrefrax" blanket was used to insulate the "hot filter" from the condenser cooling coils in the other half of the cannister. The cooled gas was then passed through a cold filter of similar construction as that used in the elutriation tests. The filtered gas was then passed into a caustic scrubber.

In carrying out the tests, the furnace(s) for the preheater(s) were turned on and air was passed through the heat exchanger(s) until the input air to the fuming reactor was at constant maximum temperature, 600-700°C. Nitrogen was then used to replace this for the actual run. The dust was added batchwise into the feeder which was then sealed, then feeding was begun.

In the case of the fluidized bed feeder, a large flow of nitrogen approx. 1 scfm, was used to fluidize the dust and carry the dust into the reactor. A timed solenoid valve on the drop tube into the reactor was used to control the flow of dust admitted to the reactor.

In the case of the pocket feeder, the speed of the variable speed drive was adjusted to raise or lower the feed rate. Vibrators were used on the fluidized bed feeder and on the pocket feeder and nitrogen was used intermittently to pressurize the pocket feeder and hence promote dust flow into the carrier flow of cold nitrogen passing into the fluidized bed.

(FUMING TESTS, Cont'd.)

At the end of each run, the various weights of feed, "ash", and products were obtained and then analyzed for  $\text{As}_2\text{O}_3$ . The coils in the condenser, various fittings, and the fibrefrax blanket, isolating the hot filter from the cooling coils, were washed in hot water and the washings were analyzed also for arsenic.

## RESULTS AND DISCUSSION

A summary of the elutriation tests is shown in Figure 2. It is interesting that even at 0.50 m/sec space velocity only 50% of the dust is lost in 10 minutes, from a  $-14 + 65 \mu$  sand bed. This is quite encouraging and led to our choice of a fluid bed for the fuming reactor.

The results of the fuming tests are summarized in Table II. As can be seen from the table, the major problem in this work was feeding of the dust at the required low rate of 1-5 g/min. The dust as seen from Table I is extremely fine and has a very narrow particle size distribution, 100% between 1 and 10  $\mu$ m. Another factor that contributes to the difficulty in feeding of the dust is the electrostatic charge on the particles. Since a non-conducting plastic tube was necessary to see the progress of feeding the material, the charged dust particles stuck to the plastic and to each other. Discharging of the particles with a grounded wire mesh in the hopper was of some help but it did not eliminate the problem.

Despite the difficulty in feeding the dust, there were some encouraging results from the tests. The most important result is that the product purity is very good at +99%  $\text{As}_2\text{O}_3$ . Some of the analyzed impurities are shown in Table IV below. There was visible gangue contamination of the product from the Fibrefrax filter paper. It gave off a white powdery material during heating.

TABLE IV: Chemical Analyses of Product Arsenious Oxide

<u>Material</u>	<u>%Fe</u>	<u>%Zn</u>	<u>%Sb</u>
Product from Test #3	0.009	0.002	-
Product from Test #4	0.006	<0.001	0.06

One other interesting observation was that the fuming fluidizer was quite clean after each test showing no sign of deposits in and around the dust entry point. The sand bed was virtually the same after each run as at the beginning with no sign of "ash" fusing to the bed.

(RESULTS AND DISCUSSION, Cont'd.)

The hot filter worked quite well. The bunsen burner flame playing on the copper shell maintained the temperature at 250-300°C which kept the inner surfaces of the cannister free of solid  $\text{As}_2\text{O}_3$ . The ash did penetrate the fiberglass cloth in Test 3 and 4 but not the Fibrefrax felt paper filter.

The product was collected in the cold filters and in the condenser but the particle size in the condenser was visibly coarser than that in the cold filters. Unfortunately all crystals were mixed together so a quantitative measurement is unavailable. In addition, in Test #4 the crystals found between the SS support plate and the fibrefrax blanket were quite coarse, 1/16-1/8", and dense. This suggests that high temperatures, relative to the cold filter, and high  $\text{As}_2\text{O}_3$  vapour concentrations are required for dense crystalline product. A small number of nucleation sites will occur with a low degree of supercooling of the vapour.

The "buildup" of ash and dust in the hot filter cannister in Test #4 was brought about by intermittent overfeeding of the dust to the fuming fluidizer. Insulation of the walls\* with unfumed dust prevented heat transfer inwards and  $\text{As}_2\text{O}_3$  condensed from the vapour accounting for the 68%  $\text{As}_2\text{O}_3$  in the so called ash from the hot filter. In Test #3 the ash contained only 1.7%  $\text{As}_2\text{O}_3$ .

The mass balances for  $\text{As}_2\text{O}_3$  for Tests 3 and 4 shown in Table V, are not very good since the unaccounted is 25-28% input. Mechanical losses from the feeder during charging and during "clean-up" after each run probably accounts for this problem, since the scrubber liquid contained only 0.25 g/L arsenic, which did not change from test #3 to Test #4.

The fuming of  $\text{As}_2\text{O}_3$  from Giant Yellowknife Baghouse Dust would appear to be technically feasible. The actual maximum treatment rate of dust under given conditions of temperature and gas flow has not been determined. However further tests should give a rough indication of this rate. Small batches of dust 10-30 g will be blown into the fuming reactor over e.g. 5-10 min. The presence of unfumed dust in the hot filter will indicate overfeeding for a given condition of gas flowrate and temperature.

Since the work indicates that a fluid bed reactor might be suitable for fuming  $\text{As}_2\text{O}_3$  from the dust, the FNV nickel chloride hydrolyzer could be used as a model in designing a fuming reactor. Apparently FNV has used propane firing with success at approx. 850°C. They are now using butane. The feeding of the dust and the "hot filter" would then remain as the largest problems. The powder dispensers such as those designed by IRSID for lime powder injection into the BOF might be applicable to feeding the GYK dust.

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Attach.

  
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\* Hot filter-casing walls

TABLE II: Modifications of Laboratory Fuming Apparatus

Test #1	Test #2	Test #3	Test #4
1. Furnace set at 900°C to preheat nitrogen gas	Furnace set at 1100°C	Same as in #2	Same as in #2
2. Normal plumbing, black iron parts in "leg"	Same as in #1 but more insulation on leg	Black iron parts in leg replaced with Stainless Steel and welded all except union	Same as in #3
3. Feeder 2" $\phi$ plastic tube with plastic funnel, with 1/2" side arm to fluidizer, air vibrator on  Solenoid valve on side arm open at intervals	Funnel replaced with flat hearth disc with 5 jets, 2 holes in each 1/16" $\phi$ + 10 g silica -10 + 14 mesh  Solenoid valve on side arm open at intervals	Same feeder but more silica sand, 120 g -14 + 65 mesh  Vibrator on, solenoid open all the time, 1/2" gate valve on filter to force flow dust into fuming reactor	Pocket feeder, variable speed drive  Feed in 1" above T-1  Vibrator on
4. No heating of cross leg	Same as in #1	Add 2 heating elements to cross leg, connected in series	Heating element in parallel for more heat  Two cold filters on condenser with pressure gauge
5. Cold filters, one layer of Polyester cloth on each filter	Two layers of cloth, one Polyester and one Polypropylene felt, on each filter	Same as in #2	Same as in #2
6. Hot filter, one layer of fibrefrax felt	Same as in #1	One fibrefrax felt and fibre-glass cloth FSG25	Same as in #3 Cloth FSG40

TABLE V: Mass Balance on As<sub>2</sub>O<sub>3</sub> for Tests #3 & #4

Description	Test #3			Test #4		
	Wt. g or L	Assay		Wt. g or L	Assay	
		Wt. % or g/L As <sub>2</sub> O <sub>3</sub>	Units (g)		Wt. % As <sub>2</sub> O <sub>3</sub>	Units (g)
Feed	42.3	91.5	38.7	501.5	91.5	458.9
<u>Products:</u>						
Condenser	(7.7)			(114.6)		
Cold Filter	(14.5)			(53.0)		
Condenser + Coldfilter	22.2	99.9	22.18	167.6	99.1	166.09
Ash + Dust	13.1	1.72	0.225	186.0	68.0	126.48
Scrubber Sol'n.	6.0 L	0.28	1.70	6.0 L	0.25)	
Wash Sol'n.	2.0 L	1.72	<u>3.44</u>	2.0 L	23.9	<u>47.8</u>
Total			27.55			340.37
Unaccounted			11.15			118.53
% Unaccounted			28.81			25.80

TABLE III: Summary of Results of Fuming Tests

Description	Test #1	Test #2	Test #3	Test #4
Feeder	2" Cone Bottom Fluidizer	2" Fluidizer, Flat Hearth, 5 capped nozzles, 2 1/16 holes in each +10 g -10 + 14 M sand	→ +120 g -10 + 14 M sand, + valve to restrict gas flow to cold filter	Pocket Feeder + N <sub>2</sub> Pressurization
Run Time (mm) min	105	141	51	96
Temp. Fuming Reactor (°C)				
#1 Bottom	351 - 370	555 - 626	528 - 564	726 - 784
#2 Top	241 - 250	357 - 456	328 - 404	422 - 460
Temp. Inlet Hot Filter	(163) - 203	(155) - 289	132 - 282	237 - 266
Flow N <sub>2</sub> - (scfm)				
Reactor	0.85 - 1.06	0.96 - 98	1	1
Feeder	0.34 - 1.58	0.39 - 1.11	1.81 - 206	0.24 - 0.34
Max ΔP <sub>1</sub> ΔP <sub>2</sub> ΔP <sub>3</sub> (Psig)	0,0,-	0,0,-	0,2,-	5,5,0
*Space Velocity Max (ft/sec)	7.6	9.9	9.4	11.8
Feed, Total (g)	?	?	42.3	501.5
Rate g/min	?	?	0.8	5.2
Product Purity (% As <sub>2</sub> O <sub>3</sub> )	-	85.7	99.9	99.1
Comments:	Feeding problems, dust agg'd, fluid'n changed for worse, "Rat holing", hangups, some As <sub>2</sub> O <sub>3</sub> on coils, hot filter casing clean, no buildup, ash on filter, reactor fluidizer clean, sand bed as at start	Dust agg'd, fluid'z'n poor, same as in #1, ΔP across feeder increased, Some As <sub>2</sub> O <sub>3</sub> on coils, hot filter casing clean, no buildup, ash on filter, reactor fluidizer clean, sand bed as at start	Fluid'n better but decreased with time, dust stuck to sand in feeder, very high ΔP across reactor due to high flow, hot filter worked, no As <sub>2</sub> O <sub>3</sub> in ash, copious x't'ls in condenser + cold reactor filter, reactor fluidizer clean, sand bed as at start	Overfeeding problems 50-100 g slugs, caused high ΔP across hot filter, no control over rate, hangups in hopper, sticking dust in pockets, copious x't'ls in condenser + filters, ash + unfumed dust on hot filter, reactor fluidizer clean, sand bed same as at start

\* Assume max flow and temp. in run and that feed N<sub>2</sub> no influence on space velocity



FIGURE 2: Batch Elutriation Tests on  
Giant Yellowknife Baghouse Dust  
at Room Temperature  
(1" Fluidizer, L/D = 2.4, -14 +65 M Sand)

