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**RECOVERY AND PURIFICATION OF  
ARSENIC OXIDE - GIANT MINE**

**DECEMBER 1999**

**MINING AND MINERAL SCIENCES LABORATORIES  
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**CONFIDENTIAL**

**MINING AND MINERAL SCIENCES LABORATORIES  
REPORT MMSL 99-078 (CR)**



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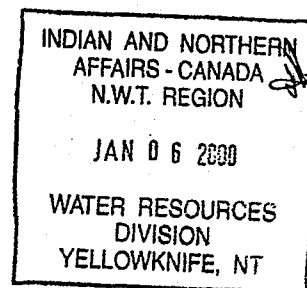
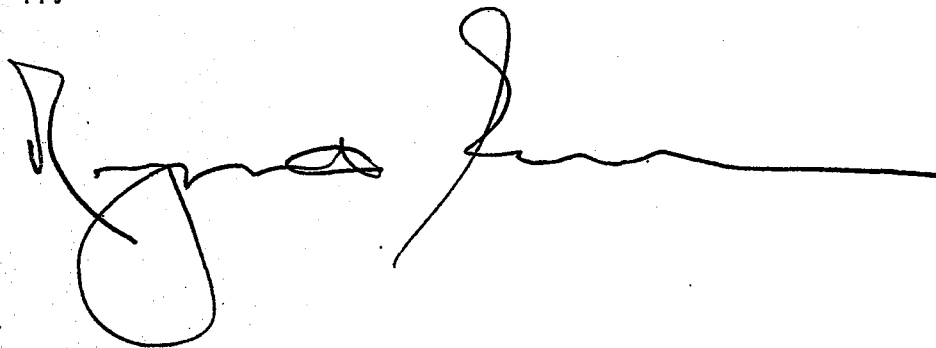
Neill,

As mentioned in my last E-Mail to you, I have ready to be sent by courier, a DRAFT version of the report. There are few gaps and these are as follows:

- Antimony analysis have to be confirmed;
- Results of Acid Leach are available at 50%. Remaining to come;
- A conclusion is being written and will be available early in January (next week); and
- No third party editing was done on the draft report.

I hope this will be of immediate help for you at this specific moment.

Happy New Year.



## INTRODUCTION

The Giant Mine, Yellowknife, Northwest Territories has been in operation for over fifty years and has produced 6,800,000 oz of gold in that period. Ore reserves, however, are nearly exhausted. In fact, at the present price of gold, calculated ore reserves are adequate for less than two years of operation. Until very recently, the mine was operated by Royal Oak Mines Inc., but that company was declared bankrupt in 1999. Accordingly, the mine is presently operating under a court-appointed receiver. At the Giant Mine, the ore is crushed, milled and then is floated to produce an arsenopyrite ( $\text{FeAsS}$ )-pyrite ( $\text{FeS}_2$ ) concentrate. The concentrate is refractory, and accordingly, is subjected to roasting in a series of two slurry-feed Dorr-Oliver roasters to drive off the sulphur as  $\text{SO}_2$  and the arsenic as  $\text{As}_2\text{O}_3$ . A two-stage roasting operation is required to ensure the total evolution of the arsenic as  $\text{As}_2\text{O}_3$  and the complete liberation of the gold for subsequent cyanidation. Gases from the roaster train are passed through a hot electrostatic precipitator to collect any carried-over calcine, which is recycled to the gold extraction circuit. The gases are then cooled, by admitting dilution air, to condense the arsenic oxide. Finally, the  $\text{As}_2\text{O}_3$  fume is collected in a baghouse, which operates with a solids collection efficiency of 99.9%. The collected  $\text{As}_2\text{O}_3$ -rich dust is easily fluidized and is subsequently transported pneumatically (1).

The  $\text{As}_2\text{O}_3$ -rich dusts produced since 1951 have been stored underground, initially in chambers in permafrost, but subsequently in chambers excavated in coherent bedrock. Presently, 265,000 tonnes of the  $\text{As}_2\text{O}_3$ -rich dust are stored underground in 15 chambers, and 8-15 t/day of new dust is accumulating. It is reported that, on average, the accumulated dust contains 76%  $\text{As}_2\text{O}_3$  and 0.54 oz/t Au; however, localized gold concentrations as high as 2 oz/t Au have been analyzed. The dust presently produced is reported to contain 89%  $\text{As}_2\text{O}_3$  (67.7% As), 1.04% Sb, 1.68% Fe and 0.14 oz/t Au. Recent mining activities have disturbed the permafrost seal in some of the chambers, and some arsenic contamination of the mine waters is occurring. Accordingly, the As-containing mine waters are pumped to the surface, where they are oxidized with

hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) and then are treated with lime ( $\text{CaO}$ ) and ferric sulphate ( $\text{Fe}_2(\text{SO}_4)_3$ ) to precipitate an insoluble amorphous iron arsenate compound for storage in a residue pond (1).

The Department of Indian Affairs and Northern Development (DIAND) has the responsibility as regulator to ensure that the environment of the Northwest Territories is protected and that human health is maintained. Accordingly, DIAND is considering various options to reduce or eliminate the potential environmental and health impacts caused by the stored  $\text{As}_2\text{O}_3$ -rich dust. One of these options is the reprocessing of the dust to recover a purified arsenic oxide product which could be used in the wood preservative industry or for other applications. Although the value of  $\text{As}_2\text{O}_3$  is only U.S. \$0.20-\$0.30/lb, the manufacture of any product would remove the arsenic from the mine site and convert it into a useful commodity. For such applications, it is claimed that the product must contain >98%  $\text{As}_2\text{O}_3$ , but less than 0.2% Sb and 0.02% Fe, although the need for such low Sb and Fe specifications for wood preservative applications is not clear.

One means to produce a marketable arsenic product from the  $\text{As}_2\text{O}_3$ -rich dust involves leaching with hot water to dissolve  $\text{As}_2\text{O}_3$  selectively, filtration to reject insoluble matter, removal of any co-solubilized Sb from solution to meet product specifications, and crystallization of a pure  $\text{As}_2\text{O}_3$  product from the aqueous medium. Preliminary testwork on the water leaching option was carried out by Royal Oak Mines (private communication of L. Connell, Royal Oak Mines Inc., 1998). Using a residence time of 30-240 minutes at 95°C, the company was able to achieve >90% As dissolution, while rejecting ~80% of the Sb to the residue. Negligible iron dissolution occurred. Over 80% of the mass of the initial dust dissolved; the residue amounted to about 17% of the initial mass and contained 39% As. The solution generated at 95°C analyzed 31 g/L As, 92 mg/L Sb and 6 mg/L Fe. The observed As solubility of 31 g/L As, however, is much less than the reported solubility of ~100 g/L  $\text{As}_2\text{O}_3$  (~75 g/L As) (2). The reasons for the low solubilities over the  $\text{As}_2\text{O}_3$ -rich dusts are not known, but they could be partly attributable to the relatively short leaching times used (<4 h) or to the low

pulp densities employed (~5% solids). Because of the low arsenic solubilities realized, the yield of  $\text{As}_2\text{O}_3$  crystals during the subsequent cooling of the solution to ambient temperature was unacceptably low. In the Royal Oak work, this problem was addressed by evaporation of the ~95°C solution to about one-third of its initial volume prior to cooling to room temperature to crystallize  $\text{As}_2\text{O}_3$ . Efforts were also made to remove part of the co-dissolved Sb using two ion exchange resins and activated carbon. The use of the resins or activated carbon allowed the crystallization of a product containing ~100%  $\text{As}_2\text{O}_3$ , <0.2% Sb and <0.05% Fe. Hence, it appears that the simple water leaching-crystallization technique can yield an  $\text{As}_2\text{O}_3$  product which satisfies the purity requirements of the marketplace. The major difficulty with this processing option seems to be the low  $\text{As}_2\text{O}_3$  solubilities produced in the leaching part of the process that result in a commercially unacceptable yield of  $\text{As}_2\text{O}_3$  crystals unless costly techniques, such as solution evaporation, are employed.

A second option to produce a pure  $\text{As}_2\text{O}_3$  product is the controlled re-sublimation of the  $\text{As}_2\text{O}_3$ -rich dust to collect a pure  $\text{As}_2\text{O}_3$  fume while leaving most of the impurities in a residue for subsequent treatment and stabilization. This option has also been pursued by Royal Oak Mines using the Warox process (1). Their test work showed that it was possible to re-sublime the  $\text{As}_2\text{O}_3$ -rich dust to yield a fume containing 99.3-99.8%  $\text{As}_2\text{O}_3$ , together with less than 0.05% Fe. Significantly, the Sb contents of the sublimates ranged from 0.13 to 0.59% Sb. Careful study of the low-Sb products, however, indicated that they tended to originate from low-Sb dusts. That is, there was little selective re-sublimation of  $\text{As}_2\text{O}_3$  relative to  $\text{Sb}_2\text{O}_3$ . Although re-sublimation can control most of the impurities in the dust, the behaviour of antimony remains a concern with this option.

Within the above framework, CANMET was contracted to investigate the production of pure  $\text{As}_2\text{O}_3$  using water leaching-crystallization and re-sublimation techniques. The initial contract was jointly funded by DIAND and Royal Oak Mines Inc. and was initiated in October, 1998. During that study, it became apparent that the originally proposed scope of work needed to be expanded, and

as a consequence, the original project was amended and extended in February, 1999. The amendment was authorized only by DIAND as Royal Oak Mines Inc. was in receivership at that time.

The scope of the original and amended project involves the production of pure  $\text{As}_2\text{O}_3$  by water leaching-crystallization and by re-sublimation of four  $\text{As}_2\text{O}_3$ -rich dust samples provided by the company. The water leaching work was intended to measure the solubility of  $\text{As}_2\text{O}_3$  in water as a function of temperature on both heating and cooling. Because low solubilities were initially measured, they were subsequently determined at temperatures up to  $200^\circ\text{C}$  with the use of a high pressure autoclave. These latter tests suggested a possible processing procedure that involves autoclave leaching, cooling of the slurry to  $95\text{--}100^\circ\text{C}$ , followed by filtration and the subsequent cooling of the solution to room temperature to crystallize pure  $\text{As}_2\text{O}_3$ . Efforts were also made to leach the  $\text{As}_2\text{O}_3$ -rich dusts in acid media. Sulphuric acid ( $\text{H}_2\text{SO}_4$ ), hydrochloric acid ( $\text{HCl}$ ) and nitric acid ( $\text{HNO}_3$ ) were evaluated for this application, and subsequently, a number of leaching parameters were examined using hydrochloric acid or nitric acid as the leaching medium. Moderate  $\text{As}_2\text{O}_3$  solubilities were realized in hydrochloric acid media, and hydrochloric acid leaching seems to offer some advantages over water leaching. Attempts were made to remove the low concentrations of co-dissolved Sb from the water and acid leach liquors using four ion exchange resins and activated carbon. These studies were done at various temperatures to try to optimize the level of antimony removal. Re-sublimation tests were carried out for different times at various temperatures and flow rates to try to enhance the vaporization of  $\text{As}_2\text{O}_3$  relative to  $\text{Sb}_2\text{O}_3$ . The use of oxidizing, neutral and reducing atmospheres was explored. The initial results on the re-sublimation of  $\text{As}_2\text{O}_3$  from the dusts indicated that the greatest selectivity of  $\text{As}_2\text{O}_3$  over  $\text{Sb}_2\text{O}_3$  occurred at the lowest temperatures, where unfortunately, mass transport of the vapour is also low. Consequently, the use of controlled re-sublimation at low temperatures was also investigated to try to obtain a high level of  $\text{As}_2\text{O}_3$  selectivity over  $\text{Sb}_2\text{O}_3$ , together with an acceptable re-sublimation rate. To complement the re-sublimation activities, a preliminary

mass spectrographic study of the vapour produced from one of the dusts was done to ascertain whether mixed  $(As_{4-x}Sb_x)O_6$  molecules were present, or whether the vapour consisted only of  $As_4O_6$  and  $Sb_4O_6$  entities (3). In support of both the water leaching and re-sublimation activities, detailed mineralogical studies were carried out on the as-received  $As_2O_3$ -rich dust samples and on some of the intermediate products. The mineralogical activities focussed on the As-, Sb- and Fe-containing phases rather than on the gold content of the dusts. This report summarizes the results of the above exploratory investigations, and also offers some observations on their implications for the recovery of a marketable  $As_2O_3$  product from the  $As_2O_3$ -rich dusts stored at the Giant Mine.

## **EXPERIMENTAL**

### **Samples**

Four samples of  $As_2O_3$ -rich dust from the Giant Mine were supplied by Royal Oak Mines Inc. Two of the dust samples were recent composites, and were identified as the January 98 Composite and the April 97 Composite. The remaining two samples were taken from the underground storage chambers, and were identified as 212 Chamber and 236 Chamber. All four samples were fine powders; three of the dusts were dry, but the 236 Chamber sample was slightly damp. All four samples, however, were used without further treatment. Reagent grade arsenious oxide (J.T. Baker, 99.95%  $As_2O_3$ ) and reagent grade antimony trioxide (J.T. Baker, 101.4%  $Sb_2O_3$ ) were sometimes used for comparative experimentation; the reagent grade oxides also were used in the as-received state.

### **Mineralogical Techniques**

X-ray powder diffraction analyses were carried out on the four as-supplied samples, on the reagent grade  $As_2O_3$ , on some of the water leach and acid leach residues, and on some of the re-sublimation products using a Rigaku automated diffractometer with a water-cooled rotating copper anode. The detection limit of most crystalline compounds by this apparatus is 1-3%, depending on the number

of phases present and their diffracting powers. Polished sections of the samples were prepared, and these were then examined using scanning electron microscopy (SEM) with energy dispersive X-ray analysis (EDX). Extensive use was made of the backscattered electron detector at high magnification to distinguish compositionally different phases. Some of the phases were analyzed quantitatively with a JEOL-8900 electron microprobe operated, generally, at 20 kV and a 20 nanoampere beam current. In some instances, loose powder mounts of the samples, wherein the material was sprinkled loosely on the stage of the electron microscope, were examined with the SEM to show the morphology of the dust particles more fully. Details of the various mineralogical procedures have been reported previously (4).

### **Solubility Measurements**

The solubility experiments were done in a 2-L reaction vessel having a tight-fitting ground glass lid to prevent water evaporation during the course of the experiments. As shown in Figure 1, a thermometer ( $\pm 0.1^\circ\text{C}$ ), a water-cooled condenser which was loosely stoppered, and a sampling tube closed at the bottom with a medium porosity glass frit were fitted in the lid of the vessel. The sampling tube with the fritted glass disk extended into the middle of the test slurry. The reaction vessel and its contents were heated in a temperature-controlled circulating oil bath regulated to better than  $\pm 0.1^\circ\text{C}$ . The actual temperature of the solution was measured by the thermometer suspended in the slurry. The reaction vessel containing ~1.5 L of solution with an excess of dust (~150 g) rested on the bottom of the oil bath, and the slurry was stirred magnetically from beneath.

The solubility measurements were performed as follows. One and a half litres of water were placed in the 2-L reaction vessel together with an excess of the  $\text{As}_2\text{O}_3$ -rich dust or reagent grade  $\text{As}_2\text{O}_3$  and  $\text{Sb}_2\text{O}_3$ . The contents were then stirred for ~3 days to attain equilibrium. Preliminary experiments showed that the dusts equilibrated slowly in water; three days were required to reach saturation at room temperature and one day was required at  $60^\circ\text{C}$ . Significantly, most of the

solubility measurements carried out previously by Royal Oak Mines used equilibration times of <4 h. When equilibrium had been attained, a clean fritted sampling tube was immersed in the liquid and was thermally equilibrated. Saturated solution was then drawn through the frit into the sampling tube by the application of suction at the open end. When the sampling tube within the reaction vessel was partly filled with the filtered solution, the suction attachment, which fitted into the open end of the sampling tube in the lid of the vessel, was removed and samples of the hot solution were taken using 5 mL glass pipettes. The pipettes were previously calibrated as a function of temperature using pure water and the same sampling procedure as that employed for the  $\text{As}_2\text{O}_3$ -saturated solutions. A solution sample was taken at each temperature. The sample was diluted directly into 100 mL or 500 mL of 2%  $\text{HNO}_3$  contained in a volumetric flask; the dilution factor was sufficiently large that crystallization of  $\text{As}_2\text{O}_3$  or  $\text{Sb}_2\text{O}_3$  never occurred. The contents of the volumetric flask were brought to volume, and the solution was analyzed for As by ICP analysis and for Sb and Fe by ICP-mass spectroscopy. In some instances, a second sample was taken at the various temperatures. These samples were discharged directly into a weighing bottle, sealed, cooled to room temperature, and weighed; this sample allowed the density of the saturated solution to be calculated.

After the samples were taken, the solution temperature was increased to 100°C in 10°C increments, with samples being taken at each incremental temperature. The solution was then cooled to ~95°C, and subsequently, in 10°C increments to room temperature or to 5°C to obtain saturation curves on both heating and cooling. Agreement between the data obtained on heating and cooling is necessary if "equilibrium" solubilities are to be reported.

The solubilities were calculated from the measured concentrations, the dilution factor and the volume of the calibrated pipette. The densities of the saturated solutions were calculated as a function of temperature by the measured weight of the sample and the calibrated volume of the pipette.

### Autoclave Solubility-Leaching Tests

These experiments were carried out in a Parr 2-L titanium autoclave fitted with a glass liner and titanium internals. The  $\text{As}_2\text{O}_3$ -rich dust and water were placed in the autoclave, which was sealed and then heated rapidly to the desired reaction temperature of 125-200°C. Because of the high temperatures used in the autoclave solubility experiments, solution saturation was achieved in just a few hours. In this regard, retention times were measured from the moment that the operating temperature was reached, although it is recognized that some dissolution occurs during the heat-up period. The experiments were done without an applied gas pressure; that is, the tests were effected only under the steam pressure generated by the system. At the end of the reaction period, the autoclave was rapidly cooled (~5 min) to 95-100°C by the use of the internal water cooling coil. Of course, at temperatures <100°C, the pressure in the autoclave is that of the surrounding atmosphere. The autoclave was opened at 95-100°C, and the slurry was rapidly transferred to a solubility reactor heated to 95°C in its oil bath. The solubility reactor was sealed, and then equilibrated for 2-3 days at 95°C. A sample was taken for a solubility determination and the vessel was then cooled to room temperature. The procedure for determining the solubilities is described above.

In those experiments intended to produce  $\text{As}_2\text{O}_3$  crystals, the autoclave slurry at 95-100°C was filtered hot using a Buchner vacuum filter with Whatman No. 3 paper; the apparatus was preheated to ensure that the temperature of the filtrate was always ~95°C. Following filtration, the solution was transferred to a beaker and was allowed to cool to room temperature while being gently agitated. It was found that  $\text{As}_2\text{O}_3$  crystals formed spontaneously and without the need for  $\text{As}_2\text{O}_3$  seed additions. When crystallization was complete (~16 h), the product was filtered using Whatman No. 3 paper. The various precipitates were collected, dried at 110°C, weighed, and sometimes, analyzed.

### **Antimony Removal by Ion Exchange or Activated Carbon**

Four commercial ion exchange resins were selected on the basis of previous experiments carried out by Royal Oak Mines and on information published in the open literature. These resins were considered to be the most promising for the removal of antimony from concentrated  $\text{As}_2\text{O}_3$ -containing media under neutral or acid pH conditions. Samples of the resins were obtained from the manufacturers, and Table 1 presents a list of the four ion exchange resins used. The resins were hydrated for 24 h, washed with copious amounts of water and then used without additional treatment.

Table 1 - Ion Exchange Resins Tested for Antimony Removal

Resin	Type	Manufacturer
Gravex GR-2-0	Sulphonic	Graver Technologies
Amberlite IRC 120	Sulphonic	Rohm and Haas
Amberlite IRC 50	Carboxylic	Rohm and Haas
Duolite C467	Aminophosphonic	Rohm and Haas

Equilibrium extraction experiments were carried out by contacting appropriate amounts of the individual resins with the Sb-containing  $\text{As}_2\text{O}_3$ -rich solution, while applying gentle mechanical agitation. For most experiments, the amount of resin is expressed as the "wet settled volume", as is common practice in ion exchange applications. In some experiments, however, where a direct comparison with activated carbon was desired, the actual resin weight was used instead of the resin volume. In all the experiments, the extent of antimony removal was calculated from the Sb concentration of the aqueous solution before and after its contact with the resin. Total Sb was analyzed, and no effort was made to differentiate Sb(III) from Sb(V).

In addition to the four ion exchange resins given in Table 1, an activated carbon sample, Hycarb 6X12, was also evaluated for its Sb removal potential. The activated carbon was washed with water prior to use.

### Re-Sublimation of the $\text{As}_2\text{O}_3$ -Rich Dusts

The overall objective of this part of the investigation was to re-sublime the as-received  $\text{As}_2\text{O}_3$ -rich dusts to produce a pure  $\text{As}_2\text{O}_3$  product. These experiments were carried out using a horizontal tube furnace which was 60 cm in length. A fused silica tube 90 cm long by 60 mm outside diameter was positioned in the tube furnace such that ~25 cm of the tube protruded on one side. The  $\text{As}_2\text{O}_3$ -rich dust was weighed and placed in an alumina crucible which was positioned in the constant temperature ( $\pm 2^\circ\text{C}$ ) hot zone of the furnace. Rubber stoppers were used to seal the ends of the tube, and the stoppers were fitted with ports for the entry and exit of gases and for a temperature measuring thermocouple. Gas was sometimes passed through the tube to help transport the volatilized  $\text{As}_2\text{O}_3$  to the cooler part of the tube where it condensed as a "ring" on the surface of the glass. At the completion of the experiment, the tube was removed from the furnace and was cooled. The condensed oxide product was removed and analyzed. The re-sublimation experiments were carried out at various temperatures and, sometimes, for different degrees of sublimation. Comparative tests were done using slowly-flowing oxygen, inert gas and hydrogen to assess the influence of the gaseous atmosphere on the relative transport of  $\text{As}_2\text{O}_3$  and  $\text{Sb}_2\text{O}_3$ . Finally, screening experiments were done wherein the dust was heated with As metal to try to remove any vapourized antimony oxide from the gas stream prior to the condensation of the  $\text{As}_2\text{O}_3$ . To complement the vapourization studies and to provide a fundamental rationale for the co-sublimation of antimony at temperatures significantly below the  $1550^\circ\text{C}$  boiling point of  $\text{Sb}_2\text{O}_3$ , a preliminary mass spectrographic study of the vapours was carried out. This work was sub-contracted to the University of Montreal and was based on the use of a Micromass Autospec-OA time-of-flight mass spectrometer operating in the EI mode at 70 eV. The samples were heated to  $300^\circ\text{C}$  on a solid probe to vapourize the arsenic and antimony oxide species into the mass spectrometer.

### Arsenic Solubility in Acid Media

The intent of these experiments was to evaluate the possibility of enhancing the solubility of  $\text{As}_2\text{O}_3$  over the  $\text{As}_2\text{O}_3$ -rich dusts by the use of acid media in place of water. All of these scoping experiments were done using the 236 Chamber sample. The acid leaching experiments were carried out in 2-L glass reaction kettles contained in a temperature-controlled oil bath. One litre of the acid solution was heated to  $95^\circ\text{C}$ , and at that point, a known mass of the  $\text{As}_2\text{O}_3$ -rich dust from the 236 Chamber was added and was stirred for the duration of the experiment, generally 2 h. The acids initially evaluated were 2.0 M HCl, 2.0 M  $\text{HNO}_3$  and 1.0 M  $\text{H}_2\text{SO}_4$  (i.e., 2.0 M  $\text{H}^+$ ). For the experiments using the different acids, solution samples were taken at various time intervals by drawing the liquid through an in-line filter. The collected solutions were stored in a water bath at  $95^\circ\text{C}$ . Subsequently, they were accurately pipetted, diluted to a known volume and finally analyzed to assess the extent of arsenic dissolution as a function of time. Based on these initial tests, nitric acid ( $\text{HNO}_3$ ) and hydrochloric acid (HCl) were selected for further experimentation. The parameters investigated in this latter part of the study were the acid concentration (1.0, 2.0, 3.0 and 4.0 M  $\text{HNO}_3$ ; 0.75, 2.0 and 3.0 M HCl) the leaching temperature ( $95^\circ\text{C}$ ,  $75^\circ\text{C}$  and  $55^\circ\text{C}$ ) and the solids content of the slurry (75 g/L and 175 g/L solids). For the latter experiments, solution sampling as a function of time was not done. Rather, leaching was carried out for a constant 2-h period. At that time, the slurry was filtered while hot, and the filtrate was collected. The leach residue was then water-washed, dried at  $110^\circ\text{C}$ , and analyzed by X-ray powder diffraction methods. The filtrate was then cooled to room temperature to allow  $\text{As}_2\text{O}_3$  to crystallize. The  $\text{As}_2\text{O}_3$  crystals were filtered, dried at  $\sim 60^\circ\text{C}$ , weighed and also analyzed by X-ray diffraction methods. In some tests, the solution remaining after filtration of the  $\text{As}_2\text{O}_3$  crystals was neutralized to pH 9.0 with solid, reagent grade  $\text{Na}_2\text{CO}_3$  to try to precipitate additional  $\text{As}_2\text{O}_3$ . The resulting precipitate was filtered, washed, dried at  $110^\circ\text{C}$ , and then analyzed by X-ray powder diffraction techniques to identify the arsenic compound precipitated.

## RESULTS AND DISCUSSION

### Samples

The four samples of  $\text{As}_2\text{O}_3$ -rich dust supplied by Royal Oak Mines were analyzed at CANMET; the results are presented in Table 2. The recent composite samples have As contents in excess of 66% As ( $>87\% \text{As}_2\text{O}_3$ ), but also contain modest amounts of Sb, Fe, Al, Ca, Mg and Si. The two samples from the underground chambers have much lower As contents (56-58% As), but also contain minor amounts of Sb, Fe, Al, Ca, Mg and Si. The Sb content is a concern for the marketing of any  $\text{As}_2\text{O}_3$  product, and the Sb contents of the as-received dusts are variable. They range from 0.30 to 2.13% Sb. The data of Table 2 support the view that the stockpiled dusts have higher gold values than the dusts presently being generated.

Table 2 - Chemical Compositions of the Four  $\text{As}_2\text{O}_3$ -rich Dust Samples Provided by Royal Oak Mines (wt %)

Element	January 98 Composite	April 97 Composite	212 Chamber	236 Chamber
As	68.2, 68.7	66.7, 69.6	56.2	58.0
Sb	0.87, 0.84	1.48, 1.49	2.13	0.30
Fe	1.23, 1.17	0.78, 0.81	2.62	1.66
Al	0.64, 0.64	0.33, 0.33	1.06	0.92
Ca	--	--	--	--
Mg	0.24, 0.26	0.13, 0.13	0.37	0.36
Si	1.05, 1.09	0.61, 0.61	2.07	1.81
Au(ppm)	2.2	2.4	11.0	5.1

### Mineralogical Characterization of the Four Dust Samples

X-ray diffraction analyses of the two lots of reagent grade arsenic oxide indicated only  $\text{As}_2\text{O}_3$  having the cubic arsenolite structure. Analogous X-ray diffraction analyses of the four dust samples showed them to be very similar, *inter alia*, despite the minor differences in chemical composition indicated in Table 2. The X-ray diffraction patterns of the four dust samples are presented in Appendix 1. All four samples consist of major amounts of  $\text{As}_2\text{O}_3$  of the arsenolite

structure, together with minor amounts of a chlorite-type mineral (chamosite?) having the general formula of  $(\text{Fe,Al,Mg})_6(\text{Si,Al})_4\text{O}_{10}(\text{OH})_8$ . A trace quantity of muscovite,  $\text{KAl}_2(\text{Si}_3\text{Al})\text{O}_{10}(\text{OH,F})$ , is also evident. Quartz is also present in trace amounts, but the detection of quartz is made difficult because the strong line of quartz overlaps that of muscovite. The amounts of chlorite, muscovite and quartz, relative to arsenolite, vary somewhat from sample to sample, but arsenolite is always the dominant phase. The four dust samples were supplied in large plastic pails. To investigate the possibility of sample segregation within the pails, X-ray diffraction analyses of material from the top and bottom of each pail were carried out. The spectra from the top and bottom of a given pail were identical, and the implication is that the supplied samples themselves are relatively homogeneous.

#### **Characterization of the January 98 Composite Sample**

Detailed SEM-EDX study of the dust provided considerably more insight into the phases present. Figure 2 presents a general view of the January 98 Composite sample. This sample consists mostly of  $\text{As}_2\text{O}_3$  which commonly occurs as 1-3  $\mu\text{m}$  free crystals or as somewhat larger crystal agglomerates. Many of the  $\text{As}_2\text{O}_3$  crystals demonstrate an octahedral morphology characteristic of arsenolite. The large and darker appearing particles are chlorite (chamosite ?) or quartz. Tiny bright appearing grains of iron oxide having a minor As content are also present. The EDX study of the  $\text{As}_2\text{O}_3$  particles commonly detected trace amounts of Sb; similar studies showed the chlorite to contain major amounts of Mg, Al and Si, along with traces of Fe, K and Na.

Figure 3 is a backscattered electron micrograph of the same sample as seen in polished section. The arsenolite ( $\text{As}_2\text{O}_3$ ) occurs abundantly as tiny bright appearing crystals or crystal aggregates. The EDX study of the  $\text{As}_2\text{O}_3$  commonly detected a trace-to-minor amount of Sb. The larger platy crystals are chlorite, whereas the dark appearing irregular grains are usually quartz. Of note are a number of 1-3  $\mu\text{m}$  calcium arsenate particles having a minor or trace S content. Occasionally, large masses of  $\text{As}_2\text{O}_3$  or  $(\text{As,Sb})_2\text{O}_3$  are detected, and one such

mass is illustrated in Figure 4. Although the majority of the image shown in Figure 4 consists of 1-3  $\mu\text{m}$  euhedral  $\text{As}_2\text{O}_3$  crystals and somewhat larger grains of As-bearing iron oxide, a  $>50 \mu\text{m}$  mass of  $\text{As}_2\text{O}_3$  having a trace Sb content is also evident. Such masses of  $\text{As}_2\text{O}_3$  are moderately abundant in this sample, but nevertheless, constitute only a small percentage of the total  $\text{As}_2\text{O}_3$  present. The mechanism of formation of these large masses of  $\text{As}_2\text{O}_3$  is not known, but they may represent spalled accumulations from the roof or ducting of the baghouse.

Rarer masses of  $(\text{Sb,As})_2\text{O}_3$  are also detected, and one such occurrence is shown in Figure 5. The mass is 40-50  $\mu\text{m}$  in size, and is irregular in form. Its brighter appearance reflects the elevated Sb content of this phase, which seems to be an As-bearing Sb-rich oxide; i.e.,  $(\text{Sb,As})_2\text{O}_3$ . The mechanism by which such Sb-rich oxide particles form in the baghouse is unknown.

An iron arsenic-oxide phase occurs in minor amounts in the January 98 Composite sample, and the iron arsenic-oxide ranges in composition from As-bearing iron oxide to compositions approaching iron arsenate. Figure 6 illustrates a  $\sim 20 \mu\text{m}$  mass of iron oxide containing minor amounts of As as well as traces of Sb. The iron oxide particle has a banded texture that may reflect the cyclical oxidation of a grain of pyrite. Variations in the grey level of the iron oxide reflect local variations in the As content. In this instance, it seems that the morphology of the original iron oxide particle has been faithfully preserved. Figure 7 illustrates a  $\sim 100 \mu\text{m}$  porous mass of iron arsenate that is associated with a large mass of  $\text{As}_2\text{O}_3$  and numerous tiny  $\text{As}_2\text{O}_3$  crystals. The iron arsenate phase contains major amounts of iron and arsenic, as well as a minor-to-major S content. It is likely an iron arsenate phase having a significant sulphate ion substitution for arsenate. The morphology suggests a complex formation mechanism that likely involves the diffusion of both As and Fe.

The SEM-EDX investigation of the January 98 Composite sample also detected trace amounts of a number of other phases. These phases are Mg silicate, Fe-Ca-Al silicate-sulphate, K-Mg-Al silicate, Na-Mg-Al silicate, Ca-Na-Mg-Al silicate, K-Al silicate (feldspar) and  $\text{PbSO}_4$ .

Quantitative electron microprobe analyses were carried out on the As- and Sb-containing phases in the January 98 Composite sample, and the detailed analyses are presented in Appendix 2.

Table 3 summarizes the electron microprobe data obtained for the major  $\text{As}_2\text{O}_3$  phase. Thirty-nine particles of  $\text{As}_2\text{O}_3$  were analyzed, and these particles included both the abundant tiny  $\text{As}_2\text{O}_3$  crystals and the less common large masses of  $\text{As}_2\text{O}_3$ . Aside from its modest Sb content, the  $\text{As}_2\text{O}_3$  is relatively pure, with only trace amounts of Fe and Pb being detected as impurities. Its general formula is  $(\text{As,Sb})_2\text{O}_3$ . Given the abundance of the  $\text{As}_2\text{O}_3$  phase in this sample and its high As and Sb content relative to the bulk composition of the dust (Table 2), it is clear that the  $\text{As}_2\text{O}_3$  phase is the dominant As and Sb carrier in the January 98 Composite sample.

Table 3 - Electron Microprobe Determined Composition of the  $\text{As}_2\text{O}_3$  Phase in the January 98 Composite Sample (wt %)

Element	Average Composition	Range
As	75.03	63.04 - 82.11
Sb	1.31	0.00 - 5.38
Fe	0.18	0.00 - 2.01
Pb	0.07	0.00 - 1.88
O	23.41	17.63 - 32.55

Table 4 presents the electron microprobe-determined compositional data for the Sb-rich oxide phase present in the January 98 Composite sample.

Although this phase is a minor constituent of the dust, it has a high Sb and As content; that is, the phase is possibly best formulated as  $(\text{Sb,As})_2\text{O}_3$ . The Sb-rich oxide seems to be relatively pure; only trace amounts of Fe and Pb are detected in its structure.

Table 4 - Electron Microprobe Determined Composition of the (Sb,As)<sub>2</sub>O<sub>3</sub> Phase in the January 98 Composite Sample (wt %)

Element	Average Composition	Range
Sb	44.65	40.50 - 47.77
As	35.16	28.94 - 38.95
Fe	0.05	0.01 - 0.09
Pb	0.01	0.00 - 0.07
O	20.12	14.41 - 30.52

The January 98 Composite sample contains both an As-bearing iron oxide phase as well as an iron arsenate phase. Table 5 summarizes the compositional data obtained on nine particles of the As-bearing iron oxide. The iron oxide phase contains major amounts of Fe and O, in addition to minor contents of As and Sb, and traces of Pb and S. The compositional data suggest a hematite-like compound, but complementary structural studies would be needed to corroborate this conclusion. Table 6 summarizes the analogous data obtained from 16 particles of the iron arsenate phase. This material has a relatively low Fe content but relatively high levels of As and S, which likely are present as AsO<sub>4</sub> and SO<sub>4</sub>. There are also trace amounts of Sb, Pb, Ca and Bi detected. The composition suggests an iron arsenate compound having a significant degree of sulphate substitution for arsenate, but it should be appreciated that some of the analyses may be biased by intimately admixed As<sub>2</sub>O<sub>3</sub>.

Table 5 - Electron Microprobe Determined Composition of the As-bearing Iron Oxide Phase in the January 98 Composite Sample (wt %)

Element	Average Composition	Range
Fe	59.11	57.51 - 61.11
As	4.68	3.15 - 5.87
Sb	1.67	1.15 - 2.10
Pb	0.10	0.05 - 0.17
S	0.04	0.00 - 0.08
O	34.39	33.67 - 35.23

Table 6 - Electron Microprobe Determined Composition of the Iron Arsenate Phase in the January 98 Composite Sample (wt %)

Element	Average Composition	Range
Fe	10.15	1.78 - 38.33
As	15.95	7.92 - 21.63
Sb	0.27	0.06 - 0.87
S	3.57	0.23 - 7.41
Pb	0.10	0.04 - 0.19
Ca	0.73	0.04 - 4.39
Bi	0.03	0.00 - 0.07
O	69.16	52.44 - 77.45

#### **Characterization of the April 97 Composite Sample**

In terms of its chemical composition (see Table 2) the April 97 Composite sample is similar to the January 98 Composite sample, although there are minor differences in the Sb contents as well as those of Fe, Al, Ca, Mg and Si. Both samples are more rich in As than the two samples collected from the storage chambers.

The SEM-EDX studies also indicated many similarities between the April 97 Composite sample and the January 98 Composite sample. In this regard, Figure 8 shows the general appearance of the April 97 Composite sample as seen in a loose powder mount. The sample consists predominantly of 1-3  $\mu\text{m}$  crystals of  $\text{As}_2\text{O}_3$ , many of which have well developed external crystal faces. The EDX analysis showed major amounts of As, together with traces of Sb. The larger, irregular particles are chlorite, which consists mostly of Fe, Mg, Al, Si and O. Comparison with Figure 2 indicates that the general morphologies of the April 97 Composite and January 98 Composite samples are very similar.

Figure 9 shows the April 97 Composite sample in more detail and in polished section. Most of the field consists of 1-3  $\mu\text{m}$  crystals of  $\text{As}_2\text{O}_3$ , which generally occur free but sometimes are intergrown into larger agglomerates. The large, bright appearing particle in the centre of the image is  $(\text{As,Sb})_2\text{O}_3$  containing about 13% Sb. As noted above, the mechanism by which such large arsenic-antimony oxide particles form is not known. The figure also indicates a

number of small particles of quartz that passed through the electrostatic precipitator circuit and were collected in the baghouse along with the  $\text{As}_2\text{O}_3$ . Figure 10 shows an even larger mass of  $(\text{As,Sb})_2\text{O}_3$ , the growth of which has sometimes encapsulated other species. Such large particles of  $(\text{As,Sb})_2\text{O}_3$  presumably formed on the walls or ducting of the baghouse where they accumulated to a considerable thickness before becoming dislodged and reporting with the baghouse dust. The large mass of  $(\text{As,Sb})_2\text{O}_3$  is associated with smaller crystals of  $\text{As}_2\text{O}_3$ , as well as with particles of quartz and chlorite. Notable is the presence of two  $\sim 10\ \mu\text{m}$  particles of iron-calcium oxide containing only trace amounts of As or Sb. Figure 11 illustrates a  $\sim 5\ \mu\text{m}$  compact particle of iron arsenate as well as a  $\sim 10\ \mu\text{m}$  mass of  $(\text{As,Sb})_2\text{O}_3$ , both of which are associated with 1-3  $\mu\text{m}$  crystals of  $\text{As}_2\text{O}_3$ . Figure 12 illustrates a  $10\ \mu\text{m}$  zoned particle which was shown by EDX analysis to contain major amounts of Ca, As, S and O, as well as a trace amount of Fe. This phase is believed to be calcium arsenate-sulphate, and a number of such particles were identified in the April 97 Composite sample. The porous region of this figure is mounting medium.

In addition to the principal species discussed above, the detailed SEM-EDX studies also identified trace amounts of K-Al silicate and Na-Al silicate; these phases likely represent fragments of different feldspars which passed through the electrostatic precipitator circuit into the baghouse. A few particles of calcium oxide containing trace amounts of Fe, As and Sb were also noted.

Quantitative electron microprobe analyses were carried out on the principal As- and Sb-containing phases in the April 97 Composite sample. The detailed electron microprobe results are presented in Appendix 3.

Twenty-four particles of the  $\text{As}_2\text{O}_3$  phase were analyzed with the electron microprobe, and the results are summarized in Table 7. The material is relatively pure  $\text{As}_2\text{O}_3$  which contains only 0.23% Sb, in addition to trace amounts of Fe, Pb, Ca and S. The Sb content of the dominant  $\text{As}_2\text{O}_3$  phase in the April 97 Composite sample is lower than that of the  $\text{As}_2\text{O}_3$  crystals in the January 98 Composite sample, despite the higher total Sb content of the April 97 Composite sample.

Table 7 - Electron Microprobe Determined Composition of the  $\text{As}_2\text{O}_3$  Phase in the April 97 Composite Sample (wt %)

Element	Average Composition	Range
As	76.29	71.45 - 82.70
Sb	0.23	0.00 - 0.54
Fe	0.18	0.02 - 0.52
Pb	0.04	0.00 - 0.52
Ca	0.04	0.00 - 0.19
S	0.03	0.00 - 0.18
O	23.15	16.97 - 28.15

In addition to the "normal"  $\text{As}_2\text{O}_3$  crystals, the April 97 Composite sample contains a number of arsenic oxide particles having an elevated Sb content, but not containing enough Sb to constitute  $(\text{Sb,As})_2\text{O}_3$ . Eight of these Sb-rich  $\text{As}_2\text{O}_3$  particles were analyzed with the electron microprobe, and the results are given in Table 8. The average Sb content is 13.55%, a high enough concentration to warrant the differentiation of this phase from the "normal"  $\text{As}_2\text{O}_3$  crystals which contain only 0.23% Sb. A small amount of iron, along with traces of Pb, Ca, Bi and S, is also detected in this material.

Table 8 - Electron Microprobe Determined Composition of the Sb-rich  $\text{As}_2\text{O}_3$  Phase in the April 97 Composite Sample (wt %)

Element	Average Composition	Range
As	59.59	50.84 - 68.70
Sb	13.55	12.14 - 15.09
Fe	0.42	0.07 - 1.62
Pb	0.05	0.00 - 0.15
Ca	0.13	0.00 - 0.21
Bi	0.01	0.00 - 0.03
S	0.05	0.00 - 0.21
O	26.18	17.99 - 32.16

The April 97 Composite sample also contains an Sb-As oxide phase in which the amount of Sb equals or exceeds that of As; i.e., the phase has the general formula of  $(\text{Sb,As})_2\text{O}_3$ . Thirty-two particles of the  $(\text{Sb,As})_2\text{O}_3$  phase were

analyzed quantitatively with the electron microprobe, and the results are presented in Table 9. Comparison with the data of Table 4 suggests that the  $(\text{Sb,As})_2\text{O}_3$  phase in the April 97 Composite sample is very similar to that of the analogous phase in the January 98 Composite sample. In particular, the Sb and As contents of the  $(\text{Sb,As})_2\text{O}_3$  phase are virtually identical in the two samples.

Table 9 - Electron Microprobe Determined Composition of the  $(\text{Sb,As})_2\text{O}_3$  Phase in the April 97 Composite Sample (wt %)

Element	Average Composition	Range
Sb	44.55	42.43 - 46.72
As	35.94	30.71 - 42.45
Fe	0.14	0.00 - 0.84
Pb	0.06	0.00 - 0.34
Ca	0.01	0.00 - 0.11
S	0.84	0.50 - 1.23
O	18.44	11.40 - 25.25

The SEM-EDX studies identified both an iron arsenate phase and a calcium arsenate-sulphate phase having a high Sb content. Electron microprobe analyses of four grains of each phase were carried out, and the data are summarized in Table 10 and Table 11, respectively. The iron arsenate phase contains major amounts of both As and Sb, but has only a very minor Ca and S content. This phase is clearly an iron arsenate-antimonate compound, although As always exceeds Sb in the structure. In contrast, the calcium arsenate-sulphate phase seems to have a major Sb content, but contains only minor amounts of Fe. It would be more fully described as a calcium-iron sulphate-antimonate-arsenate.

Table 10 - Electron Microprobe Determined Composition of the Iron Arsenate Phase in the April 97 Composite Sample (wt %)

Element	Average Composition	Range
Fe	38.17	18.35 - 55.31
As	9.04	6.69 - 11.48
Sb	5.20	0.58 - 9.28
Pb	0.12	0.00 - 0.25
Ca	0.82	0.00 - 2.40
Bi	0.00	-
S	0.85	0.04 - 1.55
O	45.77	37.09 - 57.08

Table 11 - Electron Microprobe Determined Composition of the Calcium Arsenate-Sulphate Phase in the April 97 Composite Sample (wt %)

Element	Average Composition	Range
Ca	11.47	10.22 - 12.26
Fe	2.24	1.91 - 2.55
Sb	15.43	11.15 - 20.28
As	8.46	7.73 - 9.57
S	8.93	7.41 - 9.89
Pb	0.14	0.12 - 0.16
O	53.30	50.27 - 56.27

A few particles of calcium oxide were detected during the SEM-EDX study of this sample, and four particles of this phase were analyzed by the electron microprobe. The results are given in Table 12. The phase is mostly calcium oxide, although there is a modest level of Fe present. Only minor amounts of As and Sb are detected. The low As and Sb contents, coupled with the rarity of this phase, make the calcium oxide a relatively unimportant carrier of either As or Sb in this sample.

Table 12 - Electron Microprobe Determined Composition of the Calcium Oxide Phase in the April 97 Composite Sample (wt %)

Element	Average Composition	Range
Ca	19.08	17.68 - 20.33
Fe	5.60	5.29 - 6.05
As	0.93	0.24 - 1.85
Sb	0.41	0.25 - 0.57
Pb	0.00	
S	0.52	0.20 - 0.80
O	73.43	72.67 - 74.54

### **Characterization of the 212 Chamber Sample**

Table 2 indicates that the 212 Chamber sample has the highest Si, Al, Ca, Mg and Fe contents of any of the dusts studied. Accordingly, it would be expected that this sample contains more of the silicate impurity species. The sample also has the highest Sb content of any of the samples, and should, therefore, contain more Sb-rich arsenic oxide species.

Figure 13 illustrates the general appearance of the 212 Chamber sample as seen in a loose powder mount. Most of the sample consists of 1-3  $\mu\text{m}$  crystals of  $\text{As}_2\text{O}_3$ , and many of the crystals show the development of octahedral crystal faces. A number of somewhat larger silicate particles are present in this sample, and Figure 13 shows several such grains of chlorite and muscovite.

A detailed SEM-EDX study of the 212 Chamber sample was carried out using polished section mounts, and Figure 14 illustrates the general appearance of the sample. Most of the bright appearing grains in the photomicrograph are  $\text{As}_2\text{O}_3$  or  $\text{As}_2\text{O}_3$  having a modest Sb content. Most of the darker grey grains are chlorite or other silicate species, but some of these particles are As-bearing iron oxide or As-bearing calcium oxide containing minor amounts of Fe and traces of S and Si. Relative to the two more recently collected samples discussed above, the 212 Chamber sample seems to have a higher Ca content. Figure 15 provides another general view of the dust from the 212 Chamber. Although a number of tiny bright appearing crystals of  $\text{As}_2\text{O}_3$  are evident, there is also a large number of darker appearing particles. Various fragments of quartz and

chlorite are evident, as is the situation for all the samples studied. Numerous particles of iron arsenate occur, and these tend to be present as  $\sim 10 \mu\text{m}$  free grains. Of interest is a  $\sim 10 \mu\text{m}$  particle of As-bearing iron oxide that is rimmed by very bright  $(\text{Sb,As})_2\text{O}_3$ . A large grain of muscovite is rimmed by an Fe-Ca sulphate-arsenate phase. Relative to the initial two samples studied, there are more silicate and oxide phases present in the 212 Chamber sample, and several of these minerals seem to have undergone at least superficial alteration during roasting-gas cleaning.

Occasionally, large fragments of Sb-rich arsenic oxide or  $(\text{Sb,As})_2\text{O}_3$  are detected. Figure 16 illustrates one such occurrence. The bright appearing particle in the centre of the image has a high Sb content; it is likely  $(\text{Sb,As})_2\text{O}_3$ . The particle is  $20 \times 40 \mu\text{m}$  in size. Such large particles were evident in the samples studied above, but the mechanism of formation of such large grains in the baghouse circuit is unknown. In Figure 16, the  $(\text{Sb,As})_2\text{O}_3$  particle is associated with tiny light grey crystals of  $\text{As}_2\text{O}_3$  and slightly larger, and darker grey, particles of silicates and iron oxide or calcium oxide.

The 212 Chamber sample contains both iron arsenate and As-bearing iron oxide; the As-bearing iron oxide may be an intermediate phase in the formation of iron arsenate, although additional work would be required to substantiate this hypothesis. Figure 17 illustrates a large mass of iron arsenate, although smaller, typically  $\sim 10 \mu\text{m}$  particles, are more common. The large mass of iron arsenate seems to envelop smaller grains of  $\text{As}_2\text{O}_3$ , and the implication is that the iron arsenate mass "grew" in the baghouse system. In addition, there are two large grains of  $\text{CaSO}_4$  evident in the image, and these particles support the contention that the dust in the 212 Chamber is Ca-rich. Figure 18 illustrates a  $10 \times 12 \mu\text{m}$  particle of As-bearing iron oxide that may reflect an oxidized pyrite crystal. The grain is distinctly zoned and contains major Fe and O, with minor As and a trace quantity of Sb. Also shown is a smaller, dark appearing grain of  $\text{As}_2\text{O}_3$  containing minor amounts of Fe and Sb that is probably a reaction product between fine iron oxide particles and the  $\text{As}_2\text{O}_3$  crystals.

Quantitative electron microprobe analyses were carried out on the As- and Sb-bearing phases present in the 212 Chamber sample. The detailed analyses are presented in Appendix 4, and the results are summarized below.

Table 13 presents the average electron microprobe-determined composition of 31  $\text{As}_2\text{O}_3$  crystals present in the 212 Chamber sample. This oxide consists mostly of As, but contains a modest amount of Sb that is consistent with the bulk Sb content of this sample given in Table 2. In addition, there is a modest amount of Fe detected, as well as traces of Pb, Ca, Si and S; some of the detected impurity content could arise from secondary fluorescence from associated impurity phases. That is, the  $\text{As}_2\text{O}_3$  crystals are so small that penetration of the electron beam is likely.

Table 13 - Electron Microprobe Determined Composition of the  $\text{As}_2\text{O}_3$  Crystals in the 212 Chamber Sample (wt %)

Element	Average Composition	Range
As	76.01	70.24 - 82.42
Sb	1.19	0.08 - 3.52
Fe	0.56	0.25 - 1.72
Pb	0.03	0.00 - 0.22
Ca	0.05	0.00 - 0.30
Si	0.05	0.00 - 0.52
S	0.04	0.00 - 0.15
O	22.04	16.39 - 29.05

In addition to the "normal"  $\text{As}_2\text{O}_3$  crystals, which contain ~1% Sb, there are arsenic oxide particles which have elevated contents of Sb. In the January 98 Composite and April 97 Composite samples discussed above, the amount of Sb often exceeded that of As, such that the phase could be described as  $(\text{Sb,As})_2\text{O}_3$ . In the 212 Chamber sample, the Sb content of the arsenic oxide, although significant, generally does not exceed that of As. Consequently the Sb-rich oxide in the 212 Chamber sample is best described as  $(\text{As,Sb})_2\text{O}_3$ . Table 14 summarizes the electron microprobe data obtained on the  $(\text{As,Sb})_2\text{O}_3$  phase.

The material has a significant As and Sb content, and also contains trace amounts of Fe, Pb and S.

Table 14 - Electron Microprobe Determined Composition of the (As,Sb)<sub>2</sub>O<sub>3</sub> Phase in the 212 Chamber Sample (wt %)

Element	Average Composition	Range
As	50.46	42.57 - 56.72
Sb	24.44	20.93 - 31.10
Fe	0.33	0.12 - 1.49
Pb	0.03	0.00 - 0.11
S	0.01	0.00 - 0.02
O	24.71	17.14 - 27.82

The 212 Chamber sample contains numerous ~10  $\mu\text{m}$  particles of As-bearing iron oxide as well as an iron arsenate phase exhibiting a modest level of SO<sub>4</sub> substitution for AsO<sub>4</sub>. The electron microprobe-determined composition of the As-bearing iron oxide phase, as based on the analysis of 16 particles, is given in Table 15, from which it is apparent that modest amounts of both As and Sb are incorporated in the iron oxide. Other impurity elements, including S, are present only in trace amounts. Table 16 summarizes the analogous compositional data for the iron arsenate phase, as based on the analysis of 21 particles of this material. The iron arsenate phase has a significant content of both Fe and As, and also contains a minor amount of S, which is probably present as SO<sub>4</sub> replacing AsO<sub>4</sub> in the iron arsenate structure. Significantly, there are only trace amounts of Sb and other impurity elements in this material.

Table 15 - Electron Microprobe Determined Composition of the As-bearing Iron Oxide in the 212 Chamber Sample (wt %)

Element	Average Composition	Range
Fe	52.41	44.90 - 57.53
As	8.54	3.51 - 14.88
Sb	3.09	0.77 - 9.89
Pb	0.38	0.16 - 0.78
Ca	0.26	0.00 - 0.99
S	0.33	0.02 - 3.02
O	34.95	30.98 - 40.27

Table 16 - Electron Microprobe Determined Composition of the Iron Arsenate Phase in the 212 Chamber Sample (wt %)

Element	Average Composition	Range
Fe	12.60	11.34 - 13.21
As	31.67	26.19 - 37.33
Sb	0.49	0.38 - 0.73
Pb	0.72	0.50 - 1.10
Ca	0.86	0.64 - 3.46
S	1.58	1.12 - 2.21
O	51.99	45.84 - 57.46

The 212 Chamber sample contains a trace amount of an iron-calcium arsenate phase, and three particles of this compound were analyzed by the electron microprobe. As Table 17 indicates, this phase has a relatively low Sb content; it contains a minor amount of Pb as well as a trace of S.

Table 17 - Electron Microprobe Determined Composition of the Iron-Calcium Arsenate Phase in the 212 Chamber Sample (wt %)

Element	Average Composition	Range
Fe	6.62	5.08 - 9.59
Ca	3.03	2.03 - 4.12
As	35.21	29.93 - 41.34
Sb	1.09	1.75 - 2.05
Pb	2.55	2.30 - 3.00
S	0.48	0.08 - 1.30
O	50.17	47.39 - 52.37

A few particles of a Ca-Fe-As oxide phase were detected in the 212 Chamber sample, and three of the particles were analyzed by the electron microprobe. Table 18 summarizes the data obtained, and shows that this oxide consists mostly of Ca, Fe and As, together with trace amounts of Pb, Sb and S. The iron-calcium arsenate phase and the Ca-Fe-As oxide phase are rare constituents of the 212 Chamber sample, and accordingly, are relatively unimportant overall As carriers despite their high As contents.

Table 18 - Electron Microprobe Determined Composition of the Ca-Fe-As Oxide Phase in the 212 Chamber Sample (wt %)

Element	Average Composition	Range
Ca	13.93	8.39 - 18.65
Fe	8.75	8.03 - 9.93
As	10.75	1.38 - 20.76
Sb	0.53	0.06 - 0.82
Pb	0.24	0.06 - 0.34
S	0.50	0.04 - 1.08
O	65.26	60.29 - 69.85

#### Characterization of the 236 Chamber Sample

Unlike the other three  $\text{As}_2\text{O}_3$ -rich dusts supplied, the 236 Chamber sample was slightly moist when received, and the water content must affect its analysis to some extent. Nevertheless, the compositional data of Table 2 suggest that

the 236 Chamber sample is similar to that of the 212 Chamber material in that it has a comparatively low As content, together with high levels of Fe, Si, Al, Ca and Mg. The 236 Chamber sample has the lowest Sb content of the four samples studied. The implication is that this sample will contain an abundance of low-Sb  $\text{As}_2\text{O}_3$  crystals and numerous silicate and oxide particles.

Figure 19 shows the general morphology of the 236 Chamber sample as examined in a loose powder mount. The material consists of abundant  $\text{As}_2\text{O}_3$  crystals ranging in size from  $1\text{ }\mu\text{m}$  to over  $10\text{ }\mu\text{m}$ . The large size of some of the  $\text{As}_2\text{O}_3$  crystals differentiates this sample from the others studied. In many instances, the  $\text{As}_2\text{O}_3$  crystals have well developed external crystal faces, and the crystals are sometimes intergrown into small clusters. Various silicate species are detected in this sample, and Figure 19 shows a small particle of chlorite as well as a larger fragment of muscovite.

Figure 20 illustrates the general morphology of the 236 Chamber sample as seen in polished section. The bright particles are mostly crystals of  $\text{As}_2\text{O}_3$ , and these range in size from  $1\text{ }\mu\text{m}$  to  $10\text{ }\mu\text{m}$ . The darker appearing particles are mostly silicates, and these include quartz, chlorite and muscovite. Figure 21 shows two of the larger  $\text{As}_2\text{O}_3$  crystals in greater detail, and also illustrates a  $\sim 10\text{ }\mu\text{m}$  particle of As-bearing iron oxide. Most of the dark appearing particles in the image are quartz, chlorite and muscovite. The large  $\text{As}_2\text{O}_3$  crystals are somewhat porous and seem to be intergrowths of two or three individual crystals. The euhedral habit of the large  $\text{As}_2\text{O}_3$  crystals suggests that they developed directly in the vapour phase, as the gas from the roaster circuit was cooled with dilution air.

Figure 22 illustrates a particle of As-bearing iron oxide as well as grains of quartz, chlorite and muscovite. The SEM-EDX analyses of the quartz detected only Si and O. Complementary quantitative electron microprobe analyses of three quartz particles indicated  $\sim 40\%$  Si and  $\sim 59\%$  O; only traces of K, Fe, Mg, Ca and Al were detected, and these trace impurity contents could originate from secondary fluorescence from closely associated impurity phases. Similar SEM-EDX analyses of the chlorite phase indicated Fe, Mg, Al, Si and O, together with

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occasional traces of K. Electron microprobe analysis of nine particles of the chlorite detected 13-18% Fe, 7-11% Mg, 6-11% Al and 7-11% Si. The composition, together with the X-ray diffraction analyses, confirms that the chlorite mineral is chamosite. The SEM-EDX analyses of the muscovite indicated major amounts of K, Al, Si and O, together with trace quantities of Mg and Fe. Quantitative electron microprobe analyses of five grains of this mineral found 3-7% K, 10-17% Al and 13-21% Si. The compositional range, together with the X-ray diffraction results, confirm the presence of muscovite rather than a potassium feldspar.

Various other species were also detected in trace amounts during the SEM-EDX study of the 236 Chamber sample. These species include iron arsenate, calcium arsenate containing only trace Fe, K-Na-Mg-Al silicate, Ca-Mg-Al silicate, Na-K-Al silicate, iron oxide containing minor Cr and As, apatite and pyrite.

The principal As- and Sb-containing phases in the 236 Chamber sample were analyzed quantitatively with the electron microprobe. The individual analyses are given in Appendix 5, and the various results are summarized below.

Table 19 summarizes the electron microprobe analyses of 60 particles of  $\text{As}_2\text{O}_3$  in the 236 Chamber sample. The  $\text{As}_2\text{O}_3$  contains a major amount of As, but only a trace quantity of Sb. The low Sb content is consistent with low overall Sb content of this dust that is given in Table 2. There is also a trace amount of Fe present, but other impurities are nearly absent.

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Table 19 - Electron Microprobe Determined Composition of the  $\text{As}_2\text{O}_3$  Crystals in the 236 Chamber Sample (wt %)

Element	Average Composition	Range
As	76.78	71.43 - 79.87
Sb	0.19	0.00 - 0.99
Fe	0.27	0.08 - 1.13
Pb	0.02	0.00 - 0.13
Ca	0.01	0.00 - 0.09
Si	0.05	0.00 - 0.62
S	0.01	0.00 - 0.11
O	22.64	18.73 - 28.39

As noted above, a number of particles of As-bearing iron oxide are detected in the 236 Chamber sample, and 17 of these grains were analyzed quantitatively with the electron microprobe. Table 20 summarizes the results obtained. The iron oxide has a minor As content, but contains only a trace amount of Sb. Other impurity elements are detected only in low concentrations, but the presence of a small amount of Cr in the iron oxide in this sample is distinctive.

Table 20 - Electron Microprobe Determined Composition of the As-bearing Iron Oxide in the 236 Chamber Sample (wt %)

Element	Average Composition	Range
Fe	53.24	40.88-66.34
As	7.67	1.97 - 14.90
Sb	0.56	0.18 - 0.90
Pb	0.22	0.02 - 0.57
Ca	0.15	0.00 - 0.49
Cr	0.36	0.00 - 6.18
S	0.01	0.00 - 0.04
O	37.77	31.45 - 49.95

### Comparison of the Four As<sub>2</sub>O<sub>3</sub>-Rich Dust Samples

From a mineralogical point of view, the four As<sub>2</sub>O<sub>3</sub>-rich dust samples are similar in that they contain, with only a few minor exceptions, the same species and in approximately the same amounts. These species are indicated qualitatively in Table 21. The two more recent samples, the January 98 Composite and the April 97 Composite, have high As<sub>2</sub>O<sub>3</sub> contents and contain relatively small amounts of the impurity silicate phases. In contrast, the two older samples from the storage caverns, 212 Chamber and 236 Chamber, have relatively high silicate contents that possibly reflect less than optimum functioning of the electrostatic precipitators at the time of their production. The Sb contents of the dusts vary from 0.3 to 2.13% Sb, and this variation affects the Sb content of the As<sub>2</sub>O<sub>3</sub> crystals, as well as the amount and composition of the (Sb,As)<sub>2</sub>O<sub>3</sub> phase present. In all cases, the dominant As carrier is As<sub>2</sub>O<sub>3</sub>, which normally occurs as tiny euhedral crystals, but sometimes is present as large irregular masses. A minor amount of the As is also present as an Fe-bearing iron oxide or iron arsenate phase, in which the arsenic content varies from 1.9 to 38% As. The Ca arsenate or Ca-Fe arsenate phases are only minor As carriers in any of the dust samples. Most of the Sb in the samples is present in solid solution in the As<sub>2</sub>O<sub>3</sub> crystals. The Sb content of the As<sub>2</sub>O<sub>3</sub> varies from 0% to 47%. The more Sb-rich compositions are actually (Sb,As)<sub>2</sub>O<sub>3</sub>, and this phase often occurs as large irregular masses. A minor amount of the total Sb substitutes for As in the iron arsenate phase, and traces of Sb occur in the Ca arsenate, Ca-Fe arsenate and Fe arsenate-sulphate phases. Unlike As and Sb, most of the iron in the samples occurs in silicate form (chlorite) and as iron oxide. Lesser amounts of the total iron are present as iron arsenate or Ca-Fe arsenate, which contains 5-10% Fe.

Table 21 - Qualitative Comparison of the Phases Present in the Four  $\text{As}_2\text{O}_3$ -rich Dust Samples

Species	January 98 Composite	April 97 Composite	212 Chamber	236 Chamber
$\text{As}_2\text{O}_3$	major	major	major	major
Fe arsenate	minor	minor	minor	minor
$(\text{Sb,As})_2\text{O}_3$	minor	minor	minor	trace
Ca-Fe arsenate	trace	trace	minor	trace
Fe arsenate-sulphate	trace	trace	trace	very rare
Iron oxide	trace	trace	trace	trace
Ca-Fe oxide	very rare	trace	trace	very rare
Ca sulphate	very rare	very rare	trace	very rare
Chlorite	minor	minor	minor	minor
Quartz	trace	trace	trace	trace
Muscovite	minor	minor	minor	minor

### Solubility Measurements

The aqueous solubility of two lots of reagent grade  $\text{As}_2\text{O}_3$  was determined over the temperature range from 20°C to 100°C. The results obtained are presented graphically in Figure 23 and Figure 24, and the detailed numerical data are given in Appendix 6. For the first sample, the results obtained on heating and cooling are identical, and the implication is that the measured values reflect the true equilibrium solubility of  $\text{As}_2\text{O}_3$  in water. This conclusion is further supported by the close agreement of the current data with the published values of the solubility of  $\text{As}_2\text{O}_3$  in water reported by Linke and Seidell (2). The results obtained for the second lot of reagent grade  $\text{As}_2\text{O}_3$  are slightly higher at the higher temperatures and, for this sample, there is a slight difference between the heating and cooling data at the lower temperatures. This may indicate that the lower temperature values are not truly saturated, despite the fact that equilibration periods of 2-3 days were used. X-ray diffraction analysis of the solids in equilibrium with the solution at different temperatures indicated only the cubic arsenolite form of  $\text{As}_2\text{O}_3$ .

The valence state of the dissolved arsenic generated from the first lot of reagent grade  $\text{As}_2\text{O}_3$  was determined by ion chromatography, and the detailed results are also presented in Appendix 6. At all temperatures, and on heating

and cooling, only trace amounts of As(V) were detected. In fact, the concentration of As(V) never exceeded 0.1 g/L As(V), despite the fact that total arsenic concentrations as high as 63 g/L As were generated during the solubility measurements. Virtually all of the arsenic is present in the trivalent form; i.e., as  $\text{As}_2\text{O}_3$ , and the As(III) does not oxidize to any significant extent throughout the solubility measurements, which commonly lasted for over 30 days.

The solubility of reagent grade  $\text{Sb}_2\text{O}_3$  was also measured to provide a comparison with the analogous data for  $\text{As}_2\text{O}_3$ . The results for the solubility of  $\text{Sb}_2\text{O}_3$  in water are summarized graphically in Figure 25, and the numerical data are presented in Appendix 6. Unlike  $\text{As}_2\text{O}_3$  which enjoys a moderate solubility in water,  $\text{Sb}_2\text{O}_3$  is nearly insoluble over the entire temperature range from 25 to 95°C. The solubility at 25°C is <10 mg/L, and even at 95°C, it is only 55 mg/L  $\text{Sb}_2\text{O}_3$ . The data reveal a small difference between the solubilities measured on heating and those determined on cooling. Although the measured differences of <10 mg/L  $\text{Sb}_2\text{O}_3$  could be due to analytical error at the low concentrations involved, equally they could reflect the partial oxidation of Sb(III) to Sb(V) during the course of the experiments. In any event, it is clear that the solubility of  $\text{Sb}_2\text{O}_3$  is at least three orders of magnitude lower than that of  $\text{As}_2\text{O}_3$ . This conclusion is also supported by the limited solubility data published for  $\text{Sb}_2\text{O}_3$  in water. For example, Linke and Seidell (2) report that the solubility of  $\text{Sb}_2\text{O}_3$  is only 8.4 mg/L at 25°C, a value in general agreement with the results of this study. The inference is that the presence of even very modest amounts of  $\text{Sb}_2\text{O}_3$  in solid solution in  $\text{As}_2\text{O}_3$  will significantly reduce the solubility of the arsenic oxide.

The solubility of  $\text{As}_2\text{O}_3$  over the four  $\text{As}_2\text{O}_3$ -rich dust samples was measured as a function of temperature on heating and cooling. The results are displayed graphically in Figures 26, 27, 28 and 29, and the detailed numerical data are presented in Appendix 6. The graphs also present the measured solubility of As over reagent grade  $\text{As}_2\text{O}_3$  for comparative purposes.

In all instances, the solubility of  $\text{As}_2\text{O}_3$  over the dust, measured on heating, is much lower than that determined over reagent grade  $\text{As}_2\text{O}_3$ . In fact, the solubility of  $\text{As}_2\text{O}_3$  over the dust at any temperature during the heating cycle

is about one-half of that over reagent grade  $\text{As}_2\text{O}_3$ , despite the fact that a 2 ½ -3 day equilibration period was employed at each temperature. Furthermore, there is a pronounced hysteresis between the data obtained on heating and those realized on cooling. The solubility increases systematically as the temperature is increased from 5°C to ~95°C. As the temperature is reduced, however, the solubility of  $\text{As}_2\text{O}_3$  remains nearly constant until the solubility curve for reagent grade  $\text{As}_2\text{O}_3$  is intersected. As the temperature is reduced further, the solubility curve then follows that of the reagent grade  $\text{As}_2\text{O}_3$ .

The solubilities of Sb over the  $\text{As}_2\text{O}_3$ -rich dusts were also measured as a function of temperature on heating and cooling. The individual solubility curves for the four  $\text{As}_2\text{O}_3$ -rich dust samples are presented in Figures 30, 31, 32 and 33; the detailed solubility data are given in Appendix 6. The measured solubilities of Sb are low at all temperatures studied; even at 95°C, the solubility is always less than 160 mg/L Sb. In many instances, the solubility of Sb over the  $\text{As}_2\text{O}_3$ -rich dust is greater than that over reagent grade  $\text{Sb}_2\text{O}_3$ . One possible explanation is that part of the Sb in the  $\text{As}_2\text{O}_3$ -rich dust is present as Sb(IV) or Sb(V) rather than as Sb(III), which is present in  $\text{Sb}_2\text{O}_3$ . The solubility data on cooling are consistently higher than the values realized on heating, and this trend could indicate some oxidation of the Sb during the experiments, although it could equally well be caused by the slow crystallization of the low concentrations of dissolved Sb(III) throughout the cooling cycle.

Although the measured solubility of  $\text{As}_2\text{O}_3$  over reagent grade  $\text{As}_2\text{O}_3$  is similar to the accepted value reported in the literature (2), the solubility of  $\text{As}_2\text{O}_3$  over the four  $\text{As}_2\text{O}_3$ -rich dust samples is much lower than that expected from the bulk composition of the dusts and their abundance of  $\text{As}_2\text{O}_3$ . One possible cause of the lower than expected solubilities could be a very slow rate of dissolution of  $\text{As}_2\text{O}_3$  from the dusts. To pursue this possibility, the solubility of reagent grade  $\text{As}_2\text{O}_3$  was measured as a function of time at 25°C and 60°C, and that of the 212 Chamber sample was measured at 95°C. The results are summarized in Figures 34 and 35, respectively. Reagent grade  $\text{As}_2\text{O}_3$  dissolves slowly, and at 25°C, ~70 h (3 days) is needed to achieve the equilibrium

solubility. At 60°C, the time to reach saturation is about 24 h. The  $\text{As}_2\text{O}_3$  dissolves relatively rapidly from the 212 Chamber sample at 95°C. Only about 8 h is needed to reach a steady state  $\text{As}_2\text{O}_3$  concentration, which does not vary significantly for more extended heating times up to 170 h. As indicated by Figure 28, however, the solubility realized over the 212 Chamber sample is not that of pure  $\text{As}_2\text{O}_3$ , despite the constancy of the value. Because equilibration times of 2 ½-3 days were used in the present experiments, and even longer times were employed at temperatures lower than 30°C, the duration of the equilibration period at each temperature is not likely the cause of the low  $\text{As}_2\text{O}_3$  solubilities from the  $\text{As}_2\text{O}_3$ -rich dust samples.

Additional support for this conclusion is provided by the data given in Table 22 that show the concentrations of As(III) and As(V) in the solutions in contact with the four  $\text{As}_2\text{O}_3$ -rich dust samples as a function of time. Clearly, the As concentration (as reflected by the sum of As(III) and As(V)) does not increase to any significant extent with increasing equilibration times, even for periods as long as 21 days.

Another possible cause of the lower solubility of arsenic from the  $\text{As}_2\text{O}_3$ -rich dust samples could be an enhanced rate of oxidation of As(III) in the presence of the diverse solid species in the dust. To evaluate this possibility, the  $\text{As}_2\text{O}_3$ -rich dust samples were equilibrated with water at room temperature for times up to 21 days, about the total duration of the individual solubility tests. Samples were taken periodically and the solutions were analyzed for As(III) and As(V) using liquid chromatography. The results, which are summarized in Table 22, indicate that the concentration of As(V) is always less than 5% of the total As concentration, and that the As(V) concentration does not increase in any significant way with increasing retention time in the presence of water, air and the various solid phases present in the dust. Clearly, the oxidation of the dissolved arsenic is not the cause of the lower than expected  $\text{As}_2\text{O}_3$  solubilities from the  $\text{As}_2\text{O}_3$ -rich dust samples.

Table 22 - Relative Concentrations of As(III) and As(V) in the Saturated Solutions Generated From the As<sub>2</sub>O<sub>3</sub>-rich Dust Samples

Sample	Temperature (°C)	Time (days)	As(III) (g/L)	As(V) (g/L)
January 98 Composite	26.3	7	9.02	0.07
		9	9.06	0.07
		21	9.55	0.08
April 97 Composite	26.4	7	13.7	0.07
		9	13.7	0.07
		21	13.8	0.09
212 Chamber	23.4	7	9.13	0.30
		9	9.09	0.30
		21	9.48	0.32
236 Chamber	26.6	7	13.9	0.33
		9	13.9	0.32
		21	14.3	0.34

The above discussion suggests that the low As<sub>2</sub>O<sub>3</sub> solubility from the As<sub>2</sub>O<sub>3</sub>-rich dust samples is an inherent property of the dust itself, and is not a consequence of the measuring technique. Further support for this hypothesis comes from Figure 36 which compares the As<sub>2</sub>O<sub>3</sub> solubilities over the four As<sub>2</sub>O<sub>3</sub>-rich dust samples, as obtained on heating only. Although all the solubilities are low relative to that of reagent grade As<sub>2</sub>O<sub>3</sub>, there are significant solubility variations among the four As<sub>2</sub>O<sub>3</sub>-rich dust samples themselves. There is some indication that the observed solubility differences are related to the total Sb content of the dust, and possibly, to the solid solution Sb content of the As<sub>2</sub>O<sub>3</sub> crystals present in the dust. In this regard, Figure 37 shows the As<sub>2</sub>O<sub>3</sub> solubility at 65°C and 95°C as a function of the total Sb content of the dust for the four As<sub>2</sub>O<sub>3</sub>-rich dust samples (Table 2), as well as for reagent grade As<sub>2</sub>O<sub>3</sub>. These solubilities were calculated by fitting the experimental solubility data obtained on heating to a second order polynomial equation of the form:

$$\text{Solubility (g/L As}_2\text{O}_3) = a + bt + ct^2 \quad (1)$$

where  $t$  is the temperature in  $^{\circ}\text{C}$ . The relevant equations are given below.

$$\text{Reagent Grade As}_2\text{O}_3: S = 14.78 + 0.0626 t + 0.00649 t^2 \quad (2)$$

$$\text{January 98 Composite: } S = 5.41 + 0.1094 t + 0.00223 t^2 \quad (3)$$

$$\text{April 97 Composite: } S = 6.12 + 0.2012 t + 0.00123 t^2 \quad (4)$$

$$\text{212 Chamber: } S = 6.31 + 0.0888 t + 0.00167 t^2 \quad (5)$$

$$\text{236 Chamber: } S = 9.77 + 0.2414 t + 0.00151 t^2 \quad (6)$$

The data given in Figure 37 suggest that the solubility of  $\text{As}_2\text{O}_3$  is related to the Sb content, and that small amounts of Sb have a significant effect on the solubility of  $\text{As}_2\text{O}_3$ . In this regard, a similar trend is observed when the solubility of  $\text{As}_2\text{O}_3$  is plotted against the solid solution Sb content of the  $\text{As}_2\text{O}_3$  crystals in the four  $\text{As}_2\text{O}_3$ -rich dust samples (see Figure 38). Presumably, the presence of Sb in the  $\text{As}_2\text{O}_3$  crystals causes a significant reduction in the thermodynamic activity of  $\text{As}_2\text{O}_3$  that, in turn, results in a reduction in the aqueous solubility of the oxide. Based on the data presented in Figures 37 and 38, the effect is most pronounced for Sb contents in the 0.0 - 0.5% Sb range, but more experimentation would be required to substantiate this hypothesis.

The mineralogical characterization studies of the four  $\text{As}_2\text{O}_3$ -rich dust samples showed that the dominant  $\text{As}_2\text{O}_3$  crystal phase has solid solution Sb contents ranging from zero to several percent. Based on the hypothesis that only the low-Sb  $\text{As}_2\text{O}_3$  crystals dissolve readily, the addition of greater amounts of solid  $\text{As}_2\text{O}_3$ -rich dust should increase the measured solubility of  $\text{As}_2\text{O}_3$  over the dust. That is, the addition of more dust to the solubility vessel consequentially results in the addition of more low-Sb  $\text{As}_2\text{O}_3$  to the system. The low-Sb  $\text{As}_2\text{O}_3$  is postulated to dissolve readily, and should, therefore yield higher  $\text{As}_2\text{O}_3$  solubilities over the dust. To test this hypothesis, various amounts of the April 97 Composite sample were added to 1L of water heated to  $70^{\circ}\text{C}$  in a 2-L solubility reactor (see Figure 1). After 96 h of agitation at  $70^{\circ}\text{C}$ , the solution was sampled, diluted and analyzed for As and Sb. The results of this part of the investigation are summarized in Table 23. When an initial pulp density of 75 g/L solids was

employed, the measured solubility over the April 97 Composite sample was 41.55 g/L  $\text{As}_2\text{O}_3$ . It is important to recognize that, based on the overall composition of the April 97 Composite sample presented in Table 2 and assuming that all of the contained As is present as  $\text{As}_2\text{O}_3$ , an initial pulp density of 75 g/L corresponds to 130% of the  $\text{As}_2\text{O}_3$  required to achieve the ~51 g/L  $\text{As}_2\text{O}_3$  solubility of reagent grade  $\text{As}_2\text{O}_3$  at 70°C. Increasing the pulp density to 150 g/L solids yields a solubility of 48.03 g/L  $\text{As}_2\text{O}_3$ , and a pulp density of 300 g/L gives a solubility of 51.76 g/L  $\text{As}_2\text{O}_3$ . The latter value is essentially that of reagent grade  $\text{As}_2\text{O}_3$  at 70°C (50.96 g/L  $\text{As}_2\text{O}_3$ ). The implication is that a large excess of the solid  $\text{As}_2\text{O}_3$ -rich dust will result in an  $\text{As}_2\text{O}_3$ -saturated solution, even during the heating cycle. In contrast, the Sb concentration of the solution appears to be nearly independent of the pulp density, and this implies the rapid saturation of all the solutions with respect to Sb.

Table 23 - The Aqueous Solubility of  $\text{As}_2\text{O}_3$  and Sb at 70°C for Various Pulp Densities of the April 96 Composite Sample

Pulp Density (g/L)	Solubility of $\text{As}_2\text{O}_3$ (g/L)	Solubility of Sb (mg/L)
75	41.55	41
150	48.03	37
225	48.87	34
300	51.76	37
Reagent Grade $\text{As}_2\text{O}_3$	50.96	-

Support for the hypothesis that only the low-Sb  $\text{As}_2\text{O}_3$  crystals dissolve readily at temperatures below 100°C comes from the mineralogical characterization of the leach residues from the above experiments done at various pulp densities. Figure 39 illustrates the general morphology of the leach residue from the test done using an initial pulp density of 75 g/L, and Figure 40 shows the morphology of the analogous residue from the experiment done using an initial pulp density of 150 g/L of the April 97 Composite sample. Both residues consist of tiny particles of  $\text{As}_2\text{O}_3$  having a low but detectable Sb content,

and of larger bright appearing particles of  $(\text{Sb,As})_2\text{O}_3$ . The darker appearing grains are chlorite, muscovite, quartz, iron oxide and iron arsenate. Relative to the unleached material (see Figures 9 and 11), the  $\text{As}_2\text{O}_3$  crystals in the water leached residues are significantly reduced in size. This reduction in particle size is believed to result from the rapid dissolution of the low-Sb  $\text{As}_2\text{O}_3$  crystals. In contrast, the  $(\text{Sb,As})_2\text{O}_3$  particles appear to have the same size and morphology as they possessed in the original sample. Of course, water leaching does not affect the morphology of the silicate species.

Quantitative electron microprobe analyses of the  $\text{As}_2\text{O}_3$  crystals remaining in the water leach residues were carried out. All the data are presented in Appendix 7, and the results are summarized in Table 24. The  $\text{As}_2\text{O}_3$  crystals in the as-received April 97 Composite sample have a solid solution Sb content of 0.238%. Leaching of this sample at 70°C and at a pulp density of 150 g/L solids (i.e., with a ~250% excess of the  $\text{As}_2\text{O}_3$ -rich dust), results in an increase in the average solid solution Sb content of the  $\text{As}_2\text{O}_3$  crystals to 0.819%. When a pulp density of 75 g/L solids was employed, the residual  $\text{As}_2\text{O}_3$  crystals have an average Sb content of 1.64%. The implication is that the low-Sb  $\text{As}_2\text{O}_3$  crystals dissolve readily. As the dissolution of the  $\text{As}_2\text{O}_3$  proceeds, the Sb content rises in the residual particles and, eventually, the Sb content impedes the dissolution reaction. This trend is also reflected in the expanded range of the Sb contents of the  $\text{As}_2\text{O}_3$  crystals in the leach residues, especially that from the 75 g/L pulp density experiment.

Table 24 - Electron Microprobe Determined Composition of the  $\text{As}_2\text{O}_3$  Crystals After Leaching at Various Pulp Densities and 70°C (wt %)

Condition	As Content	Range	Sb Content	Range
April 97 Composite sample	76.29	71.45-82.70	0.238	0.000-0.545
150 g/L Solids Leach	74.63	70.48-83.33	0.819	0.122-4.254
75 g/L Solids Leach	74.77	70.20-81.82	1.648	0.279-7.445

#### **High Temperature Leaching of the $\text{As}_2\text{O}_3$ -Rich Dusts**

The experiments presented above indicate that the  $\text{As}_2\text{O}_3$ -rich dusts from the Giant Mine do not dissolve extensively in water at temperatures <100°C. As a result, the concentration of  $\text{As}_2\text{O}_3$  in solution generally does not exceed 50 g/L  $\text{As}_2\text{O}_3$ , a value significantly lower than the ~90 g/L  $\text{As}_2\text{O}_3$  concentration realized at 95-100°C for reagent grade  $\text{As}_2\text{O}_3$ . Although the concentration of dissolved arsenic over the four  $\text{As}_2\text{O}_3$ -rich dusts is relatively low, it increases with increasing temperature. Accordingly, the use of temperatures >100°C should generate a more concentrated  $\text{As}_2\text{O}_3$  solution, and this option was investigated using an autoclave at 125-200°C.

Table 25 shows the  $\text{As}_2\text{O}_3$  aqueous solubilities achieved when ~100 g/L of each of the four  $\text{As}_2\text{O}_3$ -rich dusts was heated in water in an autoclave for 2 h at 125, 150, 175 and 200°C, followed by cooling to 95°C and sampling. Heating the  $\text{As}_2\text{O}_3$ -rich dusts to 125-200°C results in a significant increase in the  $\text{As}_2\text{O}_3$  solubility at 95°C, and in this regard, it should be recalled that the apparent solubility achieved by heating any of the dusts directly to 95°C is only ~45 g/L  $\text{As}_2\text{O}_3$ . Heating of the  $\text{As}_2\text{O}_3$ -rich dusts to 125°C, followed by cooling to 95°C, results in a significantly enhanced solubility relative to the solubility values realized by directly heating the slurries to 95°C. Heating to a higher temperature

results in a further  $\text{As}_2\text{O}_3$  solubility increase for the April 97 Composite and the 212 Chamber samples, but has little effect on the solubility of the January 98 Composite or 236 Chamber samples. All of the dusts yield  $\text{As}_2\text{O}_3$ -saturated solutions at 95°C, after heating to 175°C or 200°C. Furthermore, the 212 Chamber sample seems to reach saturation at 150°C, and both the January 98 Composite and 236 Chamber samples give  $\text{As}_2\text{O}_3$ -saturated solutions after heating to only 125°C.

Table 25 – Solubility of  $\text{As}_2\text{O}_3$  in Water at 95°C After a Two-hour Heating Period at Elevated Temperatures in an Autoclave (g/L  $\text{As}_2\text{O}_3$ )

Sample	Heating Temperature			
	125°C	150°C	175°C	200°C
January 98 Composite	87.1	79.7	80.9	83.8
April 97 Composite	73.9	73.9	82.6	87.1
212 Chamber	67.4	81.2	80.6	80.2
236 Chamber	85.1	84.9	80.8	81.6

Based on the data given in Table 25, the procedure outlined in Figure 41 was developed to produce a significant yield of "pure"  $\text{As}_2\text{O}_3$  crystals by hot water leaching of the  $\text{As}_2\text{O}_3$ -rich dusts. The procedure consists of leaching the  $\text{As}_2\text{O}_3$ -rich dusts in water, in an autoclave, for 2 h at 150°C and under the "neutral" atmosphere generated by the air in the vessel plus the significant steam pressure produced at the leaching temperature. After 2 h of leaching at the elevated temperature, the slurry was cooled to 95°C, and was filtered while hot. The clear,  $\text{As}_2\text{O}_3$ -saturated liquor was then cooled to room temperature to crystallize  $\text{As}_2\text{O}_3$ . An autoclave temperature of 150°C was employed, but it is possible that further test work could reduce this temperature somewhat, at least for some of the  $\text{As}_2\text{O}_3$ -rich dust samples. Also, additional refinement of the

procedure possibly could shorten the time needed to achieve  $\text{As}_2\text{O}_3$  saturation at the elevated temperatures used.

Table 26 summarizes the results obtained when the four  $\text{As}_2\text{O}_3$ -rich dust samples were leached at  $150^\circ\text{C}$  under the conditions given in Figure 41 and at various pulp densities. For any of the samples, decreasing the initial pulp density results in a higher dissolution of  $\text{As}_2\text{O}_3$  from the dust, and a lower As content in the leach residue. The maximum extraction of 93% was achieved using 48 g/L of the January 98 Composite sample, and the lowest extraction of 75% was obtained when 91 g/L of the 212 Chamber sample was treated. For any of the samples, a relatively constant level of  $\text{As}_2\text{O}_3$  dissolution is achieved for pulp densities up to  $\sim 80$  g/L, but the extractions decrease at still higher pulp densities. Clearly, the decreasing solubility reflects the point at which an  $\text{As}_2\text{O}_3$ -saturated solution is produced; any additional  $\text{As}_2\text{O}_3$ -rich dust simply accumulates in the leach residue, thereby lowering the total percentage of  $\text{As}_2\text{O}_3$  dissolved. Impurities, such as Fe, Sb and Au accumulate in the residue as the  $\text{As}_2\text{O}_3$  dissolves, and this observation is consistent with the low water solubility of these species. The Au content of most of the leach residues is commercially interesting.

Following filtration at  $\sim 95^\circ\text{C}$ , the clear filtrates were cooled to room temperature, while being gently stirred, to crystallize a pure  $\text{As}_2\text{O}_3$  product. The resulting crystals were filtered and then dried in an oven at  $110^\circ\text{C}$ . The crystals were weighed to give the yield of product, and were analyzed for As, Sb and Fe. As shown in Table 27, the yield of product depends on the degree of saturation at  $95^\circ\text{C}$ , which in turn depends on the pulp density employed in the initial leaching stage at  $150^\circ\text{C}$ . The experiments done using high pulp densities produce nearly saturated solutions at  $95^\circ\text{C}$ , and in turn, these result in high yields of the  $\text{As}_2\text{O}_3$  product. Comparison with Table 26, however, shows that the tests done at high pulp densities also result in relatively low overall levels of dust dissolution. In practice, it will be necessary to strike a compromise between the pulp density needed to give a high level of dust dissolution and that required to

produce a highly concentrated  $\text{As}_2\text{O}_3$  solution at  $95^\circ\text{C}$ . Because, in any commercial process, the cold  $\text{As}_2\text{O}_3$ -bearing solution from the crystallization of the  $\text{As}_2\text{O}_3$  product would likely be recycled as "water" to the pressure leaching stage, the optimum pulp density should be re-determined using recycled  $\text{As}_2\text{O}_3$ -bearing solutions.

Table 26 – Results Obtained by Leaching the  $\text{As}_2\text{O}_3$ -rich Dusts for 2 h at  $150^\circ\text{C}$ , Followed by Cooling and Filtration at  $\sim 95^\circ\text{C}$

Sample	Pulp Density (g/L)	Dust Dissolved (%)	Residue Composition (%)			
			As	Sb	Fe	Au (ppm)
January	94	85.7	34.5	3.3	10.1	25.9
98	87	88.5	23.7	6.4	12.4	37.6
Composite	79	91.2	14.5	3.8	15.8	40.1
	66	91.3	14.1	4.7	16.2	39.2
	57	91.2	12.4	5.0	16.3	40.1
	48	93.0	9.4	3.0	17.1	43.4
April 97	78	91.1	25.1	15.1	9.4	29.9
Composite	71	91.7	23.7	13.8	9.7	32.0
212	102	77.4	21.4	7.4	14.2	54.7
Chamber	91	75.5	18.7	7.5	15.0	56.6
236	91	88.4	11.6	1.3	17.7	57.4
Chamber	83	89.0	8.4	1.1	18.0	43.9

The  $\text{As}_2\text{O}_3$  crystals produced on cooling were shown by X-ray diffraction analysis to consist only of the cubic arsenolite form of  $\text{As}_2\text{O}_3$ ; no other phases were detected. The  $\text{As}_2\text{O}_3$  was generally of high chemical purity; in many cases the product consisted of  $>99\%$   $\text{As}_2\text{O}_3$ , and "water" is likely the major impurity as the crystals were dried at only  $60^\circ\text{C}$ . Contamination of the  $\text{As}_2\text{O}_3$  with Sb or Fe was generally low, except for those experiments where the leach solution was not saturated with  $\text{As}_2\text{O}_3$ . In many of the experiments, the Sb content was  $<0.2\%$  and the Fe content was  $<0.005\%$ . Hence, it is believed that the high temperature leaching procedure outlined in Figure 41 could yield a commercially acceptable  $\text{As}_2\text{O}_3$  product.

Table 27 – Arsenic Concentrations and the Purity of the  $\text{As}_2\text{O}_3$  Crystals Made Using the High Temperature Leaching Procedure

Sample	Pulp	[As]	[As]	Yield	$\text{As}_2\text{O}_3$	Crystals	
	Density (g/L)	at 95°C (g/L)	at 25°C (g/L)		$\text{As}_2\text{O}_3$ (%)	Sb (%)	Fe (%)
January 98 Composite	94	59.7	17.4	57.7	98.3	0.06	0.003
	87	59.0	21.0	52.5	95.8	0.03	0.005
	79	54.9	18.5	48.9	99.9	0.07	0.002
	66	46.0	17.7	37.4	99.1	0.16	0.001
	57	39.2	16.7	30.6	99.5	0.28	0.002
	48	28.5	22.0	9.7	97.9	0.97	0.005
April 97 Composite	91	62.8	21.5	51.1	98.7	0.11	0.008
	83	56.5	19.3	47.3	97.7	0.13	0.008
212 Chamber	102	55.8	19.4	47.5	98.3	0.06	0.008
	91	49.5	16.8	47.4	99.0	0.10	0.008
236 Chamber	78	61.0	18.6	50.9	97.7	0.18	0.004
	71	50.2	16.6	44.6	99.0	0.22	0.002

In the hot water leaching procedure illustrated in Figure 41, the arsenic is distributed among the  $\text{As}_2\text{O}_3$  crystals, the high temperature leach residue and the cold solution. Mass balances were carried out to determine the relative distribution of arsenic among the three phases, and the results are given in Table 28. Only 1-9% of the total arsenic remains in the high temperature leach residue, and the amount of residual arsenic increases as the pulp density increases. For the preferred range of pulp densities likely to be used in a commercial process, however, the amount of residual arsenic seems to be only 2-4% of the total. Under the same preferred pulp density conditions, about 25-30% of the total arsenic remains in the cold solution. In a commercial application, however, this solution would almost certainly be recycled as "water" for the high temperature leaching operation. Hence, negligible amounts of the total arsenic leached would report to the cold solution in a continuous operation. Accordingly, it can be concluded that 96-98% of the total arsenic would report as

As<sub>2</sub>O<sub>3</sub> crystals in a commercial hot water leaching process wherein the cold solution after As<sub>2</sub>O<sub>3</sub> crystallization was recycled.

Table 28 – Relative Arsenic Distribution in the Hot Water Leaching Procedure

Sample	Pulp Density (g/L)	Relative Arsenic Distribution		
		In Leach Residue (%)	In Cold Solution (%)	In As <sub>2</sub> O <sub>3</sub> Crystals (%)
January 98 Composite	94	7.3	25.3	67.4
	87	3.9	32.4	63.7
	79	1.8	30.9	67.3
	66	1.8	36.2	62.0
	57	1.6	39.8	58.6
	48	1.0	64.8	34.2
April 97 Composite	78	2.9	32.7	64.3
	71	2.6	32.8	64.6
212 Chamber	102	8.5	30.9	60.6
	91	7.5	28.1	64.4
236 Chamber	91	2.2	27.0	70.8
	83	1.5	30.8	67.6

#### **Mineralogical Characterization of the Hot Water Leach Residues**

Samples of the four As<sub>2</sub>O<sub>3</sub>-rich dusts were leached for 2 h at 150°C, at various pulp densities, as noted above. The slurry was rapidly cooled in the autoclave to 95-100°C, and the vessel was then opened. The hot slurry was filtered, and the solids were washed with a small amount of water to displace the nearly As<sub>2</sub>O<sub>3</sub>-saturated processing solution. The washed solids then were dried at 110°C for a few hours prior to mineralogical study.

X-ray diffraction analysis of the residue from the leaching of the January 98 Composite sample, obtained using an initial pulp density of 95 g/L solids, indicated an As<sub>2</sub>O<sub>3</sub>-type compound, quartz, chlorite, muscovite and traces of other unidentifiable species. Similar X-ray diffraction analyses of the April 97 Composite sample leached at a pulp density of 77 g/L or 85 g/L solids detected

an  $\text{As}_2\text{O}_3$ -type compound, quartz, chlorite, muscovite, hematite (maghemite?) and other unidentifiable phases.

The SEM-EDX studies of the latter two samples provided complementary mineralogical information and phase confirmation. Figure 42 shows the residue from the leaching experiment done using a pulp density of 77 g/L solids. The bright appearing grains are rich in both Sb and As; they are likely  $(\text{Sb,As})_2\text{O}_3$ , which is the  $\text{As}_2\text{O}_3$ -type compound identified by X-ray diffraction analysis. Relative to the  $(\text{Sb,As})_2\text{O}_3$  particles in the as-received  $\text{As}_2\text{O}_3$ -rich dust, the  $(\text{Sb,As})_2\text{O}_3$  particles in the leach residue are better crystallized. In fact, many of the particles show well developed external crystal forms, possibly indicating the localized dissolution-crystallization of the  $(\text{Sb,As})_2\text{O}_3$  phase. The low-Sb  $\text{As}_2\text{O}_3$  crystals prevalent in the as-received  $\text{As}_2\text{O}_3$ -rich dust are lacking, and this behaviour is not unexpected given the low pulp density used for this leaching experiment. The various dark appearing grains are mostly chlorite, muscovite or quartz, although a number of fragments of As-bearing iron oxide were also detected. Figure 43 shows the analogous leach residue from the experiment done using a pulp density of 85 g/L solids; the leach residue contains 25.1% As, 15.1% Sb and 9.4% Fe. Most of the bright appearing particles are crystals of  $(\text{Sb,As})_2\text{O}_3$ , whereas the darker appearing particles are chlorite, muscovite, quartz and As-bearing iron oxide. Both figures suggest that the low-Sb  $\text{As}_2\text{O}_3$  crystals dissolve during hot water leaching. In contrast, the Sb-rich oxides, iron oxides and silicate species are poorly soluble and accumulate in the leach residue.

X-ray diffraction analyses were also carried out on the residues from the water leaching of the 212 Chamber sample for 2 h at 150°C. The residues were generated for pulp densities ranging from 100 to 113 g/L, but all the residues were virtually identical. They consist of an  $\text{As}_2\text{O}_3$ -type compound (likely  $(\text{Sb,As})_2\text{O}_3$ ), chlorite, muscovite, quartz and iron oxide (hematite and maghemite(?)).

### Removal of Antimony From the Water Leach Solutions

Preliminary research on the water leaching option was carried out by Royal Oak Mines. In their studies, leaching was done at 100°C, and it is now known that such temperatures are inadequate to saturate the water with  $\text{As}_2\text{O}_3$ . In the Royal Oak work, the solution was evaporated to ~30% of its volume to increase the concentration of  $\text{As}_2\text{O}_3$ , and the evaporated solution was cooled to crystallize  $\text{As}_2\text{O}_3$ . In the Royal Oak work, the crystals contained 99.6%  $\text{As}_2\text{O}_3$ , 0.33 % Sb and 0.08% Fe. The Sb content was considered to be excessive, and accordingly, tests were done to remove the Sb from solution using either ion exchange or activated carbon. Those experiments, however, were inconclusive, and accordingly, additional work was done at CANMET on the use of ion exchange resins or activated carbon for antimony control.

Table 29 illustrates the composition of a solution which was made by leaching 100 g/L of the 212 Chamber sample with hot water at 100°C. The hot slurry was filtered, and the filtrate was analyzed. The solution was then cooled to room temperature and was held at room temperature for 72 h. The Sb concentration at 90°C is about that expected from the measured solubility data (Figure 32), but the solubility decreases significantly as the temperature is lowered. The measured concentration of Sb at 25°C is about three times greater than the reported solubility of antimony at 25°C which is 0.0084 g/L  $\text{Sb}_2\text{O}_3$  (5). It is not known whether this discrepancy is due to the presence of some Sb(V) in the 212 Chamber sample, or whether it reflects a persistent level of  $\text{Sb}_2\text{O}_3$  supersaturation in water. The latter explanation may be more likely as it was noted that the  $\text{Sb}_2\text{O}_3$  tended to remain in solution, in a supersaturated state, for several hours when the solution was cooled from 90°C to 25°C.

Table 29 - Composition of the Solution Resulting From the Hot Water Leaching of the 212 Chamber Sample

Time (h)	Temperature (°C)	As Concentration (g/L)	Sb Concentration (g/L)
0	90	24.5	0.0643
24	25	21.0	0.0272
72	25	21.4	0.0263

The data of Table 30 show the Sb concentrations resulting when the  $\text{As}_2\text{O}_3$ -rich dust samples were subjected to the hot water leaching procedure (Figure 41) for 2 h at 150°C, and were then cooled to 95°C and subsequently to room temperature. The Sb concentrations in the 95°C solutions vary widely, and seem to depend on both the dust sample used and its pulp density. There is an inverse correlation between the Sb and As concentrations of the hot solution, although the correlation is not precise. The Sb concentration of the solutions cooled to 25°C varies widely, from 0.029 to 0.194 g/L Sb. This observation could reflect the presence of variable amounts of Sb(V) in the dust samples, or more likely, an erratic level of  $\text{Sb}_2\text{O}_3$  supersaturation in the cooled solutions. In any event, a modest amount of Sb dissolves along with the  $\text{As}_2\text{O}_3$ , and the Sb persists in solution at room temperature.

Table 30 - Antimony Concentrations Produced in the Hot Water Leaching Procedure

Sample	Pulp Density (g/L)	Sb Concentration at 95°C (g/L)	As Concentration at 95°C (g/L)	Sb Concentration at 25°C (g/L)
January 98 Composite	94	0.075	59.7	0.030
	87	0.094	59.0	0.093
	79	0.113	54.9	0.081
	66	0.139	46.0	0.086
	57	0.154	39.2	0.080
	48	0.130	28.5	0.048
April 97 Composite	78	0.130	-	0.075
	71	0.118	-	0.068
212 Chamber	102	0.191	-	0.181
	91	0.171	-	0.194
236 Chamber	91	0.121	-	0.029
	83	0.134	-	0.049

Although Royal Oak Mines reported some success in removing Sb from water leach solutions using Gravex GR-2-0 ion exchange resin or activated carbon, their results might simply reflect the delayed crystallization of the antimony on the surface of the resin or carbon during the cooling of the solutions in their experiments. In an effort to confirm the preliminary results obtained by Royal Oak Mines, the removal of Sb was studied using the four ion exchange resins presented in Table 1 as well as Hycarb 6x12 activated carbon. As shown in Table 31, however, none of the resins extracted any significant amount of Sb from the water leach solutions at room temperature and their natural pH of ~4. For these experiments, the 212 Chamber sample was leached at 95°C in water for several hours to generate an As- and Sb-bearing solution. The leach slurry was filtered and then was cooled to room temperature for ~24 h. The cooled solution analyzed 21 g/L As and 0.0263 g/L Sb. One hundred millilitre samples of the cooled solution were contacted with 2 mL of the various resins, and the mixtures were gently agitated overnight at room temperature using an orbital

shaker. The solutions were then analyzed for As and Sb; the results are summarized in Table 31. None of the resins is effective for extracting Sb from the water leach solution; less than 10% of the total Sb was removed in any of the experiments.

Table 31 - Concentrations of As and Sb After Prolonged Contact With Different Ion Exchange Resins

Resin	As Concentration (g/L)	Sb Concentration (g/L)
Feed solution	21	0.0263
Gravex GR-2-0	21	0.0247
Amberlite IRC-120	21	0.0266
Amberlite IRC-50	21	0.0024
Duolite C-467	21	0.0232

Efforts were also made to extract the Sb from a hot aqueous leach solution. For these experiments, the 212 Chamber sample was leached for 2 h at 150°C in the autoclave. The autoclave was cooled to 95-100°C, and the slurry was then filtered hot. The filtrate was transferred to a reaction vessel which was maintained at 95°C. Next, 250 mL samples of the hot solution were contacted with 10-g portions of three of the resins and with activated carbon, and the resin-solution slurries were held at ~95°C for 2 h prior to sampling to assess the extent of Sb adsorption. The results of these experiments are summarized in Table 32. The results possibly suggest a minor amount of As extraction, but the differences in As concentration could be the consequence of minor, and undetected, changes in the volume of the samples during the 2-h equilibration period (compare the analyses for the "blank" test). Clearly, none of the ion exchange resins extracts any significant amount of Sb. In contrast, activated carbon seems to adsorb a minor amount of Sb, but unfortunately, the carbon co-sorbs a more significant amount of the associated arsenic. Of the reagents tested, however, only the activated carbon seems to offer any potential for Sb removal from the As<sub>2</sub>O<sub>3</sub>-containing water leach solutions.

Table 32 - Extraction of Antimony From the Hot (95°C) Water Leach Solutions

Resin	Concentration of the Hot Solution Before the Addition of the Resin or Carbon		Concentration of the Hot Solution After 2 h of Contact with the Resin or Carbon	
	As	Sb	As	Sb
Blank	56.6	0.243	55.5	0.235
Gravex GR-2-0	56.6	0.243	55.4	0.231
Duolite C-467	56.8	0.237	55.9	0.231
Hycarb 6x12	56.8	0.237	52.7	0.168

The generally negative extraction results are probably related to the aqueous chemistry of antimony. If any cationic resin is to extract antimony, the element must be present as a cationic species. It is known that Sb(V) does not form water soluble cationic species. Although Sb(III) can form an antimonyl ion,  $\text{SbO}^+$ , the species exists only in acid media (6). Consequently, it is unlikely that antimony can be extracted with cationic exchange resins unless the pH of the solution is lowered. To pursue this option, the 212 Chamber sample was leached in hot water, the slurry was filtered and the filtrate was cooled to room temperature. One hundred millilitre aliquots of the solution were acidified to pH 3.0 or pH 1.0 with  $\text{H}_2\text{SO}_4$ . The acidified solutions were contacted with 2 mL of the various resins and agitated gently for 2 h prior to sampling. The results of these studies are given in Table 33, in which the Sb concentrations should be compared to the initial concentration of 0.0245 g/L Sb. Minimal Sb extraction occurs at pH 3.0, but a more significant level of extraction is observed at pH 1.0. In particular, the Duolite C-467 ion exchange resin extracted >90% of the Sb from the pH 1.0 solution. Although acidification coupled with ion exchange likely could be used to control antimony, the use of strongly acid media is not compatible with the simple hot water leaching option outlined in Figure 41.

Table 33 - Extraction of Antimony by Various Ion Exchange Resins at Room Temperature and pH 3.0 or pH 1.0

Resin	pH	Sb Concentration in Raffinate (g/L)	Total Sb Extracted (%)
Gravex GR-2-0	3.0	0.0246	0.0
Amberlite IRC-120	3.0	0.0238	0.2
Amberlite IRC-50	3.0	0.0232	2.1
Duolite C-467	3.0	0.0236	0.5
Gravex GR-2-0	1.0	0.0229	11.9
Amberlite IRC-120	1.0	0.0229	11.9
Amberlite IRC-50	1.0	0.0226	13.1
Duolite C-467	1.0	0.0022	91.7

#### Re-Sublimation of the $\text{As}_2\text{O}_3$ -Rich Dusts

The underlying objective of this part of the study is to produce a marketable  $\text{As}_2\text{O}_3$  product by the re-sublimation of the as-received  $\text{As}_2\text{O}_3$ -rich dusts. The index of success of the re-sublimation process is the purity of the re-sublimed  $\text{As}_2\text{O}_3$ , and to a lesser extent, the total percentage of arsenic sublimed. As will be seen, iron and the silicate minerals do not sublime, and as a result, Sb is the only impurity of any significance in the re-sublimed  $\text{As}_2\text{O}_3$  product.

The effect of the re-sublimation temperature and the sublimation atmosphere (oxidizing, neutral or reducing) on the As/As+Sb ratio of the re-sublimed oxide product is illustrated in Table 34 and Figure 44. These data were generated for the 212 Chamber sample which analyzes 45.20% As and 2.13% Sb. It is apparent that the purity of the oxide depends directly on the temperature. As the re-sublimation temperature increases from 250°C to 550°C, the As/As+Sb ratio decreases from ~99.5% to 96-97%. Significantly, the Sb contents of the re-sublimed oxide are 0.15 - 0.66% Sb; accordingly, many of the re-sublimed products contain >0.2% Sb and might be difficult to market. All of the products made at temperatures greater than the boiling point of  $\text{As}_2\text{O}_3$  (~350°C) had unacceptably high Sb contents. It also appears that the gaseous atmosphere affects the relative purity of the re-sublimed oxide, although there is some scatter of the data obtained. Oxidizing atmospheres, such as those

produced using air or  $O_2$  gas, seem to yield the highest purity products, and the products made under oxidizing conditions were consistently white. Neutral atmospheres ( $N_2$  or He gases) and reducing atmospheres ( $H_2$  gas) seem to yield oxide products having similar purities. Although the products made using neutral atmospheres were also white, those generated under reducing conditions were consistently grey. Nevertheless, X-ray diffraction analysis showed all the re-sublimed oxide products to consist only of  $As_2O_3$  having the cubic arsenolite structure. Based on the results presented above, the decision was made to carry out all subsequent re-sublimation tests using an air atmosphere.

The results presented above indicate that low sublimation temperatures yield higher purity oxide products. Accordingly, the re-sublimation reaction was monitored as a function of time at  $259^\circ C$  using the 212 Chamber sample, which analyzes 56.2% As and 2.13% Sb. Figure 45 illustrates the extent of reaction, reported as the percentage of the original sample mass remaining unsublimed, as a function of the heating time at  $259^\circ C$ . The sublimation takes place in a nearly linear manner for the first 5-6 h of heating, but subsequently, the reaction slows and eventually levels off after about 10 h of heating. Still longer heating times have no significant effect on the amount of sample sublimed. Ultimately, about 25% of the original mass of the sample remains in the residue at  $259^\circ C$ . Table 35 presents the compositional data of the various samples produced in the heating experiments outlined in Figure 45. In total, about 75% of the initial mass of the sample sublimes; however, 93% of the initially present arsenic sublimes under the same conditions. This reflects the significantly higher volatility of  $As_2O_3$  relative to the silicate minerals and iron oxide impurities present in the sample. As will be shown later, the residual ~7% As is present in the residue as poorly volatile  $(Sb,As)_2O_3$  and iron arsenate ( $Fe_2As_4O_{12}$ ) species. The compositional data indicate that the initially re-sublimed oxide has the highest purity and that the purity decreases with prolonged heating times. In fact, there is an almost linear correlation between the  $As_2O_3$  content of the re-sublimed oxide with the duration of heating, at least for heating periods up to 20 h. Likely,

Table 34 - Influence of Temperature and the Type of Atmosphere on the Purity of the Re-sublimed Arsenic Oxide

Temperature ( C)	Oxidizing Atmosphere		Neutral Atmosphere		Reducing Atmosphere	
	As/As+Sb	Sb (wt %)	As/As+Sb	Sb (wt %)	As/As+Sb	Sb (wt %)
299	99.46	0.16	99.47	0.39	-	-
	99.80	0.15	-	-	-	-
	99.78	0.39	-	-	-	-
	99.67	0.24	-	-	-	-
	99.10	0.66	-	-	-	-
272	-	-	99.47	0.41	-	-
386	-	-	97.61	1.82	-	-
400	-	-	97.50	1.85	-	-
450	-	-	97.18	2.18	-	-
459	98.35	1.41	-	-	-	-
472	-	-	-	-	96.27	2.91
475	-	-	97.54	1.89	-	-
513	-	-	-	-	97.00	2.31
519	-	-	96.97	2.33	-	-
545	98.67	0.96	-	-	-	-

a heating time of 10-12 h would be required to re-sublime most of the arsenic present in the 212 Chamber sample at 259°C.. For such heating periods, it appears that the re-sublimed oxide would contain ~99.6%  $\text{As}_2\text{O}_3$  and ~0.4% Sb. Clearly, only the very initial products meet the stipulated antimony content of <0.2% Sb.

Table 35 – Effect of Heating Time at 259°C on the Percentage of the Total Arsenic Sublimed From the 212 Chamber Sample, and the Purity of the Re-sublimed Product

Heating Time (h)	Residue Mass (% of Initial)	As Sublimed (% of Initial)	Product Composition	
			$\text{As}_2\text{O}_3$ (%)	Sb (%)
0	100	0	—	—
2	79.5	32.1	99.80	0.15
4	60.1	—	—	—
6	44.5	69.9	99.71	0.21
8	32.6	—	—	—
10	29.2	—	—	—
12	28.2	91.9	99.57	0.31
20	27.8	93.3	99.46	0.39
30	27.0	—	—	—

Although most of the re-sublimation studies were done using the 212 Chamber sample, a direct comparison of the thermal behaviour of all four  $\text{As}_2\text{O}_3$ -rich dusts was made. For these comparative tests, samples of each dust were heated at 545°C for 10 h in static air. The sublimates were collected and analyzed; the results are summarized in Table 36. In all instances, the re-sublimation process yields a relatively pure oxide product, which consistently has a higher As/As+Sb percentage relative to the starting material. In fact, Figure 46 suggests that there is a nearly linear relationship between the percentage of As/As+Sb in the re-sublimed product and As/As+Sb percentage of the original  $\text{As}_2\text{O}_3$ -rich dust. This behaviour implies that the Sb in all the  $\text{As}_2\text{O}_3$ -rich dusts behaves in a similar manner during thermal processing. Accordingly, the trend identified in Figure 46 might be useful for predicting the re-sublimation response

of other dust samples. The re-sublimation procedure reduces the Sb content of the  $\text{As}_2\text{O}_3$  product, but aside from the 236 Chamber sample, all the re-sublimed products have Sb contents greater than the specified 0.2% Sb limit. The Au analyses also presented in Table 36 demonstrate that gold collects almost quantitatively in the re-sublimation residues. As a consequence, all of the re-sublimed oxide products contained  $<0.03$  ppm Au, the detection limit of the analytical procedure used.

Table 36 – Purities of the Re-sublimed  $\text{As}_2\text{O}_3$  Products Made When the Four  $\text{As}_2\text{O}_3$ -rich Dusts were Heated to  $545^\circ\text{C}$  for 10 h in Static Air

Sample	% As/As+Sb in Sample	% As/As+Sb in Product	% As in Product	% Sb in Product	ppm Au in Product
January 98 Composite	98.74	99.57	72.3	0.31	16.82
April 97 Composite	97.83	98.90	70.4	0.78	28.84
212 Chamber	96.35	98.67	71.4	0.96	42.90
236 Chamber	99.49	99.94	71.7	0.04	26.64

The results presented above indicate a modest level of  $\text{Sb}_2\text{O}_3$  re-sublimation along with that of  $\text{As}_2\text{O}_3$ , despite a significant difference in the vapour pressures of the two end-member oxides. The limited mass spectrographic work carried out in support of the project helps to explain the unexpected transport of  $\text{Sb}_2\text{O}_3$  along with  $\text{As}_2\text{O}_3$ . It is known that both  $\text{As}_2\text{O}_3$  and  $\text{Sb}_2\text{O}_3$  exist as dimers in the vapour state; i.e., the vapour species are predominantly  $\text{As}_4\text{O}_6$  and  $\text{Sb}_4\text{O}_6$  (3). The mass spectrographic analysis of the sublimed vapour from the 212 Chamber sample detected the presence of mixed As-Sb oxide species, notably,  $\text{As}_3\text{SbO}_6$ . Such mixed As-Sb oxide species presumably have a vapour pressure lower than that of end-member  $\text{As}_2\text{O}_3$ , but

significantly higher than that of end-member  $\text{Sb}_2\text{O}_3$ . The result is the significant transport of  $\text{Sb}_2\text{O}_3$  at temperatures much lower than those needed to vapourize end-member  $\text{Sb}_2\text{O}_3$ . As a result, the re-sublimed  $\text{As}_2\text{O}_3$  has a significant Sb content although the As/As+Sb ratio of the sublimate is consistently higher than that of the starting  $\text{As}_2\text{O}_3$ -rich dust.

In an effort to eliminate Sb from the mixed vapour species, such as  $\text{As}_3\text{SbO}_6$ , the  $\text{As}_2\text{O}_3$ -rich dust was mixed with metallic arsenic and was re-sublimed in a flowing  $\text{H}_2$  gas stream. The intent was to reduce the amount of Sb transported by utilizing the following reaction.



To investigate this option, 2.21 g of 99.99% As metal powder was mixed with 8.78 g of the 212 Chamber dust, and the mixture was heated for 16 h at  $545^\circ\text{C}$ . A hydrogen atmosphere was employed to prevent the possible oxidation of the elemental arsenic. The presence of the arsenic metal increases the  $\text{As}_2\text{O}_3$  content of re-sublimation product, but only to a limited extent. With the arsenic metal present in the system, the re-sublimed product contained 98%  $\text{As}_2\text{O}_3$ ; in the absence of the arsenic metal the product consisted of 97%  $\text{As}_2\text{O}_3$ . Although the presence of arsenic metal improves the quality of the re-sublimed  $\text{As}_2\text{O}_3$  product, the improvement is not likely great enough to warrant the pursuit of this option.

### **Mineralogical Investigations of the Sublimation Residues**

Mineralogical studies were carried out on the residues produced by heating the January 98 Composite, the April 97 Composite and 236 Chamber samples for 10 h at  $545^\circ\text{C}$  in static air, and the 212 Chamber sample for 20 h at  $610^\circ\text{C}$  in static air.

X-ray diffraction analyses of the four sublimation residues were carried; the individual diffraction patterns are presented in Appendix 8. The sublimation residue from the January 98 Composite sample consists of an  $\text{As}_2\text{O}_3$ -type

compound, iron arsenate ( $\text{Fe}_2\text{As}_4\text{O}_{12}$ ), anhydrite ( $\text{CaSO}_4$ ) and quartz. The corresponding residue from the heating of the April 97 Composite sample consists of an  $\text{As}_2\text{O}_3$ -type compound, iron arsenate ( $\text{Fe}_2\text{As}_4\text{O}_{12}$ ), anhydrite ( $\text{CaSO}_4$ ), quartz and muscovite. A trace amount of As metal was also detected in this sample, but the presence of elemental arsenic requires independent confirmation. The sublimation residue from the heating of the 236 Chamber sample was shown to contain abundant muscovite, chlorite, quartz and iron oxide ( $\text{FeO}_3$ ), in addition to the  $\text{As}_2\text{O}_3$ -type compound, iron arsenate ( $\text{Fe}_2\text{As}_4\text{O}_{12}$ ) and anhydrite ( $\text{CaSO}_4$ ). A trace amount of  $\text{Fe}_8(\text{AsO}_4)_6(\text{OH})_6 \cdot 5\text{H}_2\text{O}$  may also be present, but the occurrence of this phase requires confirmation. The sublimation residue from the heating of the 212 Chamber sample was shown by X-ray diffraction analysis to consist of hematite, maghemite ( $\text{Fe}_2\text{O}_3$ ), anhydrite ( $\text{CaSO}_4$ ), chlorite, muscovite, K-feldspar and quartz. Significantly, the  $\text{As}_2\text{O}_3$ -type compound and iron arsenate ( $\text{Fe}_2\text{As}_4\text{O}_{10}$ ) were not detected in this sublimation residue by X-ray diffraction analysis. Their absence could be a consequence of the somewhat higher temperature ( $610^\circ\text{C}$  versus  $545^\circ\text{C}$ ) and longer heating time (20 h versus 10 h) used to generate this material.

Figure 47 shows the general appearance of the sublimation residue from the January 98 Composite sample. The residue clearly has sintered into 100-500  $\mu\text{m}$  in aggregates which have irregular outlines. The brighter masses are rich in As, but also contain minor amounts of O, as well as traces of Sb and Fe. This phase is presumably  $(\text{As,Sb})_2\text{O}_3$ , although the presence of finely dispersed As metal cannot be precluded. The darker appearing masses are mostly silicates, iron arsenate and iron oxide; one such occurrence is shown in more detail in Figure 48. The sintered agglomerate contains bright appearing particles of iron arsenate (presumably  $\text{Fe}_2\text{As}_4\text{O}_{12}$ ), as well as darker appearing grains of  $\text{CaSO}_4$ , muscovite and quartz. The various particles clearly have sintered together, but there is not an obvious agglomerating phase. In some instances, the particles may be sintered by  $(\text{Sb,As})_2\text{O}_3$ . Overall, the SEM-EDX studies of this sublimation residue showed it to consist of major iron arsenate ( $\text{Fe}_2\text{As}_4\text{O}_{12}$  or  $\text{FeAs}_2\text{O}_3$  having a minor Sb content),  $\text{CaSO}_4$ , muscovite and quartz. The iron

arsenate phase commonly contains trace amounts of Ca, K, S, Si and Al, but some of the impurities detected may originate from dispersed silicate grains. Minor amounts of  $\text{As}_2\text{O}_3$ , containing traces of Sb, were present along with trace amounts of elemental As. Isolated particles of calcium arsenate having a minor Fe and trace S content were detected, and a few grains of Na-Al silicate were evident.

The SEM-EDX study of the sublimation residue from the heating of the April 97 Composite sample showed this material to be similar to the residue from the January 98 Composite sample. Figure 49 shows the detailed texture of the sublimation residue from the April 97 Composite sample. This material consists of bright grains of iron arsenate ( $\text{Fe}_2\text{As}_4\text{O}_{12}$  or  $\text{FeAs}_2\text{O}_3$ ) having a minor Sb content. The abundance of the iron arsenate species reflects their significant stability at elevated temperatures. A few particles rich in Sb, As and Fe may be iron arsenate-antimonate compounds. Most of the grey appearing particles in Figure 49 are muscovite, whereas the dark grains are  $\text{CaSO}_4$  or quartz. Other species detected in trace amounts include As-bearing iron oxide and elemental arsenic. Minor amounts of  $\text{As}_2\text{O}_3$  having a low Sb content were also detected, and as shown in Figure 50, the  $\text{As}_2\text{O}_3$  phase tends to occur as large masses enveloping smaller particles of iron arsenate, muscovite and quartz.

The 236 Chamber sample contains less  $\text{As}_2\text{O}_3$  but more silicate and oxide species than the January 98 Composite and April 97 Composite samples. As a result, the sublimation residue from the heating of the 236 Chamber sample contains more abundant oxide and silicate species. Figure 51 illustrates a number of iron arsenate ( $\text{Fe}_2\text{As}_4\text{O}_{12}$ ) particles which are associated with needle-like grains of  $\text{As}_2\text{O}_3$  containing minor amounts of Fe. Particles of  $\text{CaSO}_4$ , about  $10\text{ }\mu\text{m}$  in size and having a modest As content, and dark appearing fragments of quartz are also evident. Most of the grey appearing grains are chlorite or muscovite. Other species detected in this sample include As-bearing iron oxide, calcium arsenate having a minor Fe content, and possibly, elemental arsenic.

Of the resublimed samples, the 212 Chamber sample was heated to be highest temperature and for the longest time. Despite this fact, the sublimation

residue from the heating of the 212 Chamber dust still contained a few large fragments of arsenic oxide. One such fragment is illustrated in Figure 52 which shows a ~500  $\mu\text{m}$  mass of arsenic oxide agglomerating various silicate and iron arsenate particles. EDX analysis showed the arsenic oxide to contain minor amounts of O, but only traces of Sb. The reasons for the persistence of large arsenic oxide masses at temperatures well above the sublimation temperature of  $\text{As}_2\text{O}_3$  (465°C) are not clear. It may be that the oxide has a reduced O content and, consequently, a lower vapour pressure than stoichiometric  $\text{As}_2\text{O}_3$ .

Alternatively, it is possible that the large masses form during the removal of the sample from the furnace, although the morphology really does not support such a mechanism. Figure 53 shows a number of the finer grain constituents of the sublimation residue from the 212 Chamber sample in greater detail. The bright appearing grains are either iron oxide or iron arsenate. In this regard, both  $\text{Fe}_2\text{As}_4\text{O}_{12}$  and  $\text{Fe}_2\text{As}_4\text{O}_{12}$  containing trace Sb were detected. A few tiny bright grains of  $(\text{Sb,As})_2\text{O}_3$  were also evident, but this phase is surprisingly rare given the abundance of Sb in the 212 Chamber sample. Figure 53 illustrates some particles of Ca arsenate having a modest Fe content and of  $\text{CaSO}_4$ . Most of the dark appearing particles are chlorite, muscovite or quartz. Figure 54 provides a different view of the same sublimation residue. In addition to the phases identified above, the figure illustrates several particles of  $\text{CaSO}_4$  and quartz, as well as a few grains of a rare Fe-Zn arsenate phase containing minor Sb. The fine grained residues presented in Figures 53 and 54 illustrate the non-volatile phases which are concentrated when the arsenic-antimony oxide species are sublimed.

## CONCLUSIONS

## ACKNOWLEDGEMENTS

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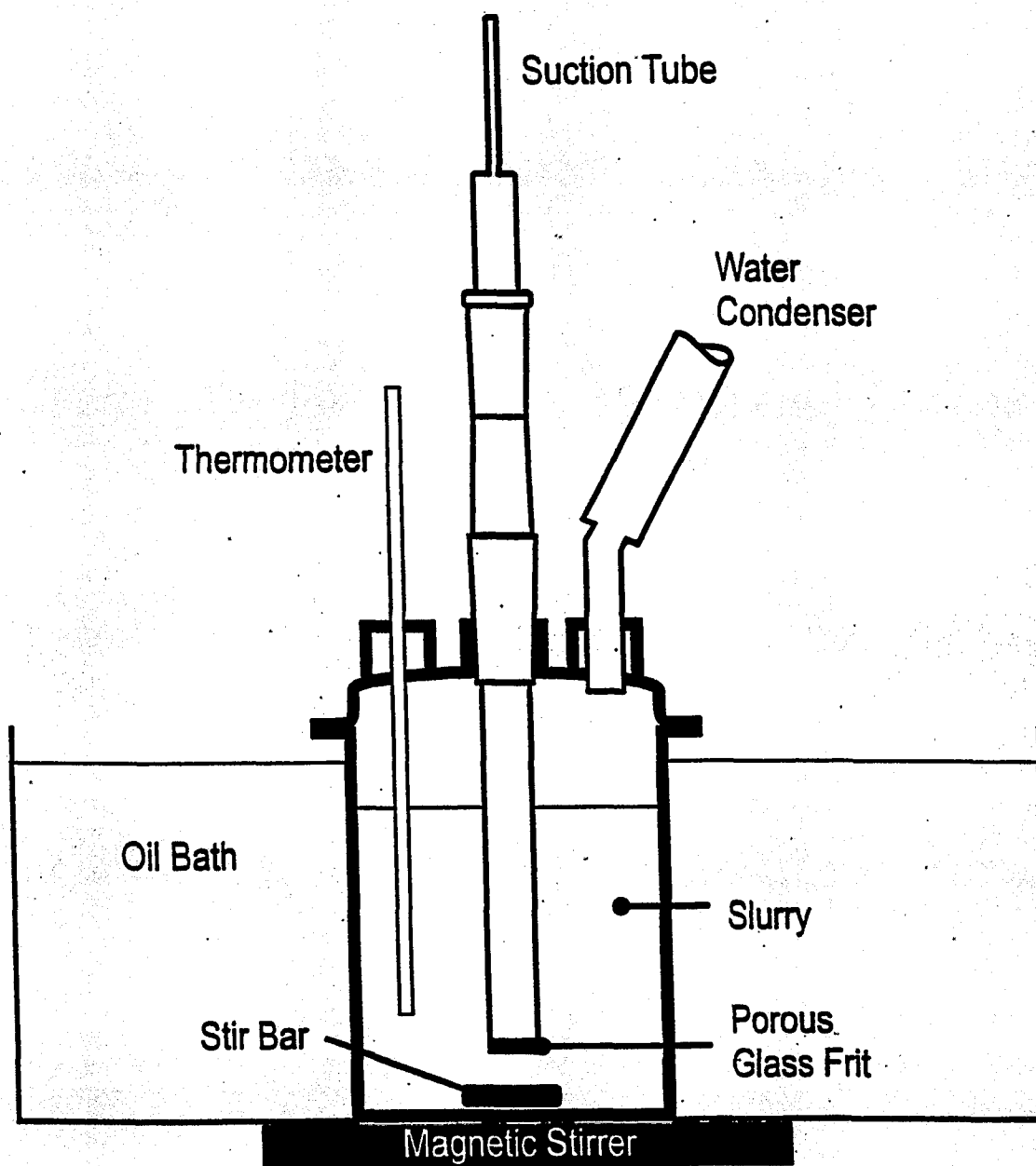


Fig. 1 - Schematic representation of the apparatus used to measure the solubilities of the  $\text{As}_2\text{O}_3$ -rich dusts.

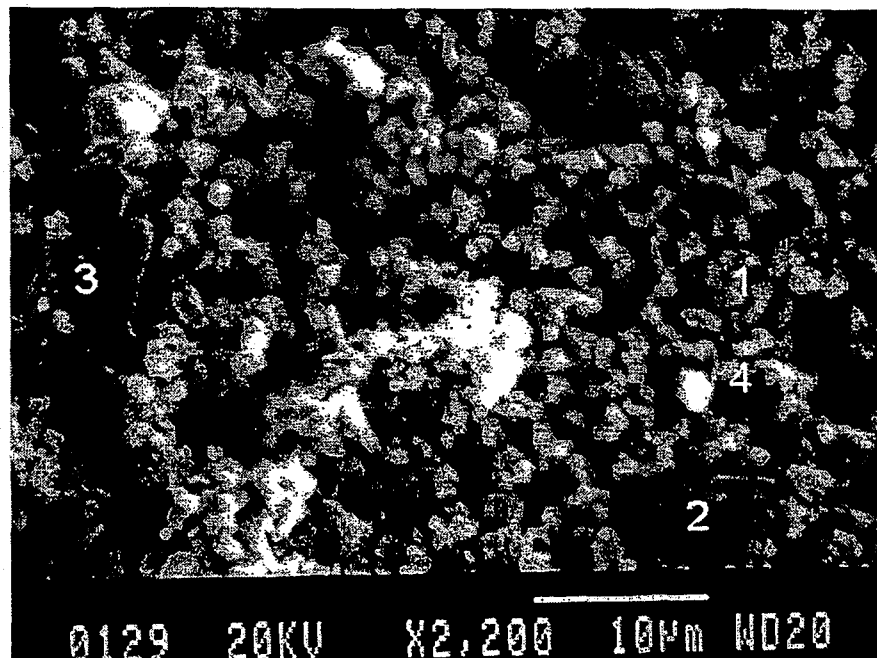


Fig. 2 - Secondary electron micrograph of a loose powder mount of the January 98 Composite sample that shows the general appearance of this material. 1-  $\text{As}_2\text{O}_3$ , 2- chlorite, 3- quartz, 4- As-bearing iron oxide

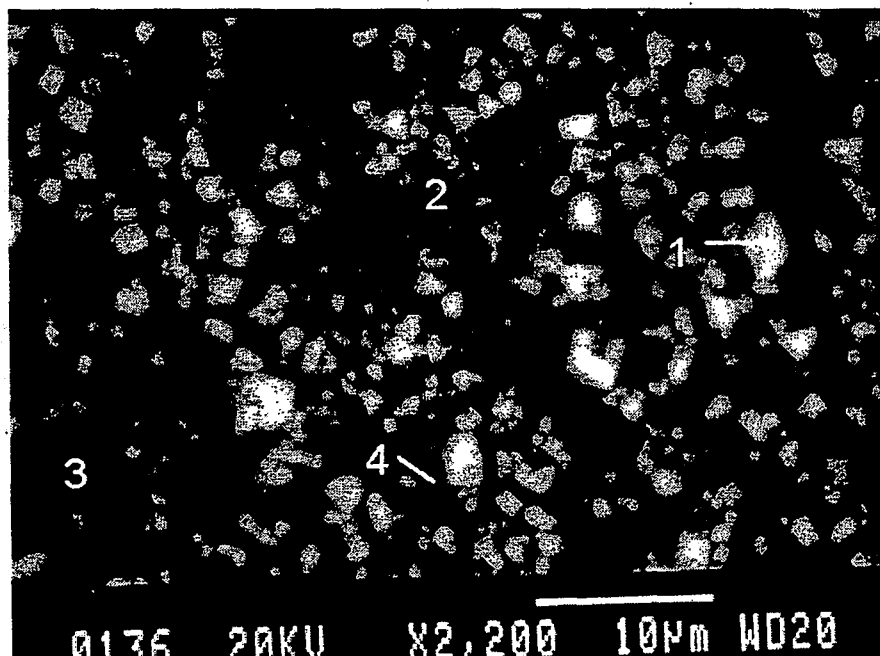


Fig. 3 - Backscattered electron micrograph of a polished section of the January 98 Composite sample. 1-  $\text{As}_2\text{O}_3$ , 2- chlorite, 3- quartz, 4- Ca arsenate

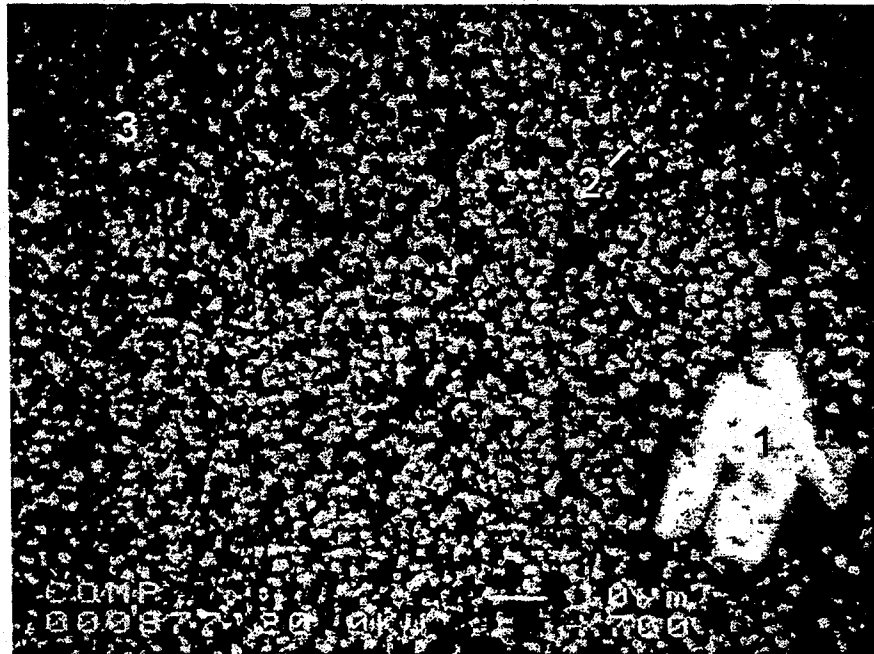


Fig. 4 - Backscattered electron micrograph illustrating a large mass of  $\text{As}_2\text{O}_3$  in the January 98 Composite sample. 1- large mass of  $\text{As}_2\text{O}_3$ , 2-  $\text{As}_2\text{O}_3$ , 3- As-bearing iron oxide

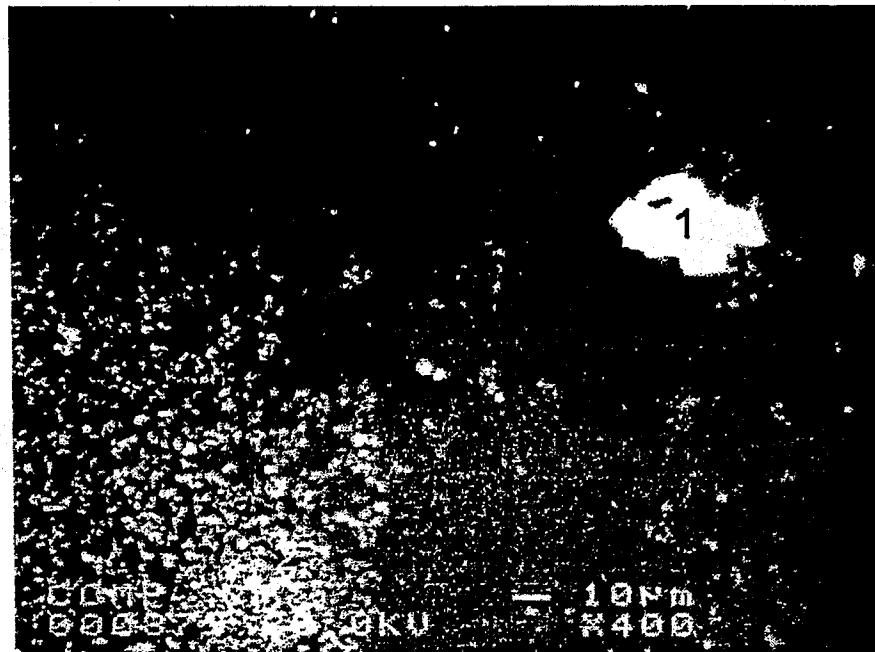


Fig. 5 - Backscattered electron micrograph showing a large mass of As-bearing Sb-rich oxide. 1- Sb-rich oxide, 2-  $\text{As}_2\text{O}_3$

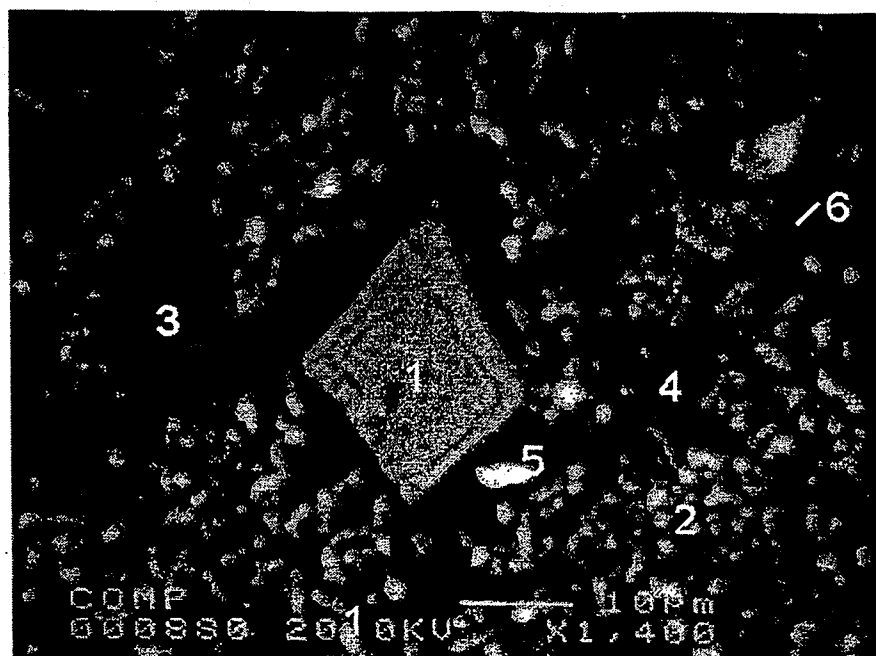


Fig. 6 - Backscattered electron micrograph of an As-bearing iron oxide particle in the January 98 Composite sample. 1- As-bearing iron oxide, 2-  $\text{As}_2\text{O}_3$ , 3- muscovite, 4- quartz, 5- rare grain of  $\text{PbSO}_4$ , 6- chlorite

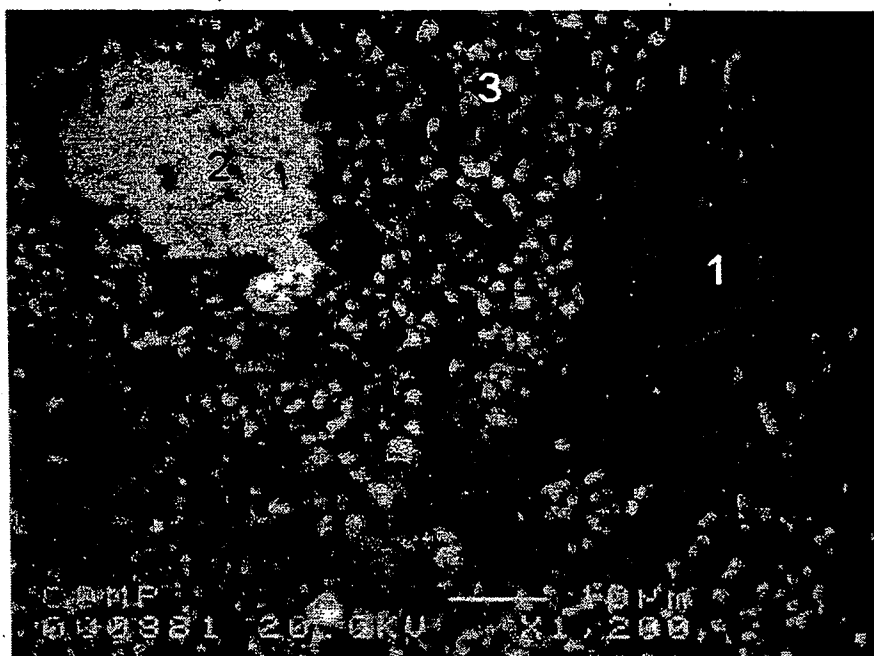


Fig. 7 - Backscattered electron micrograph of the iron arsenate phase in the January 98 Composite sample. 1- iron arsenate, 2- large mass of  $\text{As}_2\text{O}_3$ , 3-  $\text{As}_2\text{O}_3$

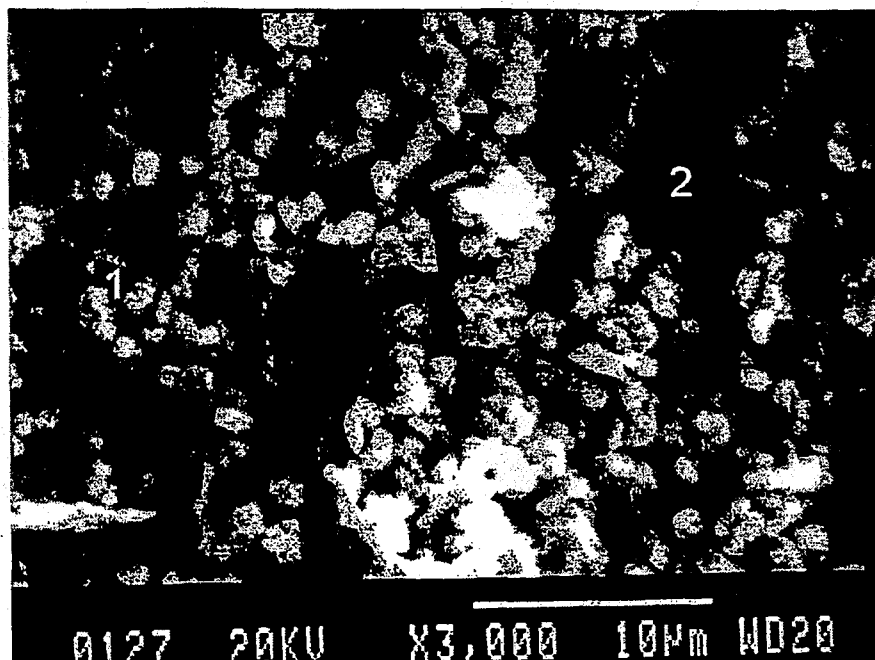


Fig. 8 - Secondary electron micrograph of a loose powder mount of the April 97 Composite sample that shows the general appearance of this material.  
1-  $\text{As}_2\text{O}_3$ , 2- chlorite

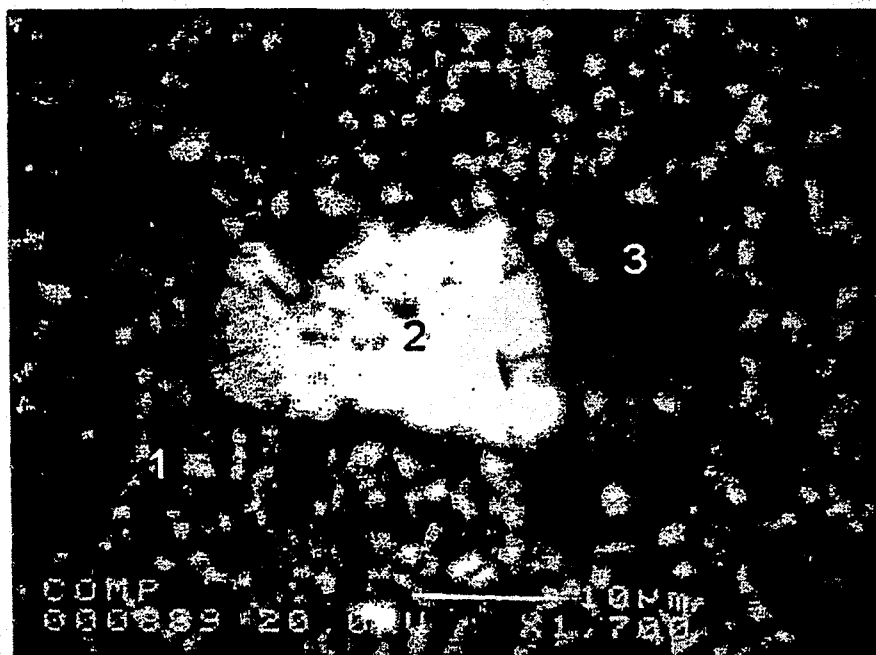


Fig. 9 - Backscattered electron micrograph showing the detailed morphology of the April 97 Composite sample. 1-  $\text{As}_2\text{O}_3$ , 2-  $(\text{As,Sb})_2\text{O}_3$  containing 13% Sb, 3- quartz

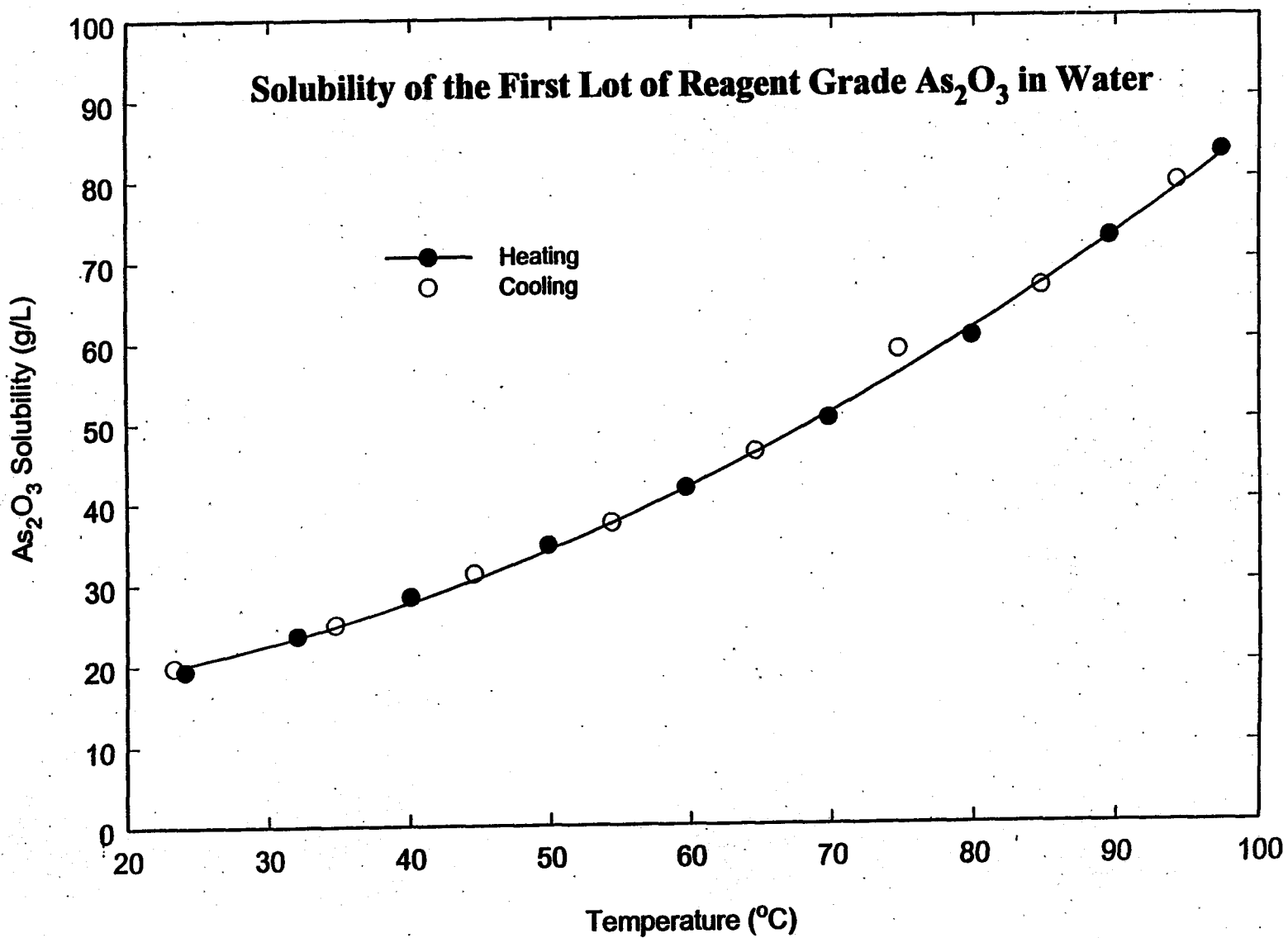


Fig. 23 - Experimentally determined solubility of  $\text{As}_2\text{O}_3$  over the first lot of reagent grade  $\text{As}_2\text{O}_3$ .



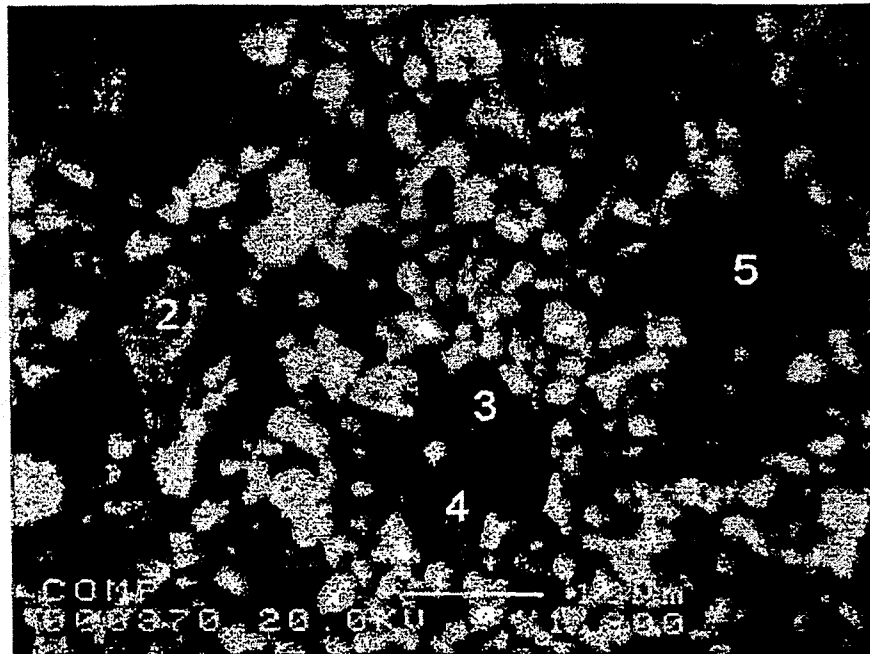


Fig. 22 - Backscattered electron micrograph showing the presence of As-bearing iron oxide and various silicate species in the 236 Chamber sample.  
1-  $\text{As}_2\text{O}_3$ , 2- As-bearing iron oxide, 3- quartz, 4- chlorite, 5- muscovite

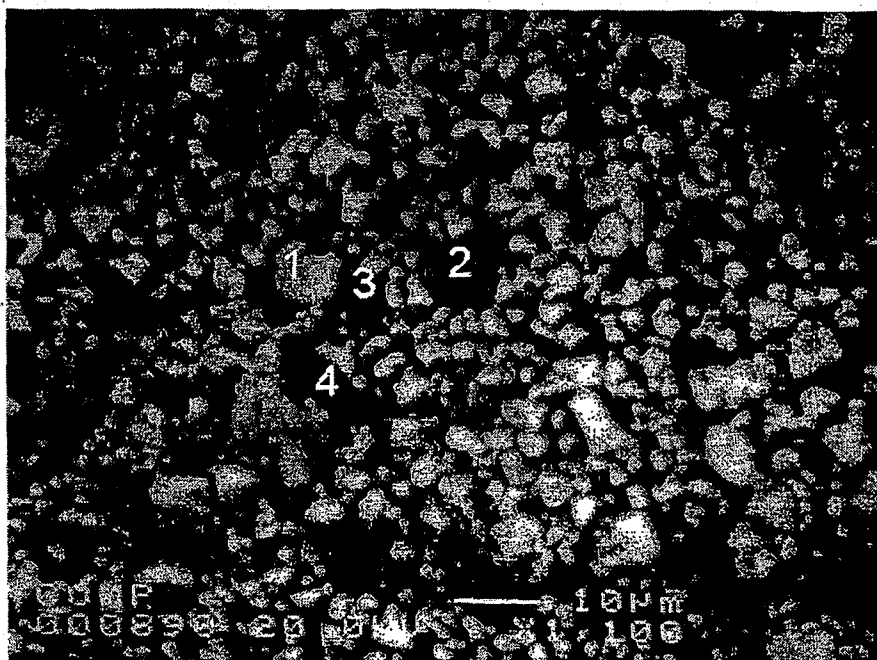


Fig. 20 - Backscattered electron micrograph showing the general morphology of the 236 Chamber sample in a polished section. 1-  $\text{As}_2\text{O}_3$ , 2- quartz, 3- chlorite, 4- muscovite

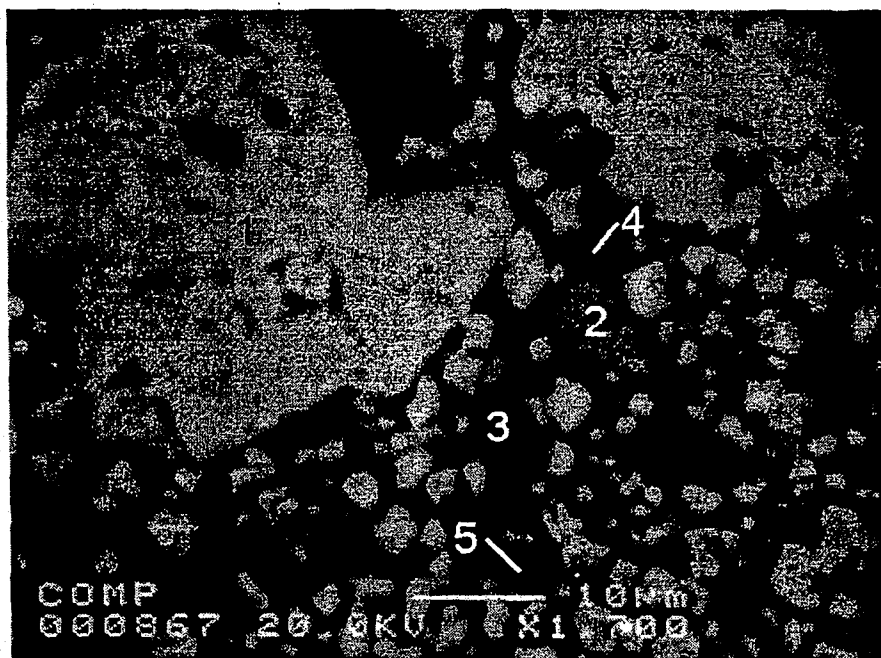


Fig. 21 - Backscattered electron micrograph showing the detailed morphology of the large  $\text{As}_2\text{O}_3$  crystals in the 236 Chamber sample. 1-  $\text{As}_2\text{O}_3$ , 2- As-bearing iron oxide, 3- quartz, 4- chlorite, 5- muscovite

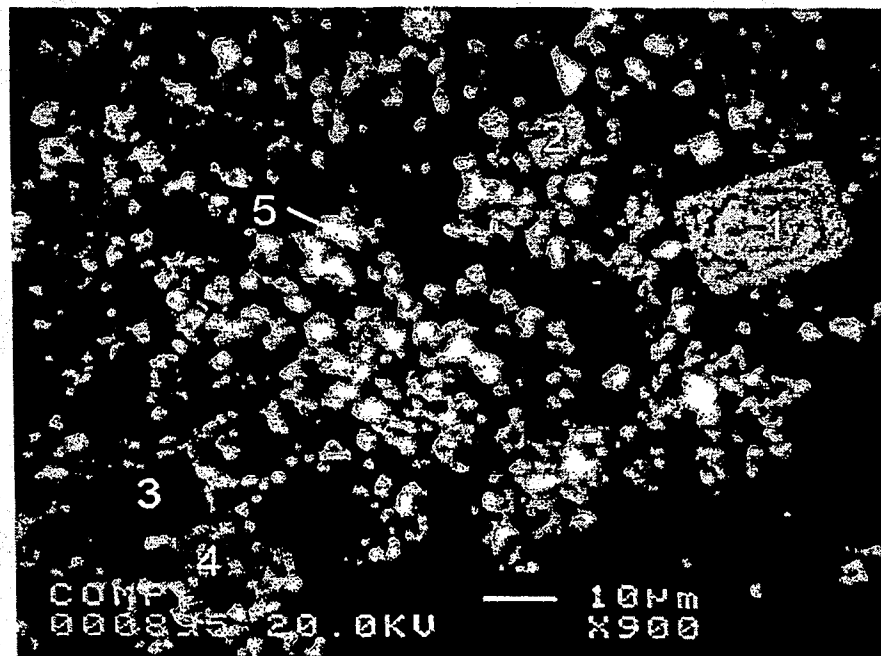


Fig. 18 - Backscattered electron micrograph showing a large particle of As-bearing iron oxide in the 212 Chamber sample. 1- As-bearing iron oxide, 2-  $\text{As}_2\text{O}_3$  with minor Fe and Sb, 3- quartz, 4- chlorite, 5-  $\text{As}_2\text{O}_3$

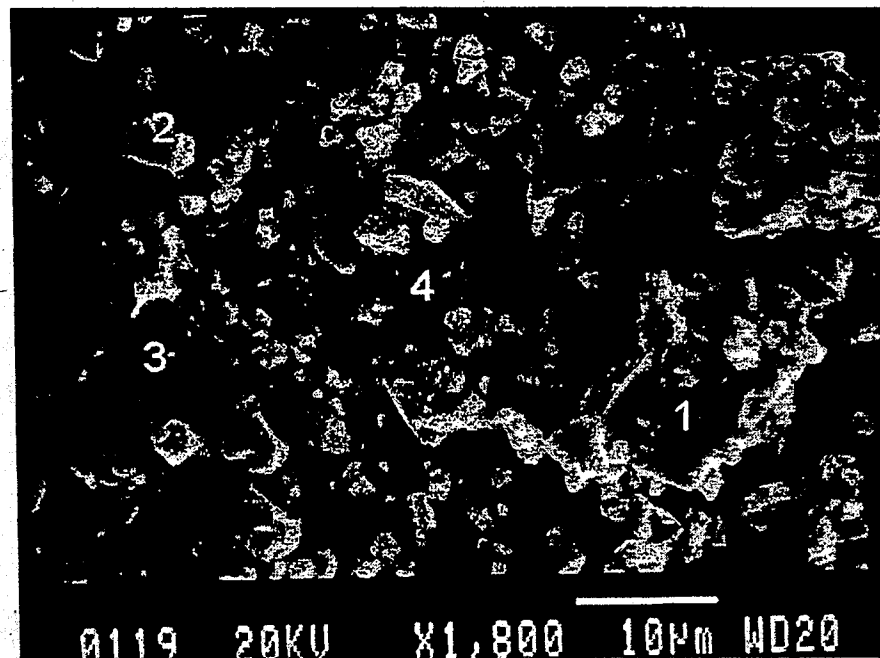


Fig. 19 - Secondary electron micrograph showing the general morphology of the 236 Chamber sample as seen in a loose powder mount. 1-  $\text{As}_2\text{O}_3$ , 2- chlorite, 3- muscovite, 4- As-bearing iron oxide

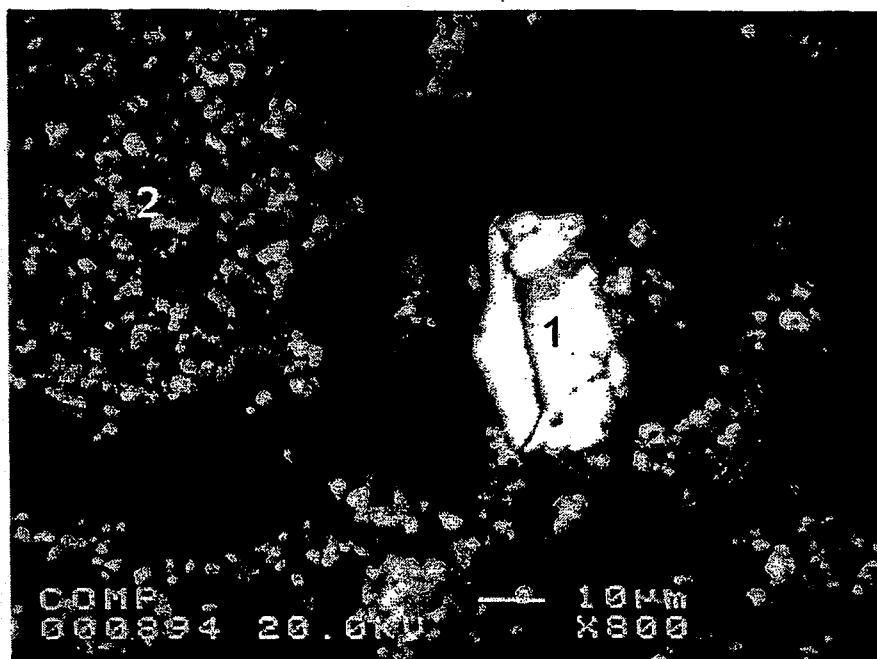


Fig. 16 - Backscattered electron micrograph of a large mass of  $(\text{Sb,As})_2\text{O}_3$  in the 212 Chamber sample. 1-  $(\text{Sb,As})_2\text{O}_3$ , 2-  $\text{As}_2\text{O}_3$  + silicates

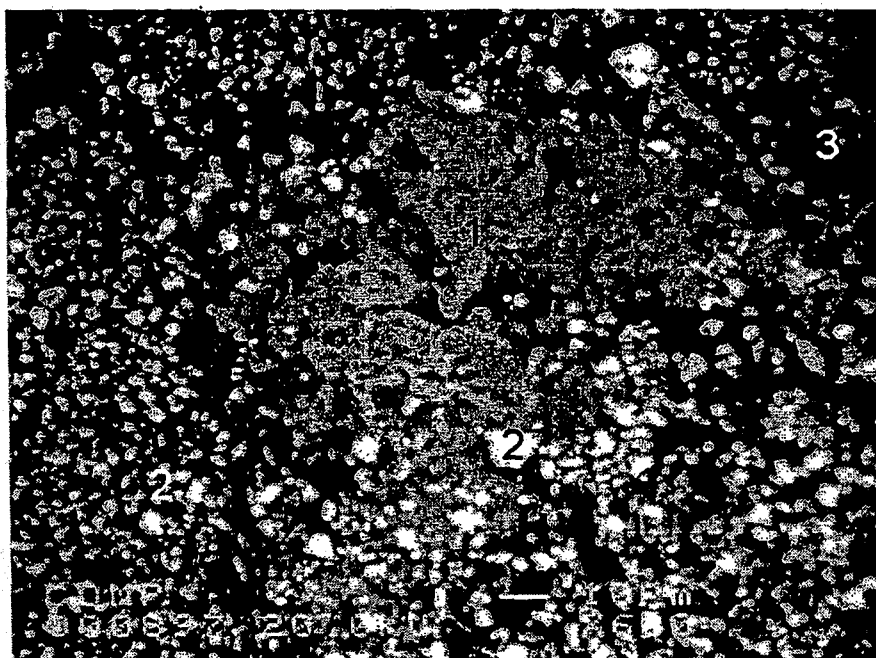


Fig. 17 - Backscattered electron micrograph showing a mass of iron arsenate in the 212 Chamber sample. 1- iron arsenate, 2-  $\text{As}_2\text{O}_3$ , 3- calcium sulphate

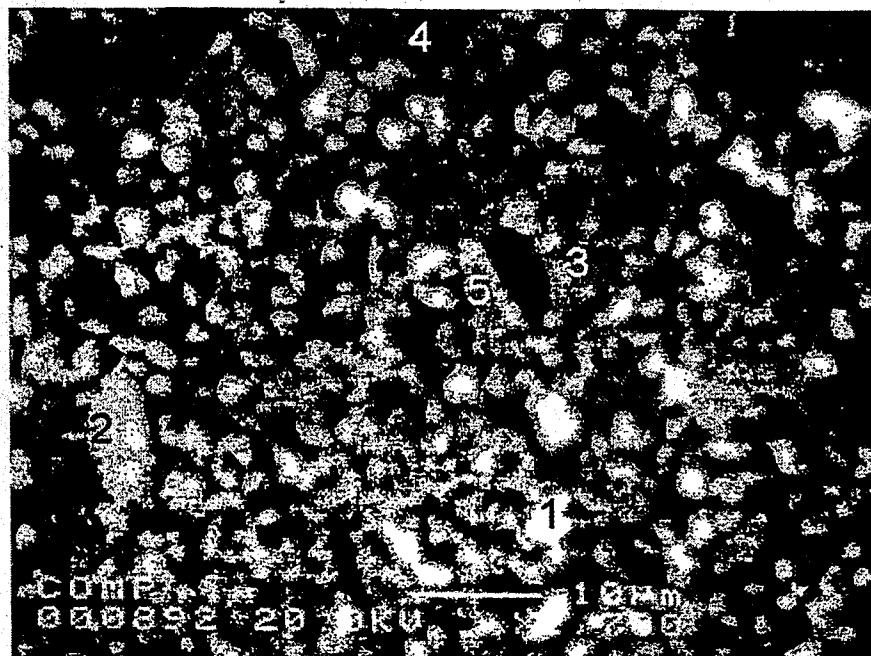


Fig. 14 - Backscattered electron micrograph showing the general appearance of the 212 Chamber sample in polished section. 1-  $\text{As}_2\text{O}_3$ , 2- As-bearing iron oxide, 3- As-bearing Ca-Fe oxide, 4- muscovite, 5- chlorite

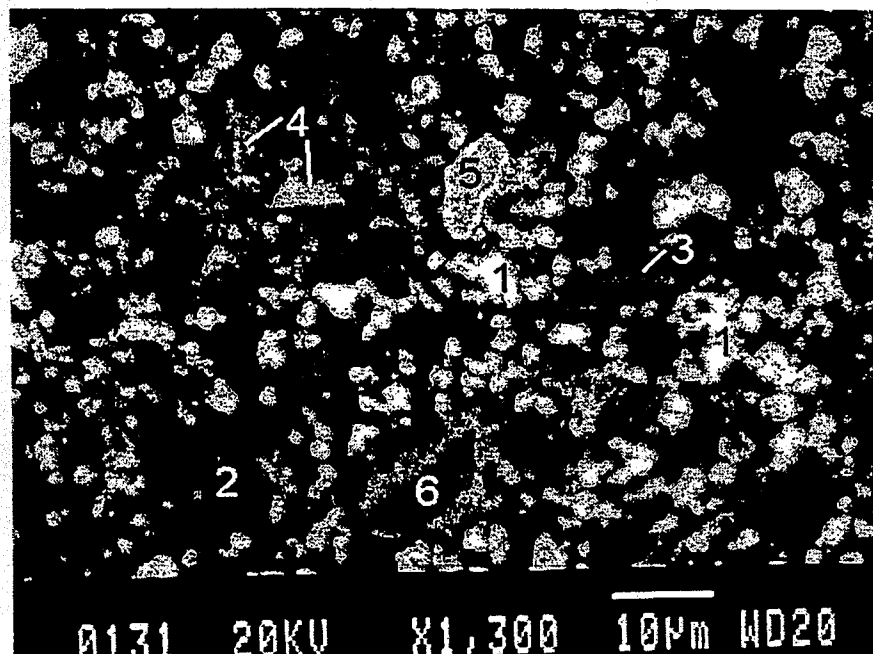


Fig. 15 - Backscattered electron micrograph showing the general appearance of the 212 Chamber sample. 1-  $\text{As}_2\text{O}_3$ , 2- quartz, 3- chlorite, 4- Fe arsenate, 5- As-bearing iron oxide rimmed by  $(\text{Sb,As})_2\text{O}_3$ , 6- muscovite rimmed with Fe-Ca sulphate-arsenate

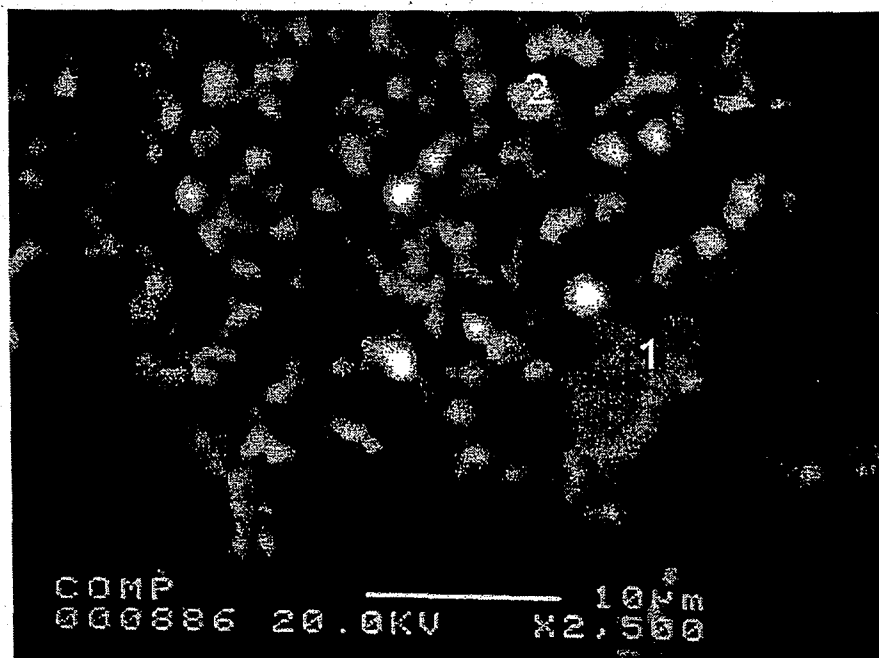


Fig. 12 - Backscattered electron micrograph illustrating a zoned particle of calcium arsenate in the April 97 Composite sample. 1- calcium arsenate-sulphate, 2-  $\text{As}_2\text{O}_3$

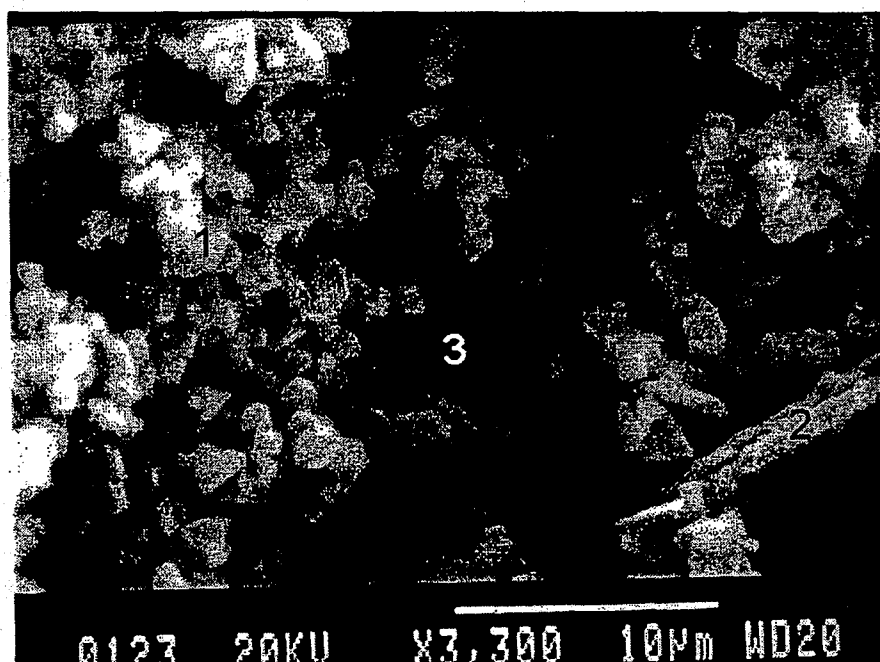


Fig. 13 - Secondary electron micrograph of a loose powder mount of the 212 Chamber sample. 1-  $\text{As}_2\text{O}_3$ , 2- chlorite, 3- muscovite

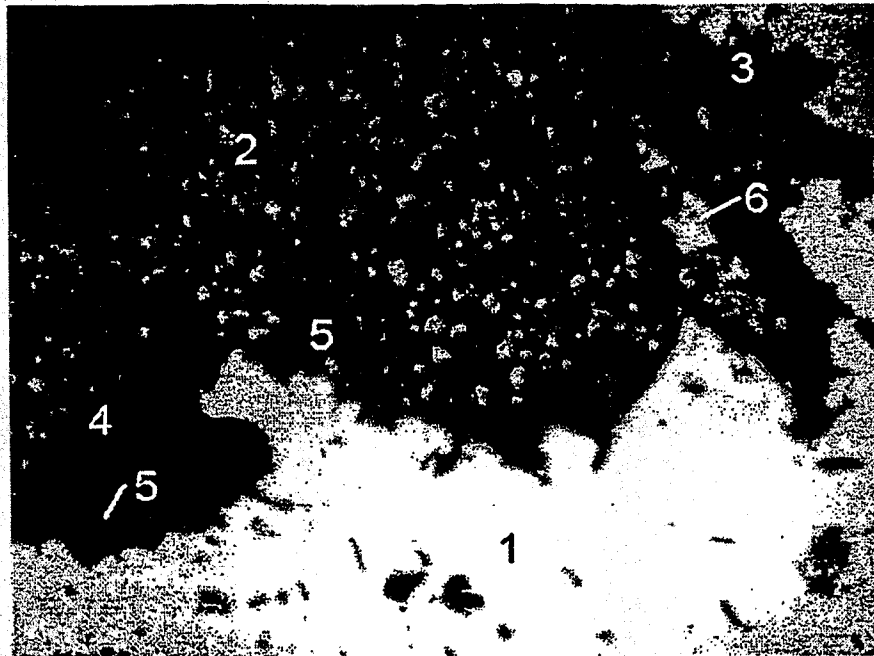


Fig. 10 - Backscattered electron micrograph of a large mass of  $(\text{As,Sb})_2\text{O}_3$  oxide in the April 97 Composite sample. 1-  $(\text{As,Sb})_2\text{O}_3$ , 2-  $\text{As}_2\text{O}_3$ , 3- quartz, 4- chlorite, 5- Fe-Ca oxide, 6-  $(\text{As,Sb,Fe})$  oxide

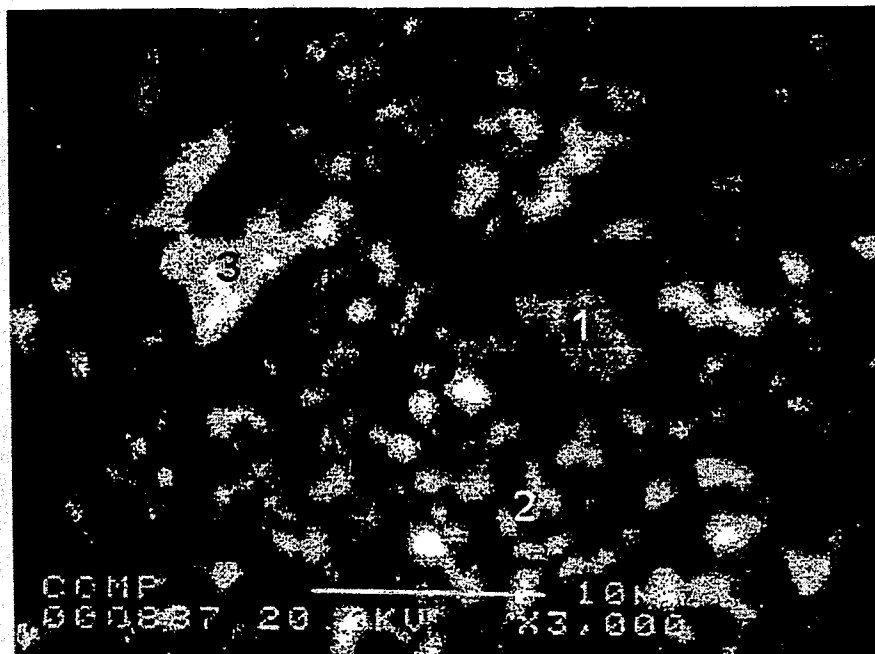


Fig. 11 - Backscattered electron micrograph showing a particle of iron arsenate in the April 97 Composite sample. 1- iron arsenate, 2-  $\text{As}_2\text{O}_3$ , 3-  $(\text{As,Sb})_2\text{O}_3$

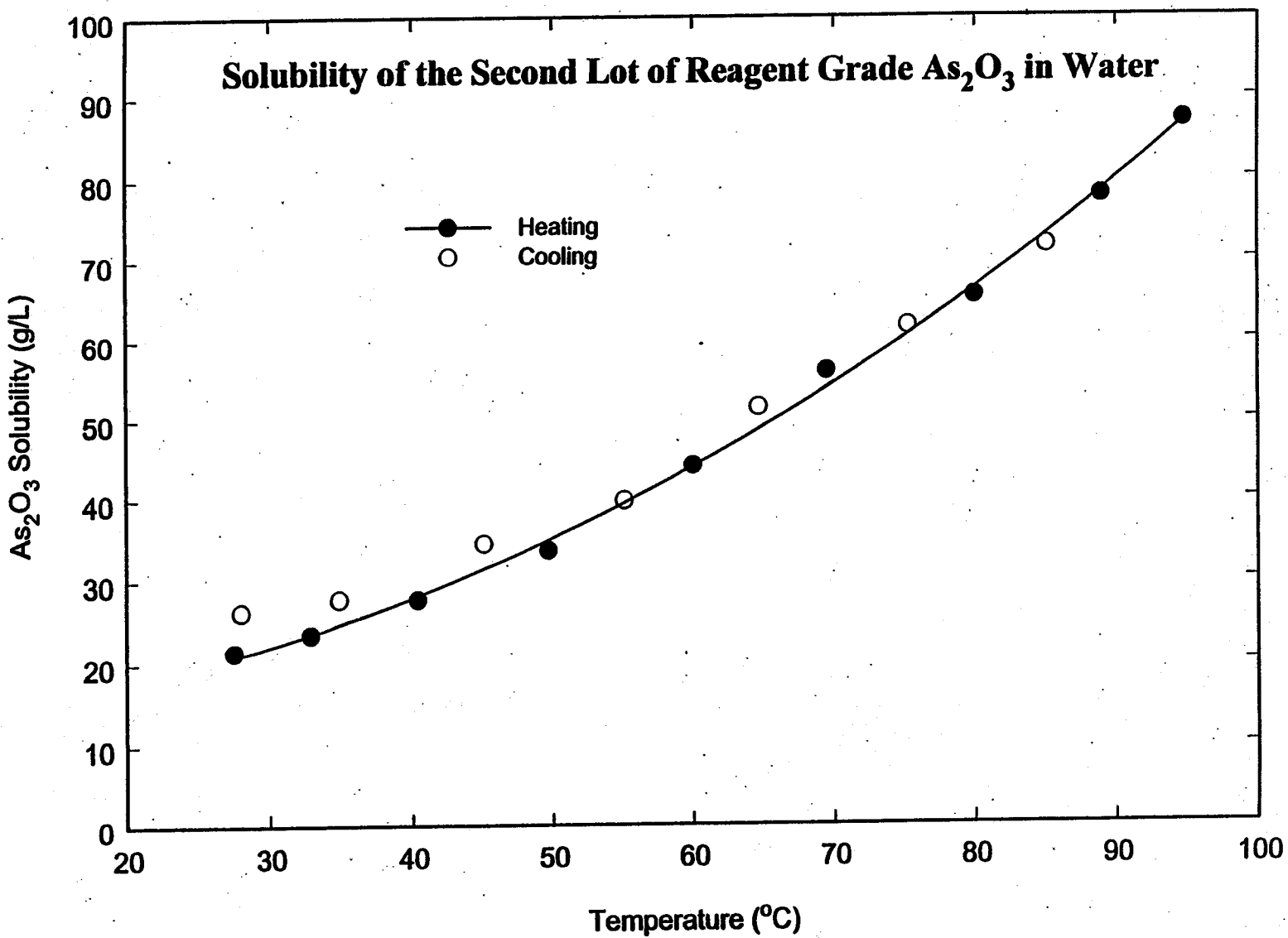


Fig. 24 - Experimentally determined solubility of  $\text{As}_2\text{O}_3$  over the second lot of reagent grade  $\text{As}_2\text{O}_3$ .



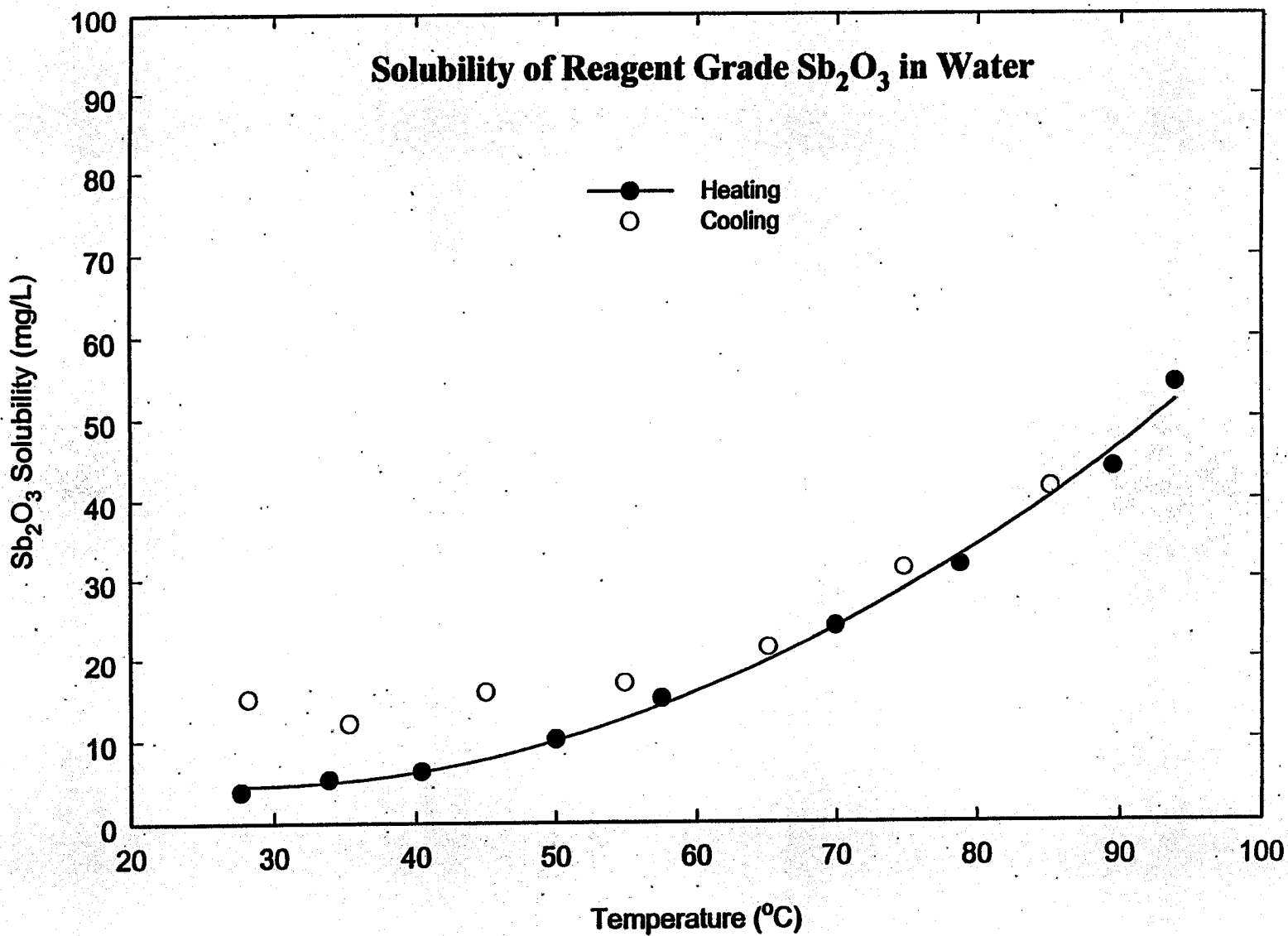


Fig. 25 - Experimentally determined solubility of  $\text{Sb}_2\text{O}_3$  over reagent grade  $\text{Sb}_2\text{O}_3$ .



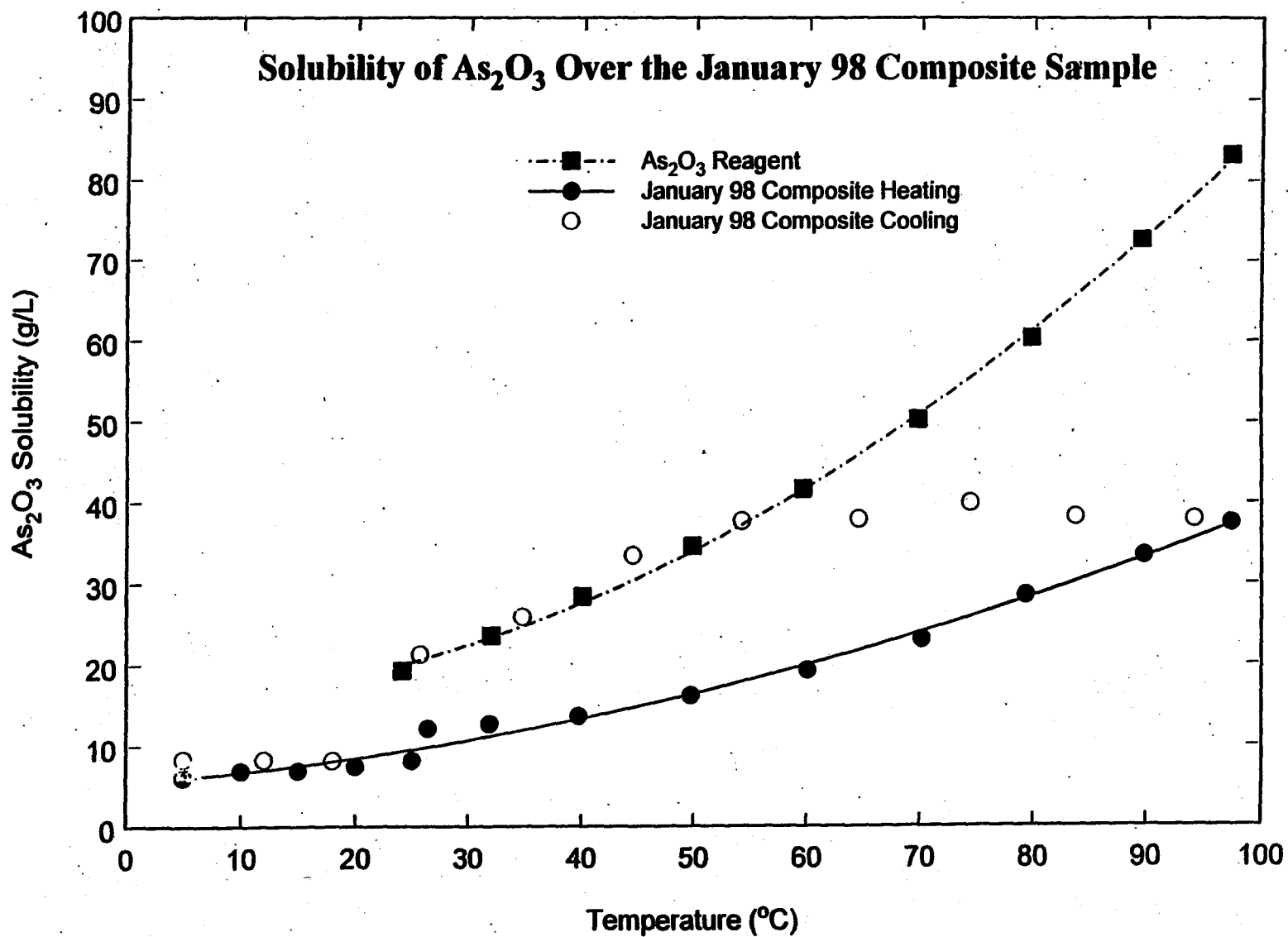


Fig. 26 - Experimentally determined solubility of  $\text{As}_2\text{O}_3$  over the January 98 Composite sample.



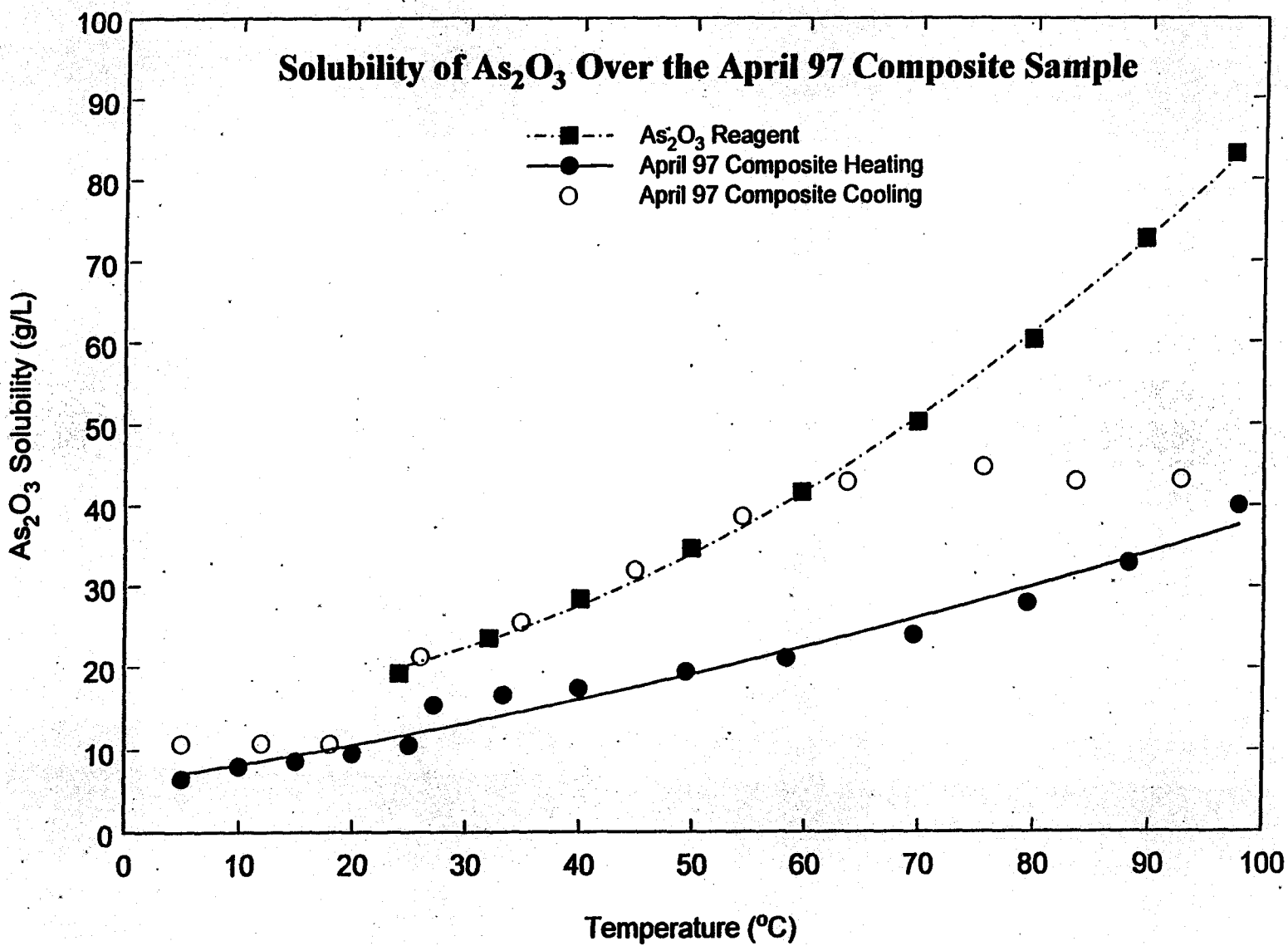


Fig. 27 - Experimentally determined solubility of  $\text{As}_2\text{O}_3$  over the April 97 Composite sample.



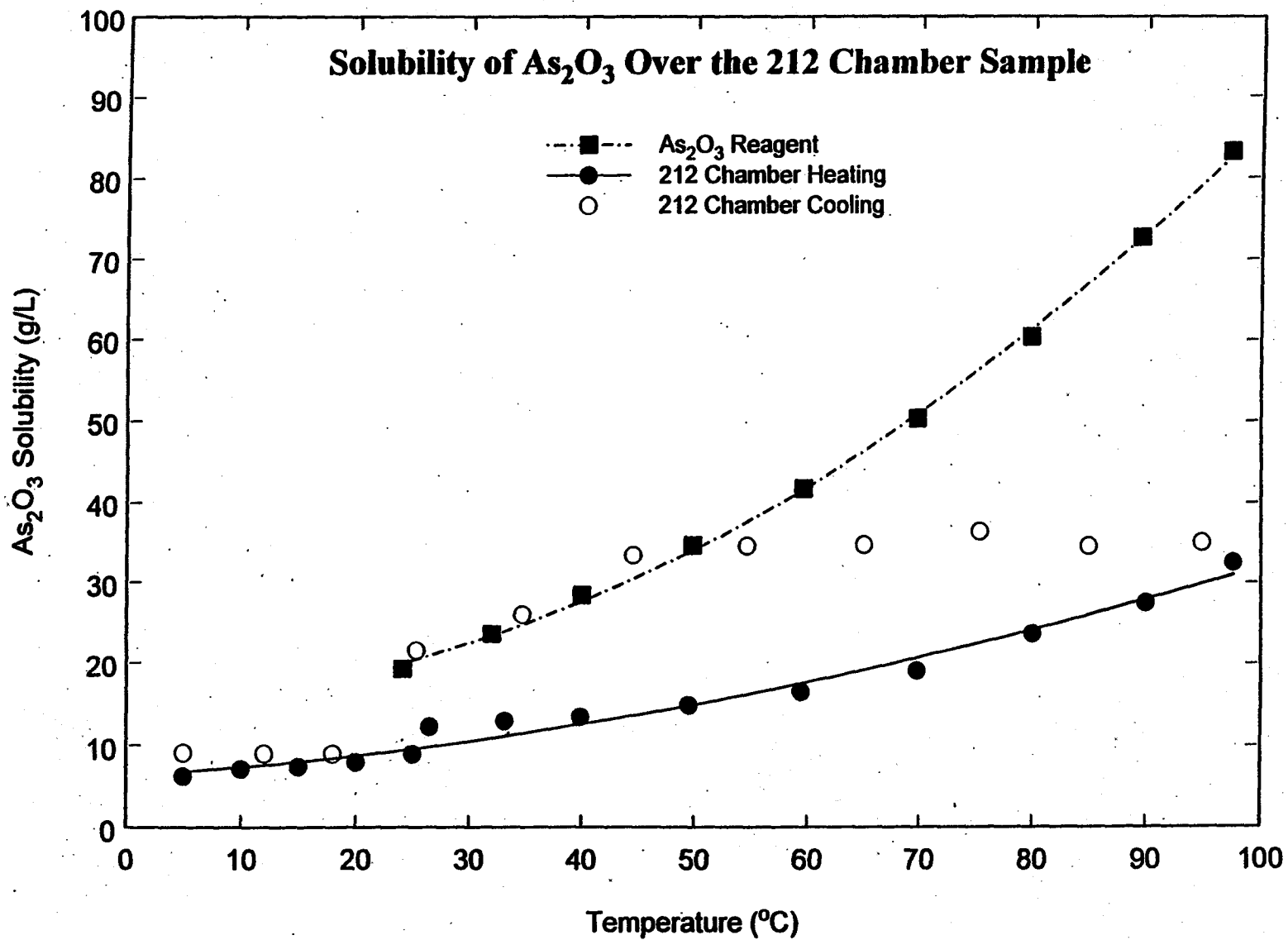


Fig. 28 - Experimentally determined solubility of  $\text{As}_2\text{O}_3$  over the 212 Chamber sample.



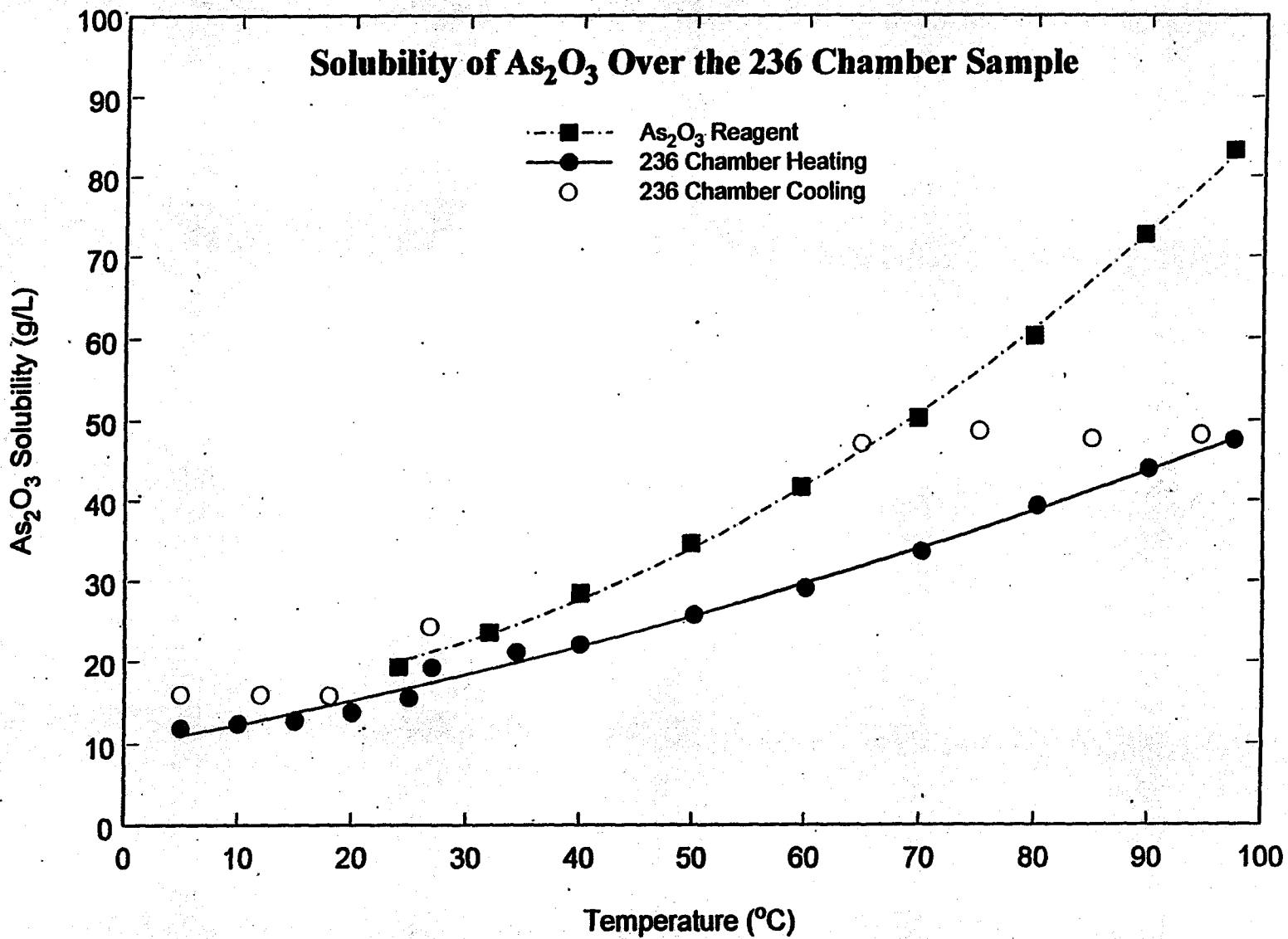


Fig. 29 - Experimentally determined solubility of  $\text{As}_2\text{O}_3$  over the 236 Chamber sample.



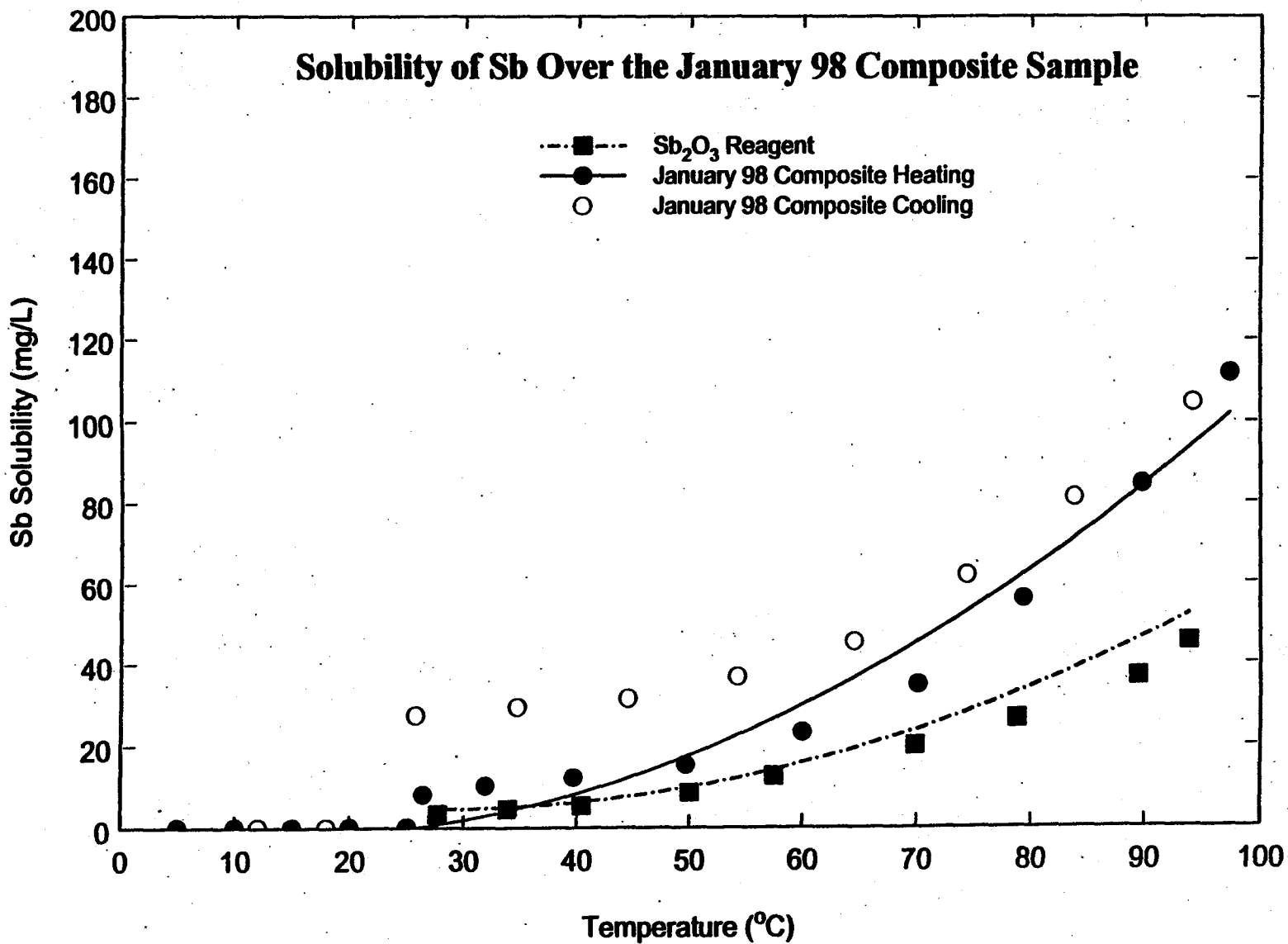


Fig. 30 - Experimentally determined solubility of Sb over the January 98 Composite sample.



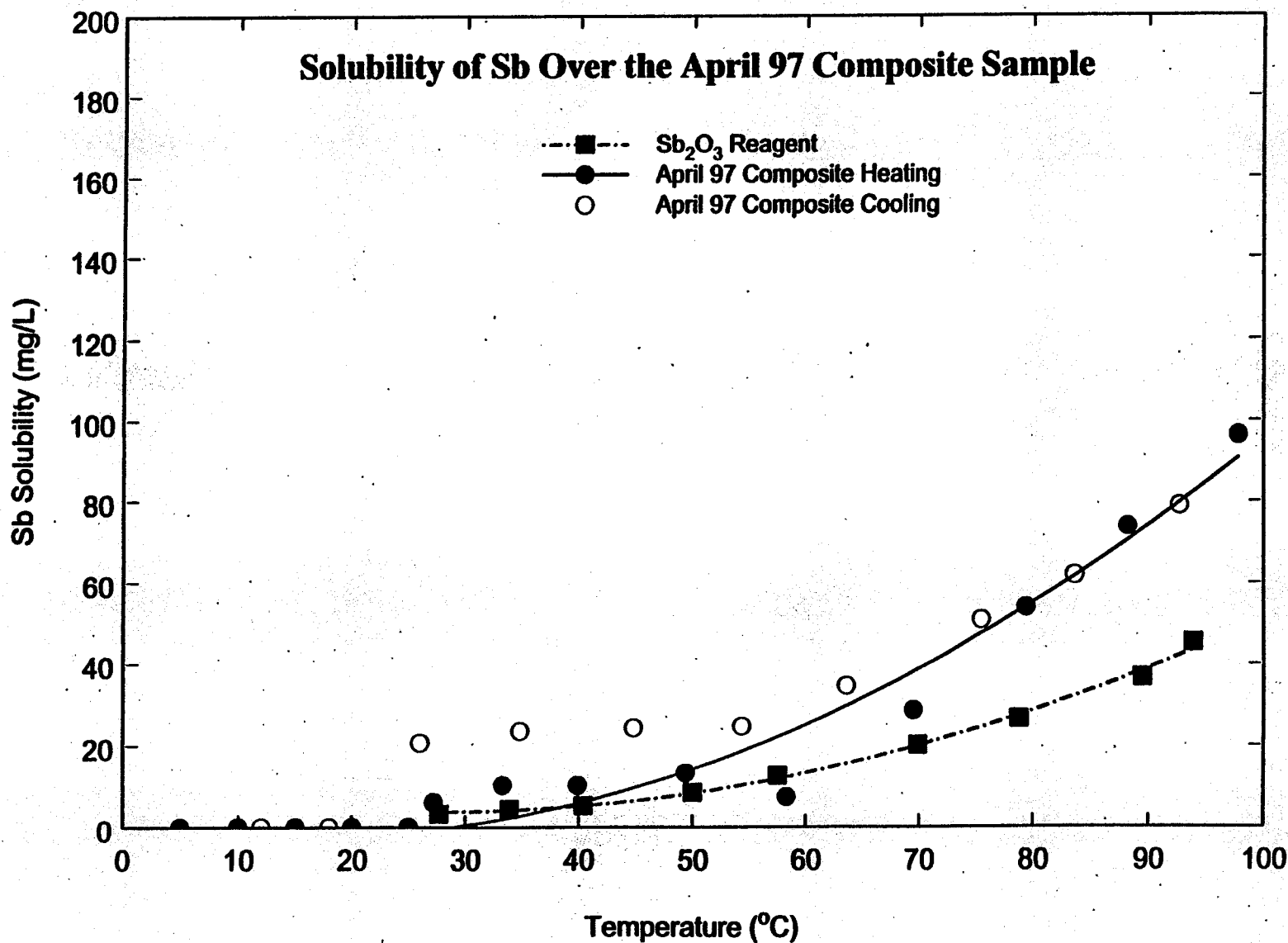


Fig. 31 - Experimentally determined solubility of Sb over the April 97 Composite sample.



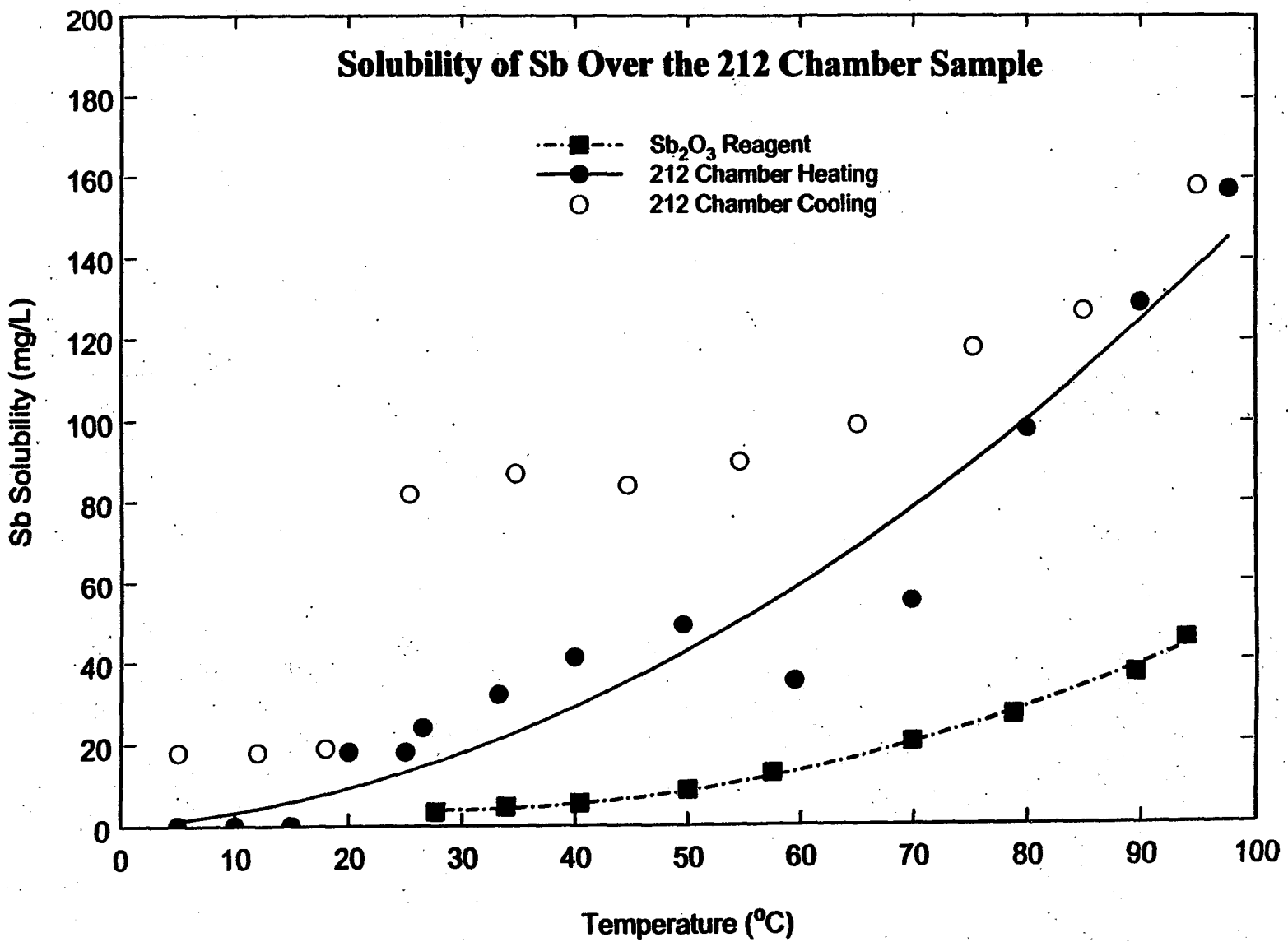


Fig. 32 - Experimentally determined solubility of Sb over the 212 Chamber sample.



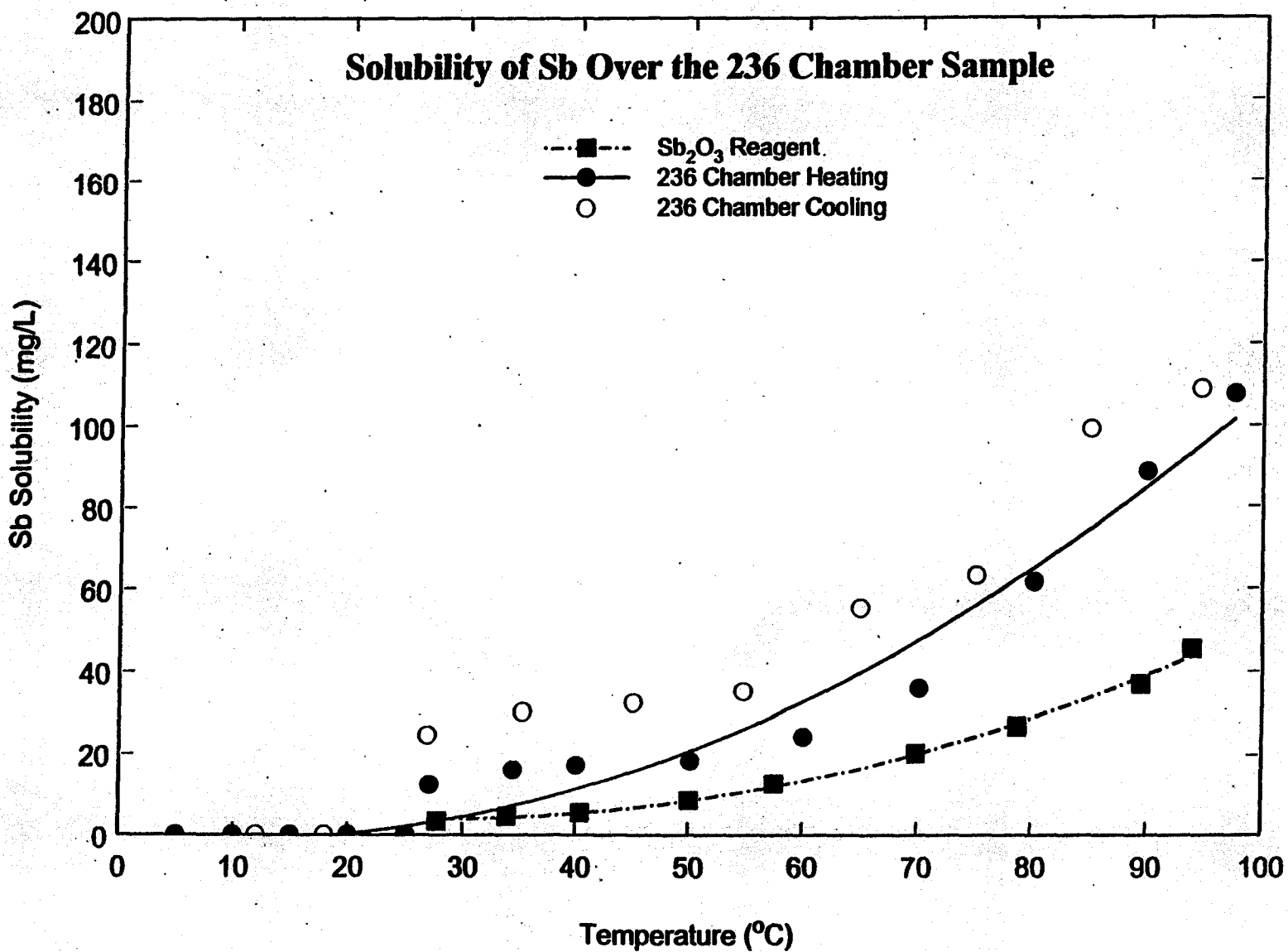


Fig. 33 - Experimentally determined solubility of Sb over the 236 Chamber sample.



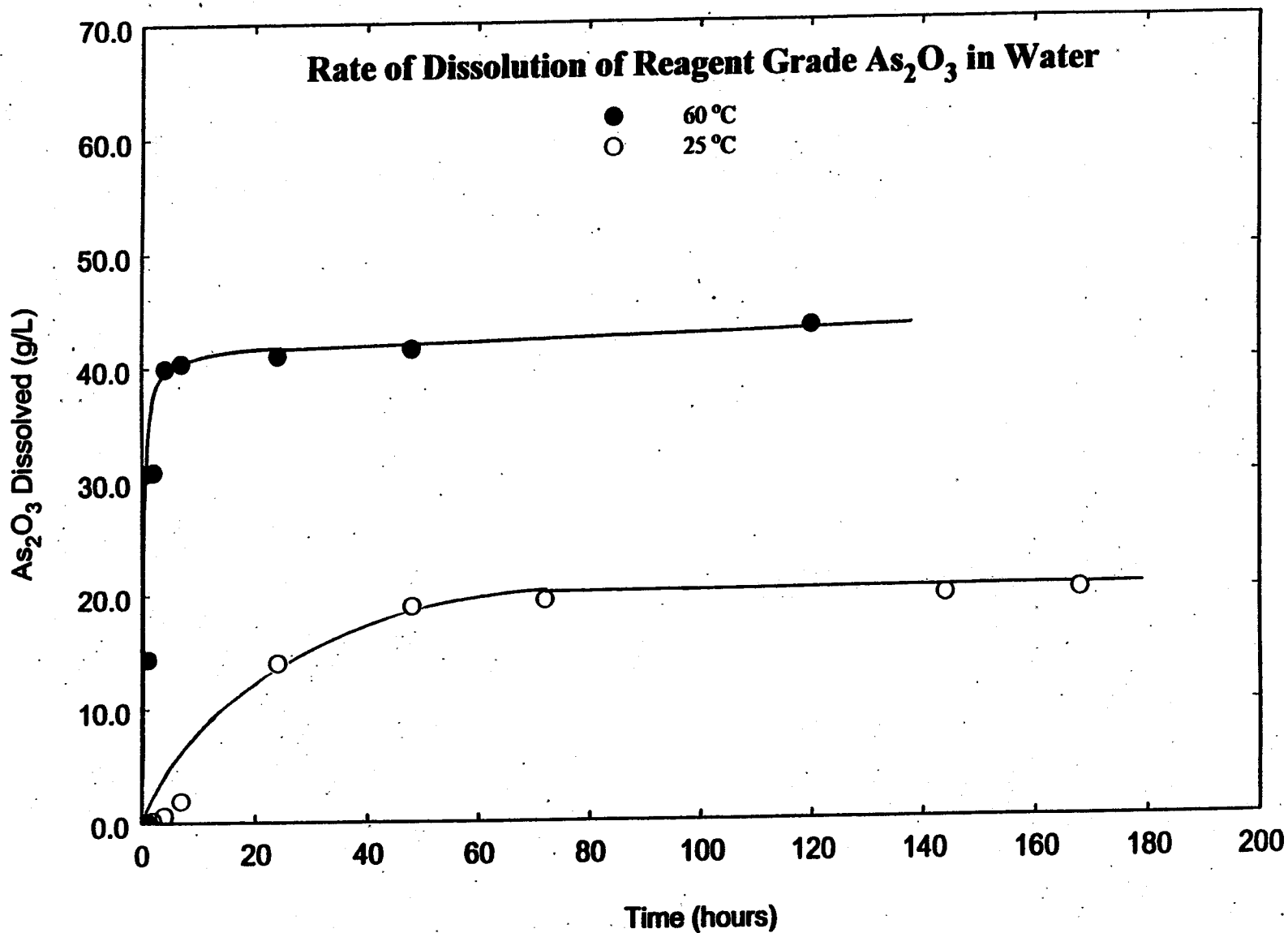


Fig. 34 - The solubility of  $\text{As}_2\text{O}_3$  from reagent grade  $\text{As}_2\text{O}_3$  in water as a function of time at 25°C and 60°C.



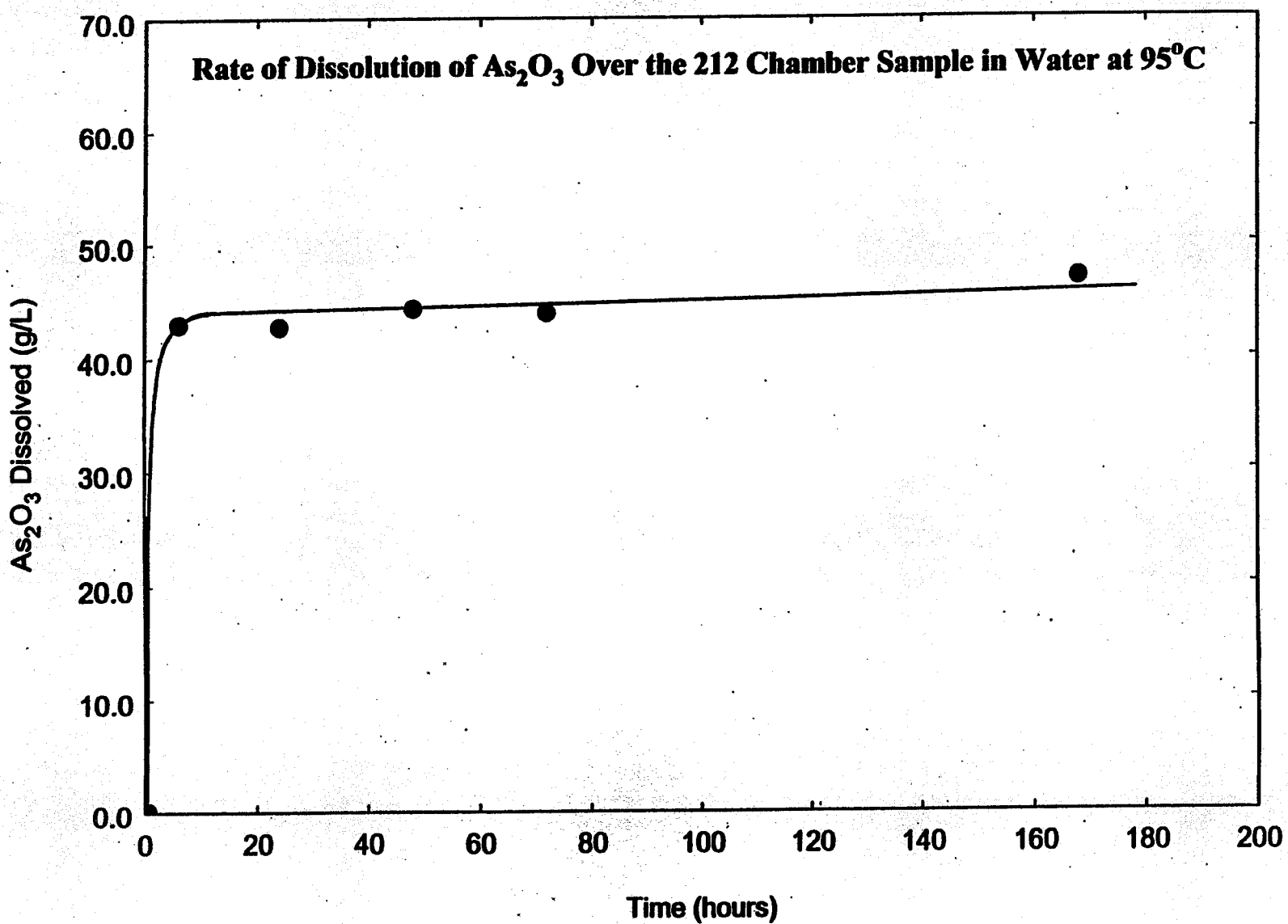


Fig. 35 - The solubility of  $\text{As}_2\text{O}_3$ , from the 212 Chamber sample, in water as a function of time at 95°C.



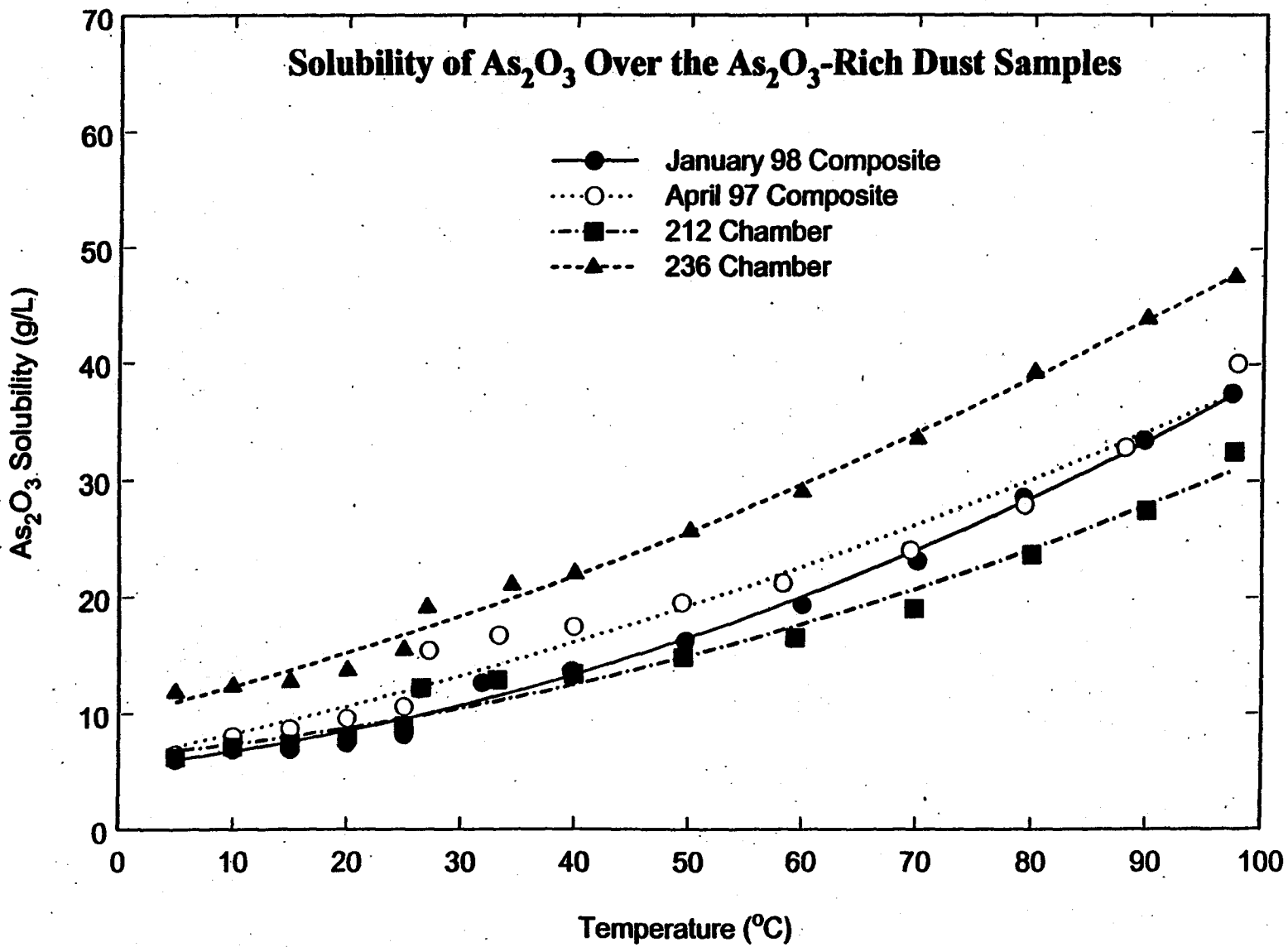


Fig. 36 - Comparison of the solubilities of  $\text{As}_2\text{O}_3$  over the four  $\text{As}_2\text{O}_3$ -rich dust samples as a function of temperature, as obtained on heating only.



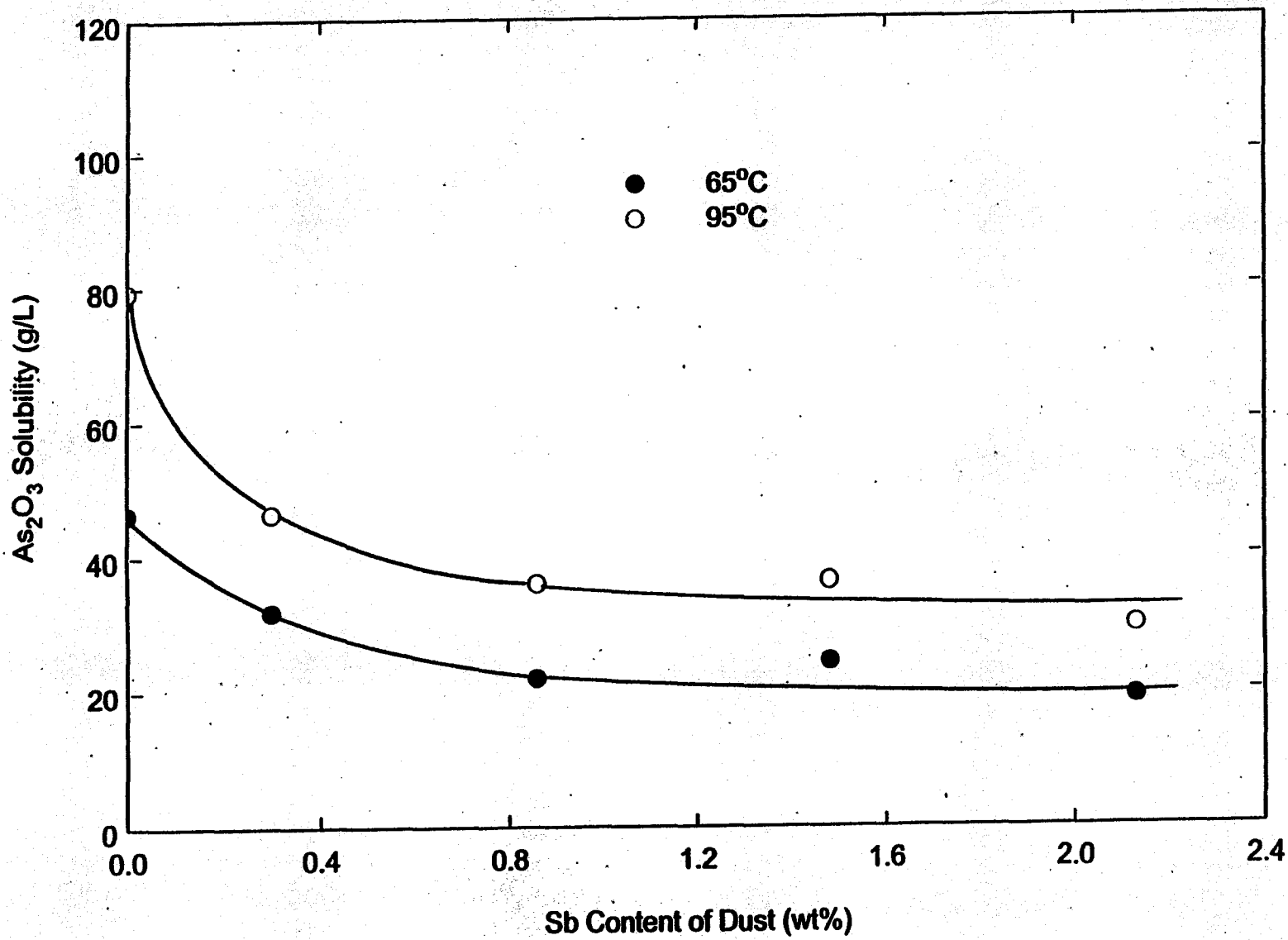


Fig. 37 - Solubility of As<sub>2</sub>O<sub>3</sub> over the four As<sub>2</sub>O<sub>3</sub>-rich dust samples as a function of the Sb content of the dust.



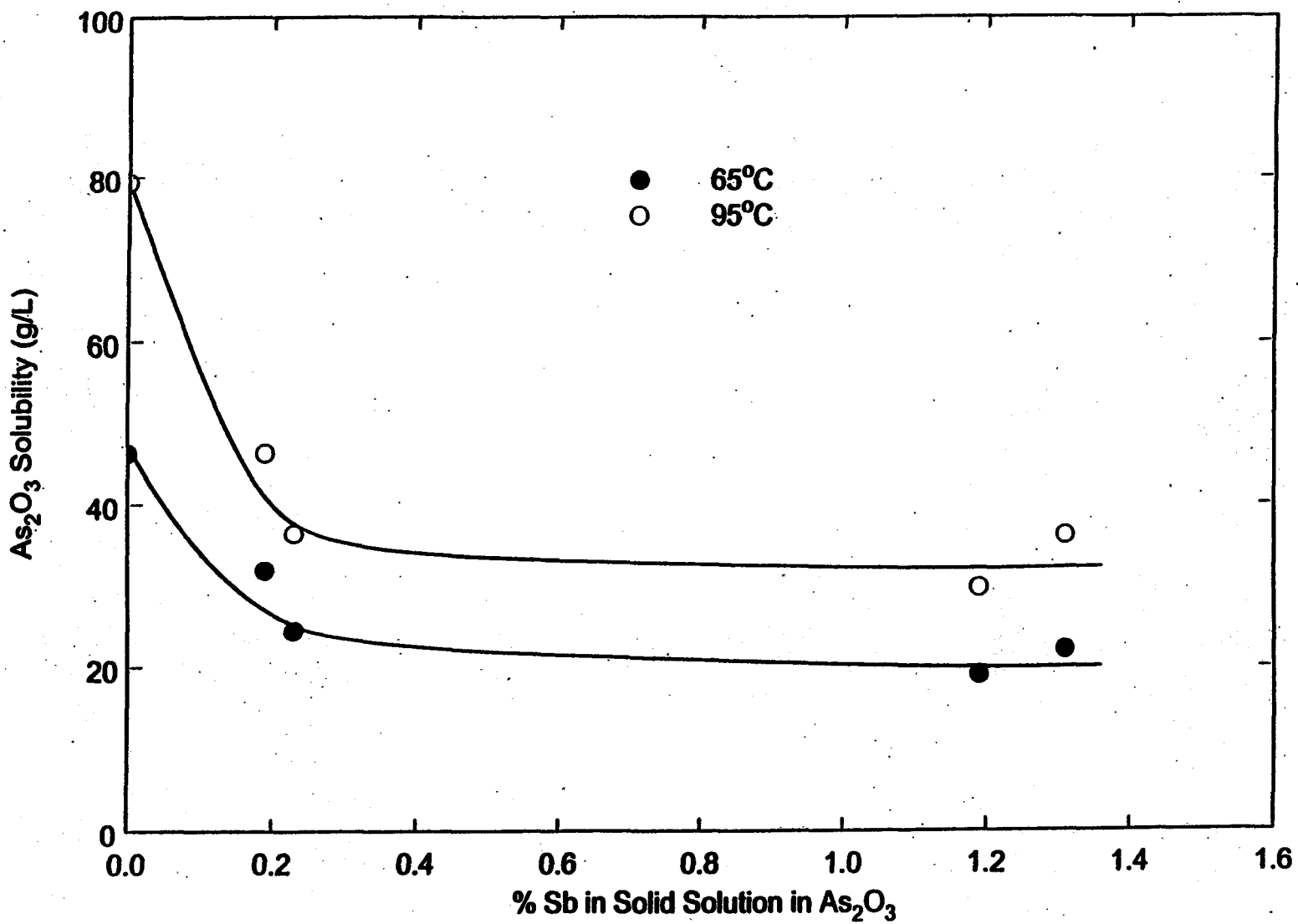


Fig. 38 - Solubility of  $\text{As}_2\text{O}_3$  over the four  $\text{As}_2\text{O}_3$ -rich dust samples as a function of the solid solution Sb content of the  $\text{As}_2\text{O}_3$  crystals in the dust.



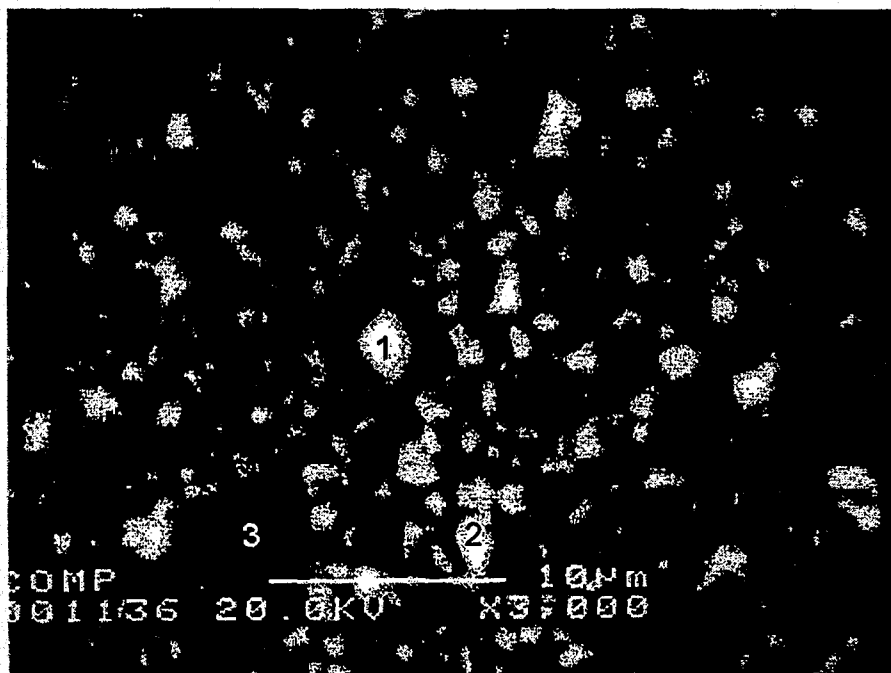


Fig. 39 – Backscattered electron micrograph showing the general morphology of the April 97 Composite sample after water leaching at 70 °C at a pulp density of 75 g/L solids. 1-  $\text{As}_2\text{O}_3$ , 2-  $(\text{Sb,As})_2\text{O}_3$ , 3- silicates

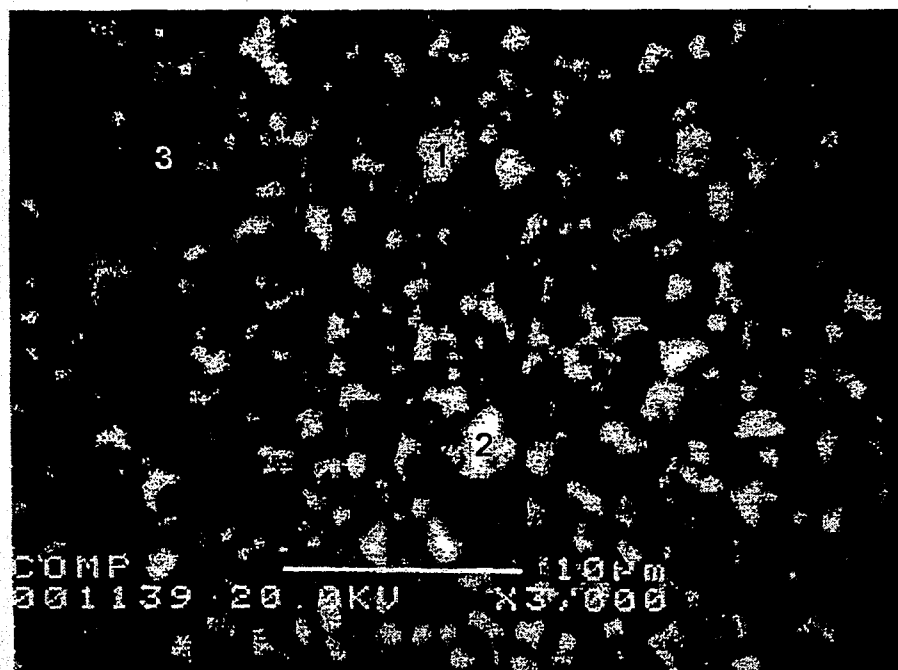


Fig. 40 - Backscattered electron micrograph showing the general morphology of the April 97 Composite sample after water leaching at 70 °C at a pulp density of 150 g/L solids. 1-  $\text{As}_2\text{O}_3$ , 2-  $(\text{Sb,As})_2\text{O}_3$ , 3- silicates

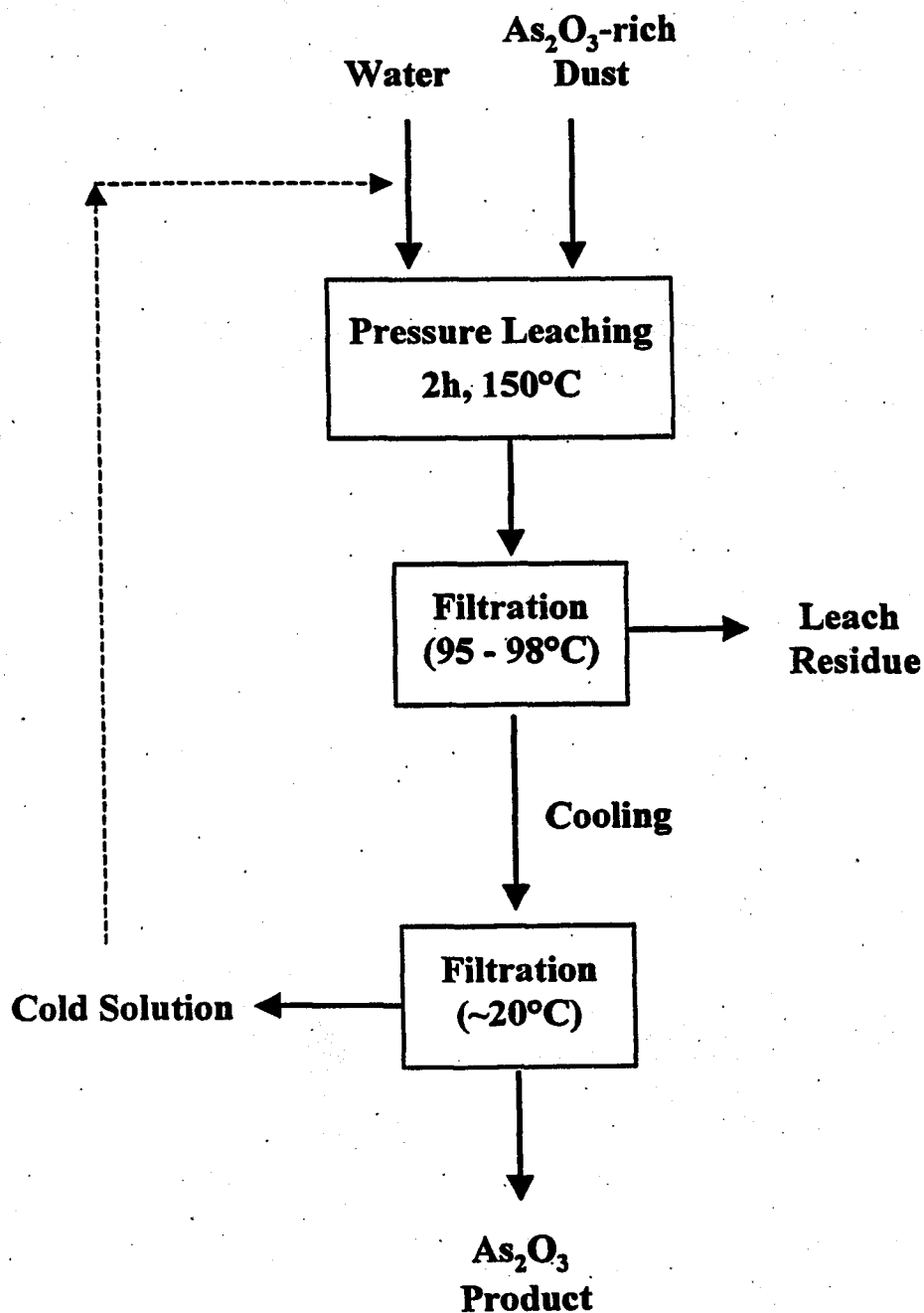


Fig. 41 - Schematic representation of the hot water leaching procedure used to produce an As<sub>2</sub>O<sub>3</sub>-saturated solution at 95°C, from which pure As<sub>2</sub>O<sub>3</sub> can be crystallized.

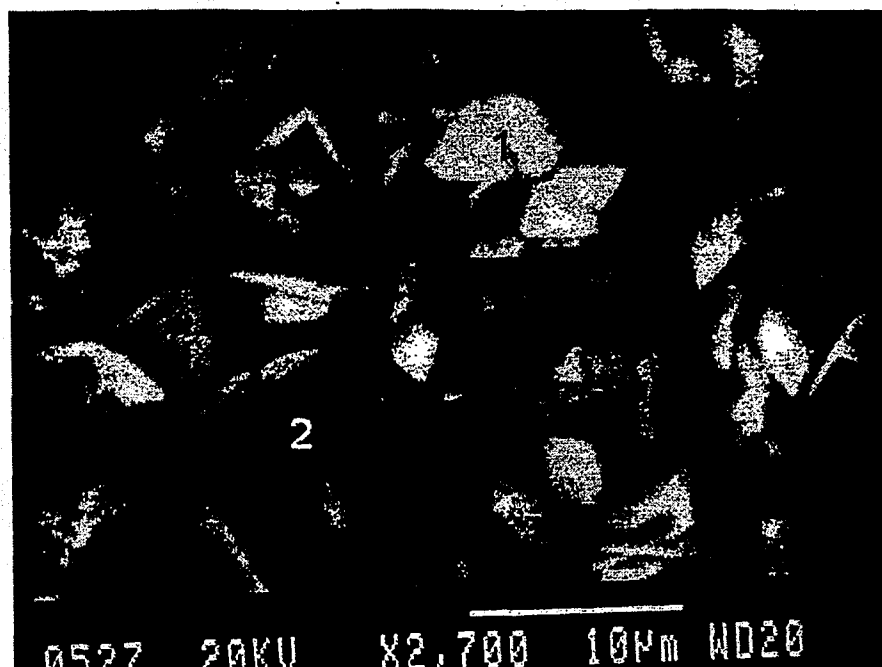


Fig. 42 - Backscattered electron micrograph of the residue from the leaching of 77 g/L of the April 97 Composite sample for 2 h at 150°C.  
1- recrystallized  $(\text{Sb,As})_2\text{O}_3$ , 2- chlorite and muscovite

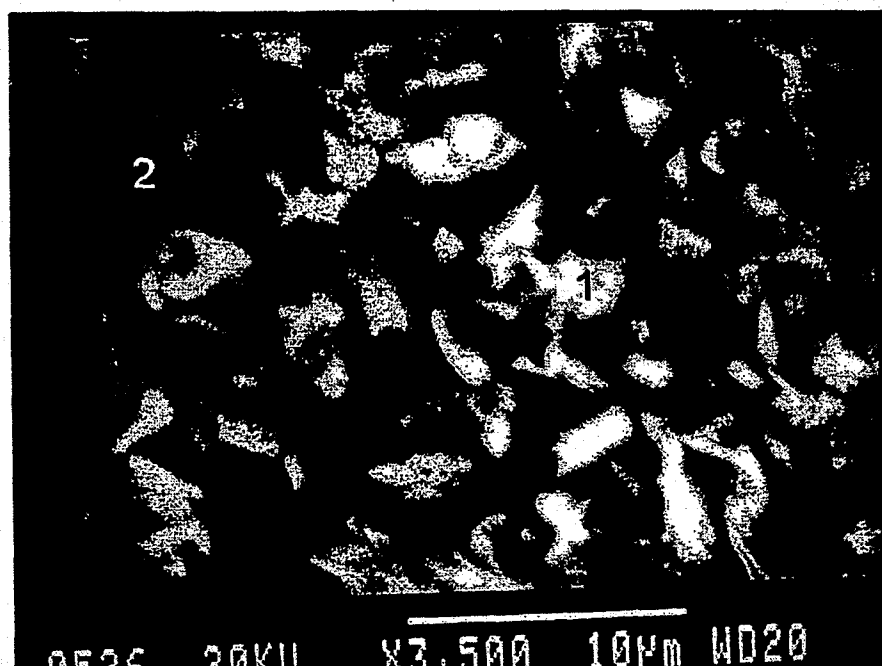


Fig. 43 - Backscattered electron micrograph of the residue from the leaching of 85 g/L of the April 97 Composite sample for 2 h at 150°C.  
1- recrystallized  $(\text{Sb,As})_2\text{O}_3$ , 2- chlorite and muscovite

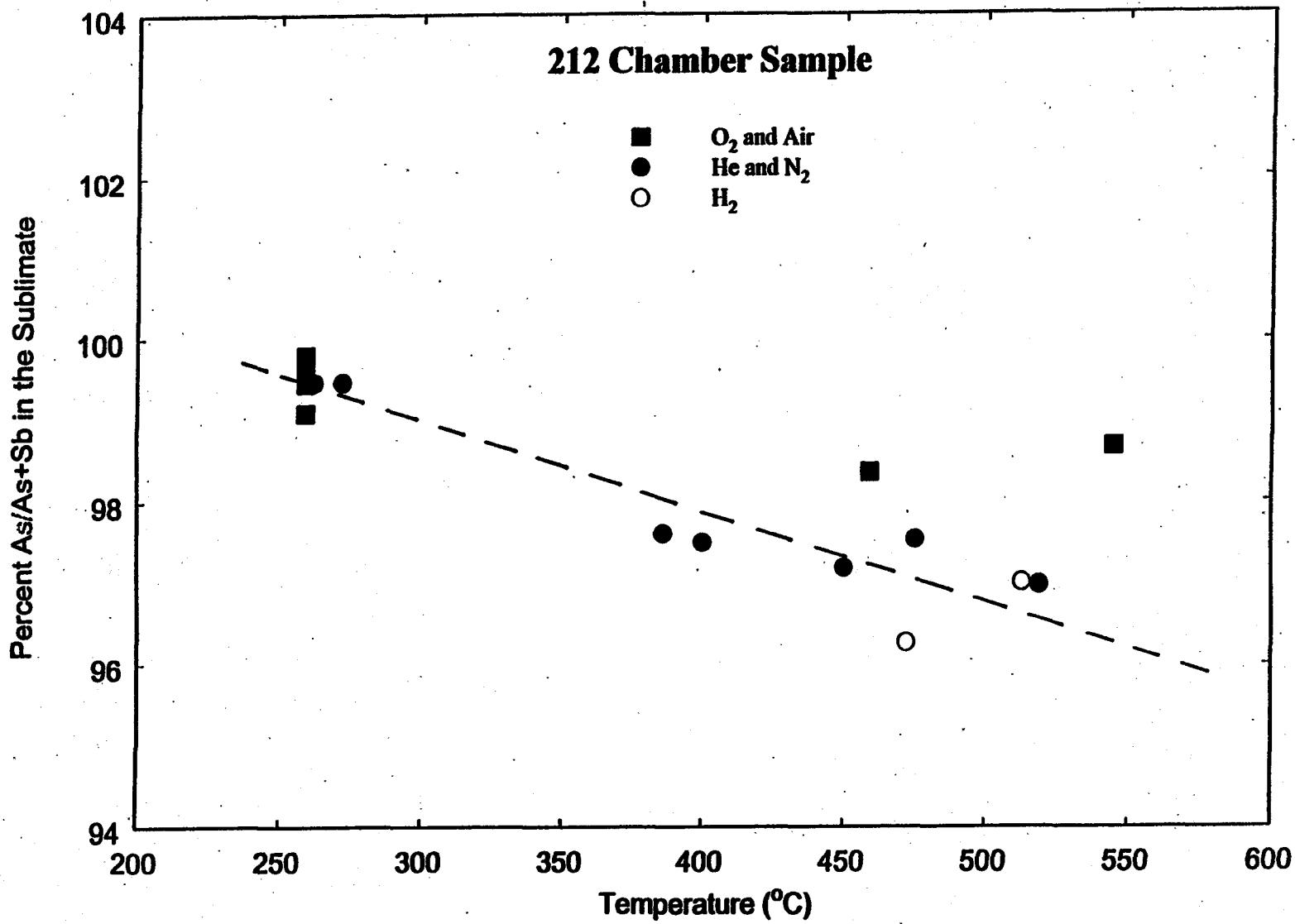


Fig. 44 - Effect of temperature and the sublimation atmosphere on the As/As+Sb ratio of the re-sublimed 212 Chamber sample.



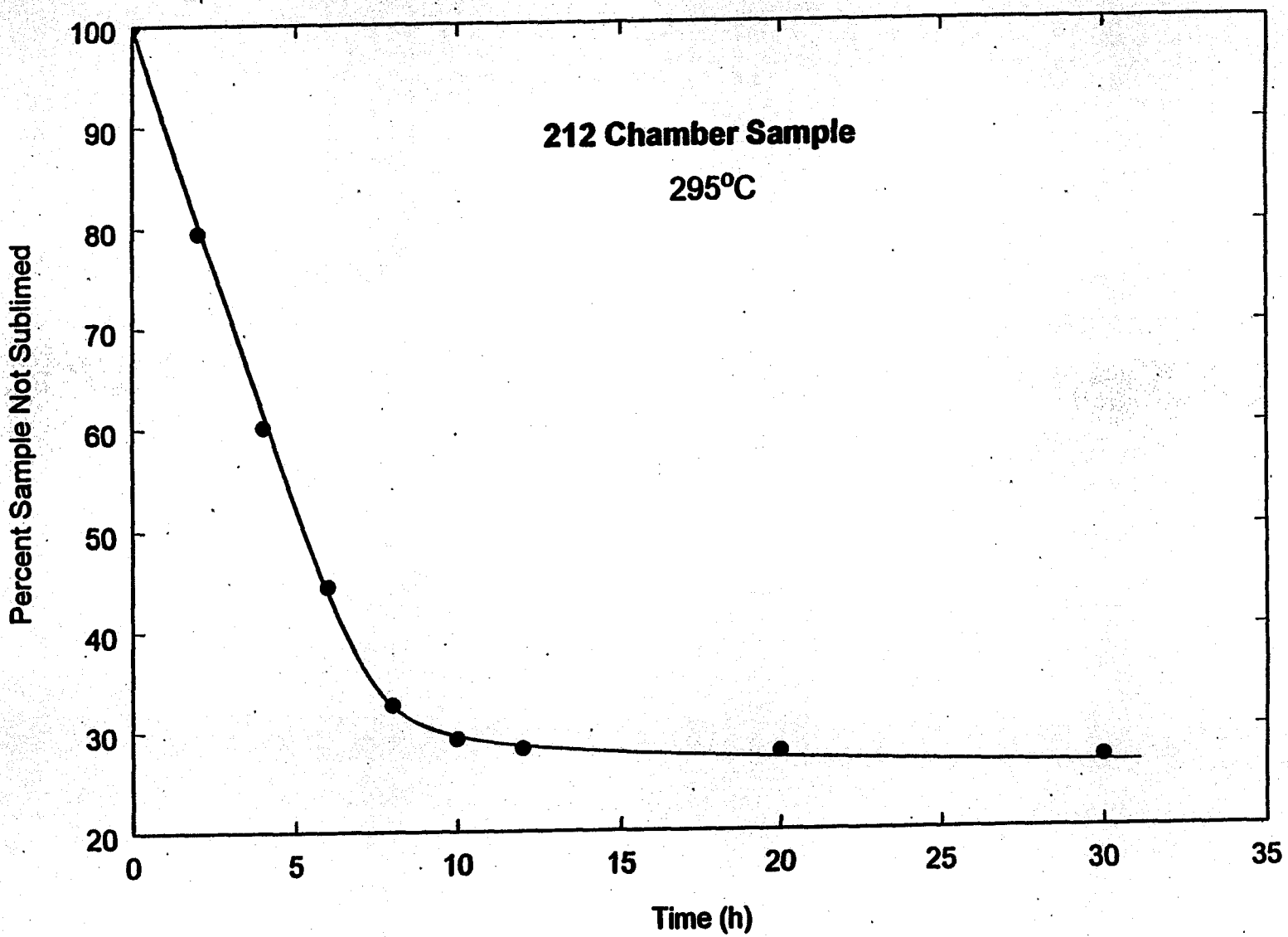


Fig. 45 - Extent of re-sublimation of the 212 Chamber sample as a function of heating time at 259°C.



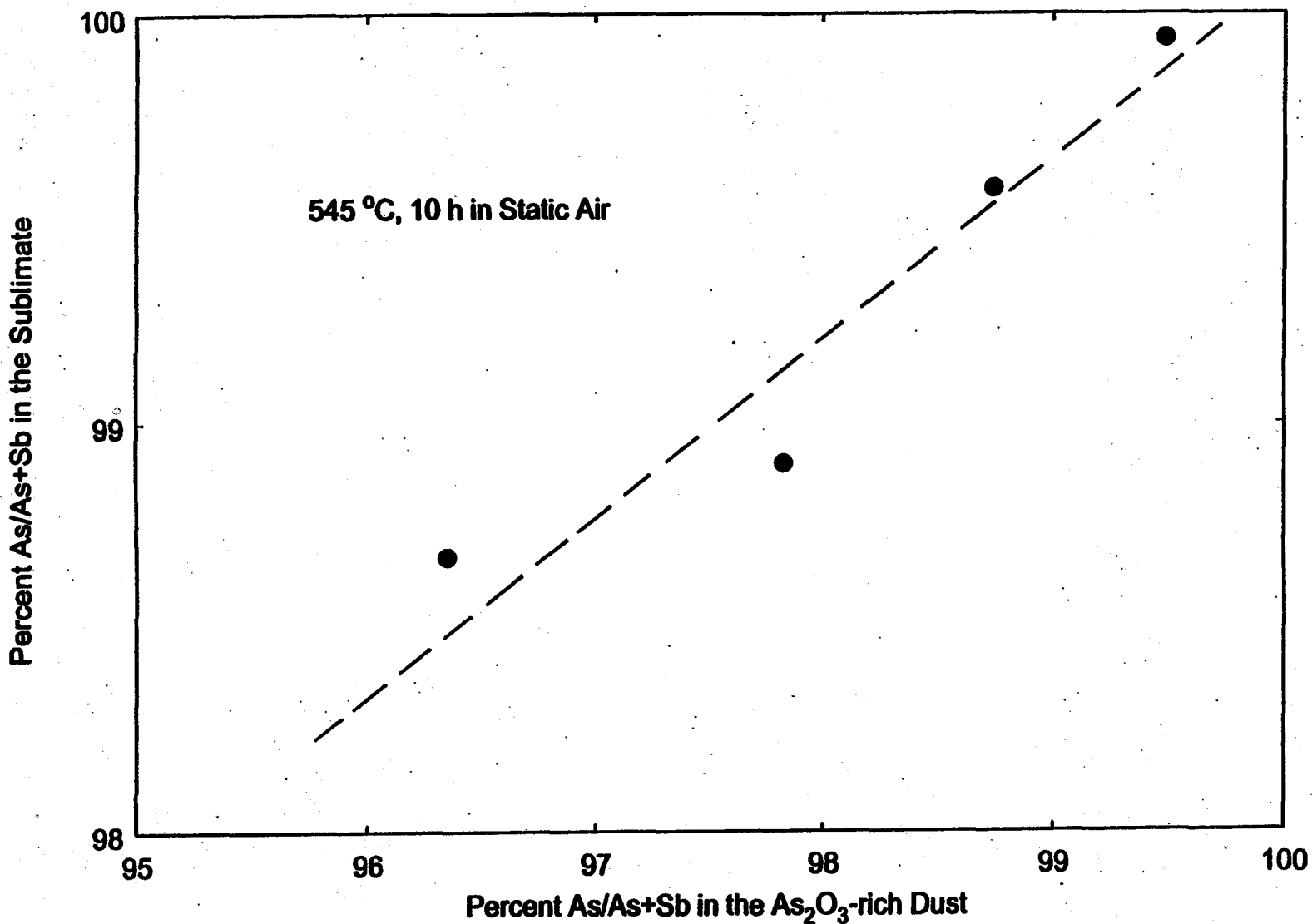


Fig. 46 - Relationship between the As/As+Sb percentage of the  $\text{As}_2\text{O}_3$ -rich dust and the As/As+Sb percentage of the re-sublimation product made at 545°C in air.

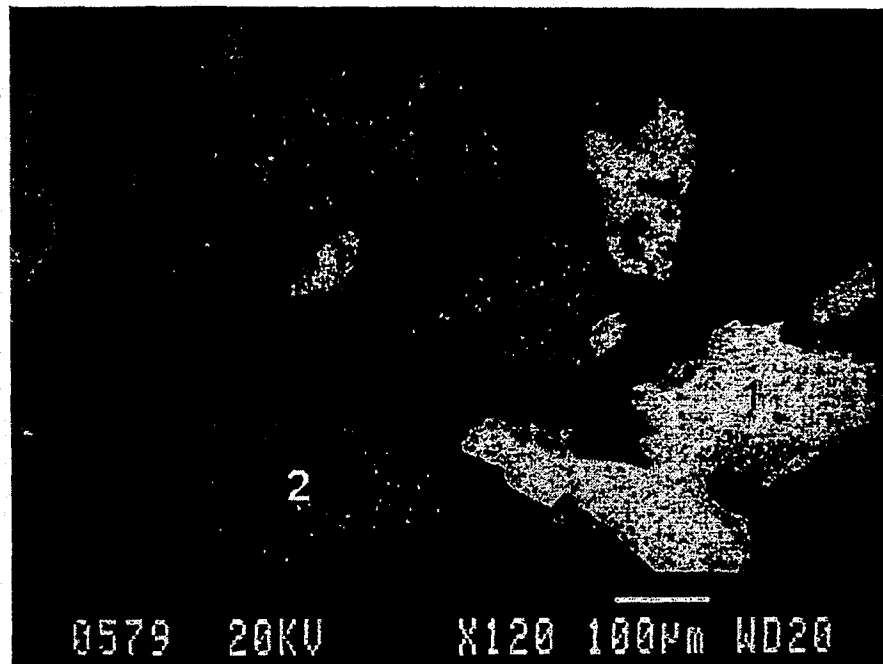


Fig. 47 - Backscattered electron micrograph showing the sublimation residue after heating the January 98 Composite sample at 545°C.  
1-  $(\text{As,Sb})_2\text{O}_3$ , 2- clusters of silicates and iron arsenate

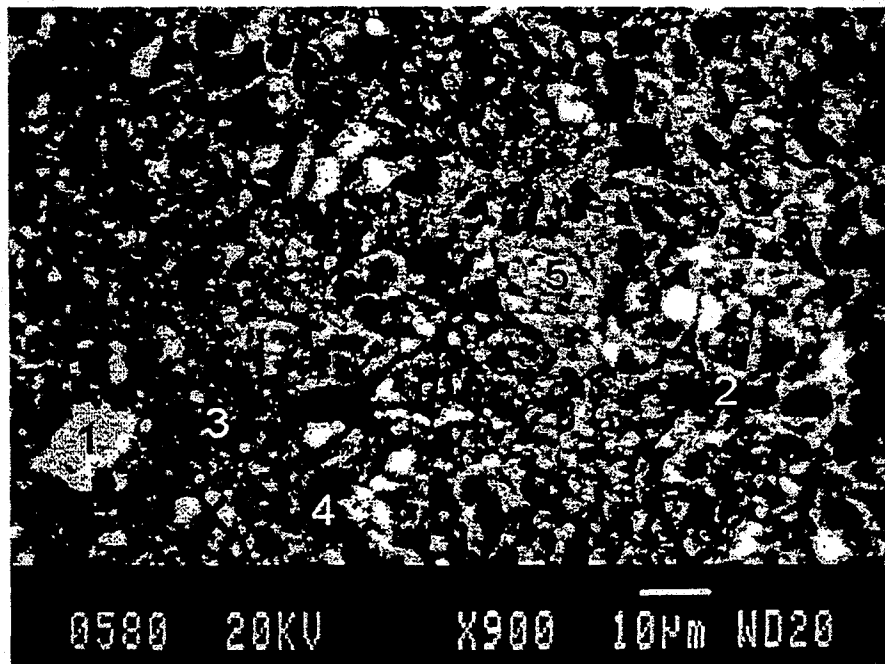


Fig. 48 - Backscattered electron micrograph showing the detailed texture of the agglomerates present in the sublimation residue from the January 98 Composite sample. 1- iron arsenate, 2-  $\text{CaSO}_4$ , 3- muscovite, 4- quartz, 5- mixture of iron arsenates and silicates, 6-  $\text{CaSO}_4$

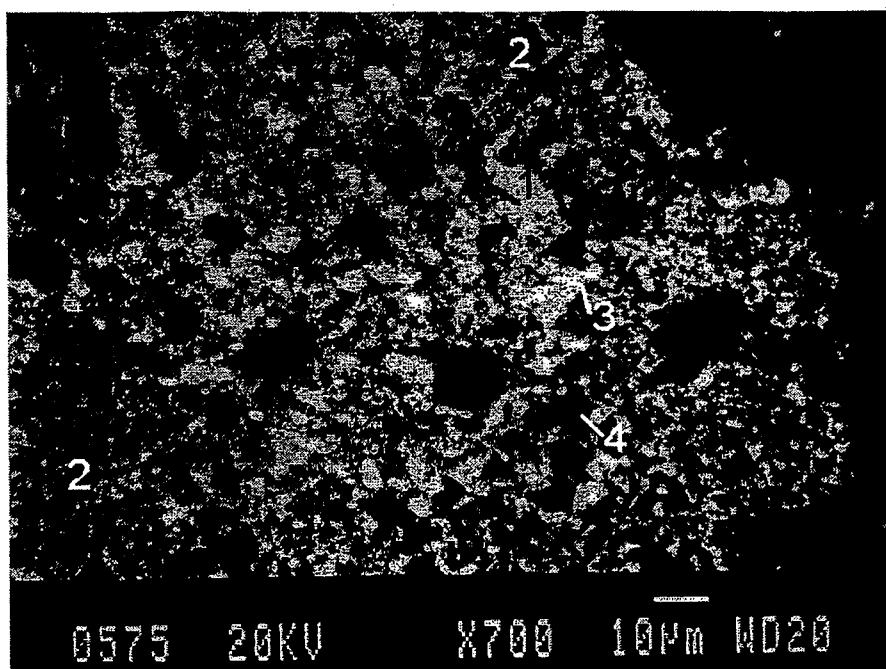


Fig. 49 - Backscattered electron micrograph of the sublimation residue from heating the April 97 Composite sample at 545°C. 1- iron arsenate, 2- muscovite, 3- iron arsenate-antimonate (bright rim), 4- quartz

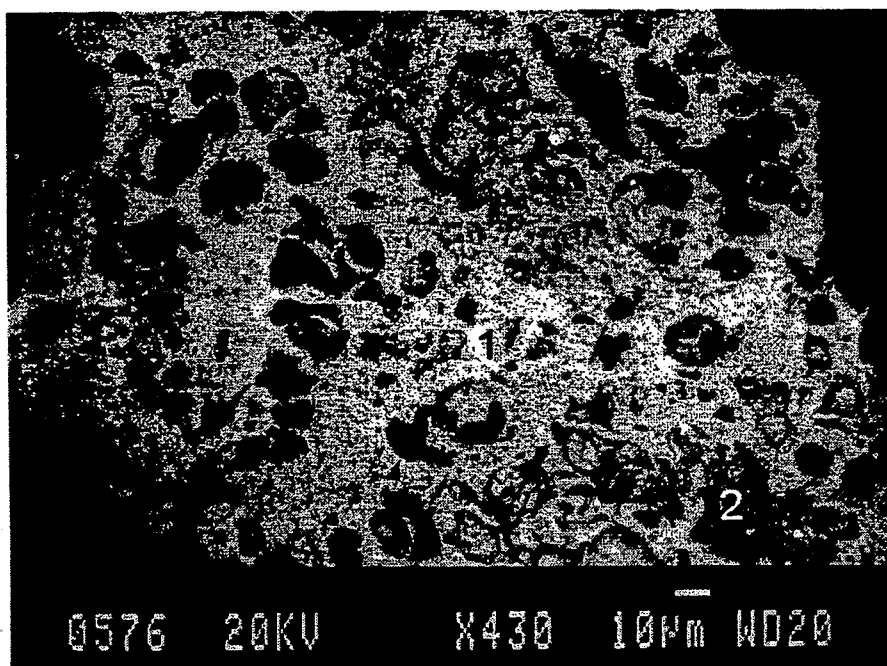


Fig. 50 - Backscattered electron micrograph illustrating the morphology of  $\text{As}_2\text{O}_3$  in the sublimation residue from the heating of the April 97 Composite sample. 1-  $\text{As}_2\text{O}_3$ , 2- muscovite (grey)

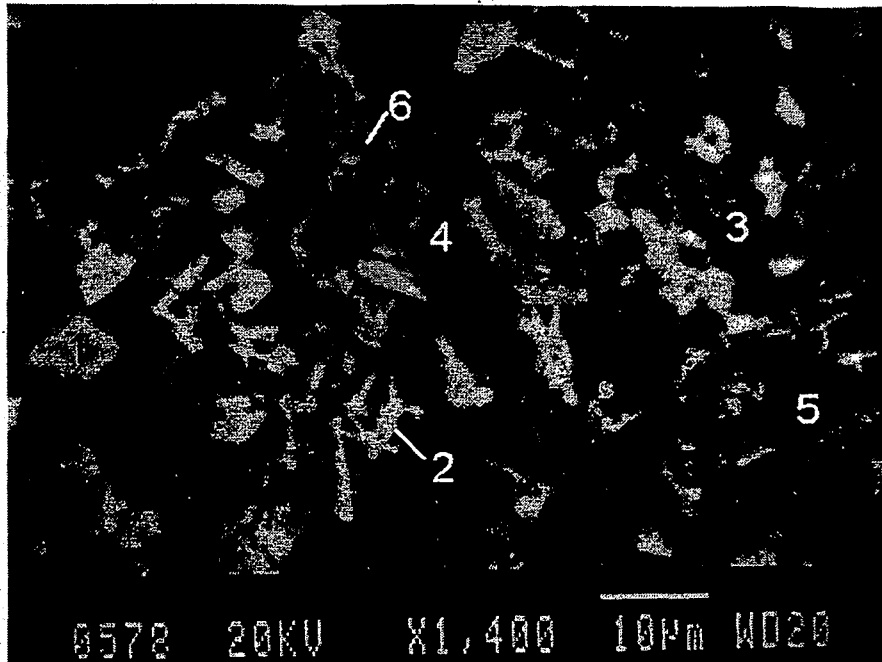


Fig. 51 - Backscattered electron micrograph of the sublimation residue from heating the 236 Chamber sample to 545 °C. 1- iron arsenate, 2- $\text{As}_2\text{O}_3$ , 3-  $\text{CaSO}_4$ , 4- quartz, 5- chlorite, 6- muscovite

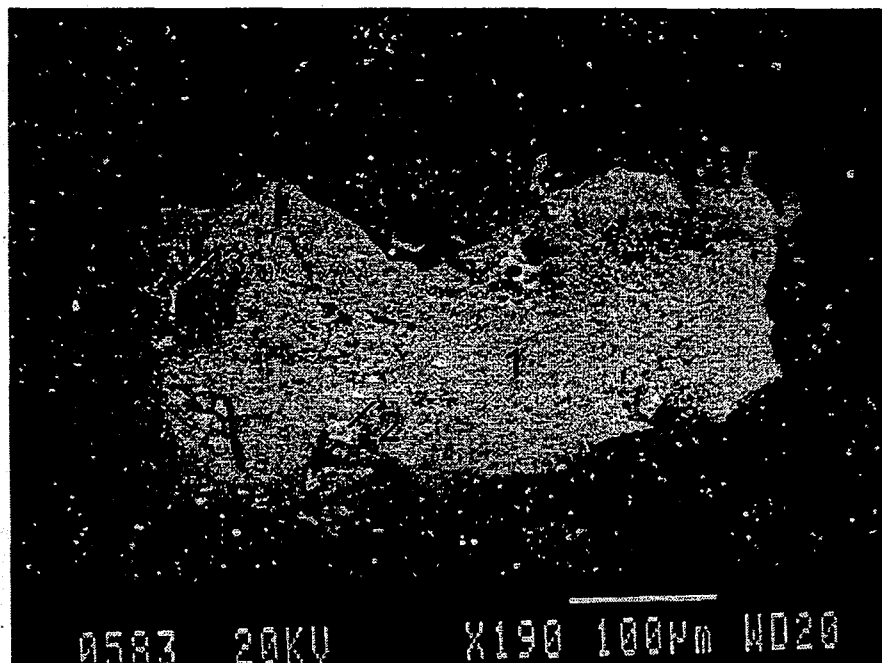


Fig. 52 - Backscattered electron micrograph of a large mass of arsenic oxide in the sublimation residue from heating the 212 Chamber sample to 610°C. 1- arsenic oxide, 2- iron arsenate, 3- silicates

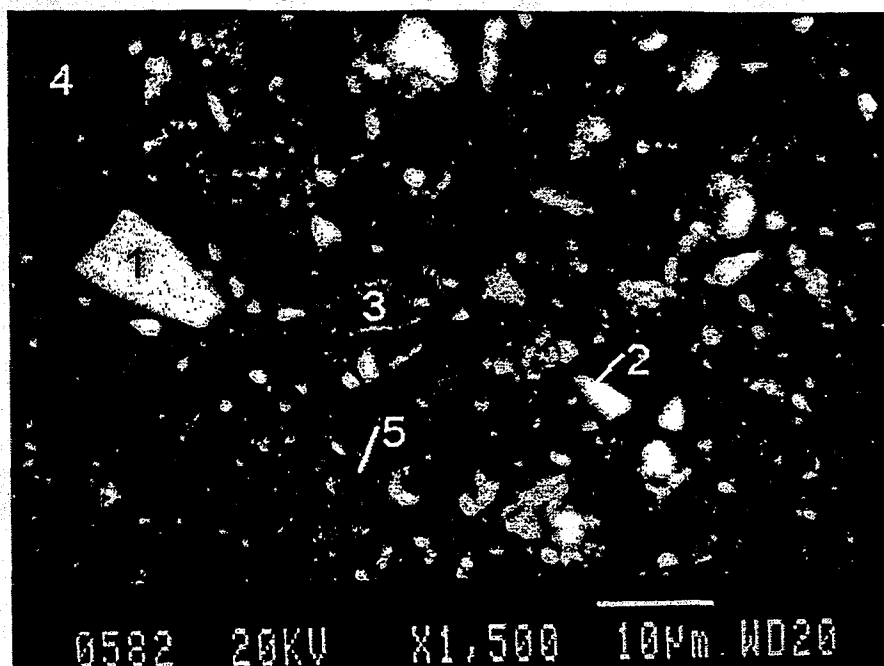


Fig. 53 - Backscattered electron micrograph showing a number of tiny particles in the sublimation residue from the heating of the 212 Chamber sample.  
1- iron oxide, 2- iron arsenate, 3- Ca arsenate, 4- chlorite, 5- muscovite

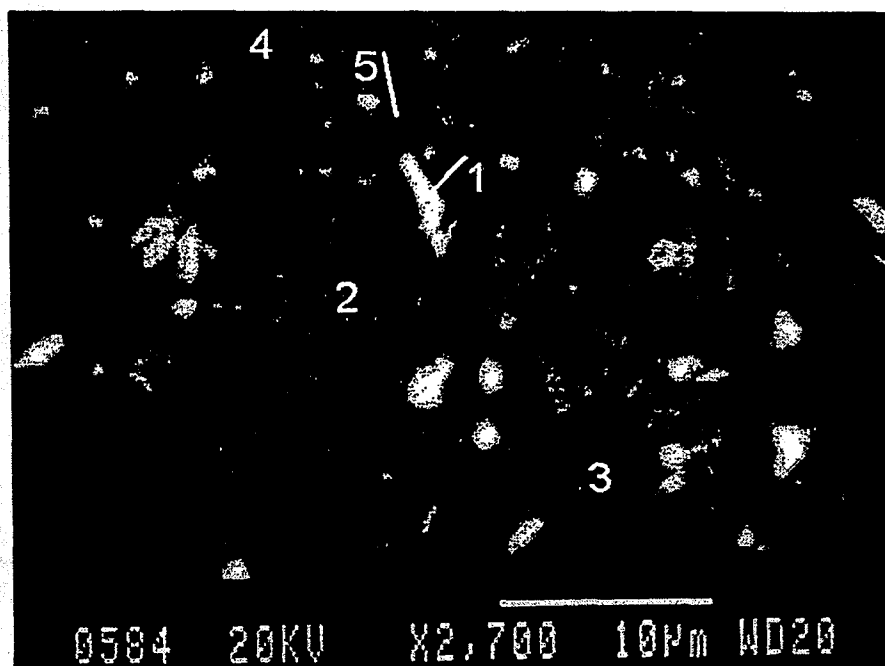


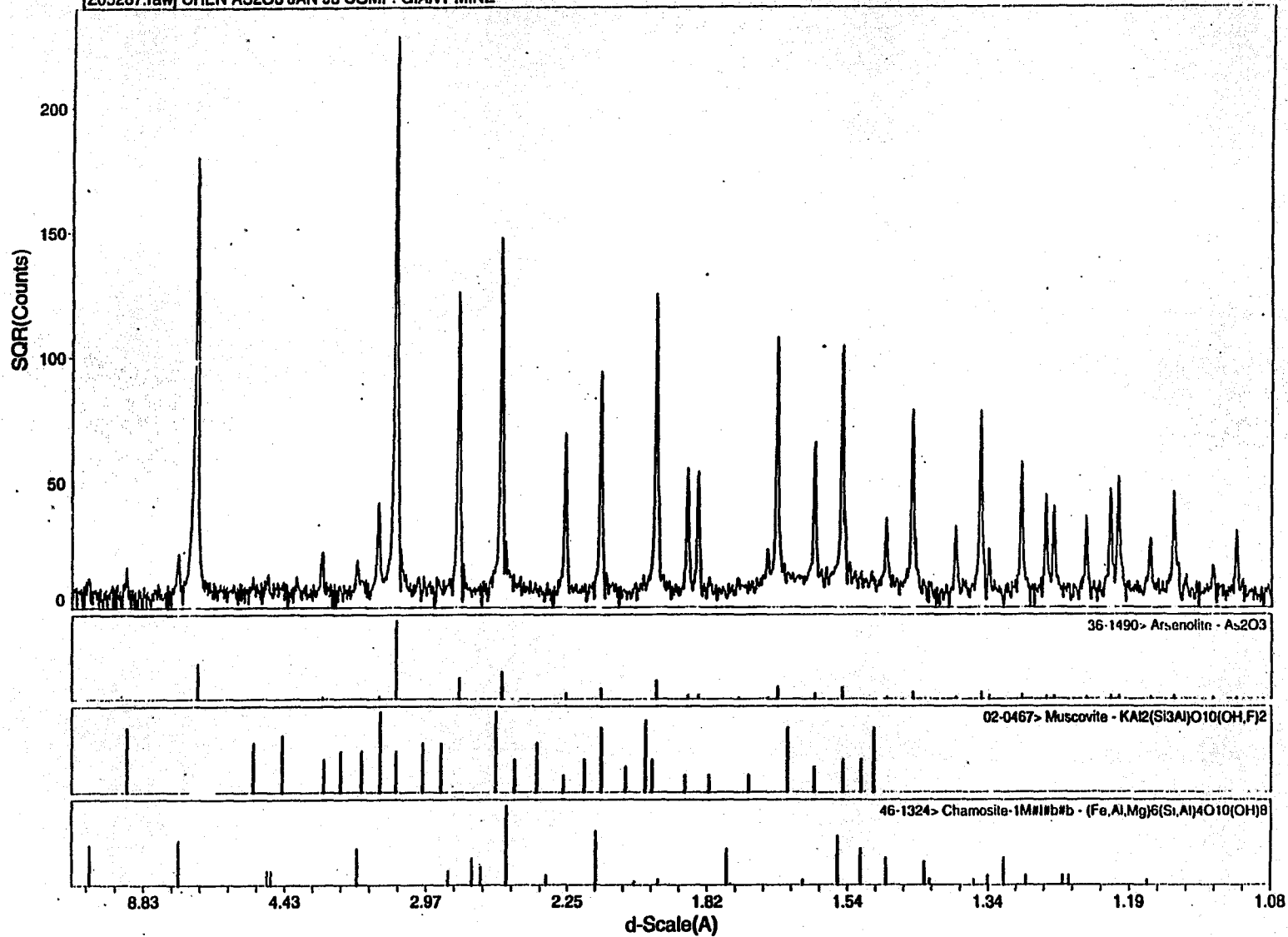
Fig. 54 - Backscattered electron micrograph showing the fine particles present in the sublimation residue from the heating of the 212 Chamber sample.  
1- iron arsenate, 2-  $\text{CaSO}_4$ , 3- quartz, 4- chlorite, 5- muscovite

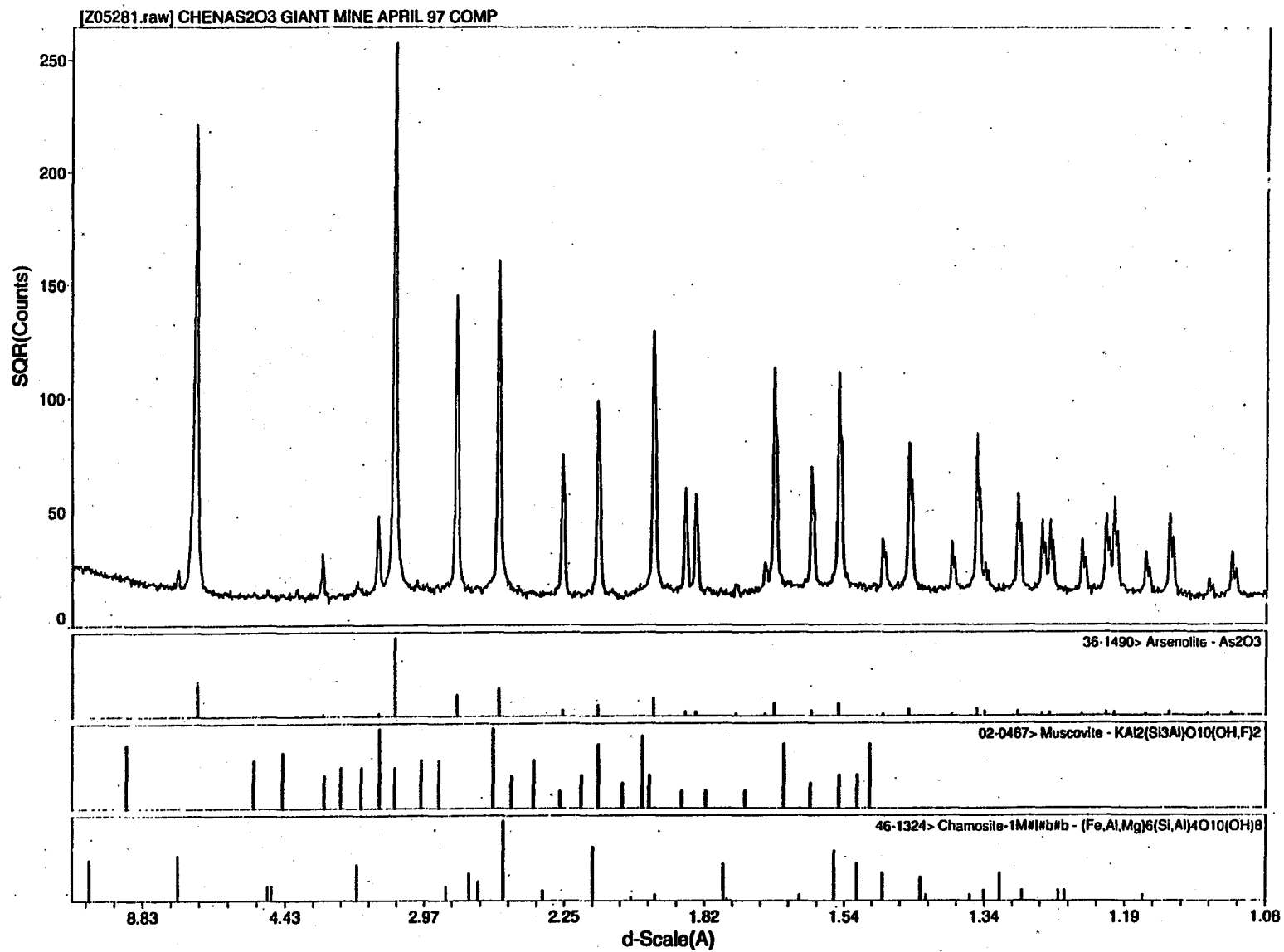
## **APPENDIX 1**

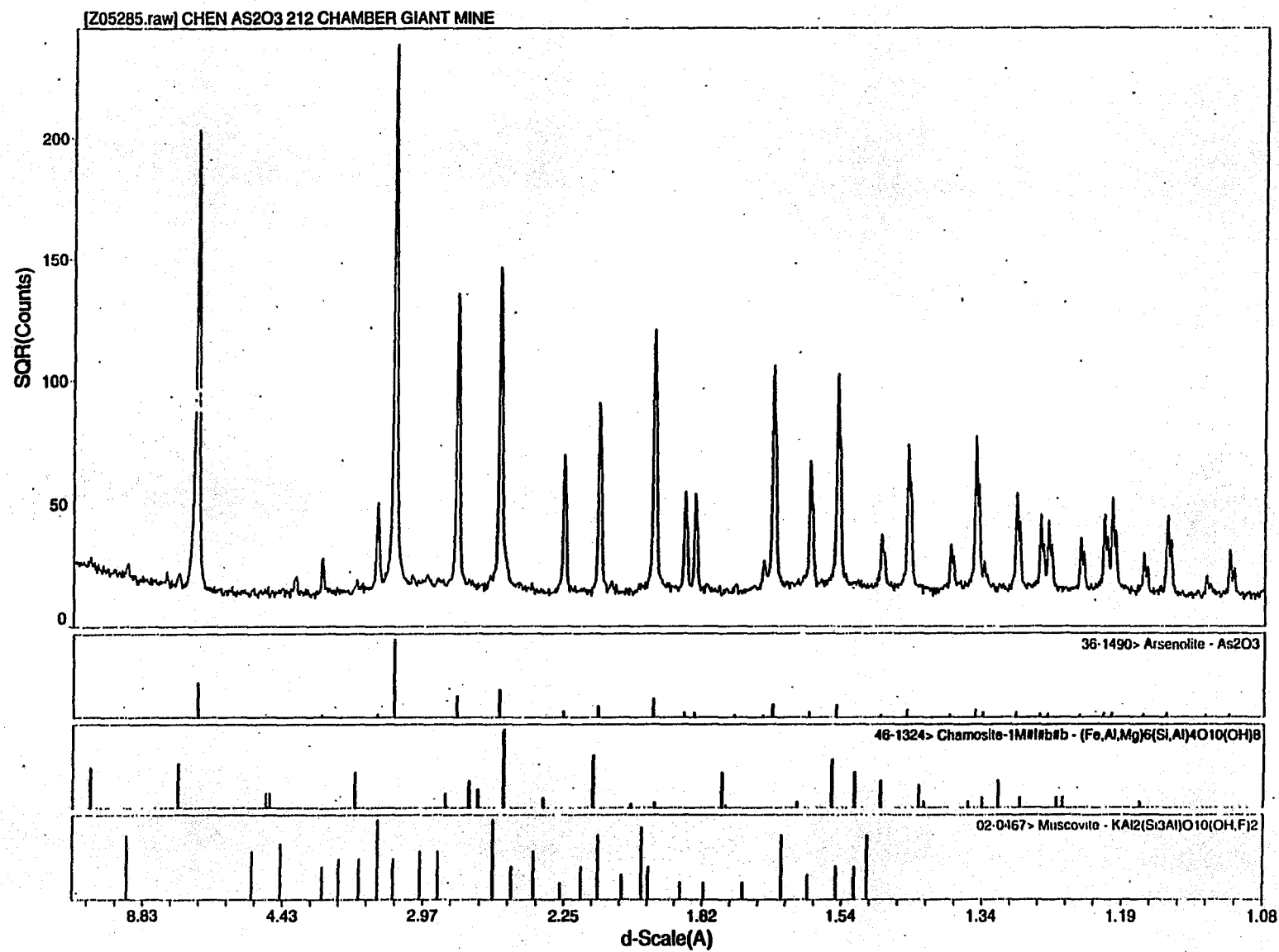
### **X-ray Diffraction Patterns of the Four Dust Samples**

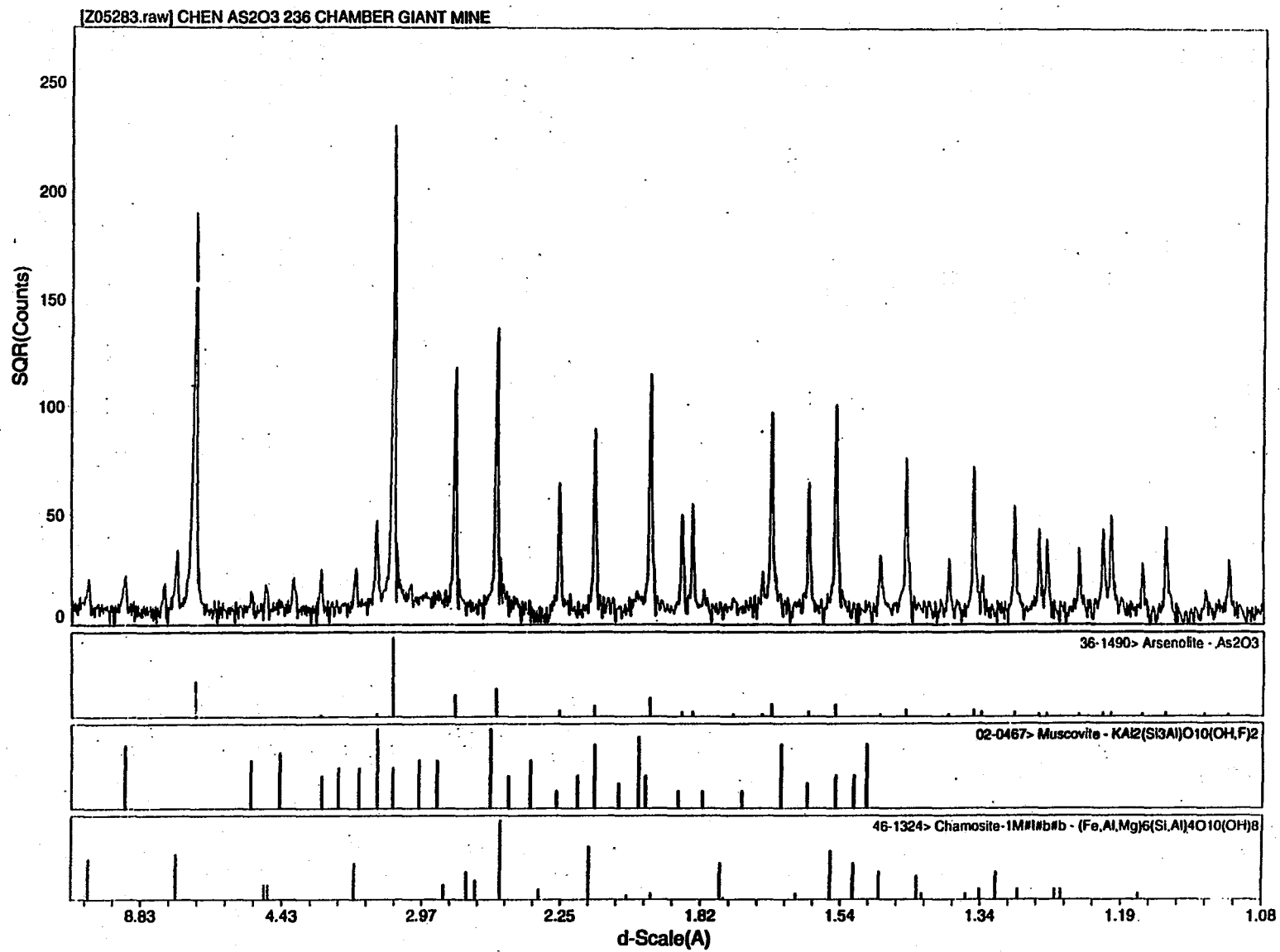


[Z05287.raw] CHEN AS2O3 JAN 98 COMP. GIANT MINE









## **APPENDIX 2**

### **Detailed Electron Microprobe Analyses of the Arsenic Carriers in the January 98 Composite Sample**



As<sub>2</sub>O<sub>3</sub> Phase

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Weight percent		Group : CHEN				Sample : As2O3-Giant		Page 1
Element	64	65	66	67	68	73	74	77
Sb	0.565	0.473	0.749	0.391	0.252	0.824	-	-
As	78.306	81.706	77.828	77.738	71.230	70.437	76.266	78.963
Fe	0.148	0.087	0.154	0.233	0.066	0.459	0.135	0.136
Pb	-	-	0.072	0.072	0.066	0.020	0.021	0.012
S	-	-	-	-	-	-	-	-
Cr	-	-	-	-	-	-	-	-
O	20.981	17.734	21.197	21.566	28.386	28.260	23.578	20.889
Ca	-	-	-	-	-	-	-	-
Bi	-	-	-	-	-	-	-	-
Si	-	-	-	-	-	-	-	-
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Weight percent		Group : CHEN				Sample : As2O3-Giant		
Element	79	80	81	82	93	94	95	96
Sb	0.219	1.236	-	0.249	0.068	0.185	0.096	0.137
As	74.593	78.837	74.456	74.419	71.324	76.143	80.717	72.961
Fe	0.079	0.063	0.058	0.040	0.592	0.083	0.096	0.129
Pb	-	0.049	-	-	-	-	-	-
S	-	-	-	-	-	-	-	-
Cr	-	-	-	-	-	-	-	-
O	25.109	19.815	25.486	25.292	28.016	23.589	19.091	26.773
Ca	-	-	-	-	-	-	-	-
Bi	-	-	-	-	-	-	-	-
Si	-	-	-	-	-	-	-	-
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Weight percent		Group : CHEN				Sample : As2O3-Giant		
Element	98	99	100	101	103	104	106	107
Sb	1.890	2.189	1.708	4.028	0.142	0.485	0.358	0.004
As	74.045	67.162	69.288	63.048	79.110	78.778	80.137	81.485
Fe	0.104	0.076	0.118	0.295	0.010	0.096	0.091	0.016
Pb	-	-	0.109	0.076	0.076	-	0.016	0.003
S	-	-	-	-	-	-	-	-
Cr	-	-	-	-	-	-	-	-
O	23.961	30.573	28.777	32.553	20.662	20.641	19.398	18.492
Ca	-	-	-	-	-	-	-	-
Bi	-	-	-	-	-	-	-	-
Si	-	-	-	-	-	-	-	-
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000



# PROTECTED BUSINESS INFORMATION

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Weight percent		Group : CHEN				Sample : As2O3-Giant		Page 2
Element	108	109	110	111	112	113	114	115
Sb	5.244	4.470	4.815	4.700	4.220	5.010	5.383	0.093
As	70.591	74.667	66.640	73.101	76.093	68.165	75.553	73.885
Fe	0.321	0.113	2.015	0.103	0.075	0.078	0.164	0.051
Pb	1.886	0.040	0.080	-	-	-	0.022	-
S	-	-	-	-	-	-	-	-
Cr	-	-	-	-	-	-	-	-
O	21.958	20.710	26.450	22.096	19.612	26.747	18.878	25.971
Ca	-	-	-	-	-	-	-	-
Bi	-	-	-	-	-	-	-	-
Si	-	-	-	-	-	-	-	-
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Weight percent		Group : CHEN				Sample : As2O3-Giant	
Element	116	117	118	119	120	121	122
Sb	0.125	0.190	0.204	0.098	0.086	0.229	0.109
As	77.097	82.115	79.724	75.853	75.723	74.496	73.427
Fe	0.057	0.065	0.069	0.018	0.066	0.263	0.066
Pb	-	-	-	0.026	0.026	-	-
S	-	-	-	-	-	-	-
Cr	-	-	-	-	-	-	-
O	22.721	17.630	20.003	24.005	24.099	25.012	26.398
Ca	-	-	-	-	-	-	-
Bi	-	-	-	-	-	-	-
Si	-	-	-	-	-	-	-
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Weight percent		Group : CHEN			Sample : As2O3-Giant
Element	Minimum	Maximum	Average	Sigma	
Sb	-	5.383	1.313	1.843	
As	63.048	82.115	75.028	4.449	
Fe	0.010	2.015	0.177	0.324	
Pb	-	1.886	0.069	0.300	
S	-	-	-	-	
Cr	-	-	-	-	
O	17.630	32.553	23.413	3.722	
Ca	-	-	-	-	
Bi	-	-	-	-	
Si	-	-	-	-	
Total	100.000	100.000	100.000	0.000	
No. of data 39					



$(\text{Sb,As})_2\text{O}_3$  Phase

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Weight percent		Group : CHEN				Sample : As2O3-Giant		Page 1
Element	83	84	85	86	87	88	89	90
Sb	43.796	43.086	45.125	46.538	44.369	47.773	45.406	45.197
As	31.385	32.624	35.882	38.956	36.116	37.141	37.692	35.961
Fe	0.039	0.019	0.042	0.088	0.043	0.070	0.049	0.022
Pb	0.066	-	-	-	-	-	-	-
S	-	-	-	-	-	-	-	-
Cr	-	-	-	-	-	-	-	-
O	24.714	24.271	18.951	14.418	19.472	15.016	16.853	18.820
Ca	-	-	-	-	-	-	-	-
Bi	-	-	-	-	-	-	-	-
Si	-	-	-	-	-	-	-	-
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Weight percent		Group : CHEN		Sample : As2O3-Giant		
Element	91	92	Minimum	Maximum	Average	Sigma
Sb	44.779	40.505	40.505	47.773	44.657	1.968
As	36.910	28.946	28.946	38.956	35.161	3.148
Fe	0.076	0.029	0.019	0.088	0.048	0.023
Pb	-	-	-	0.066	0.007	0.021
S	-	-	-	-	-	-
Cr	-	-	-	-	-	-
O	18.235	30.520	14.418	30.520	20.127	4.975
Ca	-	-	-	-	-	-
Bi	-	-	-	-	-	-
Si	-	-	-	-	-	-
Total	100.000	100.000	100.000	100.000	100.000	0.000
No. of data 10						



## As-bearing Iron Arsenate

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Weight percent	Group : CHEN				Sample : As2O3-Giant				Page 1
Element	70	71	72	211	212	213	214	215	
Sb	1.154	1.244	1.217	2.103	2.065	1.623	1.912	1.831	
As	3.721	3.266	3.159	5.417	5.522	5.184	5.871	4.972	
Fe	61.043	60.595	61.119	57.519	58.305	58.833	58.428	57.798	
Pb	0.097	0.068	0.070	0.148	0.073	0.152	0.056	0.097	
S	-	-	-	0.047	0.051	0.075	0.057	0.070	
Cr	-	-	-	-	-	-	-	-	
O	33.985	34.827	34.435	34.766	33.984	34.133	33.676	35.232	
Ca	-	-	-	-	-	-	-	-	
Bi	-	-	-	-	-	-	-	-	
Si	-	-	-	-	-	-	-	-	
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000	

Weight percent		Group : CHEN		Sample : As2O3-Giant	
Element	216	Minimum	Maximum	Average	Sigma
Sb	1.889	1.154	2.103	1.671	0.376
As	5.005	3.159	5.871	4.680	1.022
Fe	58.385	57.519	61.119	59.114	1.412
Pb	0.179	0.056	0.179	0.104	0.044
S	0.047	-	0.075	0.039	0.030
Cr	-	-	-	-	-
O	34.495	33.676	35.232	34.393	0.495
Ca	-	-	-	-	-
Bi	-	-	-	-	-
Si	-	-	-	-	-
-----					
Total	100.000	100.000	100.000	100.000	-
No. of data 9					



## Iron Arsenate Phase

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Weight percent		Group : CHEN				Sample : As2O3-Giant		Page 1
Element	218	219	220	221	222	223	224	225
Sb	0.065	0.146	0.332	0.239	0.224	0.382	0.215	0.173
As	17.388	17.115	12.752	15.636	17.843	12.973	15.710	16.825
Fe	9.849	10.061	6.943	14.154	7.959	6.307	9.540	10.100
Pb	0.057	0.137	0.098	0.130	0.081	0.066	0.165	0.190
S	4.824	7.413	2.976	4.226	4.195	2.652	3.545	5.126
Cr	-	-	-	-	-	-	-	-
O	67.752	60.667	76.774	65.460	69.639	77.425	70.558	65.299
Ca	0.065	4.392	0.125	0.082	0.059	0.195	0.260	2.233
Bi	-	0.069	-	0.073	-	-	0.007	0.054
Si	-	-	-	-	-	-	-	-
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Weight percent		Group : CHEN				Sample : As2O3-Giant		
Element	226	227	228	229	230	231	232	233
Sb	0.271	0.406	0.114	0.317	0.215	0.209	0.231	0.876
As	16.656	20.243	18.526	21.637	14.585	12.066	17.371	7.924
Fe	10.111	9.686	8.624	2.193	8.167	8.706	1.781	38.332
Pb	0.101	0.099	0.076	0.090	0.042	0.066	0.108	0.133
S	4.689	4.857	4.692	0.997	3.423	2.919	0.439	0.236
Cr	-	-	-	-	-	-	-	-
O	68.080	64.606	66.763	74.566	73.347	75.803	77.456	52.441
Ca	0.045	0.052	1.152	0.142	0.176	0.204	2.581	0.058
Bi	0.047	0.051	0.053	0.058	0.045	0.027	0.033	-
Si	-	-	-	-	-	-	-	-
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Weight percent		Group : CHEN			Sample : As2O3-Giant
Element	Minimum	Maximum	Average	Sigma	
Sb	0.065	0.876	0.276	0.184	
As	7.924	21.637	15.953	3.350	
Fe	1.781	38.332	10.157	8.093	
Pb	0.042	0.190	0.102	0.040	
S	0.236	7.413	3.576	1.878	
Cr	-	-	-	-	
O	52.441	77.456	69.165	6.822	
Ca	0.045	4.392	0.739	1.259	
Bi	-	0.073	0.032	0.027	
Si	-	-	-	-	
Total	100.000	100.000	100.000	0.000	



## **APPENDIX 3**

### **Detailed Electron Microprobe Analyses of the Arsenic Carriers in the April 97 Composite Sample**



As<sub>2</sub>O<sub>3</sub> Phase

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Weight percent	Group : CHEN				Sample : As2O3-Giant			Page 1
Element	124	128	132	135	138	181	183	184
Sb	0.314	0.283	0.043	-	-	0.432	0.545	0.288
As	71.451	76.482	78.023	78.387	79.112	78.019	77.481	78.598
Fe	0.056	0.093	0.353	0.051	0.069	0.122	0.221	0.320
Pb	-	-	-	0.066	-	0.041	0.076	0.064
S	-	0.039	0.021	0.014	0.017	0.015	0.057	0.086
Cr	-	-	-	-	-	-	-	-
O	28.151	23.089	21.560	21.474	20.802	21.371	21.620	20.554
Ca	0.028	0.014	-	0.008	-	-	-	0.090
Bi	-	-	-	-	-	-	-	-
Si	-	-	-	-	-	-	-	-
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Weight percent	Group : CHEN				Sample : As2O3-Giant			
Element	185	186	187	188	190	191	192	194
Sb	0.257	0.323	0.523	0.366	0.287	0.410	0.531	-
As	73.244	72.670	73.804	78.201	82.705	80.481	74.779	73.096
Fe	0.526	0.426	0.317	0.190	0.030	0.028	0.313	0.184
Pb	0.070	0.170	0.088	0.114	-	0.042	0.084	0.016
S	0.040	0.114	0.112	0.005	0.003	0.009	0.025	0.069
Cr	-	-	-	-	-	-	-	-
O	25.776	26.127	25.055	21.086	16.975	19.030	24.229	26.442
Ca	0.087	0.170	0.101	0.038	-	-	0.039	0.193
Bi	-	-	-	-	-	-	-	-
Si	-	-	-	-	-	-	-	-
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Weight percent	Group : CHEN				Sample : As2O3-Giant			
Element	195	196	197	204	205	207	209	210
Sb	-	0.006	0.292	0.110	-	0.345	0.013	0.333
As	76.555	78.131	75.405	78.008	71.834	73.841	77.855	72.840
Fe	0.351	0.037	0.091	0.033	0.526	0.075	0.033	0.075
Pb	0.004	0.066	0.029	0.027	-	0.012	-	0.043
S	0.069	0.018	-	-	0.181	0.019	0.016	0.002
Cr	-	-	-	-	-	-	-	-
O	22.861	21.695	24.178	21.822	27.329	25.708	22.070	26.692
Ca	0.160	0.047	0.005	-	0.130	-	0.013	0.015
Bi	-	-	-	-	-	-	-	-
Si	-	-	-	-	-	-	-	-
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000



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Weight percent		Group : CHEN		Sample : As2O3-Giant		Page 2	
Element	Minimum	Maximum	Average	Sigma			
Sb	-	0.545	0.238	0.190			
As	71.451	82.705	76.292	2.944			
Fe	0.028	0.526	0.188	0.163			
Pb	-	0.170	0.042	0.044			
S	-	0.181	0.039	0.046			
Cr	-	-	-	-			
O	16.975	28.151	23.154	2.810			
Ca	-	0.193	0.047	0.062			
Bi	-	-	-	-			
Si	-	-	-	-			
-----							
Total	100.000	100.000	100.000	0.000			
No. of data 24							

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*Sb-Rich As<sub>2</sub>O<sub>3</sub> Phase*

Weight percent		Group : CHEN			Sample : As2O3-Giant			Page 1
Element	126	127	130	131	198	199	200	201
Sb	12.145	15.091	14.693	14.919	13.316	12.794	13.121	12.318
As	60.555	60.291	57.803	50.848	59.585	54.435	68.707	64.521
Fe	0.885	0.107	0.075	1.624	0.117	0.336	0.107	0.181
Pb	0.027	-	-	0.158	0.029	0.114	0.063	0.021
S	0.063	0.025	0.018	0.217	-	0.067	0.009	0.008
Cr	-	-	-	-	-	-	-	-
O	26.299	24.472	27.411	31.628	26.936	32.166	17.993	22.610
Ca	0.026	0.014	-	0.606	0.017	0.088	-	0.304
Bi	-	-	-	-	-	-	-	0.037
Si	-	-	-	-	-	-	-	-
-----								
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Weight percent		Group : CHEN		Sample : As2O3-Giant	
Element	Minimum	Maximum	Average	Sigma	
Sb	12.145	15.091	13.550	1.187	
As	50.848	68.707	59.593	5.543	
Fe	0.075	1.624	0.429	0.552	
Pb	-	0.158	0.052	0.057	
S	-	0.217	0.051	0.072	
Cr	-	-	-	-	
O	17.993	32.166	26.189	4.634	
Ca	-	0.606	0.132	0.217	
Bi	-	0.037	0.005	0.013	
Si	-	-	-	-	
<hr/>					
Total	100.000	100.000	100.000	0.000	
No. of data 8					



(Sb,As)<sub>2</sub>O<sub>3</sub>

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Weight percent		Group : CHEN				Sample : As2O3-Giant		Page 1
Element	140	141	142	143	144	145	146	147
Sb	45.633	43.482	43.907	45.878	42.855	45.013	43.809	44.892
As	37.987	36.043	32.523	35.877	36.761	39.460	39.234	39.160
Fe	0.221	0.207	0.625	0.232	0.154	0.141	0.132	0.153
Pb	0.304	0.312	0.285	0.344	0.058	0.022	0.077	0.022
S	0.879	0.967	0.875	0.623	0.727	0.798	0.801	0.898
Cr	-	-	-	-	-	-	-	-
O	14.866	18.931	21.714	16.942	19.445	14.566	15.947	14.875
Ca	0.110	0.058	0.071	0.104	-	-	-	-
Bi	-	-	-	-	-	-	-	-
Si	-	-	-	-	-	-	-	-
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Weight percent		Group : CHEN				Sample : As2O3-Giant		
Element	148	149	150	151	152	153	154	155
Sb	44.593	46.242	45.309	43.101	45.296	46.099	44.308	43.363
As	36.261	34.370	37.696	35.448	42.452	41.041	35.134	35.569
Fe	0.186	0.843	0.158	0.081	0.090	0.079	0.128	0.075
Pb	0.041	0.004	0.021	0.008	0.028	0.028	0.015	0.013
S	0.918	0.747	1.011	0.925	0.728	0.807	1.023	0.868
Cr	-	-	-	-	-	-	-	-
O	18.001	17.794	15.805	20.437	11.406	11.946	19.392	20.112
Ca	-	-	-	-	-	-	-	-
Bi	-	-	-	-	-	-	-	-
Si	-	-	-	-	-	-	-	-
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Weight percent		Group : CHEN				Sample : As2O3-Giant		
Element	156	157	158	159	160	161	162	163
Sb	42.547	42.430	44.359	44.224	46.634	45.104	43.136	44.154
As	35.744	36.355	36.134	32.764	37.442	34.918	33.076	33.840
Fe	0.073	0.084	0.040	0.131	0.135	0.068	0.046	0.052
Pb	0.013	0.005	0.018	0.051	0.015	0.087	0.074	0.142
S	0.785	0.709	0.872	0.851	0.589	1.239	0.947	0.962
Cr	-	-	-	-	-	-	-	-
O	20.838	20.417	18.577	21.979	15.185	18.584	22.721	20.850
Ca	-	-	-	-	-	-	-	-
Bi	-	-	-	-	-	-	-	-
Si	-	-	-	-	-	-	-	-
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000



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Weight percent		Group : CHEN				Sample : As2O3-Giant			Page 2
Element	164	165	166	167	168	169	170	171	
Sb	44.315	43.796	43.124	45.819	46.776	44.387	44.277	46.770	
As	36.273	33.075	30.711	41.406	35.104	35.265	32.131	30.944	
Fe	0.046	0.072	0.057	0.079	0.067	-	0.020	0.055	
Pb	-	0.035	0.018	0.063	0.016	-	-	0.080	
S	0.984	0.706	0.838	0.780	0.502	0.962	1.048	0.618	
Cr	-	-	-	-	-	-	-	-	
O	18.382	22.316	25.252	11.853	17.535	19.386	22.524	21.533	
Ca	-	-	-	-	-	-	-	-	
Bi	-	-	-	-	-	-	-	-	
Si	-	-	-	-	-	-	-	-	
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000	

Weight percent		Group : CHEN			Sample : As2O3-Giant	
Element	Minimum	Maximum	Average	Sigma		
Sb	42.430	46.776	44.551	1.243		
As	30.711	42.452	35.944	2.883		
Fe	-	0.843	0.142	0.168		
Pb	-	0.344	0.069	0.099		
S	0.502	1.239	0.843	0.152		
Cr	-	-	-	-		
O	11.406	25.252	18.441	3.396		
Ca	-	0.110	0.011	0.030		
Bi	-	-	-	-		
Si	-	-	-	-		
Total	100.000	100.000	100.000	0.000		
No. of data 32						



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*As-, Sb-bearing Iron Oxide*

Weight percent		Group : CHEN				Sample : As2O3-Giant		Page 1	
Element	133	206	240	241	Minimum	Maximum	Average	Sigma	
Sb	2.075	0.588	9.285	8.889	0.588	9.285	5.209	4.521	
As	7.984	6.690	10.017	11.481	6.690	11.481	9.043	2.125	
Fe	49.972	55.313	29.040	18.359	18.359	55.313	38.171	17.407	
Pb	0.054	-	0.176	0.254	-	0.254	0.121	0.115	
S	0.043	0.316	1.554	1.518	0.043	1.554	0.858	0.791	
Cr	-	-	-	-	-	-	-	-	
O	39.872	37.093	49.031	57.089	37.093	57.089	45.771	9.107	
Ca	-	-	0.897	2.405	-	2.405	0.826	1.135	
Bi	-	-	-	0.005	-	0.005	0.001	0.002	
Si	-	-	-	-	-	-	-	-	
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	0.000	
No. of data 4									



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*Ca Arsenate-Sulphate Phase*

Weight percent		Group : CHEN				Sample : As2O3-Giant		Page 1	
Element	178	179	180	239	Minimum	Maximum	Average	Sigma	
Sb	17.191	13.102	20.287	11.158	11.158	20.287	15.434	4.097	
As	8.350	8.188	9.579	7.731	7.731	9.579	8.462	0.789	
Fe	1.918	2.465	2.062	2.550	1.918	2.550	2.249	0.306	
Pb	0.160	0.144	0.158	0.127	0.127	0.160	0.147	0.015	
S	8.780	9.642	7.416	9.895	7.416	9.895	8.933	1.118	
Cr	-	-	-	-	-	-	-	-	
O	52.304	54.351	50.274	56.274	50.274	56.274	53.301	2.588	
Ca	11.297	12.108	10.224	12.265	10.224	12.265	11.474	0.935	
Bi	-	-	-	-	-	-	-	-	
Si	-	-	-	-	-	-	-	-	
-----									
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	-	
No. of data 4									

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*Calcium Oxide Phase*

Weight percent		Group : CHEN				Sample : As2O3-Giant		Page 1	
Element	176	177	235	236	Minimum	Maximum	Average	Sigma	
Sb	0.575	0.565	0.256	0.264	0.256	0.575	0.415	0.179	
As	1.263	1.854	0.242	0.375	0.242	1.854	0.933	0.763	
Fe	5.290	5.363	6.051	5.734	5.290	6.051	5.609	0.353	
Pb	-	-	-	0.002	-	0.002	0.001	0.001	
S	0.650	0.803	0.437	0.205	0.205	0.803	0.524	0.260	
Cr	-	-	-	-	-	-	-	-	
O	74.541	73.147	72.678	73.356	72.678	74.541	73.431	0.793	
Ca	17.681	18.268	20.330	20.064	17.681	20.330	19.086	1.310	
Bi	-	-	0.006	-	-	0.006	0.002	0.003	
Si	-	-	-	-	-	-	-	-	
-----									
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	-	
No. of data 4									



## **APPENDIX 4**

### **Detailed Electron Microprobe Analyses of the Arsenic Carriers in the 212 Chamber Sample**



As<sub>2</sub>O<sub>3</sub> Phase

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Weight percent		Group : CHEN				Sample : As <sub>2</sub> O <sub>3</sub> -Giant		Page 1
Element	265	271	272	273	274	276	277	278
Sb	0.979	1.411	2.360	0.544	0.313	0.844	2.998	0.157
As	70.242	70.853	70.287	75.898	77.990	73.076	78.202	79.328
Fe	0.935	0.924	0.527	0.602	0.500	1.723	0.603	0.822
Pb	0.014	-	-	0.005	-	0.088	0.128	0.084
S	0.094	0.044	-	0.053	0.020	0.083	0.051	0.036
Cr	-	-	-	-	-	-	-	-
O	27.554	26.742	26.807	22.865	21.173	23.886	17.962	19.531
Ca	0.182	0.026	0.019	0.033	0.004	0.300	0.056	0.042
Bi	-	-	-	-	-	-	-	-
Si	-	-	-	-	-	-	-	-

Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000
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Weight percent		Group : CHEN			Sample : As2O3-Giant			
Element	280	292	293	295	302	305	306	307
Sb	0.080	3.524	0.863	1.022	0.682	1.609	0.378	2.845
As	70.319	75.295	79.063	80.226	73.812	79.775	76.108	76.990
Fe	0.484	0.343	0.636	0.257	0.754	0.395	0.317	0.377
Pb	-	0.033	0.225	0.124	0.021	0.029	-	-
S	0.027	0.057	0.075	0.031	0.068	0.038	0.017	0.031
Cr	-	-	-	-	-	-	-	-
O	29.058	20.670	19.016	18.288	24.663	18.154	23.167	19.726
Ca	0.032	0.078	0.122	0.052	-	-	0.013	0.031
Bi	-	-	-	-	-	-	-	-
Si	-	-	-	-	-	-	-	-

Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000
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Weight percent		Group : CHEN				Sample : As2O3-Giant		
Element	310	311	312	313	333	334	336	337
Sb	1.340	1.079	1.309	0.831	2.396	2.159	0.841	0.329
As	70.349	73.523	77.994	82.427	71.014	71.878	72.676	80.878
Fe	0.497	0.268	0.408	0.324	0.570	0.360	0.449	0.339
Pb	0.051	-	-	0.003	-	0.013	0.028	-
S	0.039	0.025	0.017	0.004	0.024	0.006	0.034	0.158
Cr	-	-	-	-	-	-	-	-
O	27.688	25.105	20.270	16.395	25.953	25.584	25.966	18.087
Ca	0.036	-	0.002	0.016	0.043	-	0.006	0.209
Bi	-	-	-	-	-	-	-	-
Si	-	-	-	-	-	-	-	-
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000
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Weight percent		Group : CHEN			Sample : As2O3-Giant			Page 2
Element	338	342	345	346	347	348	351	
Sb	0.539	2.395	1.155	0.385	0.577	0.631	0.534	
As	74.644	75.411	75.283	78.806	81.081	81.522	81.534	
Fe	0.556	1.498	0.386	0.409	0.516	0.373	0.479	
Pb	0.059	-	0.050	0.022	-	-	-	
S	0.097	0.035	0.028	0.049	0.009	0.010	0.073	
Cr	-	-	-	-	-	-	-	
O	23.991	20.270	22.926	20.126	17.598	17.352	16.802	
Ca	0.114	-	0.029	0.040	0.037	0.012	0.053	
Bi	-	-	-	-	-	-	-	
Si	-	0.391	0.143	0.163	0.182	0.100	0.525	
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	

Weight percent		Group : CHEN		Sample : As2O3-Giant
Element	Minimum	Maximum	Average	Sigma
Sb	0.080	3.524	1.197	0.910
As	70.242	82.427	76.016	3.909
Fe	0.257	1.723	0.569	0.329
Pb	-	0.225	0.032	0.051
S	-	0.158	0.043	0.034
Cr	-	-	-	-
O	16.395	29.058	22.044	3.749
Ca	-	0.300	0.051	0.069
Bi	-	-	-	-
Si	-	0.525	0.049	0.122
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Total	100.000	100.000	100.000	0.000
No. of data 31				



*As<sub>2</sub>O<sub>3</sub> Phase*

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Weight percent		Group : CHEN				Sample : As2O3-Giant		Page 1
Element	275	279	288	289	296	301	303	304
Sb	1.021	1.620	2.513	3.400	1.806	6.599	3.140	4.956
As	58.067	58.341	65.540	54.565	59.154	65.030	60.973	67.111
Fe	1.062	0.673	0.708	1.208	0.063	0.614	0.620	0.789
Pb	-	0.025	-	-	-	0.011	0.049	0.060
S	0.049	0.094	0.019	0.107	0.016	0.027	0.058	0.029
Cr	-	-	-	-	-	-	-	-
O	39.708	39.134	31.162	40.470	38.952	27.697	34.555	26.990
Ca	0.093	0.113	0.058	0.250	0.009	0.022	0.605	0.065
Bi	-	-	-	-	-	-	-	-
Si	-	-	-	-	-	-	-	-
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Weight percent		Group : CHEN				Sample : As2O3-Giant	
Element	309	335	339	341	343	350	
Sb	3.822	1.836	1.114	1.625	1.319	1.828	
As	62.170	69.817	62.117	68.103	65.600	66.392	
Fe	0.766	0.617	0.345	0.238	0.160	0.352	
Pb	-	0.038	-	0.066	-	0.091	
S	0.066	0.065	0.098	0.157	0.030	0.040	
Cr	-	-	-	-	-	-	
O	33.045	27.505	35.372	29.564	31.879	31.003	
Ca	0.131	0.122	0.059	0.013	0.012	0.012	
Bi	-	-	-	-	-	-	
Si	-	-	0.895	0.234	1.000	0.282	
Total	100.000	100.000	100.000	100.000	100.000	100.000	

Weight percent		Group : CHEN			Sample : As2O3-Giant
Element	Minimum	Maximum	Average	Sigma	
Sb	1.021	6.599	2.614	1.618	
As	54.565	69.817	63.070	4.435	
Fe	0.063	1.208	0.587	0.328	
Pb	-	0.091	0.024	0.031	
S	0.016	0.157	0.061	0.041	
Cr	-	-	-	-	
O	26.990	40.470	33.360	4.763	
Ca	0.009	0.605	0.112	0.157	
Bi	-	-	-	-	
Si	-	1.000	0.172	0.342	
Total	100.000	100.000	100.000	0.000	



*(As,Sb)<sub>2</sub>O<sub>3</sub> Phase*

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Weight percent      Group : CHEN      Sample : As2O3-Giant      Page 1

Element	314	315	316	317	318	320	321
Sb	24.603	24.612	22.194	23.718	31.108	20.938	23.966
As	47.398	56.720	52.440	54.573	42.577	51.417	48.142
Fe	0.170	1.495	0.121	0.129	0.171	0.129	0.142
Pb	-	-	0.003	-	-	0.114	0.071
S	-	0.025	0.006	0.009	-	0.010	-
Cr	-	-	-	-	-	-	-
O	27.829	17.148	25.236	21.571	26.144	27.392	27.679
Ca	-	-	-	-	-	-	-
Bi	-	-	-	-	-	-	-
Si	-	-	-	-	-	-	-
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Weight percent      Group : CHEN      Sample : As2O3-Giant

Element	Minimum	Maximum	Average	Sigma
Sb	20.938	31.108	24.448	3.229
As	42.577	56.720	50.467	4.793
Fe	0.121	1.495	0.337	0.511
Pb	-	0.114	0.027	0.047
S	-	0.025	0.007	0.009
Cr	-	-	-	-
O	17.148	27.829	24.714	3.981
Ca	-	-	-	-
Bi	-	-	-	-
Si	-	-	-	-

Total      100.000      100.000      100.000      0.000  
No. of data 7



## As-bearing Iron Oxide

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Weight percent		Group : CHEN				Sample : As2O3-Giant		Page 1
Element	267	268	281	282	283	284	286	287
Sb	2.603	2.228	9.899	7.119	7.244	7.262	1.228	1.336
As	9.505	9.744	3.820	3.513	9.188	7.645	10.200	5.790
Fe	52.541	51.492	54.219	56.888	47.949	50.239	44.902	57.538
Pb	0.674	0.529	0.169	0.255	0.418	0.340	0.275	0.676
S	0.187	0.197	0.038	0.023	0.051	0.045	3.027	0.077
Cr	-	-	-	-	-	-	-	-
O	33.936	35.145	31.855	32.202	35.007	34.364	40.274	34.415
Ca	0.554	0.665	-	-	0.143	0.105	0.094	0.168
Bi	-	-	-	-	-	-	-	-
Si	-	-	-	-	-	-	-	-
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Weight percent		Group : CHEN				Sample : As2O3-Giant		
Element	294	297	298	299	324	325	326	331
Sb	1.697	0.777	0.918	1.755	1.566	1.478	1.503	0.853
As	10.005	6.857	10.100	14.886	8.747	9.579	9.370	7.774
Fe	55.489	53.219	47.717	45.202	54.921	56.178	55.977	54.132
Pb	0.782	0.290	0.227	0.178	0.371	0.361	0.310	0.371
S	0.041	0.427	0.792	0.240	0.070	0.052	0.066	0.066
Cr	-	-	-	-	-	-	-	-
O	30.987	38.108	39.816	37.673	34.163	32.178	32.596	36.571
Ca	0.999	0.322	0.430	0.066	0.162	0.174	0.178	0.233
Bi	-	-	-	-	-	-	-	-
Si	-	-	-	-	-	-	-	-
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Weight percent		Group : CHEN			Sample : As2O3-Giant
Element	Minimum	Maximum	Average	Sigma	
Sb	0.777	9.899	3.092	2.955	
As	3.513	14.886	8.545	2.727	
Fe	44.902	57.538	52.413	4.097	
Pb	0.169	0.782	0.389	0.184	
S	0.023	3.027	0.337	0.744	
Cr	-	-	-	-	
O	30.987	40.274	34.956	2.833	
Ca	-	0.999	0.268	0.271	
Bi	-	-	-	-	
Si	-	-	-	-	
Total	100.000	100.000	100.000	0.000	



## Iron Arsenate

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Weight percent		Group : CHEN				Sample : As2O3-Giant		Page 1
Element	291	395	396	398	399	400	401	402
Sb	0.739	0.468	0.538	0.535	0.539	0.441	0.475	0.444
As	26.195	29.858	36.551	31.050	30.207	27.167	26.769	27.823
Fe	12.286	12.912	13.187	13.156	13.218	12.978	12.341	12.864
Pb	1.106	0.599	0.797	0.727	0.778	0.687	0.750	0.716
S	1.380	1.499	1.517	1.443	1.231	1.128	1.432	1.542
Cr	-	-	-	-	-	-	-	-
O	54.829	53.915	46.565	52.325	53.276	56.887	57.466	55.851
Ca	3.465	0.739	0.766	0.724	0.725	0.703	0.760	0.745
Bi	-	-	-	-	-	-	-	-
Si	-	0.010	0.079	0.040	0.026	0.009	0.007	0.015
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Weight percent		Group : CHEN				Sample : As2O3-Giant		
Element	403	404	405	406	407	408	409	410
Sb	0.526	0.472	0.478	0.552	0.439	0.483	0.430	0.421
As	37.339	34.839	30.144	33.352	28.275	32.681	29.246	29.950
Fe	13.034	11.686	13.122	13.085	12.184	12.987	12.164	12.662
Pb	0.739	0.732	0.785	0.838	0.724	0.819	0.728	0.654
S	1.632	1.810	1.494	1.621	1.270	1.531	1.721	1.492
Cr	-	-	-	-	-	-	-	-
O	45.847	49.521	53.230	49.779	56.436	50.674	54.965	54.111
Ca	0.764	0.807	0.716	0.742	0.646	0.766	0.717	0.690
Bi	-	-	-	-	-	-	-	-
Si	0.119	0.133	0.031	0.031	0.026	0.059	0.029	0.020
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Weight percent		Group : CHEN				Sample : As2O3-Giant		
Element	411	412	413	414	415			
Sb	0.503	0.476	0.531	0.552	0.389			
As	36.250	35.715	34.865	31.363	35.566			
Fe	12.413	12.008	12.496	12.523	11.344			
Pb	0.664	0.504	0.645	0.689	0.595			
S	1.969	2.181	1.635	1.457	2.210			
Cr	-	-	-	-	-			
O	47.430	48.241	48.951	52.593	49.076			
Ca	0.708	0.753	0.783	0.796	0.728			
Bi	-	-	-	-	-			
Si	0.063	0.122	0.094	0.027	0.092			
Total	100.000	100.000	100.000	100.000	100.000			



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Weight percent	Group : CHEN			Sample : As2O3-Giant	Page 2
Element	Minimum	Maximum	Average	Sigma	
Sb	0.389	0.739	0.497	0.073	
As	26.195	37.339	31.676	3.537	
Fe	11.344	13.218	12.602	0.526	
Pb	0.504	1.106	0.727	0.118	
S	1.128	2.210	1.581	0.277	
Cr	-	-	-	-	
O	45.847	57.466	51.998	3.535	
Ca	0.646	3.465	0.869	0.596	
Bi	-	-	-	-	
Si	-	0.133	0.049	0.041	
-----					
Total	100.000	100.000	100.000	0.000	
No. of data 21					



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*Fe-bearing As<sub>2</sub>O<sub>3</sub> Phase*

Weight percent		Group : CHEN		Sample : As <sub>2</sub> O <sub>3</sub> -Giant			Page 1
Element	327	328	330	Minimum	Maximum	Average	Sigma
Sb	2.058	1.758	1.896	1.758	2.058	1.904	0.150
As	29.934	41.343	34.366	29.934	41.343	35.214	5.752
Fe	9.599	5.080	5.206	5.080	9.599	6.628	2.573
Pb	3.000	2.309	2.346	2.309	3.000	2.552	0.389
S	0.081	0.081	1.301	0.081	1.301	0.488	0.704
Cr	-	-	-	-	-	-	-
O	52.371	47.392	50.764	47.392	52.371	50.176	2.541
Ca	2.957	2.037	4.121	2.037	4.121	3.038	1.044
Bi	-	-	-	-	-	-	-
Si	-	-	-	-	-	-	-
-----							
Total	100.000	100.000	100.000	100.000	100.000	100.000	-
-----							
No. of data	3						



## Calcium-Iron Arsenate

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Weight percent				Group : CHEN		Sample : As2O3-Giant		Page 1
Element	264	266	269	Minimum	Maximum	Average	Sigma	
Sb	0.062	0.702	0.827	0.062	0.827	0.530	0.410	
As	1.384	10.119	20.764	1.384	20.764	10.756	9.706	
Fe	9.931	8.038	8.296	8.038	9.931	8.755	1.027	
Pb	0.066	0.335	0.345	0.066	0.345	0.249	0.158	
S	0.048	0.381	1.084	0.048	1.084	0.504	0.529	
Cr	-	-	-	-	-	-	-	
O	69.852	65.666	60.290	60.290	69.852	65.269	4.793	
Ca	18.657	14.759	8.394	8.394	18.657	13.937	5.181	
Bi	-	-	-	-	-	-	-	
Si	-	-	-	-	-	-	-	
-----								
Total	100.000	100.000	100.000	100.000	100.000	100.000	0.000	
No. of data 3								



## **APPENDIX 5**

### **Detailed Electron Microprobe Analyses of the Arsenic Carriers in the 236 Chamber Sample**



As<sub>2</sub>O<sub>3</sub> Phase

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Weight percent		Group : CHEN				Sample : As <sub>2</sub> O <sub>3</sub> -Giant			Page 2
Element	49	50	51	53	57	58	59	243	
Sb	0.534	0.314	0.614	0.232	0.335	0.370	-	0.166	
As	77.000	78.211	78.880	75.289	72.403	78.069	75.750	74.988	
Fe	0.188	0.174	0.140	0.354	0.195	1.134	0.672	0.182	
Pb	-	-	0.017	0.026	-	-	-	0.081	
S	-	-	-	-	-	-	-	0.009	
Cr	-	-	-	-	-	-	-	-	
O	22.278	21.301	20.349	24.099	27.067	20.427	23.578	24.540	
Ca	-	-	-	-	-	-	-	0.034	
Bi	-	-	-	-	-	-	-	-	
Si	-	-	-	-	-	-	-	-	
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000	

Weight percent		Group : CHEN				Sample : As <sub>2</sub> O <sub>3</sub> -Giant			
Element	244	245	250	251	254	255	259	352	
Sb	-	0.066	-	0.402	0.063	0.418	-	-	
As	75.243	78.860	78.134	77.139	78.528	76.776	78.927	78.866	
Fe	0.230	0.226	0.168	0.381	0.125	0.205	0.189	0.205	
Pb	0.139	0.092	0.052	-	-	0.011	-	0.053	
S	0.008	-	0.001	0.020	0.016	-	0.010	0.004	
Cr	-	-	-	-	-	-	-	-	
O	24.341	20.734	21.621	22.035	21.268	22.582	20.874	20.762	
Ca	0.039	0.022	0.024	0.023	-	0.008	-	0.010	
Bi	-	-	-	-	-	-	-	-	
Si	-	-	-	-	-	-	-	0.100	
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000	

Weight percent		Group : CHEN				Sample : As <sub>2</sub> O <sub>3</sub> -Giant			
Element	354	356	358	359	360	362	365	366	
Sb	0.042	0.270	0.765	0.281	0.992	0.052	0.437	0.885	
As	77.077	73.469	71.413	76.127	75.802	76.455	78.141	79.656	
Fe	0.845	0.160	0.261	0.398	0.481	0.198	0.163	0.488	
Pb	-	0.025	-	-	-	-	0.029	0.004	
S	0.011	-	0.114	0.039	0.060	0.027	0.005	0.029	
Cr	-	-	-	-	-	-	-	-	
O	22.025	25.981	27.142	22.653	22.455	23.204	21.216	18.734	
Ca	-	0.017	0.092	0.016	0.023	-	0.009	0.016	
Bi	-	-	-	-	-	-	-	-	
Si	-	0.078	0.213	0.486	0.187	0.064	-	0.188	
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000	



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Weight percent		Group : CHEN				Sample : As2O3-Giant		Page 1
Element	1	3	12	21	22	23	24	25
Sb	-	0.200	0.292	-	-	-	-	0.079
As	77.198	76.000	75.906	76.555	73.349	78.201	75.431	73.491
Fe	0.105	0.190	0.487	0.251	0.225	0.262	0.349	0.219
Pb	-	0.001	0.075	0.011	-	-	-	-
S	-	-	-	-	-	-	-	-
Cr	-	-	-	-	-	-	-	-
O	22.697	23.609	23.240	23.183	26.426	21.537	24.220	26.211
Ca	-	-	-	-	-	-	-	-
Bi	-	-	-	-	-	-	-	-
Si	-	-	-	-	-	-	-	-
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Weight percent		Group : CHEN				Sample : As2O3-Giant		
Element	26	27	28	29	30	34	35	36
Sb	0.155	-	-	0.077	0.315	0.154	0.066	0.395
As	73.142	71.447	73.011	76.710	78.581	76.675	75.166	77.462
Fe	0.231	0.161	0.223	0.193	0.256	0.212	0.320	0.539
Pb	-	-	-	0.013	0.031	0.048	0.082	0.073
S	-	-	-	-	-	-	-	-
Cr	-	-	-	-	-	-	-	-
O	26.472	28.392	26.766	23.007	20.817	22.911	24.366	21.531
Ca	-	-	-	-	-	-	-	-
Bi	-	-	-	-	-	-	-	-
Si	-	-	-	-	-	-	-	-
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Weight percent		Group : CHEN				Sample : As2O3-Giant		
Element	37	38	39	41	42	43	44	48
Sb	0.097	-	-	-	-	-	-	0.380
As	79.877	78.183	75.430	79.193	78.728	77.544	76.380	79.390
Fe	0.303	0.737	0.242	0.181	0.384	0.229	0.422	0.187
Pb	0.058	-	0.014	0.019	0.029	0.045	0.037	-
S	-	-	-	-	-	-	-	-
Cr	-	-	-	-	-	-	-	-
O	19.665	21.080	24.314	20.607	20.859	22.182	23.161	20.043
Ca	-	-	-	-	-	-	-	-
Bi	-	-	-	-	-	-	-	-
Si	-	-	-	-	-	-	-	-
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000



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Weight percent		Group : CHEN				Sample : As2O3-Giant		Page 3
Element	367	368	372	373	377	379	380	382
Sb	0.307	0.408	-	0.634	-	0.008	-	0.601
As	78.799	76.277	78.897	76.546	78.947	78.610	76.003	75.375
Fe	0.356	0.151	0.102	0.186	0.172	0.199	0.217	0.144
Pb	0.041	0.001	0.050	-	-	-	-	-
S	0.013	0.015	0.017	0.021	0.019	0.007	0.033	0.016
Cr	-	-	-	-	-	-	-	-
O	20.125	23.010	20.934	22.274	20.849	21.164	23.103	23.864
Ca	0.013	0.008	-	-	0.013	0.006	0.017	-
Bi	-	-	-	-	-	-	-	-
Si	0.346	0.130	-	0.339	-	0.006	0.627	-
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Weight percent		Group : CHEN				Sample : As2O3-Giant		
Element	387	389	390	393	Minimum	Maximum	Average	Sigma
Sb	0.121	-	0.182	0.090	-	0.992	0.197	0.243
As	78.843	78.886	76.698	79.115	71.413	79.877	76.787	2.101
Fe	0.106	0.122	0.086	0.229	0.086	1.134	0.279	0.190
Pb	0.017	0.017	0.045	0.010	-	0.139	0.021	0.030
S	-	0.014	0.011	0.012	-	0.114	0.009	0.018
Cr	-	-	-	-	-	-	-	-
O	20.902	20.882	22.818	20.278	18.734	28.392	22.646	2.124
Ca	-	-	-	0.027	-	0.092	0.007	0.015
Bi	-	-	-	-	-	-	-	-
Si	0.011	0.079	0.160	0.239	-	0.627	0.054	0.126
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	0.000
No. of data 60								



## As-bearing Iron Oxide

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Weight percent	Group : CHEN				Sample : As2O3-Giant			Page 1
Element	8	9	10	16	17	31	32	242
Sb	0.717	0.662	0.810	0.902	0.787	0.537	0.654	0.711
As	14.312	14.901	9.401	9.257	6.046	8.221	9.029	10.614
Fe	40.905	40.888	43.648	54.657	57.983	57.121	55.698	52.567
Pb	0.554	0.577	0.254	0.268	0.249	0.036	0.104	0.308
S	-	-	-	-	-	-	-	0.021
Cr	-	-	6.185	-	-	-	-	-
O	43.512	42.972	39.702	34.916	34.935	34.085	34.515	35.281
Ca	-	-	-	-	-	-	-	0.498
Bi	-	-	-	-	-	-	-	-
Si	-	-	-	-	-	-	-	-
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Weight percent		Group : CHEN				Sample : As2O3-Giant		
Element	248	249	256	257	258	261	355	363
Sb	0.248	0.241	0.518	0.856	0.671	0.496	0.184	0.247
As	5.813	5.620	6.449	9.314	5.461	7.541	1.947	3.342
Fe	45.771	43.665	55.930	54.822	55.575	55.111	66.342	62.327
Pb	0.115	0.147	0.143	0.275	0.188	0.132	0.028	0.225
S	0.040	0.030	0.029	0.008	0.035	0.037	0.029	0.014
Cr	-	-	-	-	-	-	-	-
O	47.680	49.955	36.684	34.376	37.844	36.431	31.455	33.679
Ca	0.333	0.342	0.247	0.349	0.226	0.252	0.015	0.166
Bi	-	-	-	-	-	-	-	-
Si	-	-	-	-	-	-	-	-
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Weight percent	Group : CHEN		Sample : As2O3-Giant		
Element	364	Minimum	Maximum	Average	Sigma
Sb	0.361	0.184	0.902	0.565	0.235
As	3.126	1.947	14.901	7.670	3.573
Fe	62.063	40.888	66.342	53.240	7.669
Pb	0.169	0.028	0.577	0.222	0.152
S	0.021	-	0.040	0.016	0.015
Cr	-	-	6.185	0.364	1.500
O	34.063	31.455	49.955	37.770	5.255
Ca	0.197	-	0.498	0.154	0.165
Bi	-	-	-	-	-
Si	-	-	-	-	-
Total	100.000	100.000	100.000	100.000	0.000



*Various Silicate Species*

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Weight percent		Group : CHEN			Sample : Silicate-Giant			Page 1
No.	Fe	Mg	Al	Si	K	O	Ca	Total
1	0.840	0.535	14.224	16.467	4.691	63.243	-	100.000
2	0.102	0.012	0.111	39.967	0.031	59.777	-	100.000
3	0.258	0.025	0.396	43.919	0.215	55.187	-	100.000
4	13.136	11.166	11.223	10.725	0.110	52.571	1.069	100.000
5	1.172	0.447	17.346	21.235	7.104	52.661	0.035	100.000
6	0.464	0.175	0.269	40.525	0.033	58.412	0.122	100.000
7	4.587	1.140	3.667	4.912	0.897	84.230	0.567	100.000
8	9.241	3.003	3.368	4.948	0.187	78.782	0.471	100.000
9	14.217	8.564	9.542	8.552	0.049	58.964	0.112	100.000
10	16.893	8.655	10.838	9.790	0.083	53.178	0.563	100.000
11	1.893	1.081	13.505	18.193	7.288	57.913	0.127	100.000
12	4.703	4.065	10.708	12.705	3.448	63.814	0.557	100.000
13	14.816	10.738	10.760	11.334	0.231	51.869	0.252	100.000
14	13.369	7.556	8.338	8.030	0.234	61.877	0.596	100.000
15	11.607	7.245	6.861	6.579	0.222	66.708	0.778	100.000
16	18.194	6.881	11.136	10.009	0.148	53.440	0.192	100.000
17	17.539	8.979	10.863	10.206	0.022	52.334	0.057	100.000
18	14.226	9.345	10.604	9.829	0.019	55.892	0.085	100.000
19	3.383	1.998	15.390	16.338	4.968	57.849	0.074	100.000
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Minimum	0.102	0.012	0.111	4.912	0.019	51.869	-	100.000
Maximum	18.194	11.166	17.346	43.919	7.288	84.230	1.069	100.000
Average	8.455	4.822	8.903	16.014	1.578	59.932	0.298	100.000
Sigma	6.757	4.109	5.151	12.127	2.538	8.786	0.312	0.000

No. of data 19



## APPENDIX 6

**Detailed Solubility Data for Reagent Grade  $\text{As}_2\text{O}_3$   
and  $\text{Sb}_2\text{O}_3$ , as well as for the Four  
 $\text{As}_2\text{O}_3$ -rich Dust Samples**



**Vessel 1:**

**100 g/L reagent grade As<sub>2</sub>O<sub>3</sub>**

**Test 1**

Sample No.	Temp. (°C)	Volume of Liquid (ml)	Conc. of As <sup>3+</sup> In diluted sample (mg/L)	Conc. of As <sup>3+</sup> Corrected for dilution (g/L)	As <sub>2</sub> O <sub>3</sub> Concentration (g/L)	Conc. of As <sup>5+</sup> In diluted sample (mg/L)	Conc. of As <sup>5+</sup> Corrected for dilution (g/L)	Total As Conc. (mg/L)
601903-1113-1-1	24.1	4.9058	718	14.64	19.32	< 10	< 0.2	728
601903-1116-1-2	32.0	4.9087	878	17.89	23.62	< 10	< 0.2	900
601903-1117-1-3	40.0	4.9116	1057	21.52	28.41	< 10	< 0.2	1039
601903-1118-1-4	49.8	4.9151	1290	26.25	34.65	< 5	< 0.1	1301
601903-1119-1-5	59.6	4.9187	1552	31.55	41.66	< 10	< 0.2	1575
601903-1120-1-6	69.8	4.9224	1873	38.05	50.24	< 10	< 0.2	1892
601903-1123-1-7	79.9	4.9261	2250	45.68	60.31	< 10	< 0.2	2236
601903-1124-1-8	89.6	4.9296	2712	55.01	72.64	< 10	< 0.2	2674
601903-1125-1-9	97.4	4.9325	3105	62.95	83.12	< 10	< 0.2	3031
601903-1126-1-10	94.3	4.9313	2965	60.13	79.39	< 50	< 1	2910
601903-1127-1-11	84.8	4.9279	2483	50.39	66.53	< 50	< 1	2464
601903-1130-1-12	74.7	4.9242	2079	42.22	55.74	5	0.10	2107
601903-1201-1-13	64.6	4.9205	1720	34.96	46.15	4	0.08	1740
601903-1201-1-14	54.3	4.9168	1393	28.33	37.41	5	0.10	1437
601903-1201-1-15	44.5	4.9132	1162	23.65	31.23	3	0.06	1178
601903-1204-1-16	34.7	4.9096	930	18.94	25.01	4	0.08	936
601903-1207-1-17	23.3	4.9055	735	14.98	19.78	4	0.08	737

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**Vessel 1: Reagent Grade As<sub>2</sub>O<sub>3</sub>**
**Test 2**

Sample No.	Temperature (deg. C)	Weighing Dish Initial Wt. (g)	Weighing Dish Final Wt. (g)	Sample Net Weight (g)	Volume of Liquid (ml)	Density of Solution (g/ml)	Sample Dilution	Total As Conc. of Diluted Sample (mg/L)	Total As Conc. Corrected for dilution (g/L)	Conc. of As <sub>2</sub> O <sub>3</sub> (g/L)
As-1-1	27.5	88.0382	93.0484	5.0102	4.9070	1.0210	0.0098	159	16.20	21.39
As-1-2	32.9	72.5726	77.5898	5.0172	4.9090	1.0220	0.0098	175	17.82	23.53
As-1-3	40.4	72.5694	77.5888	5.0194	4.9117	1.0219	0.0098	206	20.97	27.69
As-1-4	49.7	72.5713	77.6330	5.0617	4.9151	1.0298	0.0098	251	25.53	33.71
As-1-5	60.0	72.5696	77.6363	5.0667	4.9188	1.0301	0.0098	329	33.44	44.16
As-1-6	69.5	72.5708	77.6245	5.0537	4.9223	1.0267	0.0098	418	42.46	56.06
As-1-7	80.0	72.5719	77.6345	5.0626	4.9261	1.0277	0.0099	488	49.53	65.40
As-1-8	89.0	72.5706	77.6392	5.0686	4.9294	1.0282	0.0099	581	58.93	77.81
As-1-9	94.8	72.5729	77.6494	5.0765	4.9315	1.0294	0.0099	650	65.90	87.01
As-1-10	85.1	72.5698	77.6323	5.0625	4.9280	1.0273	0.0099	535	54.28	71.67
As-1-11	75.3	72.5706	77.6186	5.0480	4.9244	1.0251	0.0098	460	46.71	61.67
As-1-12	64.7	72.5707	77.6037	5.0330	4.9206	1.0229	0.0098	384	39.02	51.52
As-1-13	55.1	72.5685	77.5911	5.0226	4.9171	1.0215	0.0098	297	30.20	39.88
As-1-14	45.1	72.5721	77.5920	5.0199	4.9134	1.0217	0.0098	257	26.15	34.53
As-1-15	34.9	56.3292	61.3408	5.0116	4.9097	1.0208	0.0098	206	20.98	27.70
As-1-16	28.0	56.3291	61.3405	5.0114	4.9072	1.0212	0.0098	195	19.87	26.23

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**Vessel 2: Reagent Grade Sb<sub>2</sub>O<sub>3</sub>**

Sample No.	Temperature (deg. C)	Weighing Dish Initial Wt. (g)	Weighing Dish Final Wt. (g)	Sample Net Weight (g)	Volume of Liquid (ml)	Density of Solution (g/ml)	Sample dilution	Total Sb Conc. of Diluted Sample (mg/L)	Total Sb Conc. Corrected for dilution (mg/L)	Conc. of Sb <sub>2</sub> O <sub>3</sub> (mg/L)	Comparison by XRD
Sb-2-1	27.7	88.0350	92.9683	4.9333	4.9212	1.0025	0.0492	0.160	3.25	3.89	
Sb-2-2	33.9	88.0345	92.9624	4.9279	4.9239	1.0008	0.4924	2.200	4.47	5.35	
Sb-2-3	40.4	88.0362	92.9545	4.9183	4.9267	0.9983	0.4927	2.600	5.28	6.32	
Sb-2-4	50.0	88.0343	92.9426	4.9083	4.9310	0.9954	0.4931	4.170	8.46	10.12	
Sb-2-5	57.5	88.0386	92.9265	4.8879	4.9342	0.9906	0.4934	6.180	12.52	14.99	
Sb-2-6	69.9	88.0398	92.9100	4.8704	4.9397	0.9860	0.1976	3.970	20.09	24.05	
Sb-2-7	78.8	88.0362	92.8778	4.8416	4.9436	0.9794	0.1977	5.240	26.50	31.72	
Sb-2-8	89.5	88.0382	92.8485	4.8103	4.9483	0.9721	0.1979	7.270	36.73	43.97	
Sb-2-9	93.9	88.0367	92.8335	4.7968	4.9502	0.9690	0.1980	8.980	45.35	54.29	Sb <sub>2</sub> O <sub>3</sub>
Sb-2-10	85.1	88.0365	92.8773	4.8408	4.9464	0.9787	0.1979	6.860	34.67	41.51	
Sb-2-11	74.8	88.0364	92.8890	4.8526	4.9418	0.9819	0.1977	5.170	26.15	31.31	
Sb-2-12	65.1	88.0341	92.9134	4.8793	4.9376	0.9882	0.1975	3.520	17.82	21.34	
Sb-2-13	54.9	88.0390	92.9497	4.9107	4.9331	0.9955	0.1973	2.790	14.14	16.93	
Sb-2-14	45.0	80.1539	85.0654	4.9115	4.9288	0.9865	0.4929	6.540	13.27	15.88	
Sb-2-15	35.3	80.1547	85.0806	4.9259	4.9245	1.0003	0.4924	4.950	10.05	12.03	
Sb-2-16	28.2	88.0335	92.9565	4.9230	4.9214	1.0003	0.4921	6.200	12.60	15.08	

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## Vessel 5: 100 g/L Jan 98 Comp

Sample No.	Temp. (°C)	Volume of Liquid (ml)	Conc. of As <sup>3+</sup> in diluted sample (mg/L)	Conc. of As <sup>3+</sup> Corrected for dilution (g/L)	As <sub>2</sub> O <sub>3</sub> Concentration (g/L)	Conc. of As <sup>3+</sup> in diluted sample (mg/L)	Conc. of As <sup>3+</sup> Corrected for dilution (g/L)	Total As Conc. (mg/L)	Conc. Of Sb in diluted sample (mg/L)	Conc. Of Sb corrected for dilution (mg/L)	Conc. Of Fe in diluted sample (mg/L)	Conc. Of Fe corrected for dilution (mg/L)
H-JAN98-5	5.0	4.9313	225	4.56	6.02	0.70	0.01	232	< 1.0	< 20	< 0.1	< 2
H-JAN98-10	10.0	4.9326	257	5.21	6.88	< 5	< 0.1	258	< 1.0	< 20	< 0.1	< 2
H-JAN98-15	15.0	4.9338	258	5.23	6.90	1.00	0.02	259	< 0.8	< 16	< 0.05	< 1
H-JAN98-20	20.0	4.9351	278	5.63	7.44	1.00	0.02	286	< 0.8	< 16	0.14	2.84
H-JAN98-25	25.0	4.9364	308	6.20	8.18	1.00	0.02	305	< 0.4	< 8	0.18	3.85
601903-1113-5-1	26.4	4.9298	452	9.17	12.11	< 10	< 0.2	454	0.40	8.11	0.09	1.83
601903-1116-5-2	31.9	4.9318	472	9.57	12.84	< 10	< 0.2	481	0.50	10.14	< 0.06	< 1.2
601903-1117-5-3	39.7	4.9350	509	10.31	13.62	< 10	< 0.2	496	0.60	12.18	< 0.04	< 0.8
601903-1118-5-4	48.7	4.9390	605	12.25	16.17	< 10	< 0.2	603	0.75	15.19	0.11	2.23
601903-1119-5-5	60.0	4.9431	723	14.63	19.31	< 10	< 0.2	755	1.14	23.06	0.10	2.02
601903-1120-5-6	70.1	4.9472	885	17.48	23.09	< 10	< 0.2	915	1.72	34.77	< 0.09	< 1.8
601903-1123-5-7	79.3	4.9509	1070	21.61	28.54	< 10	< 0.2	1088	2.77	55.95	< 0.07	< 1.4
601903-1124-5-8	89.8	4.9551	1256	25.35	33.47	< 10	< 0.2	1279	4.18	84.38	< 0.07	< 1.4
601903-1125-5-9	97.4	4.9582	1407	28.38	37.47	< 10	< 0.2	1422	5.54	111.74	< 0.07	< 1.4
601903-1126-5-10	94.2	4.9569	1426	28.77	37.98	< 50	< 1	1418	5.19	104.70	< 0.07	< 1.4
601903-1127-5-11	83.8	4.9527	1434	28.95	38.23	< 50	< 1	1416	4.02	81.17	< 0.06	< 1.2
601903-1130-5-12	74.4	4.9489	1498	30.23	39.91	13.00	0.26	1509	3.06	61.83	< 0.06	< 1.2
601903-1201-5-13	64.5	4.9449	1422	28.78	37.97	12.00	0.24	1481	2.24	45.30	< 0.06	< 1.2
601903-1201-5-14	54.2	4.9408	1412	28.58	37.73	13.00	0.26	1484	1.82	38.84	< 0.07	< 1.4
601903-1201-5-15	44.5	4.9389	1250	25.32	33.43	9.00	0.18	1305	1.58	31.60	< 0.07	< 1.4
601903-1204-5-16	34.7	4.9329	969	19.64	25.94	12.00	0.24	1010	1.45	29.39	< 0.07	< 1.4
601903-1207-5-17	25.7	4.9293	798	16.15	21.32	13.00	0.26	804	1.36	27.59	< 0.09	< 1.8
C-JAN98-18	18.0	4.9346	307	6.22	8.21	1.60	0.03	313	< 0.9	< 18	0.28	5.67
C-JAN98-12	12.0	4.9331	310	6.28	8.30	1.60	0.03	313	< 0.9	< 18	0.32	6.49
C-JAN98-6	5.0	4.9313	312	6.33	8.35	2.20	0.04	311	< 0.9	< 18	0.29	5.88



Vessel 4:

100 g/L April 97 Comp

Sample No.	Temp. (°C)	Volume of Liquid (ml)	Conc. of As <sup>3+</sup> in diluted sample (mg/L)	Conc. of As <sup>3+</sup> Corrected for dilution (g/L)	As <sub>2</sub> O <sub>3</sub> Concentration (g/L)	Conc. of As <sup>5+</sup> in diluted sample (mg/L)	Conc. of As <sup>5+</sup> Corrected for dilution (g/L)	Total As Conc. (mg/L)	Conc. Of Sb in diluted sample (mg/L)	Conc. Of Sb corrected for dilution (mg/L)	Conc. Of Fe in diluted sample (mg/L)	Conc. Of Fe corrected for dilution (mg/L)
H-APR97-5	5.0	4.8988	240	4.90	6.47	<0.5	<10	240	<1.0	<20	<0.1	<2
H-APR97-10	10.0	4.9008	298	6.08	8.03	<0.5	<10	299	<1.0	<20	<0.1	<2
H-APR97-15	15.0	4.9025	322	6.57	8.67	1.00	0.02	328	<0.8	<18	<0.05	<1
H-APR97-20	20.0	4.9043	358	7.26	9.58	1.00	0.02	363	<0.8	<18	<0.05	<1
H-APR97-25	25.0	4.9081	393	8.01	10.58	1.00	0.02	387	<0.4	<8	<0.02	<0.4
601903-1113-4-1	27.2	4.9207	574	11.68	15.40	9.00	0.18	589	0.30	6.10	<0.03	<0.8
601903-1116-4-2	33.3	4.9238	822	12.83	16.68	<10	<0.2	828	0.50	10.16	<0.06	<1.2
601903-1117-4-3	39.9	4.9266	651	13.21	17.45	<10	<0.2	638	0.50	10.15	<0.04	<0.8
601903-1118-4-4	49.4	4.9311	728	14.78	19.49	<10	<0.2	719	0.65	13.18	<0.04	<0.8
601903-1119-4-5	58.3	4.9352	792	16.05	21.19	<10	<0.2	828	0.38	7.29	<0.09	<1.8
601903-1120-4-6	69.5	4.9405	898	18.18	24.00	<10	<0.2	949	1.41	28.54	<0.09	<1.8
601903-1123-4-7	79.4	4.9451	1045	21.13	27.90	<10	<0.2	1112	2.68	53.79	<0.07	<1.4
601903-1124-4-8	88.2	4.9492	1232	24.69	32.67	<10	<0.2	1275	3.65	73.75	<0.07	<1.4
601903-1125-4-9	97.8	4.9537	1502	30.32	40.03	<10	<0.2	1478	4.77	98.29	<0.07	<1.4
601903-1126-4-10	92.7	4.9513	1620	32.72	43.20	<50	<1	1629	3.91	78.97	<0.07	<1.4
601903-1127-4-11	83.6	4.9471	1611	32.56	43.00	<50	<1	1622	3.08	61.85	<0.08	<1.2
601903-1130-4-12	75.5	4.9433	1675	33.88	44.74	11.00	0.22	1698	2.51	50.78	<0.08	<1.2
601903-1201-4-13	63.6	4.9377	1605	32.50	42.92	11.00	0.22	1675	1.71	34.63	<0.06	<1.2
601903-1201-4-14	54.3	4.9334	1445	29.29	38.67	11.00	0.22	1481	1.22	24.73	<0.07	<1.4
601903-1201-4-15	44.8	4.9289	1195	24.24	32.01	8.00	0.18	1219	1.20	24.35	<0.07	<1.4
601903-1204-4-16	34.8	4.9243	953	19.35	25.55	10.00	0.20	973	1.18	23.56	<0.07	<1.4
601903-1207-4-17	26.0	4.9202	795	16.16	21.33	10.00	0.20	810	1.02	20.73	<0.09	<1.8
C-APR97-18	18.0	4.9035538	399	8.14	10.74	1.20	0.02	398	<0.9	<18	0.06	1.22
C-APR97-12	12.0	4.9013692	401	8.18	10.80	1.30	0.03	401	<0.9	<18	0.06	1.22
C-APR97-5	5.0	4.8968205	398	8.12	10.73	1.60	0.03	397	<0.9	<18	0.06	1.22

Vessel 2: 100 g/L 212 Chamber

Sample No.	Temp. (°C)	Volume of Liquid (ml)	Conc. of As <sup>3+</sup> in diluted sample (mg/L)	Conc. of As <sup>3+</sup> Corrected for dilution (g/L)	As <sub>2</sub> O <sub>3</sub> Concentration (g/L)	Conc. of As <sup>3+</sup> in diluted sample (mg/L)	Conc. of As <sup>3+</sup> Corrected for dilution (g/L)	Total As Conc. (mg/L)	Conc. Of Sb in diluted sample (mg/L)	Conc. Of Sb corrected for dilution (mg/L)	Conc. Of Fe in diluted sample (mg/L)	Conc. Of Fe corrected for dilution (mg/L)
H-212-5	5.0	4.9112	231	4.70	6.21	8.00	0.18	240	< 1.0	< 20	< 0.1	< 2
H-212-10	10.0	4.9134	285	5.39	7.12	7.00	0.14	271	< 1.0	< 20	< 0.1	< 2
H-212-15	15.0	4.9156	275	5.59	7.39	7.00	0.14	279	< 0.8	< 16	< 0.05	< 1
H-212-20	20.0	4.9178	297	6.04	7.97	8.00	0.16	308	0.89	18	< 0.05	< 1
H-212-25	25.0	4.9200	334	6.79	8.98	9.00	0.18	338	0.88	18	< 0.02	< 0.4
601903-1113-2-1	26.5	4.9208	455	9.25	12.21	6.00	0.12	471	1.20	24	0.03	0.6
601903-1116-2-2	33.2	4.9236	481	9.77	12.90	< 10	< 0.2	497	1.60	32	< 0.06	< 1.2
601903-1117-2-3	36.9	4.9265	500	10.15	13.40	9.00	0.18	497	2.00	41	< 0.04	< 0.8
601903-1118-2-4	40.5	4.9307	553	11.22	14.81	10.00	0.20	558	2.40	49	0.05	1.0
601903-1119-2-5	59.4	4.9351	816	12.48	16.48	11.00	0.22	845	1.74	35	< 0.09	< 1.8
601903-1120-2-6	69.8	4.9398	710	14.37	18.68	12.00	0.24	757	2.74	55	< 0.09	< 1.8
601903-1123-2-7	80.0	4.9441	685	17.90	23.63	18.00	0.36	920	4.83	98	< 0.07	< 1.4
601903-1124-2-8	90.0	4.9485	1028	20.77	27.43	20.00	0.40	1037	6.39	129	< 0.07	< 1.4
601903-1125-2-9	97.6	4.9518	1219	24.62	32.50	25.00	0.50	1259	7.75	157	< 0.07	< 1.4
601903-1126-2-10	94.9	4.9507	1312	26.50	34.99	27.00	0.55	1347	7.82	158	< 0.07	< 1.4
601903-1127-2-11	85.0	4.9483	1293	26.14	34.51	26.00	0.53	1345	6.27	127	< 0.06	< 1.2
601903-1130-2-12	75.3	4.9421	1360	27.52	36.33	28.00	0.57	1382	5.76	117	< 0.06	< 1.2
601903-1201-2-13	65.0	4.9375	1299	26.31	34.74	27.00	0.55	1388	4.87	99	< 0.06	< 1.2
601903-1201-2-14	54.6	4.9330	1290	26.15	34.53	28.00	0.57	1335	4.46	90	< 0.07	< 1.4
601903-1201-2-15	44.8	4.9288	1245	25.28	33.35	27.00	0.55	1280	4.15	84	< 0.07	< 1.4
601903-1204-2-16	34.7	4.9242	970	19.70	26.01	28.00	0.57	1030	4.26	87	< 0.07	< 1.4
601903-1207-2-17	25.3	4.9201	804	16.34	21.58	28.00	0.57	824	4.05	82	< 0.09	< 1.8
C-212-18	18.0	4.9189	335	6.81	9.00	9.00	0.18	343	0.92	19	< 0.04	< 0.8
C-212-12	12.0	4.9143	335	6.82	9.00	9.00	0.18	348	0.90	18	< 0.04	< 0.8
C-212-5	5.0	4.9112	338	6.88	9.09	9.00	0.18	346	0.90	18	< 0.04	< 0.8

Vessel 3: 100 g/L 236 Chamber

Sample No.	Temp. (°C)	Volume of Liquid (ml)	Conc. of As <sup>3+</sup> in diluted sample (mg/L)	Conc. of As <sup>3+</sup> Corrected for dilution (g/L)	As <sub>2</sub> O <sub>3</sub> Concentration (g/L)	Conc. of As <sup>3+</sup> in diluted sample (mg/L)	Conc. of As <sup>3+</sup> Corrected for dilution (g/L)	Total As Conc. (mg/L)	Conc. Of Sb in diluted sample (mg/L)	Conc. Of Sb corrected for dilution (mg/L)	Conc. Of Fe in diluted sample (mg/L)	Conc. Of Fe corrected for dilution (mg/L)
H-236-5	5.0	4.9103	442	9.00	11.88	12.00	0.24	445	< 1.0	< 20	< 0.1	< 2
H-236-10	10.0	4.9127	462	9.40	12.42	13.00	0.26	465	< 1.0	< 20	< 0.1	< 2
H-236-15	15.0	4.9150	476	9.68	12.79	12.00	0.24	477	< 0.8	< 16	< 0.05	< 1
H-236-20	20.0	4.9173	513	10.43	13.77	12.00	0.24	527	< 0.8	< 16	< 0.05	< 1
H-236-25	25.0	4.9197	578	11.75	15.51	13.00	0.26	576	< 0.4	< 8	< 0.02	< 0.4
601903-1113-3-1	27.0	4.9208	639	12.99	17.15	8.00	0.16	648	0.60	12.19	0.06	1.22
601903-1116-3-2	34.4	4.3988	704	16.00	21.13	8.00	0.16	719	0.70	15.91	< 0.06	< 1.2
601903-1117-3-3	40.0	4.4003	737	16.75	22.11	10.00	0.23	721	0.75	17.04	< 0.04	< 0.8
601903-1118-3-4	50.1	4.4029	859	19.51	25.76	11.00	0.25	870	0.80	18.17	< 0.04	< 0.8
601903-1119-3-5	60.0	4.4054	971	22.04	29.10	15.00	0.34	1011	1.08	24.08	< 0.09	< 1.8
601903-1120-3-6	70.1	4.4080	1123	25.48	33.64	13.00	0.29	1188	1.58	35.84	< 0.09	< 1.8
601903-1123-3-7	80.2	4.4108	1313	29.77	39.31	15.00	0.34	1337	2.71	61.44	< 0.07	< 1.4
601903-1124-3-8	90.0	4.4131	1489	33.29	43.95	16.00	0.36	1520	3.90	88.37	< 0.07	< 1.4
601903-1125-3-9	97.5	4.4150	1592	36.06	47.61	19.00	0.43	1585	4.75	107.59	< 0.07	< 1.4
601903-1126-3-10	94.6	4.4143	1613	36.54	48.25	20.00	0.45	1623	4.60	108.74	< 0.07	< 1.4
601903-1127-3-11	85.0	4.4118	1593	36.11	47.67	21.00	0.46	1574	4.37	99.05	< 0.06	< 1.2
601903-1130-3-12	75.1	4.4093	1626	36.88	48.69	21.00	0.46	1697	2.78	63.05	< 0.06	< 1.2
601903-1201-3-13	64.9	4.4067	1573	35.70	47.13	20.00	0.45	1674	2.43	55.14	< 0.06	< 1.2
601903-1201-3-14	54.7	4.4040	1566	35.56	46.95	20.00	0.45	1608	1.54	34.97	< 0.07	< 1.4
601903-1201-3-15	45.0	4.4015	1589	36.10	47.67	20.00	0.45	1619	1.42	32.28	< 0.07	< 1.4
601903-1204-3-16	35.2	4.3990	1559	35.44	46.79	19.00	0.43	1584	1.33	30.23	< 0.07	< 1.4
601903-1207-3-17	26.8	4.3989	809	16.40	24.29	20.00	0.45	824	1.08	24.58	< 0.09	< 1.8
C-236-18	18.0	4.9164	588	11.96	15.79	13.00	0.26	584	< 0.9	< 18	0.05	1.02
C-236-12	12.0	4.9136	592	12.05	15.91	13.00	0.26	596	< 0.9	< 18	0.05	1.02
C-236-5	5.0	4.9103	591	12.04	15.69	13.00	0.26	587	< 0.9	< 18	0.05	1.02

**Vessel 6:**
**50 g/L Reagent grade As<sub>2</sub>O<sub>3</sub>**

Sample No.	Time (hours)	Temp. (°C)	Volume of Liquid (ml)	Conc. of As <sup>3+</sup> in diluted sample (mg/L)	Conc. of As <sup>3+</sup> Corrected for dilution (g/L)	As <sub>2</sub> O <sub>3</sub> Concentration (g/L)	Conc. of As <sup>5+</sup> in diluted sample (mg/L)	Conc. of As <sup>5+</sup> Corrected for dilution (mg/L)	Total As Conc. (mg/L)
	0.0	22.0		0.00	0.00	0.00	0.00	0.00	0.00
601903-EQ-1	0.5	22.0	4.9064	0.60	0.01	0.02	< 0.1	< 2	
601903-EQ-2	1.0	22.0	4.9064	1.90	0.04	0.05	< 0.1	< 2	
601903-EQ-3	2.0	22.5	4.9067	6.40	0.13	0.17	< 0.1	< 2	
601903-EQ-4	4.0	23.5	4.9074	22.00	0.45	0.59	< 5	< 100	22.00
601903-EQ-5	7.0	25.0	4.9083	71.00	1.45	1.91	< 5	< 100	69.40
601903-EQ-6	24.0	25.0	4.9083	519.00	10.57	13.98	< 10	< 200	521.00
601903-EQ-7	48.0	25.2	4.9084	703.00	14.32	18.91	< 10	< 200	727.00
601903-EQ-8	72.0	25.5	4.9086	721.00	14.69	19.39	< 10	< 200	763.00
601903-EQ-9	144.0	25.5	4.9086	730.00	14.87	19.64	< 10	< 200	739.00
601903-EQ-10	168.0	25.8	4.9088	743.00	15.14	19.98	< 10	< 200	736.00

**Vessel 6:**
**75 g/L Reagent grade As<sub>2</sub>O<sub>3</sub>**

Sample No.	Time (hours)	Temp. (°C)	Volume of Liquid (ml)	Conc. of As <sup>3+</sup> in diluted sample (mg/L)	Conc. of As <sup>3+</sup> Corrected for dilution (g/L)	As <sub>2</sub> O <sub>3</sub> Concentration (g/L)	Conc. of As <sup>5+</sup> in diluted sample (mg/L)	Conc. of As <sup>5+</sup> Corrected for dilution (mg/L)	Total As Conc. (mg/L)
					0.00	0.00		0.00	
601903-EQ60-1	1.0	59.5	4.9294	535	10.85	14.33	19	385.45	494
601903-EQ60-2	2.0	60.0	4.9297	1150	23.33	30.80	17	344.85	1125
601903-EQ60-3	4.1	60.4	4.9299	1489	30.20	39.88	24	486.82	1447
601903-EQ60-4	7.0	60.0	4.9297	1506	30.55	40.34	< 50	< 1000	1492
601903-EQ60-5	24.0	59.9	4.9296	1529	31.02	40.95	< 50	< 1000	1508
601903-EQ60-6	48.0	59.6	4.9294	1548	31.40	41.46	< 50	< 1000	1527
601903-EQ60-7	120.0	59.9	4.9296	1612	32.70	43.18		0.00	

CANMET



Vessel 1:

## 100 g/L 212 Chamber Sample Saturation Test at 95°C

Sample No.	Time (hours)	Temp. (°C)	Volume of Liquid (ml)	Conc. of As <sup>3+</sup> in diluted sample (mg/L)	Conc. of As <sup>3+</sup> Corrected for dilution (g/L)	As <sub>2</sub> O <sub>3</sub> Concentration (g/L)	Conc. of As <sup>5+</sup> in diluted sample (mg/L)	Conc. of As <sup>5+</sup> Corrected for dilution (mg/L)
	0.0	95.0	4.9316	0	0.00	0.00	0	0.00
601903-212-95-1	6.0	95.0	4.9316	1605	32.55	42.97	17	344.72
601903-212-95-2	24.0	95.4	4.9317	1595	32.34	42.70	22	446.09
601903-212-95-3	48.0	94.6	4.9314	1654	33.54	44.28	29	588.06
601903-212-95-4	72.0	94.8	4.9315	1639	33.24	43.88	36	730.00
601903-212-95-5	168.0	94.5	4.9314	1752	35.53	46.91	54	1095.02

**Vessel 4:****100 g/L Jan 98 Sample**

Sample No.	Time (days)	Temp. (°C)	Volume of Liquid (ml)	Conc. of As <sup>3+</sup> in diluted sample (mg/L)	Conc. of As <sup>3+</sup> Corrected for dilution (g/L)	As <sub>2</sub> O <sub>3</sub> Concentration (g/L)	Conc. of As <sup>5+</sup> in diluted sample (mg/L)	Conc. of As <sup>5+</sup> Corrected for dilution (mg/L)
601904-EQ4-1	7.0	26.2	4.9202	444	9.02	11.9	3.5	71.1
601904-EQ4-2	9.0	26.7	4.9205	446	9.06	12.0	3.5	71.1
601904-EQ4-3	21.0	26.0	4.9202	470	9.55	12.6	4.0	81.3

**Vessel 3:****100 g/L Apr 97 Sample**

Sample No.	Time (days)	Temp. (°C)	Volume of Liquid (ml)	Conc. of As <sup>3+</sup> in diluted sample (mg/L)	Conc. of As <sup>3+</sup> Corrected for dilution (g/L)	As <sub>2</sub> O <sub>3</sub> Concentration (g/L)	Conc. of As <sup>5+</sup> in diluted sample (mg/L)	Conc. of As <sup>5+</sup> Corrected for dilution (mg/L)
601903-EQ3-1	7.0	26.1	4.3967	604	13.7	18.1	3.4	77.3
601903-EQ3-2	9.0	26.8	4.3969	601	13.7	18.0	3.2	72.8
601903-EQ3-3	21.0	26.2	4.3967	608	13.8	18.3	4.0	91.0

CANMET



**Vessel 1:****100 g/L 212 Chamber Sample**

Sample No.	Time (days)	Temp. (°C)	Volume of Liquid (ml)	Conc. of As <sup>3+</sup> In diluted sample (mg/L)	Conc. of As <sup>3+</sup> Corrected for dilution (g/L)	As <sub>2</sub> O <sub>3</sub> Concentration (g/L)	Conc. of As <sup>5+</sup> In diluted sample (mg/L)	Conc. of As <sup>5+</sup> Corrected for dilution (mg/L)
601903-EQ1-1	7.0	23.3	4.9055	448	9.13	12.1	15.0	306
601903-EQ1-2	9.0	23.8	4.9057	446	9.09	12.0	15.0	306
601903-EQ1-3	21.0	23.0	4.9054	465	9.48	12.5	16.0	326

**Vessel 2:****100 g/L 236 Chamber Sample**

Sample No.	Time (days)	Temp. (°C)	Volume of Liquid (ml)	Conc. of As <sup>3+</sup> In diluted sample (mg/L)	Conc. of As <sup>3+</sup> Corrected for dilution (g/L)	As <sub>2</sub> O <sub>3</sub> Concentration (g/L)	Conc. of As <sup>5+</sup> In diluted sample (mg/L)	Conc. of As <sup>5+</sup> Corrected for dilution (mg/L)
601903-EQ2-1	7.0	26.5	4.9206	684	13.9	18.4	16.3	331
601903-EQ2-2	9.0	26.9	4.9208	686	13.9	18.4	16.2	329
601903-EQ2-3	21.0	26.4	4.9206	706	14.3	18.9	17.0	345



**As<sub>2</sub>O<sub>3</sub> Solubility at Variable Pulp Densities at 70°C**

Vessel: Sealed glass 2 L reaction kettle heated in oil bath  
Solution: 1.0 L Water  
Dust sample: Apr 97 Comp Dust  
Temperature: 70°C  
Sampling Time: 96 hours  
Filtration: Millipore 0.45 micron pore size  
Stirring: Magnetic stirring bar

Note: 5 drops of liquid detergent added to all slurries to reduce surface scum.

Sample No.	Pulp Density (g/L)	Total As in diluted sample (g/L)	[As] corrected for dilution (g/L)	[As <sub>2</sub> O <sub>3</sub> ] (g/L)	Total Sb in diluted sample (mg/L)	[Sb] corrected for dilution (mg/L)	Weight of Solids after Filtration (g)	Solids Composition
APR97-75	75.0	1.539	31.47	41.55	2.03	41.51	38.2	
APR97-150	150.0	1.783	36.38	48.03	1.83	37.33	99.7	
APR97-225	225.0	1.823	37.01	48.87	1.68	34.11	168.6	
APR97-300	300.0	1.921	39.20	51.76	1.80	36.73	248.5	

## **APPENDIX 7**

**Detailed Electron Microprobe Analyses of the  
As<sub>2</sub>O<sub>3</sub> Phase Remaining in the April 97  
Composite Sample After Water Leaching at  
70°C and Various Pulp Densities**

April 97 Composite, 70°C, 75 g/L Solids

Weight percent	Group : CHEN				Sample : As2O3-Residue			Page 1
Element	1	2	6	12	15	17	18	21
As	81.826	78.599	75.992	75.499	71.261	72.131	73.781	80.177
Sb	0.609	0.374	0.692	0.637	1.013	7.445	1.289	1.460
Fe	0.093	0.196	0.132	0.157	0.155	0.134	0.247	0.159
Pb	0.095	0.131	0.369	0.277	0.167	0.127	0.118	0.056
Si	0.727	-	-	-	-	-	0.899	-
O	16.650	20.700	22.815	23.430	27.404	20.163	23.666	18.148
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Element	22	24	37	38	49
As	74.277	72.814	70.944	70.209	74.530
Sb	2.069	1.912	0.279	1.654	1.996
Fe	0.270	0.178	0.150	0.312	0.189
Pb	0.234	0.159	0.030	-	0.142
Si	-	0.022	-	-	-
O	23.150	24.915	28.597	27.825	23.143
Total	100.000	100.000	100.000	100.000	100.000

Element	Minimum	Maximum	Average	Sigma
As	70.209	81.826	74.772	3.600
Sb	0.279	7.445	1.648	1.850
Fe	0.093	0.312	0.182	0.061
Pb	-	0.369	0.147	0.101
Si	-	0.899	0.127	0.307
O	16.650	28.597	23.124	3.598
Total	100.000	100.000	100.000	0.000
No. of data 13				



# PROTECTED BUSINESS INFORMATION

April 97 Composite, 70°C, 150 g/L Solids

Weight percent      Group : CHEN      Sample : As2O3-Residue      Page 1

Element	50	51	52	56	57	59	62	64
As	77.884	83.331	77.177	72.840	76.611	74.083	71.983	72.474
Sb	0.334	0.548	0.621	0.469	0.579	0.408	1.580	0.569
Fe	0.444	0.487	0.413	0.206	0.148	0.080	0.329	0.182
Pb	0.010	0.092	0.159	0.171	0.157	0.295	0.354	0.330
Si	-	-	0.195	0.046	0.612	0.186	0.208	0.558
O	21.328	15.542	21.435	26.268	21.893	24.948	25.546	25.887
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Element	67	68	69	71	81	82	88	96
As	74.889	77.129	72.399	75.007	73.137	75.008	73.238	73.702
Sb	0.655	0.716	1.074	0.625	0.948	0.566	4.254	0.629
Fe	0.268	0.231	0.435	0.184	0.088	0.119	0.208	0.102
Pb	0.708	0.901	1.279	0.101	0.179	0.157	0.058	0.201
Si	0.438	0.731	0.233	-	-	-	-	-
O	23.042	20.292	24.580	24.083	25.648	24.150	22.242	25.366
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Element	98	99	100	103	105	106	107	121
As	73.573	74.842	76.408	72.892	75.354	75.734	76.574	78.217
Sb	0.128	0.122	0.153	1.001	1.151	0.432	0.534	0.823
Fe	0.114	0.120	0.089	0.107	0.430	0.141	0.087	0.212
Pb	0.075	0.170	0.407	0.007	0.245	0.346	0.376	0.133
Si	-	-	-	-	-	-	-	-
O	26.110	24.746	22.943	25.993	22.820	23.347	22.429	20.615
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Element	122	125	126	132	137	138	147
As	73.198	75.280	72.282	74.901	70.734	70.482	72.353
Sb	1.114	0.796	0.829	1.017	0.677	0.774	1.278
Fe	0.494	0.651	0.520	0.243	0.236	0.281	0.267
Pb	0.308	0.244	0.243	0.066	0.136	0.123	0.428
Si	-	-	-	-	-	-	-
O	24.886	23.029	26.126	23.773	28.217	28.340	25.674
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Element	Minimum	Maximum	Average	Sigma
As	70.482	83.331	74.636	2.581
Sb	0.122	4.254	0.819	0.720
Fe	0.080	0.651	0.255	0.156
Pb	0.007	1.279	0.273	0.267
Si	-	0.731	0.103	0.204
O	15.542	28.340	23.913	2.554
Total	100.000	100.000	100.000	0.000
No. of data 31				



April 97 Composite, 70°C, 75 g/L Solids

Weight percent		Group : CHEN				Sample : As2O3-Residue			Page 1
Element	13	20	34	36	40	41	42	45	
As	58.280	62.449	40.916	48.939	33.805	45.710	46.559	48.021	
Sb	15.677	12.045	28.279	21.496	18.192	19.093	18.218	24.980	
Fe	0.289	0.243	0.345	0.790	7.332	10.282	0.324	0.192	
Pb	0.199	0.185	0.304	0.222	-	0.109	2.318	0.001	
Si	-	0.848	-	-	-	-	-	-	
O	25.555	24.230	30.156	28.553	40.671	24.806	32.581	26.806	
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000	

Element	46	Minimum	Maximum	Average	Sigma
As	47.673	33.805	62.449	48.039	8.474
Sb	22.648	12.045	28.279	20.070	4.896
Fe	0.246	0.192	10.282	2.227	3.807
Pb	-	-	2.318	0.371	0.738
Si	-	-	0.848	0.094	0.283
O	29.433	24.230	40.671	29.199	5.092
Total	100.000	100.000	100.000	100.000	-
No. of data 9					



April 97 Composite, 70°C, 150 g/L Solids

Weight percent		Group : CHEN				Sample : As2O3-Residue		Page 1
Element	58	61	74	110	113	114	115	
As	49.962	50.305	63.050	43.690	44.946	46.965	53.193	
Sb	11.385	12.221	18.973	25.097	30.234	29.140	28.692	
Fe	1.010	0.348	0.204	0.536	0.135	0.133	0.129	
Pb	0.198	0.061	0.147	0.249	0.317	0.175	0.170	
Si	-	-	-	0.085	0.673	0.178	0.145	
O	37.445	37.065	17.626	30.343	23.695	23.409	17.671	
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	

Element	Minimum	Maximum	Average	Sigma
As	43.690	63.050	50.302	6.513
Sb	11.385	30.234	22.249	8.064
Fe	0.129	1.010	0.356	0.325
Pb	0.061	0.317	0.188	0.080
Si	-	0.673	0.154	0.240
O	17.626	37.445	26.751	8.365
Total	100.000	100.000	100.000	0.000
No. of data 7				



## **APPENDIX 8**

### **X-ray Diffraction Patterns of the Four Sublimation Residues**

