



Recovery and Purification of Arsenic Oxide - Giant Mine

by

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Objectives

To study the recovery and production of arsenic oxide (As_2O_3) from the arsenic-rich dust produced and stored at the Giant Mine, Yellowknife

Major Tasks

- Solubility measurements on four dust samples, and comparison with measurements for reagent grade As_2O_3
- Use of autoclave leaching at $>100\text{ }^\circ\text{C}$ to achieve high As_2O_3 solubilities
- Attempted use of ion exchange to remove antimony from the leaching solutions
- Controlled vapourization of the arsenic oxide dusts to up-grade the As_2O_3 products
- Supporting mineralogical studies of the dusts and the intermediate reaction products

Costs

Phase 1:

- \$55,000 - \$44,000 DIAND
\$11,000 Royal Oak

Phase 2:

- \$25,300 - \$25,300 DIAND

Schedule

- Initial agreement (Phase 1) signed October, 1998
 - completion date March 31, 1999
- Phase 2 signed February, 1999
 - completion date April, 1999

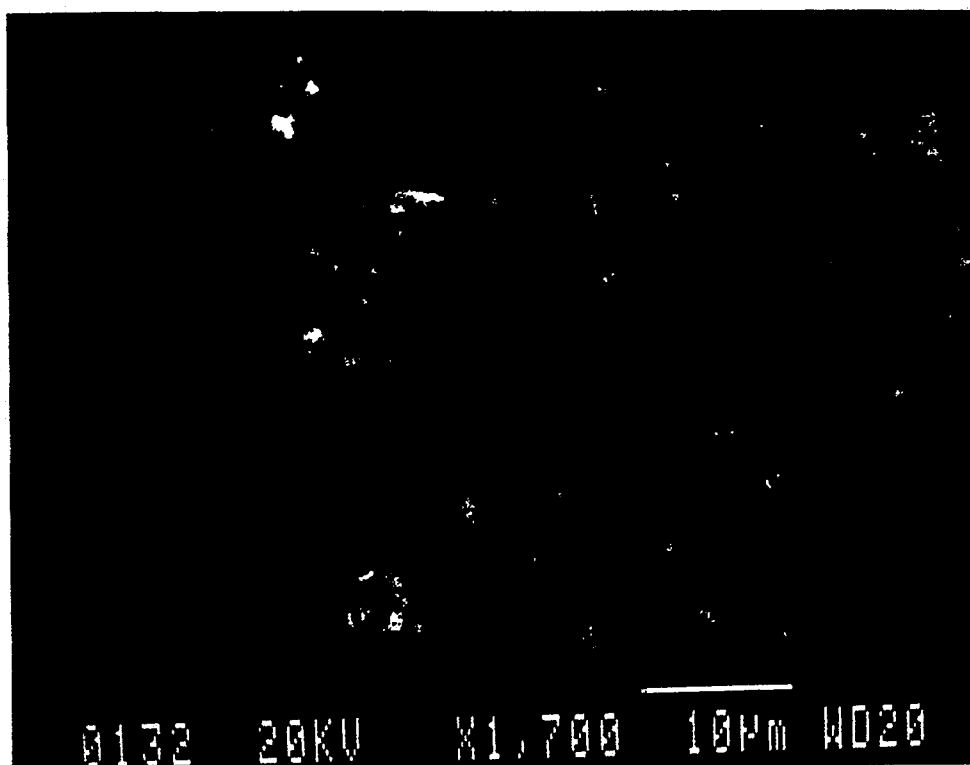
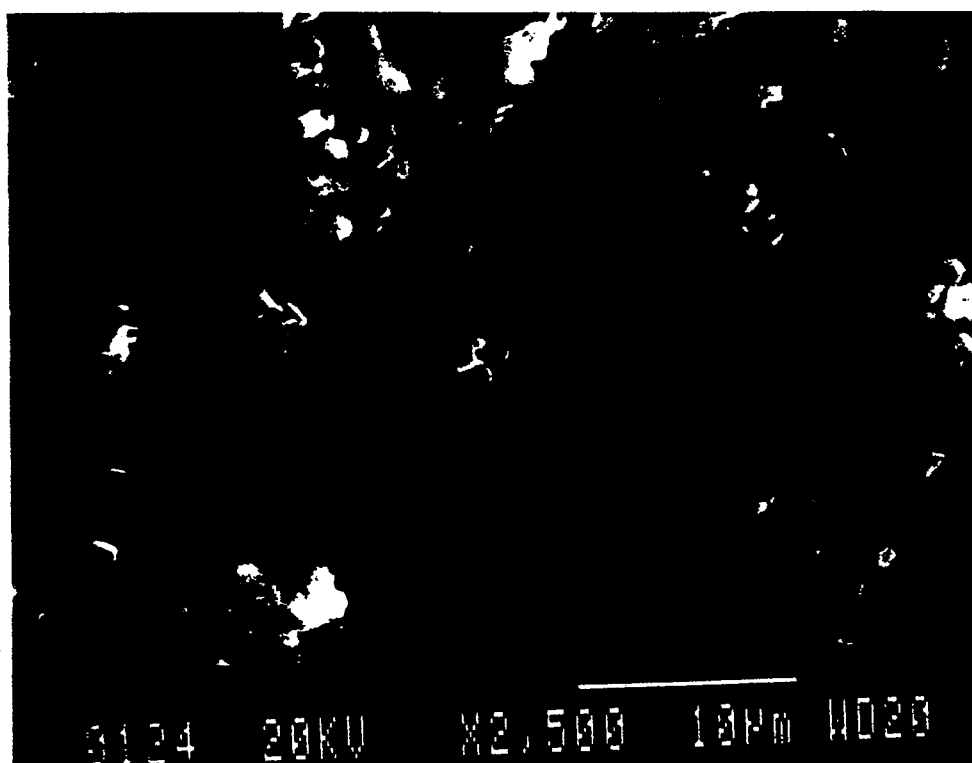
Presentation of Results

- Mineralogy
- Controlled vapourization studies
- Solubility data
- Water leaching results
- Ion exchange
- Future work (?)

Mineralogy

	Chamber 236	Chamber 212	Apr-97 Composite	Jan-98 Composite
As ₂ O ₃	M	M	M	M
(As,Sb) ₂ O ₃	t	M	M	M
Fe-AsO ₄	m	m	m	m
Ca-Fe-AsO ₄	t	m	t	t
Sb-As-Fe oxide	-	t	t	t
Ca-Fe oxide	-	t	t	-
CaSO ₄	-	t	-	-
Fe oxide	t	t	t	-
Chlorite	M	M	M	M
Quartz, mica, feldspar	t	m	t	t

M- major constituent; m- minor constituent; t- trace constituent



Chamber 212

**Average Electron Microprobe Analyses of
Phases in Sample- Chamber 212 (wt %)**

	As₂O₃	(As,Sb)₂O₃	Fe-AsO₄
As	76.02	50.47	8.55
Sb	1.20	24.45	3.10
Fe	0.57	0.34	52.41

As-, Sb- and Fe- Carriers

As-carriers:

1. As_2O_3
2. Fe arsenate, As 1.9 – 38 %
3. Ca-Fe arsenate, minor carrier

Sb-carriers:

1. As_2O_3 or $(\text{As},\text{Sb})_2\text{O}_3$, Sb substitutes for As in As_2O_3 structure
2. Fe arsenate, Sb 0.4 – 10 %
3. Ca-Fe arsenate, 0 – 0.8 %
4. Sb-As-Fe oxide, minor carrier

Fe-carriers:

1. Chlorite $(\text{Mg},\text{Fe})_5\text{Al}(\text{Si}_3\text{Al})\text{O}_{10}(\text{OH})_8$
2. As_2O_3 , Fe 0 – 2 %
3. Fe arsenate, Fe 11.3 – 66 %
4. Ca-Fe oxide, minor carrier
5. Ca-Fe arsenate, minor carrier
6. Fe oxide, minor carrier

Water Leach Residue

85g of dust (April-97 composite)

1 L at 150 °C, 2h

cooled to 95 °C, filtered

The residue: Fe- 9.4 %, As- 25.1 %, Sb- 15.1 %

Major- SbAsO_3 , chlorite

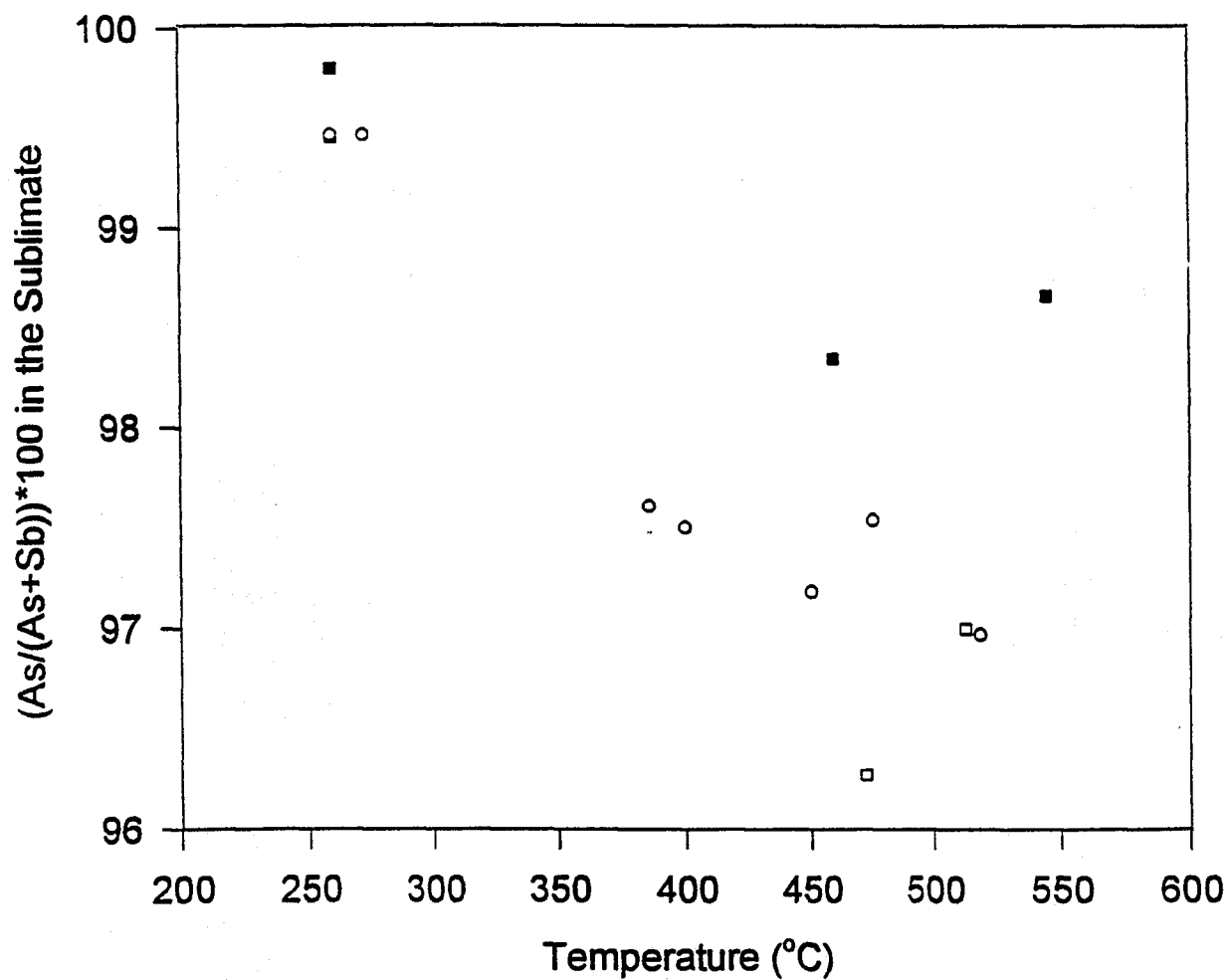
Minor- Sb-As-Fe oxide

Trace- Fe oxide, quartz and feldspar



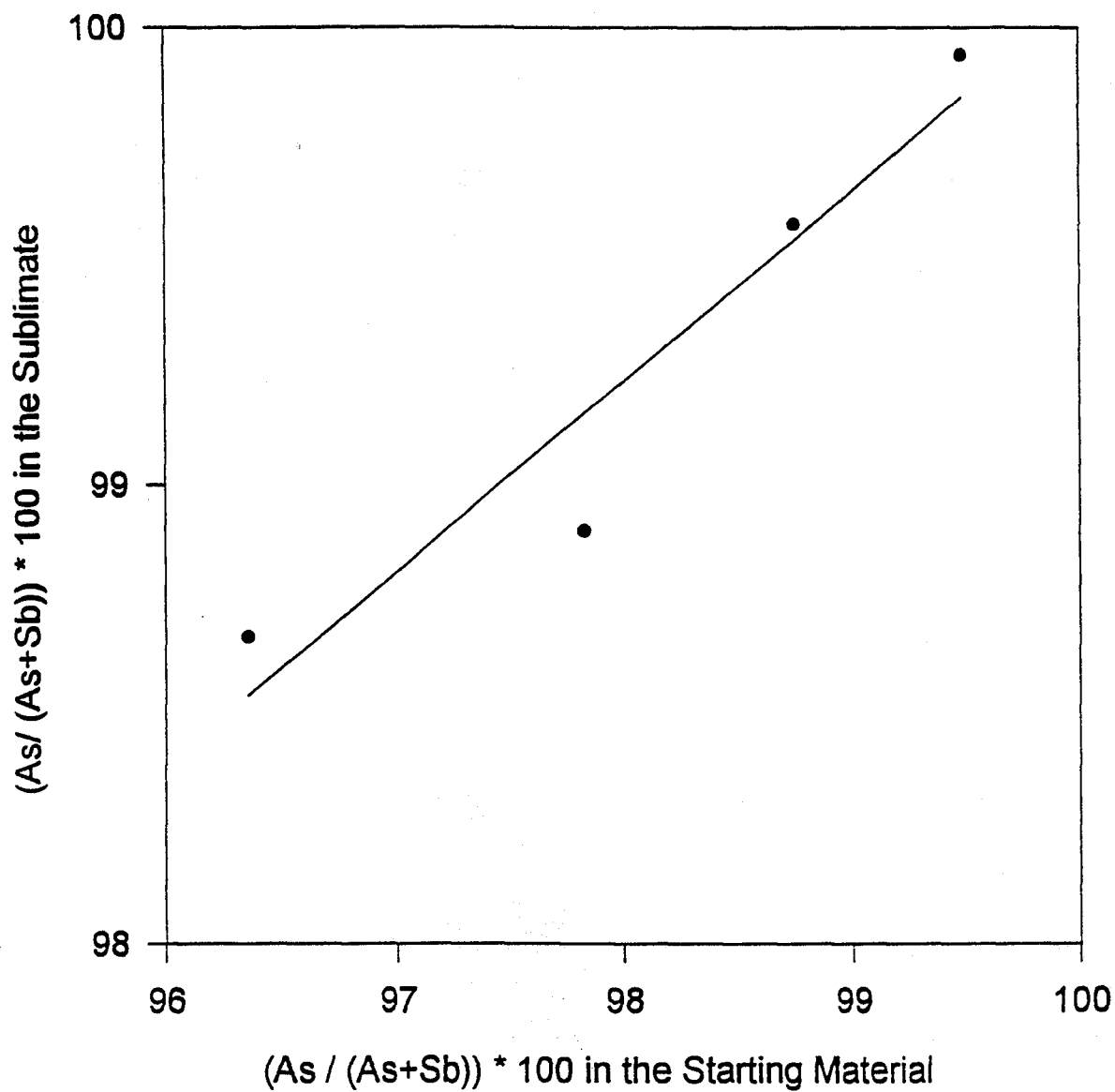
Water Leach Residue

Percentage of As the Sublimate in Function of the Temperature



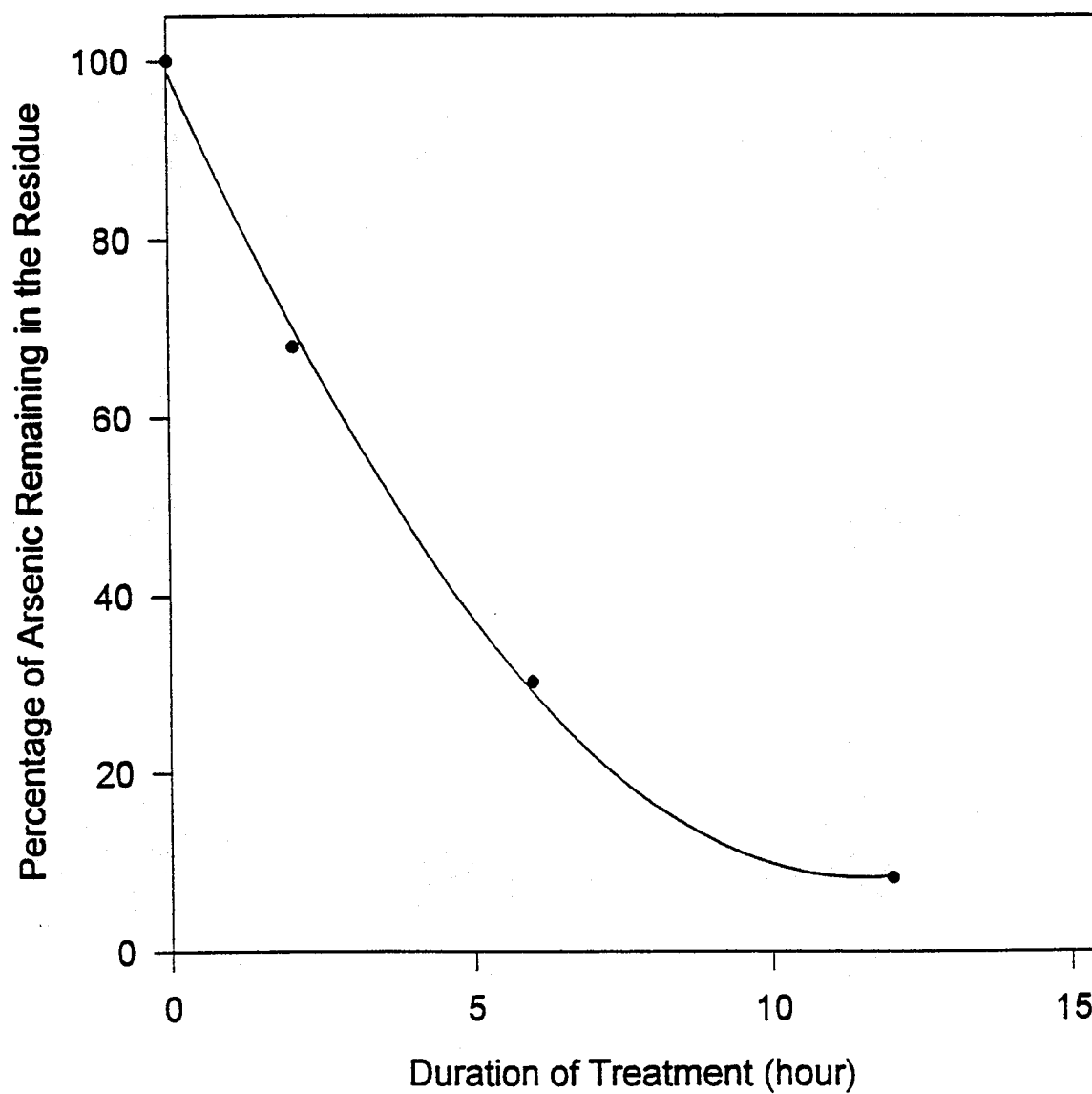
- H₂
- He and N₂
- air and O₂

As Content in the Sublimate VS As Content in the Starting Material



Treatment: heating to 545°C for 10 hours in air

Evolution of the Residue in Function of the Time



Treatment: heating to 259°C in air

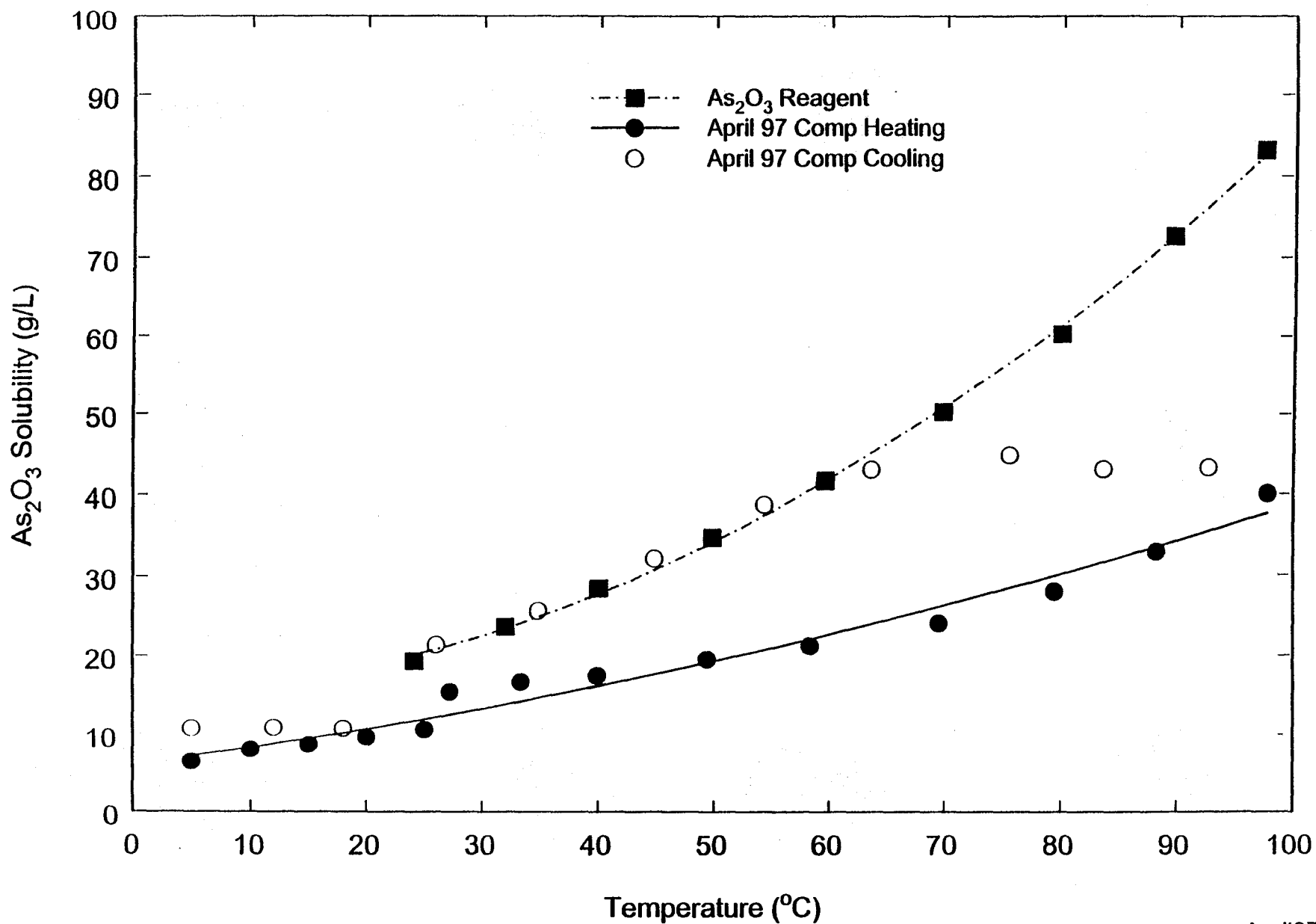
Element	Dust	Residue		Sublimate	
		analysis	partition	analysis	partition
	(%)	(%)	(%)	(%)	(%)
As	56.20	38.00	28	70.45	72
Sb	2.13	3.84	80	0.24	20

Dust 212, heated to 259°C for 6 hours in air.

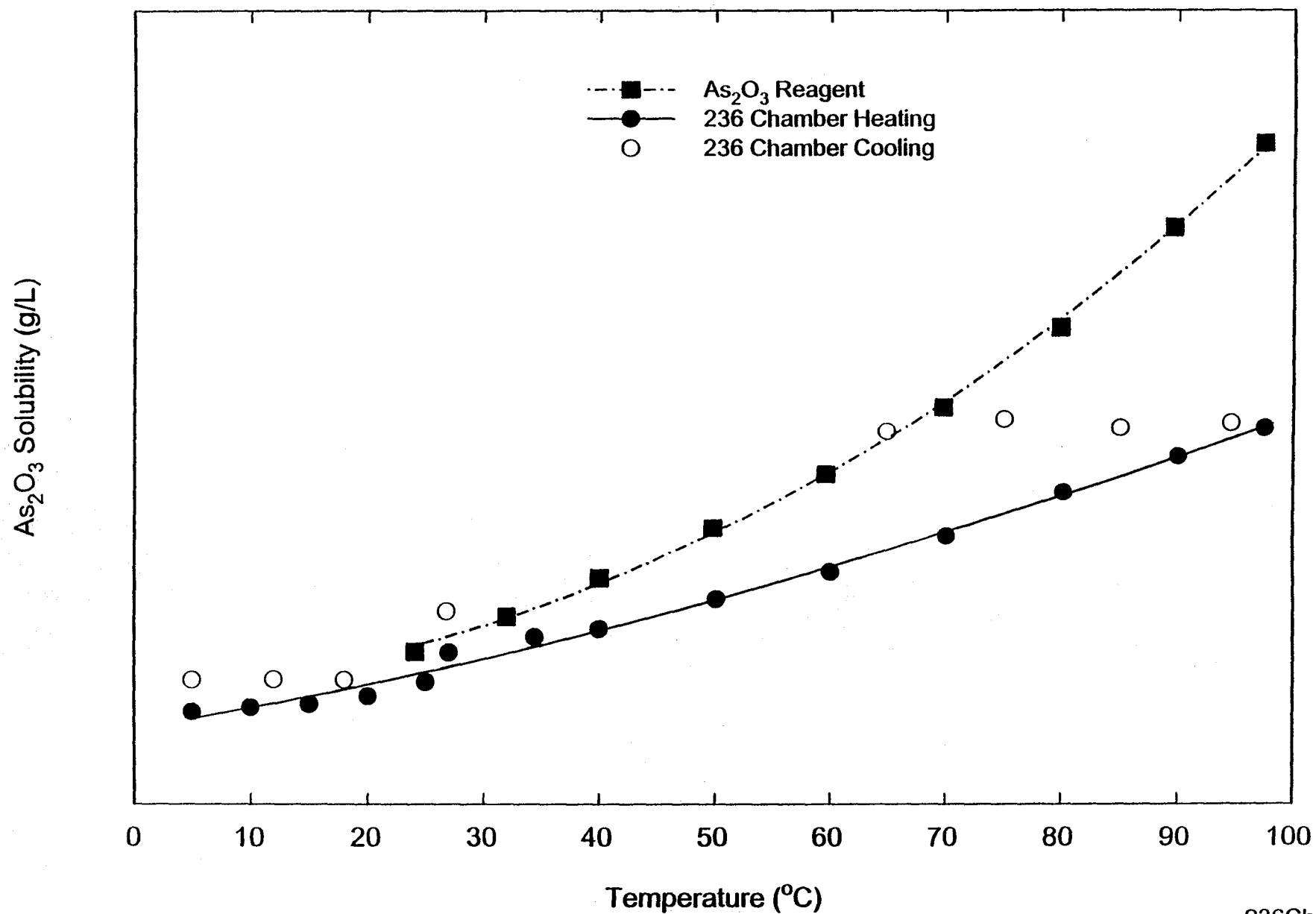
Element	Dust	Residue		Sublimate	
		analysis	partition	analysis	partition
	(%)	(%)	(%)	(%)	(%)
As	56.20	30.80	20	71.35	80
Sb	2.13	3.28	58	0.96	42
Au	10.99 ppm	42.90 ppm	100	<0.03 ppm	0

Dust 212, heated to 545°C for 10 hours in air.

Comparison of "April 97 Comp" Test Sample With Reagent Grade As_2O_3 Solubility



Comparison of "236 Chamber" Test Sample With Reagent Grade As_2O_3 Solubility



Comparison of "Jan 98 Comp" Test Sample With Reagent Grade As_2O_3 Solubility

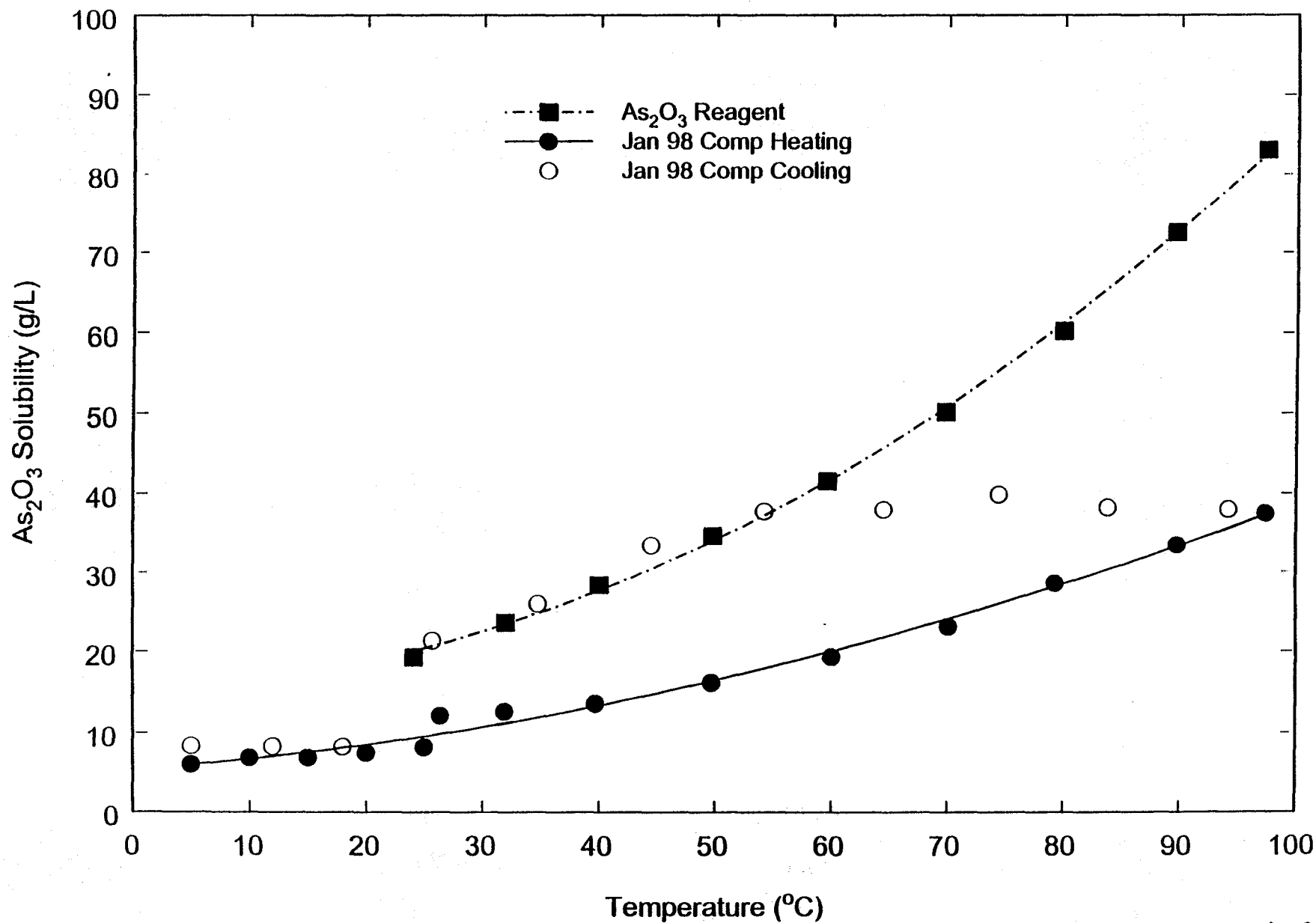


TABLE 4. Precipitation of Arsenic Trioxide from "Jan 98 Comp." Leach Solutions

Initial pulp density (% solids)	Arsenic concentration in hot solution (~95°C) g/L As	Arsenic concentration in cold solution (~ 25°C) g/L As	As ₂ O ₃ precipitate (dried at 110°C)			
			yield g/L	As ₂ O ₃ %	Sb %	Fe %
9.4	59.7	17.4	57.7	98.3	0.06	0.003
8.7	59.0	21.0	52.5	95.8	0.03	0.005
7.9	54.9	18.5	48.9	100.5	0.07	0.002
6.6	46.0	17.7	37.4	99.1	0.16	0.001
5.7	39.2	16.7	30.6	99.5	0.28	0.002
4.8	28.5	22.0	9.7	97.4	0.97	0.005

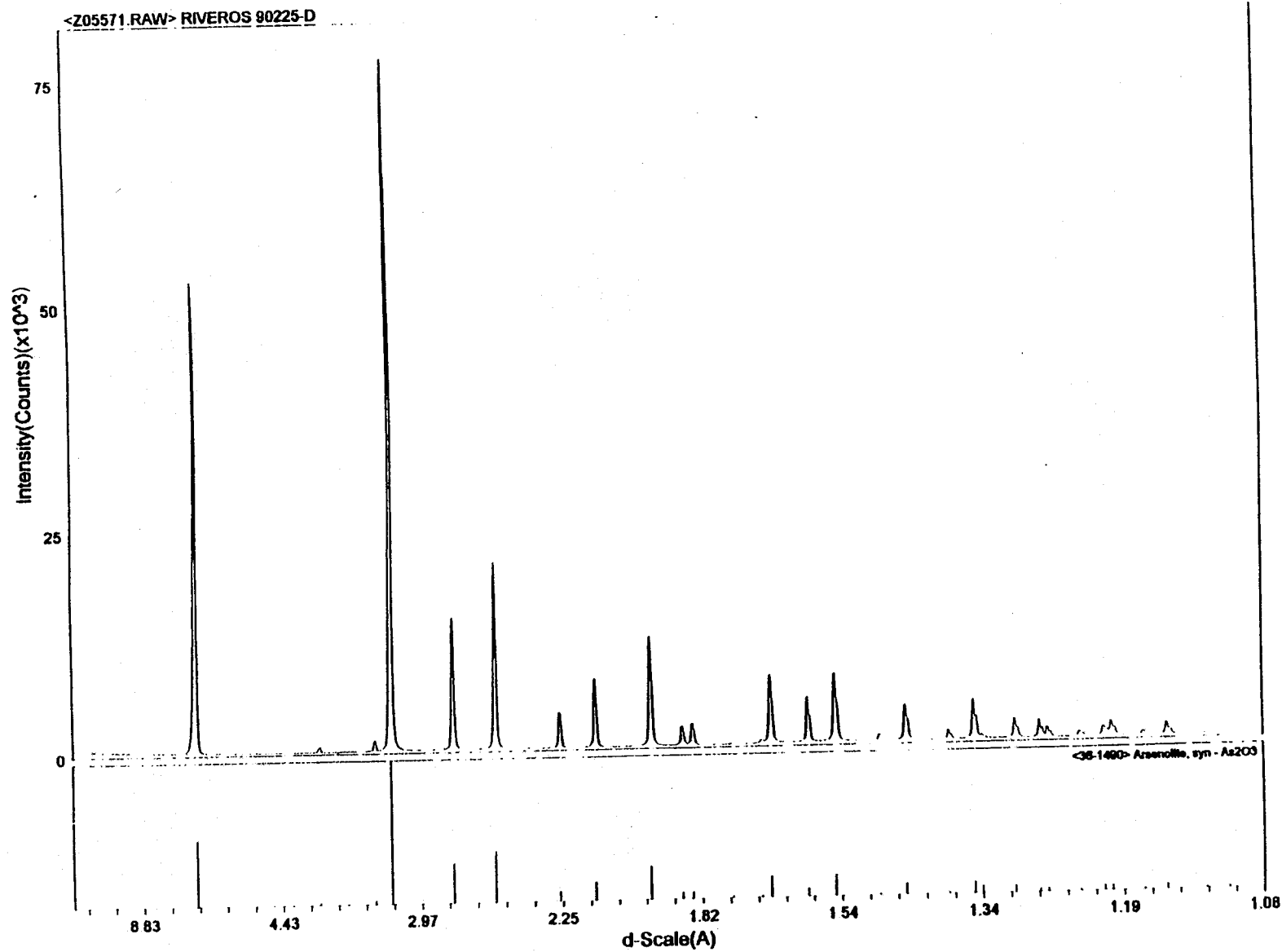


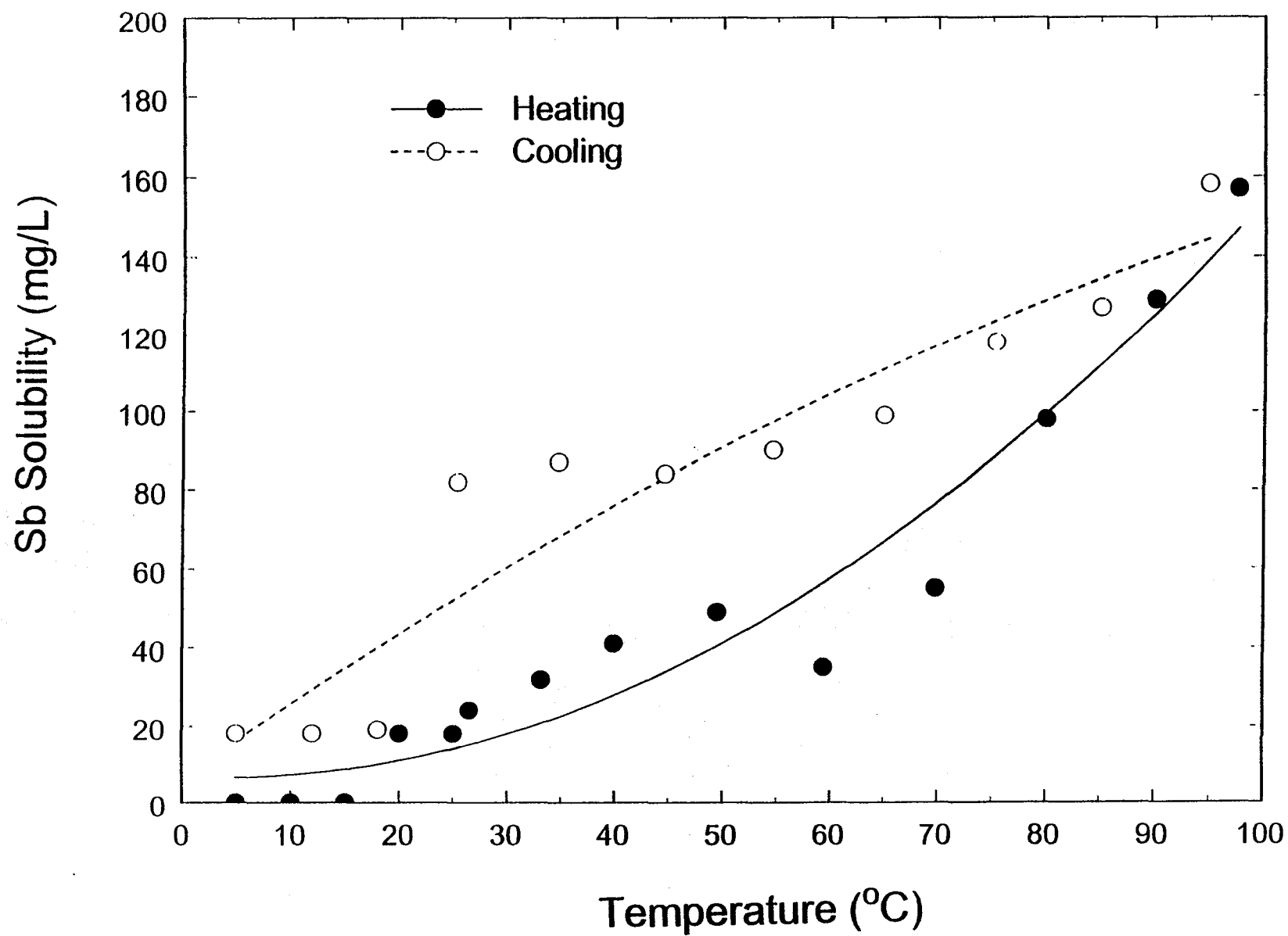
TABLE 5. Distribution of As after autoclave leaching (150°C, 2 h) and room temperature precipitation

Dust	Initial pulp density (% solids)	Distribution of arsenic		
		% As in residue	% As in solution	% As in As ₂ O ₃ product
Jan 98 Comp	9.4	7.3	25.3	67.4
Jan 98 Comp	7.9	1.8	30.9	67.3
Jan 98 Comp	4.8	1.0	64.8	34.2
Apr 97 Comp	7.8	2.9	32.7	64.3
Apr 97 Comp	7.1	2.6	32.8	64.6
Chamber 212	10.2	8.5	30.9	60.6
Chamber 212	9.1	7.5	28.1	64.4
Chamber 236	9.1	2.2	27.0	70.8
Chamber 236	8.3	1.5	30.8	67.6

TABLE 6. Deportment of Sb during the autoclave leaching of arsenic dust and precipitation of As_2O_3

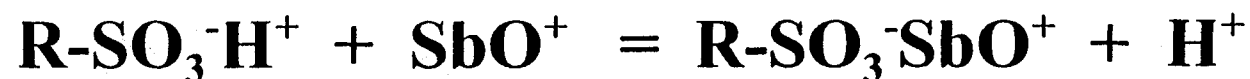
Arsenic dust	Initial pulp density % solids	Sb concentration in hot solution (~95 °C) mg/L	Sb concentration in cold solution (~25 °C) mg/L	Sb in As_2O_3 precipitate %
Jan 98 Comp	9.4	75	30	0.06
Jan 98 Comp	8.7	94	93	0.03
Jan 98 Comp	7.9	113	81	0.07
Jan 98 Comp	6.6	139	86	0.16
Jan 98 Comp	5.7	154	80	0.28
Jan 98 Comp	4.8	130	48	0.97
Apr 97 Comp	7.8	130	75	0.11
Chamber 212	10.2	191	181	0.06
Chamber 236	9.1	121	29	0.18

Solubility of Sb From 212 Chamber Sample in Water



Antimony has cationic chemistry, but only in the trivalent state. Cationic compounds of Sb(III) are mostly of the so-called “antimonyl” ion (SbO^+), which exists only in acid media.

Ion Exchange Extraction of Sb:



Antimony Removal Tests

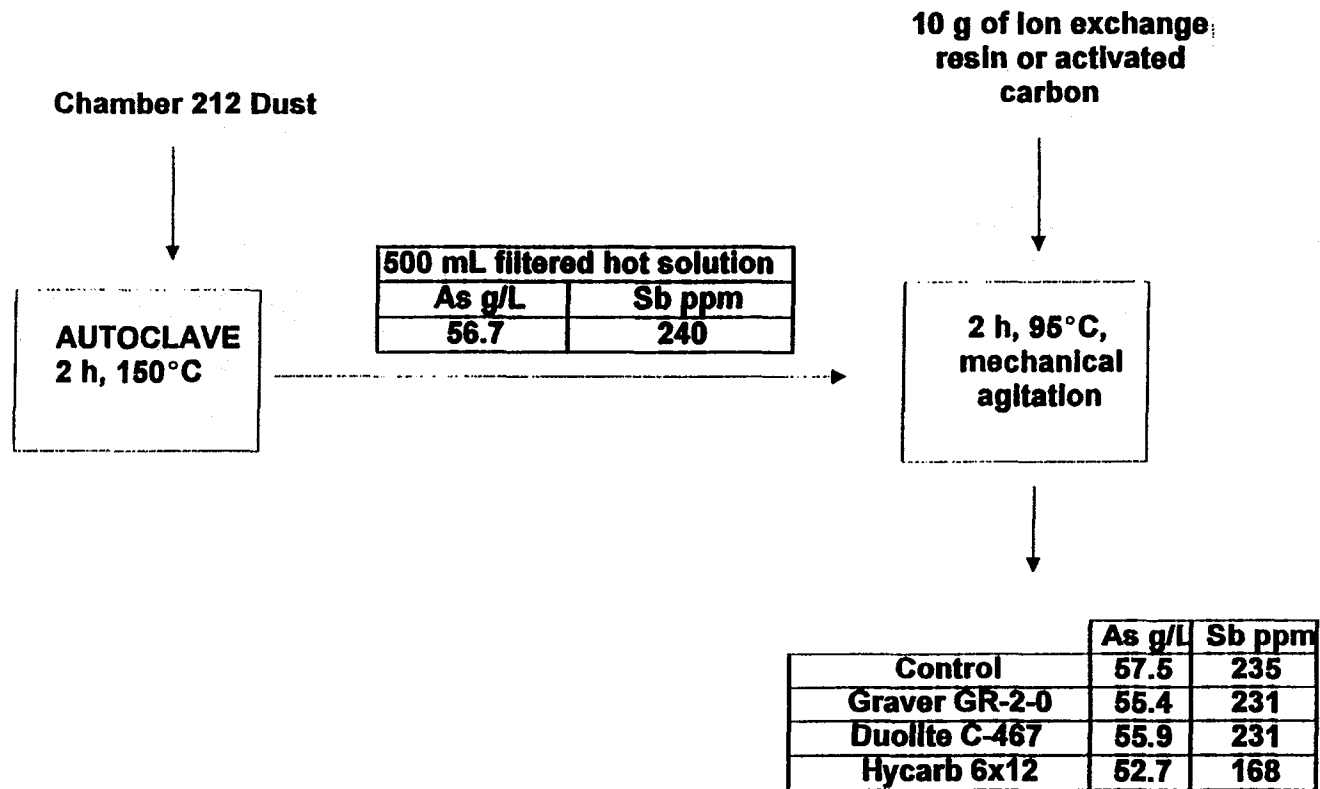


TABLE 7. Extraction of Sb by ion exchange at ~25°C. Solution: Chamber 212 dust leached at ambient pressure.

	pH	Final Sb concentration mg/L	% Sb extracted
Graver GR-2-0	3.0	24.6	0.0
Amberlite IRC-120	3.0	23.8	0.2
Amberlite IRC-50	3.0	23.2	2.1
Duolite C-467	3.0	23.6	0.5
Graver GR-2-0	1.0	22.9	11.9
Amberlite IRC-120	1.0	22.9	11.9
Amberlite IRC-50	1.0	22.6	13.1
Duolite C-467	1.0	2.2	91.7

SUMMARY

- ◆ Autoclave leaching at 125-200°C greatly improves the dissolution of the arsenic dust. The leaching of As from the dust was 97-99%.
- ◆ Gold is concentrated in the leach residue (up to ~40 ppm Au).
- ◆ Pure arsenic trioxide crystals (95-99% As_2O_3) were recovered from the leach solution.
- ◆ The contamination of the As_2O_3 product with Sb was relatively low (0.05-0.2%). If necessary, Sb could be removed from the leach solution by ion exchange, but only after acidification.

Future Work - Controlled Vapourization

- Can result in an up-graded product, but ~20% residue remains and the As_2O_3 has a high Sb content (~1%)
- Difficult to effect on commercial scale; may need a hot “filter” to separate dust from the As_2O_3 fume
- Propose to terminate these studies

Future Work - Leaching

- Use autoclave to solubilize As, cool to $<100^{\circ}\text{C}$, filter hot and cool to room temperature to collect pure As_2O_3
- Include solution recycle and optimize the process parameters; consider washing requirements and water balances
- Demonstrate gold recovery from the leach residues
- Stabilize residues after gold extraction
- Run leaching part of process continuously (separate Phase of study)

Future Work - Stabilization

- Stabilization requires arsenate (As_2O_5) and not arsenite As_2O_3
- Need to dissolve As_2O_3 and oxidize to As_2O_5
 - investigate use of air, O_2 , H_2O_2 , SO_2 -air
 - influence of As concentration, temperature, pH, oxidation potential and catalysts
 - use both fundamental techniques as well as practical condition
 - objectives are to define oxidation kinetics, identify critical process parameters and demonstrate process feasibility
 - Run preferred technique on a continuous basis to generate engineering data (separate Phase of study)

Future Work - Stabilization

- Study precipitation of iron arsenates from the oxidized solution
 - integrate scorodite ($\text{FeAsO}_4 \cdot 2\text{H}_2\text{O}$) and amorphous iron arsenate technologies
 - define effects of As concentration, acidity, temperature, neutralization agent and seed recycle
 - determine environmental stability of residues with respect to standard leachate tests
- Run preferred technique on a continuous basis to generate engineering data (separate Phase of study)

Future Work - Resources

Staff (per annum)

- 1 person full time on oxidation studies
- 1 person full time on leaching, and then stabilization
- 0.5 PY research scientist for support and immediate supervision
- 0.3 PY research scientist for mineralogical support
- 0.3 PY senior scientist involvement for project monitoring and liaison

Future Work - Resources

Schedule

- 2 year time frame for fundamental studies, parametric characterization and conceptual process development
- additional 1 year for continuous engineering studies and process definition

Comparison of "212 Chamber" Test Sample With Reagent Grade As_2O_3 Solubility

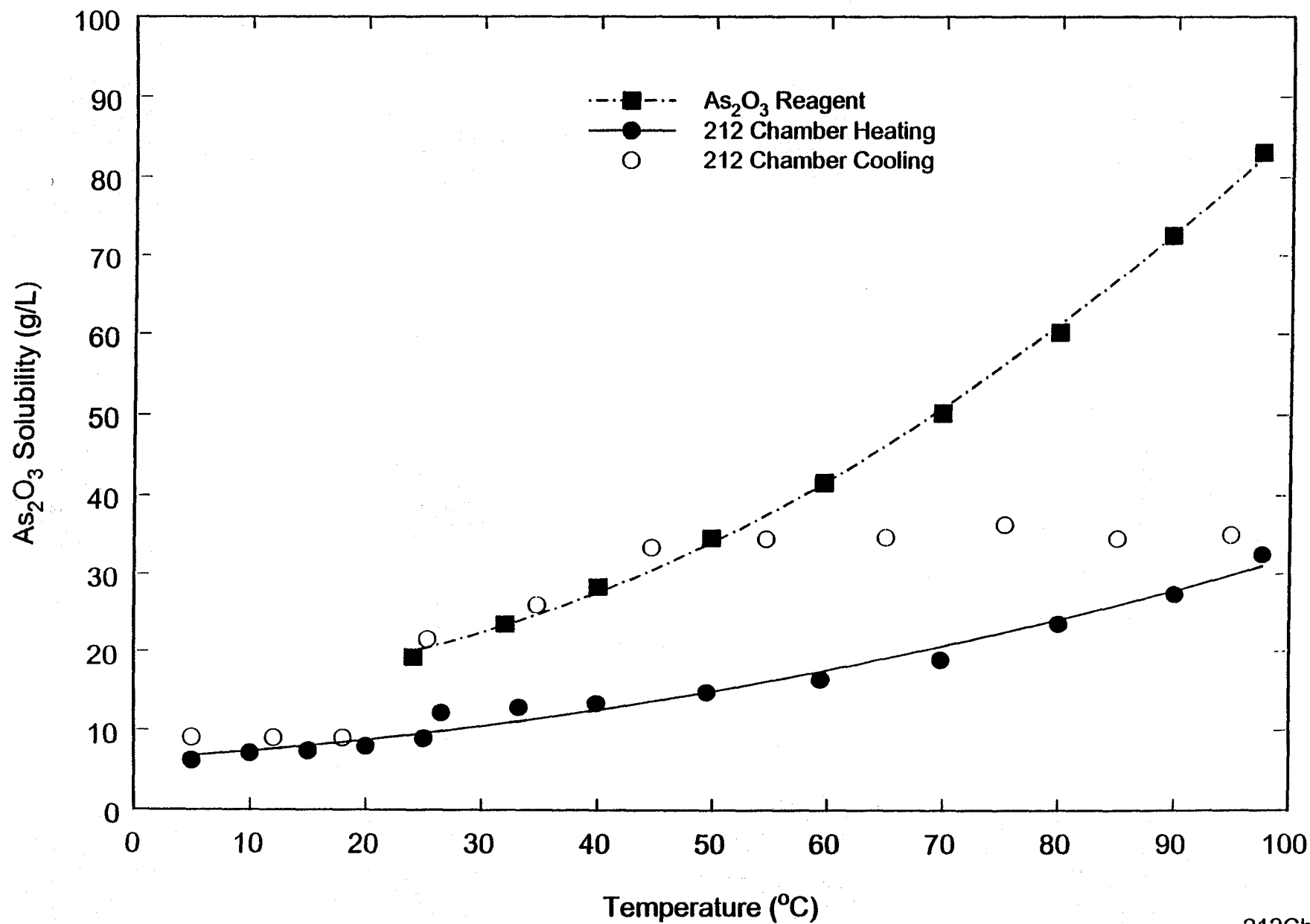


TABLE 1. Composition of 4 arsenic dust samples from Giant Mine

	As %	Sb %	Fe %	Al %	Mg %	Si %	Au ppm
Jan 98 Comp	68.5	0.9	1.2	0.6	0.3	1.1	2.2
Apr 97 Comp	68.2	1.5	0.8	0.3	0.1	0.6	2.4
Chamber 212	56.2	2.1	2.6	1.1	0.4	2.1	11.0
Chamber 236	58.0	0.3	1.7	0.9	0.4	1.8	5.1

TABLE 2. Arsenic solubility at 95°C after a 2-h pre-treatment in autoclave

Arsenic dust	Pre-treatment temperature (°C)	Arsenic in solution at 95°C	
		g/L As	g/L As ₂ O ₃
Chamber 212	125	51.1	67.4
“	150	61.5	81.2
“	175	61.1	80.6
“	200	60.8	80.2
Chamber 236	125	64.5	85.1
“	150	64.3	84.9
“	175	61.2	80.8
“	200	61.8	81.6
Reagent grade As ₂ O ₃	---	63.0	83.1

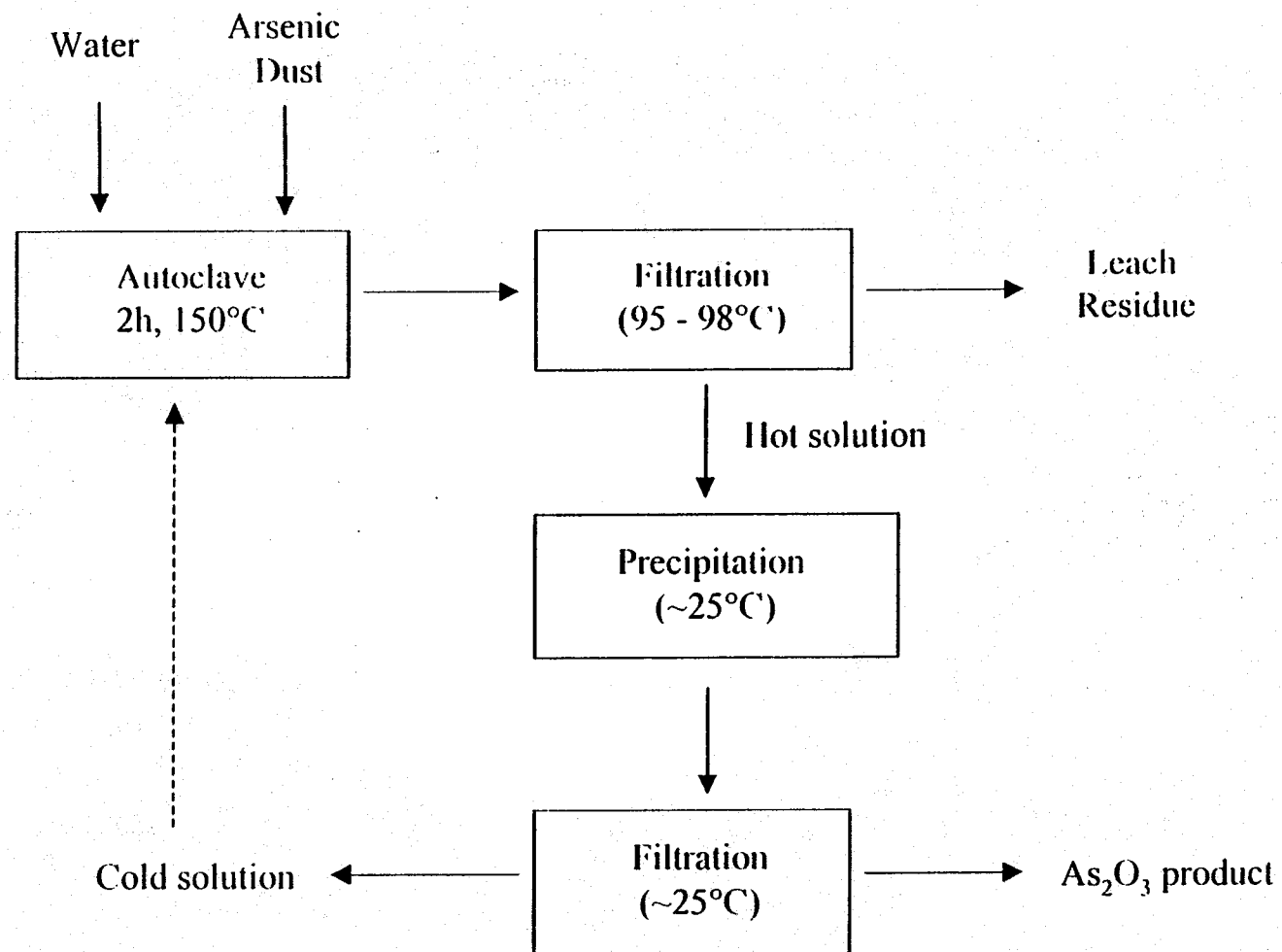


TABLE 3. Leaching of arsenic dust. Experimental conditions: 1 L water, heating in autoclave at 150°C for 2 hours, filtration at ~95°C.

Arsenic dust	Initial pulp density (% solids)	Percent of dust dissolved	Composition of the residue (dried at 110°C)			
			As %	Sb %	Fe %	Au ppm
Jan 98 Comp	9.4	85.7	34.5	3.3	10.1	25.9
Jan 98 Comp	8.7	88.5	23.7	6.4	12.4	37.6
Jan 98 Comp	7.9	91.2	14.5	3.8	15.8	40.1
Jan 98 Comp	6.6	91.3	14.1	4.7	16.2	39.2
Jan 98 Comp	5.7	91.2	12.4	5.0	16.3	40.1
Jan 98 Comp	4.8	93.0	9.4	3.0	17.1	43.4