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Flotation Process Response of Ok Tedi Fluorine Bearing Minerals

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ABSTRACT

Ore bodies at Ok Tedi contain a number of different fluorine bearing minerals. Some of these minerals report to concentrate and are responsible for the presence of the penalty element, fluorine, within the concentrate. Previous analytical work has tended to examine geological samples for content, rather than determine the metallurgical behaviour of the different mineralogical species. This investigation utilised X-Ray Diffraction combined with Scanning Electron Microscope / Electron Microprobe to identify the fluorine bearing minerals in flotation test products.

Seven fluorine bearing minerals were identified, viz., talc, phlogopite, amphibole (tremolite and actinolite), sphene, apatite, biotite and clay. Talc was found exclusively in the skarn ore type. Phlogopite and amphiboles (tremolite and actinolite) were found to occur in both skarn and porphyry ores, while sphene, apatite, biotite and clay were found only in the porphyry ores. Of the fluorine bearing minerals observed, only talc exhibited natural hydrophobicity to any significant degree. Phlogopite and the amphibole minerals were found to be hydrophilic, whilst the remaining minerals occurred in insufficient quantities to determine the flotation behaviour.

Ok Tedi copper concentrate fluorine content prior to skarn ore treatment in the mill (typically 350ppm) was previously identified as deriving from phlogopite, while talc was believed to be the source of intermittent high concentrate fluorine contents when skarn ores were treated. This paper provides supporting evidence for this belief, and reports the nature of fluorine bearing mineral flotation behaviour.

INTRODUCTION

Ok Tedi Mining Limited (OTML) mines and concentrates 600,000 tonnes of copper concentrate annually in the Star Mountains of Papua New Guinea's Western Province. The full descriptions of the mine and concentrator were presented by Stapleton, (1997) and England 1993.

The Ok Tedi deposit consists of a number of different ore bodies (Rush and Seegers, 1990). Geographically, as shown in Figure 1 (after Rush and Seegers, 1990), the deposit consists of two intrusive porphyry complexes, effectively ringed by skarn deposits. The larger of these complexes is called the Fubilan monzonite, whilst the other is called the Southern Porphyry. Skarn ore bodies associated with the intrusives include the Edinburgh, Gold Coast, Paris and the Deep Skarn. Skarn copper-gold mineralisation may be contained in massive magnetite, massive sulphide (mainly pyrite), calc-silicates or any combination of the three. Host rock siltstone contacting the intrusive may also be mined where mineralisation is of economic value.

Copper-gold concentrate consists predominantly of chalcopyrite, with sufficient digenite and bornite to render a 35% Cu concentrate grade readily attainable. Gangue contamination consists predominantly of pyrite, together with minor quantities of the ore body dependent on the host rock.

At the time of the Ok Tedi copper plant start-up (June 1987), fluorine was not considered a problem in the Ok Tedi copper concentrate, and indeed, was not assayed on a regular basis. It was not until after the introduction of skarn ores (which carry high gold and copper grades) to mill feed in June 1988 that the Japanese Smelter Pool (JSP) objected to a concentrate shipment fluorine content exceeding 1400ppm F (Shipment #16, 1455ppm). Previous shipments were estimated to have been between 300 - 400ppm F. Skarn ore treatment was discontinued for some time while research into reduction of fluorine levels was conducted.

In 1989, a large number of potential gangue depressants including guar gum, modified guar, dextrin, starch, lactic acid and synthetic polymers were evaluated. Best results were achieved with a specific CMC (carboxy methyl cellulose), Majol PSX, subsequently known as Finnifix BDA. As a CMC consumption of 800 g/t of mill feed was required in the flotation rougher stage to achieve adequate gangue depression, the CMC was added to the flotation cleaner stage at a consumption of 100 g/t of mill feed. Majol PSX was therefore added to the concentrator reagent suite for fluorine bearing mineral depression and two hourly assaying of final concentrate for fluorine instituted (Vanderdoes, 1990).

Current concentrator practice involves determining final concentrate MgO content by XRF every two hours and, using a regularly updated regression equation, estimating the fluorine content. If estimated fluorine content exceeds 900ppm for two consecutive readings (four hours), CMC is added to cleaner feed at 100 g/t (with respect to rougher feed). CMC use is discontinued when the fluorine content of the final concentrate drops below 600ppm. Above 900ppm, the use of CMC is cost effective in reducing penalty payments if it reduces fluorine content by 300 ppm. However, even whilst using CMC, two hourly fluorine content 'spikes' above 2000ppm are common. Hence, the best method of concentrate fluorine control currently available is feed blending. This involves rearranging of mill feed sources in the mine when the fluorine level in the concentrate increases. Skarn ore proportion in mill feed is currently 15% and rising.

The fluorine content of Ok Tedi copper concentrate varies typically from 400 - 900ppm in concentrates that are sent to various smelter customers. Although there is some variation in contractual specification, penalties are typically imposed in two steps (e.g., 350ppm and 850ppm) with a right of rejection (or negotiation) by the smelter at 1000 - 1500ppm.

FLUORINE

Fluorine is an undesirable element in copper smelter feed due largely to the contamination of by-product sulphuric acid (H₂SO₄) via the formation of a corrosive acid brine mixture of hydrofluoric acid (HF) and H₂SO₄ (Reist, 1992). This requires the addition of

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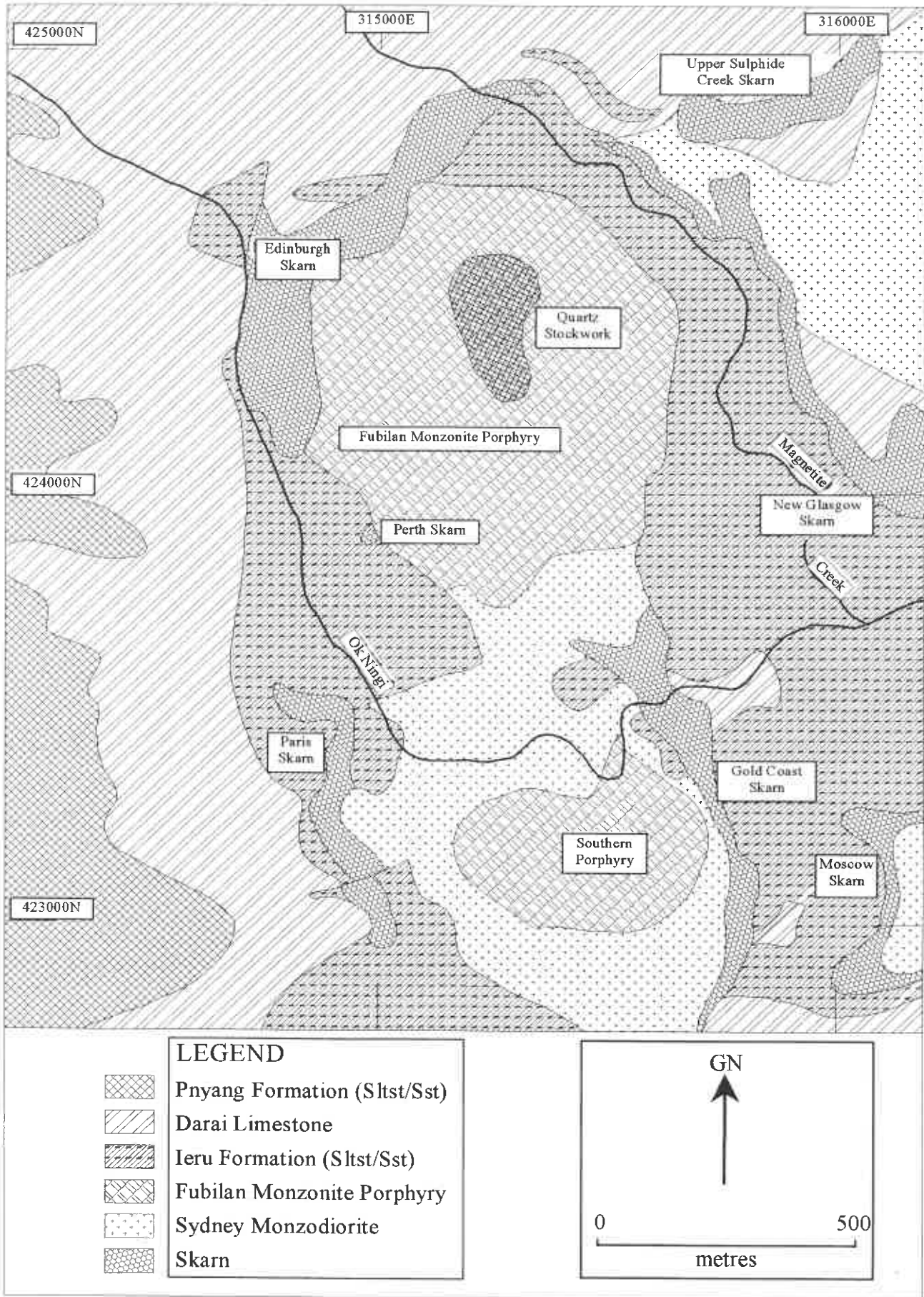


FIG 1 General Plan of Ok Tedi Orebodies (after Rush and Seegers, 1990).

sodium silicate ($\text{Na}_2\text{O-SiO}_2$) to fix and remove the fluorine as fluorosilicic acid (H_2SiF_6) from the brine mixture - an additional cost to the smelter. The corrosive acid brine may also cause accelerated corrosion of equipment in the acid plant.

Copper smelters therefore control fluorine input into their process by limiting the purchase of high fluorine bearing concentrates, by blending the high and low fluorine feeds (concentrates from different customers) and/or limiting concentrate suppliers to a specified concentration in their concentrates.

AIM OF THE WORK

The aim of the work presented in this paper was to examine the flotation process response of fluorine bearing minerals in Ok Tedi ores. This was done by performing flotation tests on a number of different ore types, and then examining the flotation products (concentrate and tailing) with both X-Ray Diffraction and Electron Microprobe to determine the presence of fluorine bearing minerals in both concentrate and tailing.

By counting sufficient particles and analysing the fluorine bearing particles for their fluorine content, an "assay" of the examined sample was calculated and compared with the chemical assay of the examined sample to demonstrate sufficient accounting for the fluorine in the sample.

Where sufficient particles of a particular fluorine bearing mineral were observed in both concentrate and tailing products, the flotation recovery of that fluorine bearing mineral was calculated. A determination was then made as to the natural hydrophobicity/hydrophilicity of the mineral by comparison of the particular mineral recovery with the total mass recovery.

Experimental

The flotation test program was conducted on samples of four different ore types. In each case, two samples were obtained of each ore type from two different locations. The samples were :

1. High talc bearing magnetite/sulphide skarn
2. High phlogopite bearing monzonite porphyry
3. Normal monzonite porphyry
4. Normal monzodiorite porphyry

Samples of each ore were dried and screened at 1.7mm. Screen oversize was reduced to less than 1.7mm by laboratory hammer mill and the two fractions combined, mixed and split into 2150gm sub-samples for subsequent flotation testwork. A preliminary grinding test used the method of Berry and Bruce (1966) to determine a relative work index for the ore and predict the grinding time for an OTML standard batch test with a grind p80 of 150 μm .

Ground slurry was transferred to a four litre flotation cell stirred with a Denver D-12 mechanism. Cell volume was made up with tap water. Flotation conditions consisted of pH adjustment with lime to pH = 11.5, followed by the addition of 20 g/t Cytec 7249 collector (a blend of diisobutyl (mono- and di-) -dithiophosphate) and Oreprep OTX-140 frother (mixed alcohols and glycols). The first concentrate was removed for 5 minutes, and a second concentrate for a subsequent five minutes. Concentrates and tailings were filtered, dried and sampled for elemental assay and microprobe examination. All elemental assays (Cu, Au & F) were provided by the OTML Mill Laboratory.

Microprobe Analysis

A total of 37 polished sections of flotation product grain mounts were analysed by microprobe at the Center for Microscopy and Microanalysis, University of Queensland, St. Lucia. The JEOL8020 Microprobe was typically operated at 15kV, with beam current of 15 nanoamps and a defocussed beam of 5.0 μm . Eleven elements (Si, Al, Fe, Mg, Ti, Ca, Mn, P, Na, K and F) were analysed by four automated spectrometers equipped with LiF, TAP, PETH and LDE crystals. The fluorine detection limit was approximately 620 ppm. Typically, a standard deviation of 0.32% (i.e. 10^5 counts) is sufficient precision for this work. Accuracy is 0.5-1.0%, relative to other instrumental factors and matrix correction errors.

Attempts to select probe points by light microscope before loading onto the probe for analysis were discouraged by the fine nature of the particles. Specific mineral analysis involved random selection of mineral grains using the SEM images, and entering the coordinates of the automated stage position of each selected particle. The number of elements analysed per particle was 11 and the number of particles per slide averaged 48.

Data analysis firstly involved identifying the individual minerals, e.g., feldspars, micas, talc, etc., followed by determination of the total quantity of each mineral. Measurement of particle size was obtained from the measurement of each probed particle. An average estimate of the particle size was thus obtained, and used to derive volume and weight percent figures. These percentiles were used to calculate the fluorine content of each fluorine bearing mineral. Summation of fluorine contents of fluorine bearing minerals was then used to estimate individual sample fluorine content. Sulphide mineral phases were identified by EDS (Energy Dispersive Spectrophotometer) and added to the total count of minerals of the particular thin section.

RESULTS

Flotation Tests

Although 35 flotation tests were conducted, only four (one from each ore type) are shown in Tables 1 through 4 for simplicity.

Examination of Tables 1 through 4 shows that fluorine is present in all samples with a minimal concentration of 1000ppm. Recoveries of fluorine are lower than the mass recoveries for the porphyry ores, but higher than the mass recoveries in the skarn ore, implying that greater amount of fluorine is associated with a naturally floating species in the skarn ore, but not in the porphyry ore.

The ratio of concentration for fluorine between concentrate and head in each test (not shown) provides further evidence for the presence of naturally floating species in the high talc bearing magnetite/sulphide skarn (Table 1). For this ore the ratio is significantly greater than one. Note that in all the other ores, the ratio of concentration approaches one. Interpretation of the flotation tests together with plant experience suggests that only the ore in Sample 1 provides a problem to the concentrator as cleaning processes in the concentrator lower the concentrate fluorine content of the other ores.

TABLE 1

Flotation of high talc bearing magnetite/sulphide skarn.

PRO-DUCT	MASS %	ASSAY			DISTRIBUTION		
		%Cu	Au(g/t)	F(ppm)	Cu	Au	F
Con 1	9.6	12.8	8.5	8916	74.2	74.6	16.7
Con 2	2.4	7.5	4.6	11578	10.9	10.1	5.4
Tail	88.0	0.28	0.19	4529	14.9	15.3	77.9
Head	100.0	1.66	1.09	5119	100	100	100

TABLE 2

Flotation of high phlogopite monzonite porphyry.

PRO-DUCT	MASS %	ASSAY			DISTRIBUTION		
		%Cu	Au(g/t)	F(ppm)	Cu	Au	F
Con 1	6.2	6.5	6.0	2823	85.4	57.4	7.7
Con 2	0.9	0.44	1.8	2933	0.8	2.5	1.2
Tail	92.9	0.07	0.28	2219	13.8	40.1	91.1
Head	100	0.47	0.65	2262	100	100	100

TABLE 3

Flotation of normal monzonite porphyry.

PRO-DUCT	MASS %	ASSAY			DISTRIBUTION		
		%Cu	Au(g/t)	F(ppm)	Cu	Au	F
Con 1	3.7	23.3	9.5	1014	88.2	63.2	3.4
Con 2	2.0	2.0	1.7	1357	4.1	6.0	2.5
Tail	94.3	0.08	0.19	1086	7.7	31.7	94.1
Head	100	0.98	0.56	1089	100	100	100

TABLE 4

Flotation of normal monzodiorite porphyry.

PRO-DUCT	MASS %	ASSAY			DISTRIBUTION		
		%Cu	Au(g/t)	F(ppm)	Cu	Au	F
Con 1	1.5	9.08	4.5	1160	71.1	54.3	1.7
Con 2	1.0	1.63	0.8	1265	8.5	6.4	1.2
Tail	97.5	0.04	0.05	1014	20.4	39.2	97.1
Head	100	0.19	0.12	1019	100	100	100

TABLE 5

Occurrence of fluorine bearing minerals in the four ore types (P = Present).

Ore type	Fluorine bearing						
	Talc	Phlog.	Amph.	Clay	Apatite	Biotite	Sphene
High talc skarn	P	P	P				
High phlogopite monzonite		P		P	P	P	
Normal monzonite		P		P	P	P	
Normal monzodiorite		P	P		P		P

TABLE 6

Distribution of high incidence fluorine bearing minerals between flotation products.

F bearing mineral	Ore type	Distribution to Product		
		Conc. 1	Conc. 2	Tailing
Talc	Skarn	18.9	6.1	75.0
Phlogopite	Skarn	5.2	0.8	94.0
"	High Phlogopite	3.8	0.6	95.6
"	Monzonite	2.1	1.0	96.8
"	Monzodiorite	1.3	0.7	97.9
Amphibole	Monzodiorite	0.4	0.3	99.3

Microprobe Analysis

Microprobe analysis identified seven fluorine bearing minerals; talc, phlogopite, amphibole (actinolite and tremolite), sphene, apatite, biotite and clay (illite and kaolinite). The presence of each fluorine bearing mineral in each ore type is shown in Table 5. Whilst phlogopite was observed in all ores, talc was limited to the skarn sample, sphene to the normal monzodiorite, and clay, apatite and biotite to the porphyry ores in general. The number of

analyses for each mineral were: talc (132), phlogopite (196), amphibole (43), clay (26), apatite (10), sphene (16) and biotite (13). Although Meinert (1995a) identified garnet and chlorite as also containing fluorine in the Ok Tedi orebodies, the current program did not detect either of these minerals. The major non-fluorine bearing phases identified were potassium feldspar, plagioclase feldspar, iron and copper sulphides, quartz, rutile, and magnetite. As previously described, the EDS component of the microprobe was employed to identify and quantify the sulphide

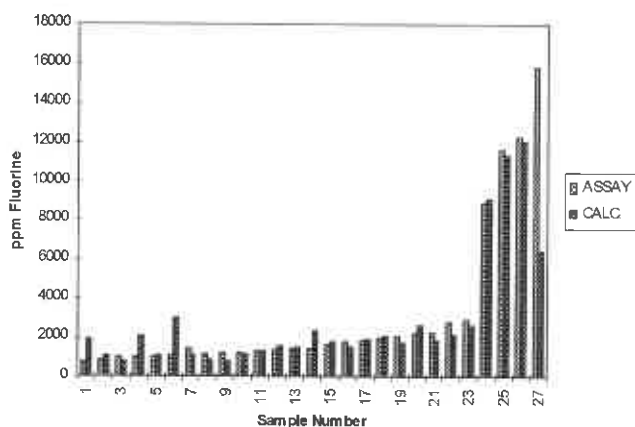


FIG 2 Comparison of Calculated and Assayed Fluorine.

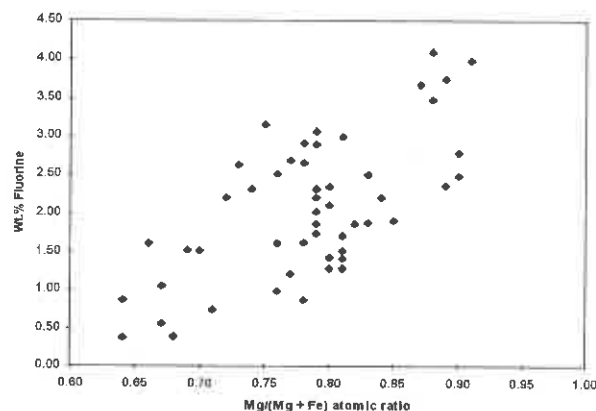


FIG 3 Fluorine content of phlogopite in normal monzonite.

phases and these were added to the total oxide and silicate phases to improve counting statistics.

Fluorine Balance.

Before interpreting the microprobe results in a quantitative manner, it is necessary to establish if all the fluorine in the samples has been accounted for by the microprobe. Figure 2 compares a calculated fluorine content, based on the proportion of fluorine bearing particles in each sample and the percentage of fluorine in the fluorine bearing particles, against the assayed fluorine content of the same sample.

It can be seen that the calculated and assay fluorine values for each product are in reasonable agreement. The exceptions are attributed to inaccuracies in the method, i.e., the low number of particles counted for true statistical significance. The general indication, however, is that majority of the fluorine bearing minerals in each product (concentrate or tailings) have been accounted for by the microprobe analysis.

DISCUSSION OF RESULTS

Observed fluorine bearing minerals

Talc - $Mg_3[Si_4O_{10}](OH,F)_2$

Talc distribution in the Gold Coast skarn was studied in detail by Maniger (1996). It was noted that talc was quite abundant near the contacts of the skarn with intrusive porphyry and siltstones, progressively decreasing towards the central part of the massive sulphide - magnetite skarn. It was also observed that where there is high volume of sulphides, disseminated talc occurs as a matrix to the sulphides.

Average fluorine content of the measured talc particles was $1.72 \pm 0.21\%$ at 95 % confidence with a range of 0.6 - 3.48%. Average talc magnesia (MgO) content was 28.2%. The large range of fluorine contents measured indicate the possibility of high fluorine talc and low fluorine talc occurrences in the skarn ore, supporting Maniger's (1996) observation that elevated assays of fluorine (up to 5000ppm) were incident in areas exposed to brecciation, shearing and faulting.

Talc is notably absent from the porphyry ores. XRD results on the skarn sample indicate that talc is the predominant silicate phase in the concentrate whilst chalcopyrite is the main sulphide mineral. Nearly 50% of the concentrate weight from Sample 1 is talc.

Table 6 shows the distribution of high incidence fluorine bearing minerals between the concentrate and tailing products of their respective samples. As with the distribution of fluorine between the flotation products of Sample 1, the recovery to each concentrate of talc in Sample 1 is significantly higher than the relative mass recovery, signifying a naturally floating mineral. It is worthy of note, however, that the total recovery of talc to concentrate is only 25%, which suggests that the talc is weakly hydrophobic.

Phlogopite - $KMg_3(Al,Si_3)O_{10}(OH,F)_2$

Phlogopite occurs in all four ore types tested, albeit in varying proportions. The average fluorine content at 95% confidence is $2.80 \pm 0.14\%$, which corresponds well with previous measurements by Doucette (1995), who reported an average of 2.81% fluorine and Meinert (1995b), who reported up to 4.8% fluorine in phlogopite. Interestingly, phlogopite in the skarn ore bears more fluorine than in the porphyry ores. Furthermore, in the porphyries, the rocks that suffered intense potassic alteration (Sample 2) bear significantly more fluorine than moderate or weakly potassic altered rocks (Samples 3 and 4), i.e.,

Sample 1: phlogopite in high talc bearing skarn	3.92 % F
Sample 2: phlogopite in high phlogopite bearing monzonite (intense potassic alteration)	3.46 % F
Sample 3: phlogopite in normal monzonite porphyry (weak potassic alteration)	2.94 % F
Sample 4: phlogopite in normal monzodiorite porphyry (weak potassic alteration)	2.53 % F

This was also noted by Mason (1993), who observed a significant increase in fluorine in micas from potassic altered rocks, ranging from 2% at low intensity up to 3 - 3.5% in medium to high intensities of potassic alteration.

Differentiation between biotite and phlogopite followed that of Mason (1993), who defined phlogopite as having a Mg/(Mg+Fe) ratio greater than 0.67. This ratio is simplified in a 'rule of

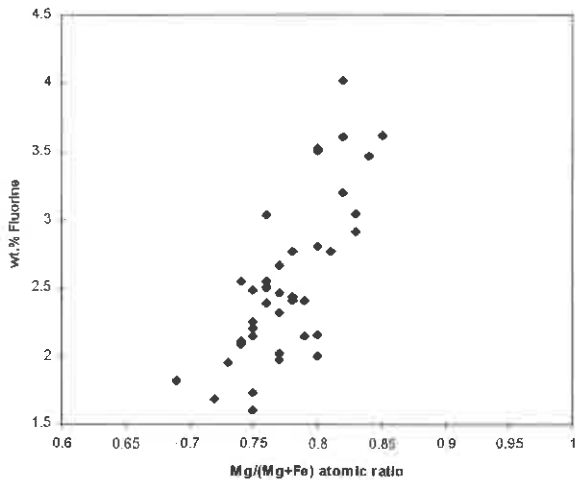


FIG 4 Fluorine content of phlogopite in monzodiorite.

thumb') to Mg:Fe = 2:1. Figures 3 to 6 depict the relationship between weight % F and Mg/(Mg + Fe) atomic ratio for the ore types tested. Phlogopite fluorine content is quite variable in normal monzonite porphyry (Figure 3) ranging from 0.5% to 4 %, whilst in normal monzodiorite (Figure 4), the range is narrowed between 1.5% to 4%.

The fluorine content of minerals in rocks that have suffered intense potassic alteration tend to be slightly higher, as seen in Figure 5 with fluorine contents ranging from 1.7 to 4.7%.

Finally, in Figure 6, it can be seen that the fluorine content of phlogopite sourced from the skarn sample ranges from 3 % to 6%. This could be due to an excess availability of magnesium (octahedral cation population) ions in the limestone units that underwent metamorphism/metasomatism to form the skarn bodies.

In all cases, it can be seen that the fluorine content increases with magnesium proportion in the phlogopite. This observation is in general agreement with the iron-fluorine avoidance rule of Munoz and Ludington (1974), i.e., that hydroxyl bearing ferromagnesium silicate minerals with high Mg/Fe ratios tend to incorporate more fluorine than comparable minerals with low Mg/Fe ratios.

Table 6 shows the distribution of phlogopite between the concentrate and tailing products for all four samples. In each case the recovery to concentrate is markedly lower than the mass recovery of the same product suggesting that phlogopite exhibits no hydrophobicity. Recovery to the concentrate is therefore by entrainment, and rejection can be achieved to a limited degree by cleaning stages in the concentrator.

Amphiboles : Actinolite - $Ca_2(Mg,Fe)_5Si_8O_{22}(OH)_2$ and Tremolite - $Ca_2Mg_5Si_8O_{22}(OH)_2$

The amphibole group of minerals (mainly tremolite and actinolite) occurs predominantly in the skarn and in monzodiorite, but is absent from the monzonite porphyry sample.

The fluorine content of the observed amphibole minerals averaged 0.5% F.

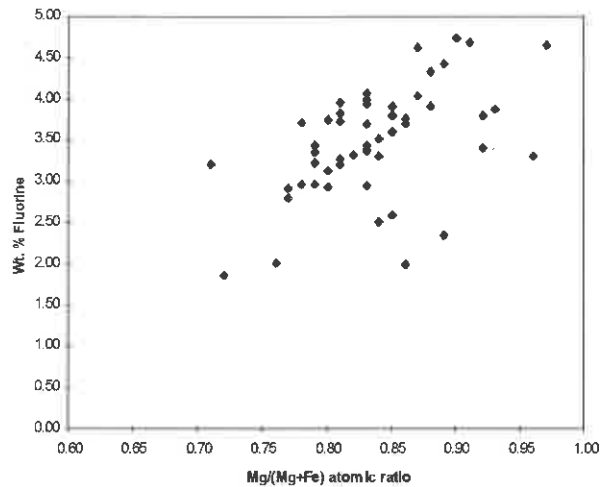


FIG 5 Fluorine content of phlogopite in intensely altered monzonite porphyry.

Sufficient amphibole minerals were only observed in the normal monzodiorite sample to allow determination of flotation behaviour. Amphibole distribution between the concentrate and tailing products of this sample is included in Table 6. As for the phlogopite, the recovery of amphibole to concentrate is significantly lower than the mass recovery, but to a greater degree than the phlogopite, suggesting that the amphiboles are more hydrophilic than the phlogopite. As with the phlogopite, recovery to the concentrate is therefore by entrainment, and rejection can be achieved to some degree by cleaning stages in the Concentrator.

Sphene - $CaTiO(SiO_4)$

Unaltered sphene was previously reported by Garry (1992) to be present in both the monzonite porphyry and within the monzodiorite.

16 analyses were made of sphene, exclusively sourced from monzodiorite porphyry exhibiting weak potassium feldspar alteration. The average fluorine content of sphene was determined as 0.466%F. 75 percent of the sphene observed was detected in the tailings with the balance in the first concentrate, suggesting that it is also a non-floating species. The contribution of sphene

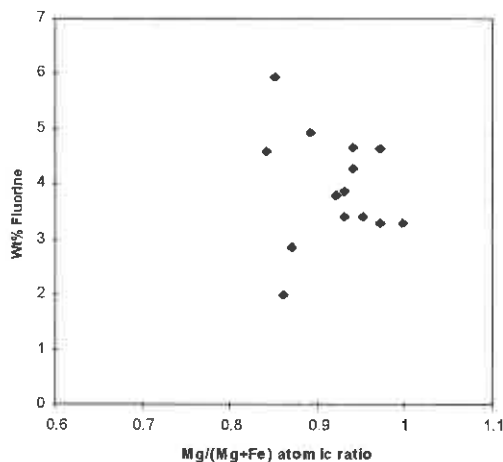


FIG 6 - Fluorine Content of phlogopite in Massive Magnetite Skarn.

to high concentrate fluorine content is insignificant due to both the low fluorine content of the mineral and the low proportion of the mineral in the orebodies. Insufficient sphene was observed to allow calculation of distribution between flotation products.

Biotite - $K(Mg,Fe)_3(Al,Si)_3O_{10}(OH,F)_2$

Biotite is a minor phase in the intrusive bearing an average of 1.2% F. It is notably absent from the high phlogopite bearing monzonite and monzodiorite porphyry, probably through alteration to phlogopite.

Similar to the sphene, insufficient biotite was observed to allow a significant calculation of distribution between flotation products. Biotite does not contribute significantly to high concentrate fluorine content due to the low proportion observed and the low fluorine content of the mineral.

Apatite - $Ca_5F(PO_4)_3$

Only eleven analyses of apatite were made, almost exclusively in monzonite porphyry. At an average fluorine content of 3.16 %F, it is the only mineral after talc and phlogopite that has a high fluorine content. Almost all apatite observed was relatively coarse (85µm), essentially 100% liberated, euhedral to subeuhedral grains. Notably, 91% of the apatite was detected in the tailings, even though the apatite content of the feed is 5% by weight. This agrees with the observations of Meinert (1995b), who also noted that despite the high fluorine content (3.5 - 4.8%) of apatite, the low proportion reporting to final concentrate limits the contribution to high concentrate fluorine content. Insufficient apatite was observed to allow a significant calculation of distribution between flotation products

Clay - Illite and Kaolinite

A total of 27 clay analyses were made, although not all clay bore fluorine, i.e. some had no fluorine or had levels below the detection limit (600ppm). XRD scans indicated illite and kaolinite to be the main fluorine bearing clay mineral phases. Of those clay minerals bearing fluorine, an average of 0.35% F was determined, with most detected in the monzonite porphyry. Mason (1993) also reported the detection of fluorine bearing clay in his work, with an average fluorine content of 0.38% F.

Although insufficient clay was observed to allow a significant calculation of distribution between flotation products, over 90% of observed clay particles were in concentrate samples. This suggests that clay is weakly hydrophobic, though significantly more observations would be needed for confirmation.

CONCLUSIONS

From this study, the following observations are made :

1. Seven fluorine bearing minerals were observed, viz., talc, phlogopite, biotite, amphiboles (actinolite, tremolite), sphene, apatite and clay (illite and kaolinite).
2. Fluorine bearing talc was only observed in the skarn sample and exhibited weak natural hydrophobicity.
3. Fluorine bearing phlogopite was present in significant quantities in all ores, but did not exhibit significant natural hydrophobicity.

4. Fluorine bearing amphiboles were present in the skarn and monzodiorite ores, but not in the monzonite ores. Where a significant concentration occurred in the monzodiorite ore, the amphiboles exhibited strong hydrophilic behaviour.
5. Fluorine bearing sphene was only observed in the monzodiorite sample. Insufficient quantities were observed to determine flotation behaviour.
6. Fluorine bearing biotite was only observed in the monzonite samples. Insufficient quantities were observed to determine flotation behaviour.
7. Fluoro-apatite was observed in all the porphyry samples. Insufficient quantities were observed to determine flotation behaviour.
8. Fluorine bearing clay was observed in the monzonite samples. Insufficient quantities were observed to determine flotation behaviour.

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