Mineralogical and Metallurgical Examination of Fluorosilicate Mineral Flotation in the Ok Tedi Circuit

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ABSTRACT

The Ok Tedi copper orebody consists of porphyry and skarn orebodies. The skarn orebodies, identified by different mineralogies, are the source of high intermittent fluorine levels in the mill concentrates. This paper discusses the results of the work undertaken to characterise the various fluorine - bearing minerals in samples of final copper concentrates and the distribution of fluorine amongst the minerals. Quantification of each mineral in mill feed and various flotation streams at Ok Tedi enables an understanding of the quantitative response of fluorine bearing minerals to flotation. The metallurgical behaviour of fluorine in the flotation process is also discussed.

INTRODUCTION

Ok Tedi Mining Limited (OTML) mines and concentrates 600 000 tonnes of copper concentrate annually in the Star Mountains of the Western Province of Papua New Guinea. Full descriptions of the mine and concentrator were presented by Stapelton and England (1993).

The Ok Tedi deposit consists of a number of different orebodies (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 orebodies associated with the orebodies include the Edinburgh, Gold Coast, Paris and the Deep skarn. Skarn copper 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.

Generally, silicate mineralogy affects sulphide ore processing in a number of ways. For example, magnesium-rich minerals in nickel ore influence nickel grade, recovery, smelter slag viscocity etc (Pietrobon et al, 1997; Wellham et al, 1992; Rhodes, 1979; Makarinsky, 1975; Oliveira et al, 1995). The minerals concerned are generally serpentines, eg lizardite, chrysotile, antigorite, chlorite and talc.

However, the impact of fluorine on copper sulphide ore processing was relatively unknown until the late-1980s when the Ok Tedi copper mine was faced with the possibility of rejection of high fluorine - bearing copper concentrate by Japanese smelters. As a result, Ok Tedi staff began studies aimed at identifying fluorine host mineralogy in the mine and developed control strategies in the mill to produce copper concentrates of acceptable fluorine concentration. It was also recognised that the level of co-operation between mine and mill production and technical personal on a daily basis was crucial to the management of the fluorine problem. This effort will continue to

form a significant part of the roles of the mine geology and mill metallurgy departments for the remainder of the mine life.

The fluorine problem at Ok Tedi is related to the development of skarn mineralisation in the ore deposit. In brief, the skarn ores formed as a consequence of the metasomatic replacement of sedimentary limestone and the igneous intrusive monzodiorite. The transfer of fluids from the intrusion into the sedimentary units, and the resulting chemical interactions between the different chemical elements, eg Ca and Mg from the limestone combining with silica from the intrusion, forming calcium silicates, etc resulted in the formation of the skarns. A number of mineralogically different skarns occur in the deposit. Endoskarns occur within the intrusives and are characterised by the assemblage of clay, orthoclase, scapolite, garnet, epidote, pyrite and chalcopyrite. Calc-silicate skarns are typified by the formation of diopsidic pyroxene and glosularite - andradite garnet with veinlets of chalcopyrite and pyrite. Massive magnetite skarns contain mainly granular magnetite with some tremolite, talc, diopside and veins or aggregates of chalcopyrite and pyrite. Massive sulphide skarns are composed of pyrite, pyrrhotite and some chalcopyrite (Davies et al, 1978). Fluorosilicate minerals in the above skarn ores have been reported in Pangum et al, (1997) and Mienert, (1995). Essentially, they are talc, epidote, clay, garnet, actinolite, tremolite and phlogopite. The porphyry rocks of monzonite and monzodiorite also contain fluorine bearing minerals namely biotite, phlogopite, clay and accessory sphene and apatite (Pangum et al, 1997). Hence, it can be seen that in a typical mill feed, where the usual practise is to blend skarn and porphyry ores, any combination of these various fluorosilicates can appear in mill feed. Their impact on final concentrate fluorine levels is entirely dependent on the flotation process response of each of these minerals, and is in essence the subject of this paper.

The fluorine content of Ok Tedi copper concentrates varies typically from 250 - 900 ppm in concentrates that are sent to various smelter customers. Although there is some variation in contractual specifications, penalties are typically imposed in two steps (eg 350 ppm and 850 ppm) with a right of rejection (or negotiation) by the smelter at 1000 - 1500 ppm (Pangum et al, 1997).

Fluorine is an undesirable element in copper smelter feed due largely to the contamination of by-product sulphuric acid (H_2SO_4) via the formation of a corrosive acid brine mixture of hydrofluoric acid (HF) and H_2SO_4 (Reist, 1992). This contamination requires the addition of sodium silicate (Na_2O-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.

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

Much of the current understanding of the fluorine problem at Ok Tedi has been developed from in-house studies and external consultancy work. Hence, information available in the public domain for the benefit of the wider scientific community is

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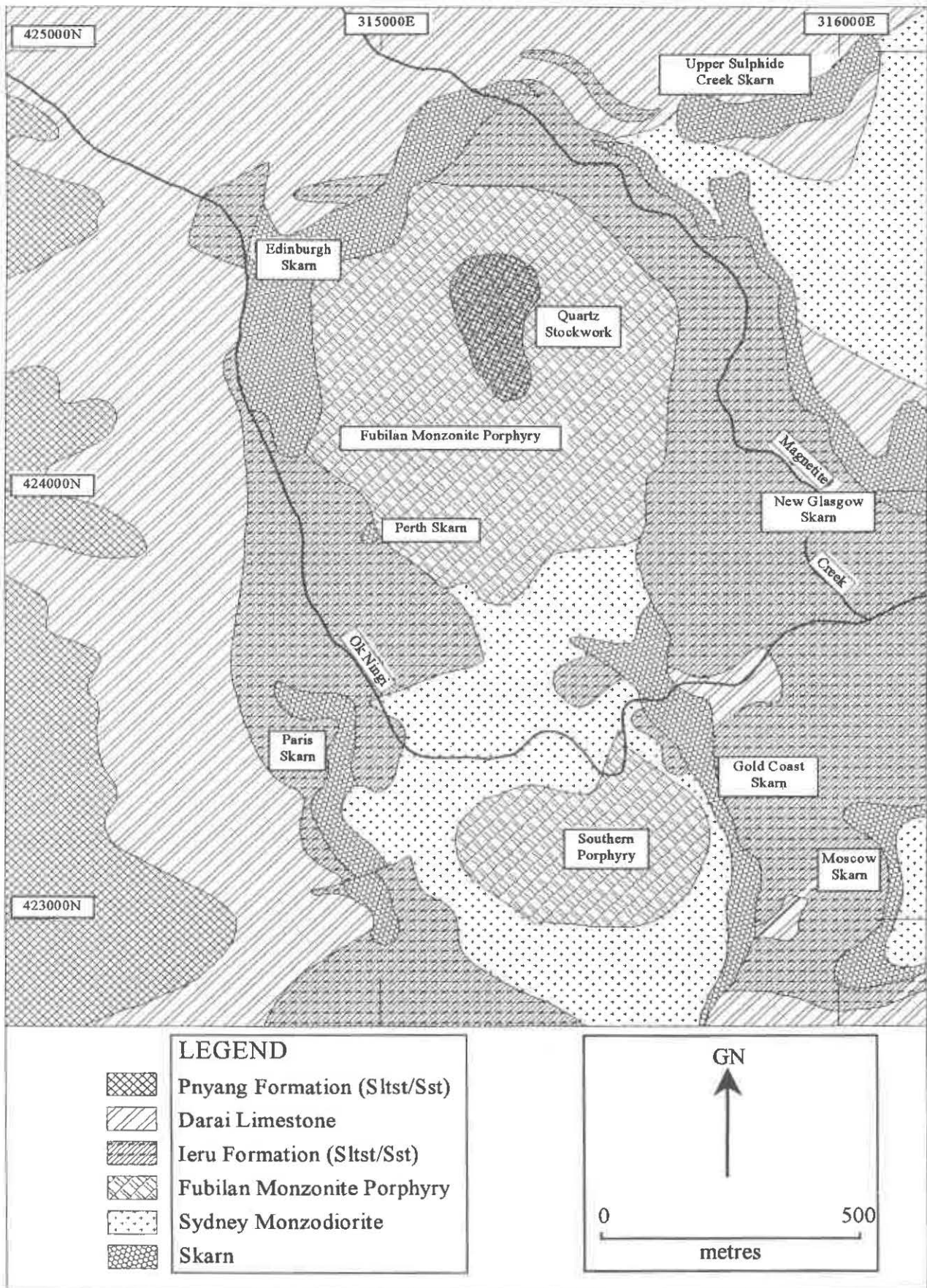
FIG 1 - General plan of Ok Tedi orebodies (after Rush and Seegers, 1990).

almost non-existent. The literature survey undertaken in the course of this study has so far failed to identify any other site elsewhere, with a similar fluorine problem.

EXPERIMENTAL

Characterisation of fluorine bearing mineralogy

The samples used in this study were sourced from nine monthly composites of final copper concentrate from the Ok Tedi concentrator. The composited samples are from the normal Ok Tedi Mill Laboratory routine sampling program. Polished grain mounts of these samples were analysed on the microprobe. 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 respective sample to demonstrate sufficient accounting of the fluorine in the sample.

Fluorine behaviour in the Ok Tedi flotation circuit

The metallurgical aspects of fluorine in the current flotation circuit were studied in a survey conducted on 20 March 1997. The nine major streams in the flotation circuit were sampled and these were subjected to typically wet and dry mass determinations, chemical assay and size by size analysis of each stream. The mass balance of the circuit was performed using the MBAL routine of the JKSIMMET software package.

Microprobe analysis

The samples used in this study were sourced from the survey taken on 20 March 97 and are the same samples used above. The quantitative assays of fluorine in the individual silicate minerals was carried out with the electron microprobe at the Centre for Microscopy and Microanalysis, The University of Queensland, St Lucia. The JEOL 8088 microprobe was typically operated at 15 kV, with beam current of 15 nanoamps and a defocused beam of 5.0 microns. Eleven elements (Si, Al, Fe, Ti, Mg, Ca, Mn, P, Na, K and F) were analysed by four automated spectrometers equiped with LiF, TAP, PET and LDE crystals. The fluorine detection limit for a single analysis is approximately 620 ppm. Typically, a standard deviation of 0.32 per cent (ie 105 counts) is sufficient precision for this work. Accuracy of the data collected is 0.5 - 1.0 per cent relative to other instrumental factors and matrix correction factors.

Attempts to select analysis points by optical microscopy before loading on to the microprobe were made difficult by the fine size of the particles. Mineral analysis involved random selection of mineral grains using the SEM images with a record of coordinates for the stage position of each selected particle. This approach allowed confirmation of mineralogy by SEM methods at a later time if necessary. Eleven elements were analysed per cycle on 50 particles per polished slide.

Data analysis firstly involved identifying the individual minerals (eg feldspar, mica and talc) followed by the determination of the total sum of each mineral phase. Particle size was obtained from the measurement of an SEM image of each probed particle. An averaged particle size was thus derived and used to compute volume and weight percent values. These values were used to calculate the fluorine content of each fluorosilicate mineral according to Equation 1:

$$F \text{ ppm} = \text{Mass \%} \times \text{Unit F content (\%)} \times 100 \qquad (1)$$

Summation of the fluorine contents for these minerals was then used to estimate the fluorine content of individual samples. Sulphide mineral phases were identified by EDS (Energy Dispersive Spectrophotometer) and added to the total mineral count of each particular thin section.

Quantitative XRD

Quantification of major mineral phases and, in particular, the fluorosilicate minerals, is necessary in order to define the response to flotation, ie recovery. This approach was made possible by the use of SIROQUANT computer software, developed by the CSIRO. Basically, the method involves a pattern fitting exercise whereby powder data of a multi-phase sample is generated by XRD. A model describing the material in terms of phase composition factors, unit cell contents and line shape character is used to simulate the diffraction pattern and the model is adjusted until the calculated pattern matches the measured pattern. The measured intensity is directly proportional to the calculated intensity and the proportionality constant is the phase scale factor converted to phase concentration. The samples of this study were the same as those used in above.

RESULTS AND DISCUSSION

Characterisation of fluorine bearing mineralogy

Of all the fluorosilicate minerals identified in the Ok Tedi orebody (Pangum *et al*, 1997), only talc, phlogopite, biotite, clay and amphiboles (actinolite) were detected in the monthly composite samples. Figure 2 shows the comparison between the bulk chemical assay obtained by the OTML Chemistry Laboratory and the microprobe 'assay' as described in section on microprobe analysis. There is reasonable agreement between the two assays, demonstrating sufficient accounting of all fluorine bearing minerals in the sample.

Months of high fluorine (January, July and November) are due to high talc content (4.7 wt per cent in July) and phlogopite with high abundance of fluorine (January and November) at 4.2 per cent F average. The months of high fluorine, however, do not translate to high skarn ore in feed as noted by Lauder (1997). In July 1996, skarn ore constituted 8.2 per cent of mill feed, but a high fluorine (839 ppm) concentrate was produced whereas in November 1996, skarn ore made up 19.7 per cent of mill feed and fluorine in concentrate was 748 ppm. Furthermore in May 1996, skarn ore comprised 12.4 per cent mill feed, but the fluorine content in the final concentrate was 574 ppm. This variation can be attributed to various factors including natural variation in fluorine content of talc and phlogopite from different areas in the orebody, variation in talc and phlogopite proportions in different skarns, and within each skarn type, eg massive sulphide, massive magnetite and/or operational variations in the cleaning circuit.

The quantities of fluorosilicate minerals identified in the composite samples with their respective fluorine contents are listed in Table 1. Using these data, the fluorine contribution by each fluorosilicate to final concentrate fluorine was calculated according to Equation 1 and tabulated in Table 2. In all calculations, the mass percentage of each mineral has been

TABLE 1

Mineral	Fluorine (%)	Mass % of fluorosilicate in final concentrate		
		Minimun	Average	Maximum
Talc	1.75	0.8	2.3	4.7
phlogopite	3.1	0.2	0.45	0.64
biotite	1.36	0.2	0.38	0.6
clay	0.32	0.2	0.51	1.3
amphibole	0.52	0.2	0.36	0.5

Mineral	Flourine (ppm)		
	Minimum	Average	Maximum
Talc	140	399	822
phlogopite	62	136	198
biotite	27	51	82
clay	6	16	42
amphibole	10	19	26
Total	245	621	1170

varied while the per cent F was kept constant. This has been done to highlight the fact that fluorosilicate response to flotation is not dependent on fluorine abundance but is entirely dependent on the efficiency of the flotation circuit to maximise rejection of fluorosilicate minerals.

On average, talc is the major contributor (64 per cent) to the fluorine content, by virtue of its relatively high mass percent and high unit fluorine content. It's natural hydrophibic character enables it to float unaided, resulting in such relatively high amounts in the concentrate. Phlogopite accounts for 22 per cent (average) of the fluorine and the balance is spread amongst the other minerals.

Quantitative XRD

Quantitative X-ray diffraction studies were undertaken in order to identify the full suite of minerals in each sample and then to quantify the process response of fluoro-silicate minerals.

Quantitative X-ray methods suffer from the lack of X-ray intensity generated from mineral phases that occur in trace or minor amounts (<1 per cent). This low signal to noise ratio affects the accuracy of data interpretation so other techniques are employed to improve or correct the data. In this work, the quantitative XRD data has been compared with that derived from empirical calculations using the microprobe data. Generally, both methods give comparable mass percent values for the major phases. However, the minor phases (<1 per cent) cannot be adequately determined.

The results of the quantitative XRD work on the plant survey samples are summarised in Table 3.

In Table 3, mass of talc in final concentrate is 4.2 per cent which is in agreement with the previously reported value of 4.7 per cent calculated from microprobe data. This mass represents a 3.3 per cent talc recovery into final concentrate. For phlogopite, due to an extremely low abundance, insufficient X-ray counts were registered to adequately derive a quantitative figure. The amount reported in Table 3 (0.6 per cent) has been determined from microprobe data and equates to 0.15 per cent recovery into final concentrate. In terms of fluorine accountability, on the basis of the above numbers including clay (illite), the calculated fluorine amounts to 783 ppm which agrees with the bulk assay of 776 ppm F.

Chalcopyrite and digenite recoveries are close to 90 per cent. Feed and concentrate Cu is calculated at 0.62 per cent and 36.05 per cent Cu which are in reasonable agreement with bulk assays of 0.8 per cent and 39 per cent Cu respectively. The difference is due to minor amounts in bornite and other Cu minerals.

A summary of mineralogical analysis obtained by three different methods are given in Table 4. This comparison indicates that Siroquant data are similar to typical Ok Tedi final copper concentrate mineralogical break-up data (Lauder, 1996) and to that obtained by microprobe (Pangum *et al*, 1997).
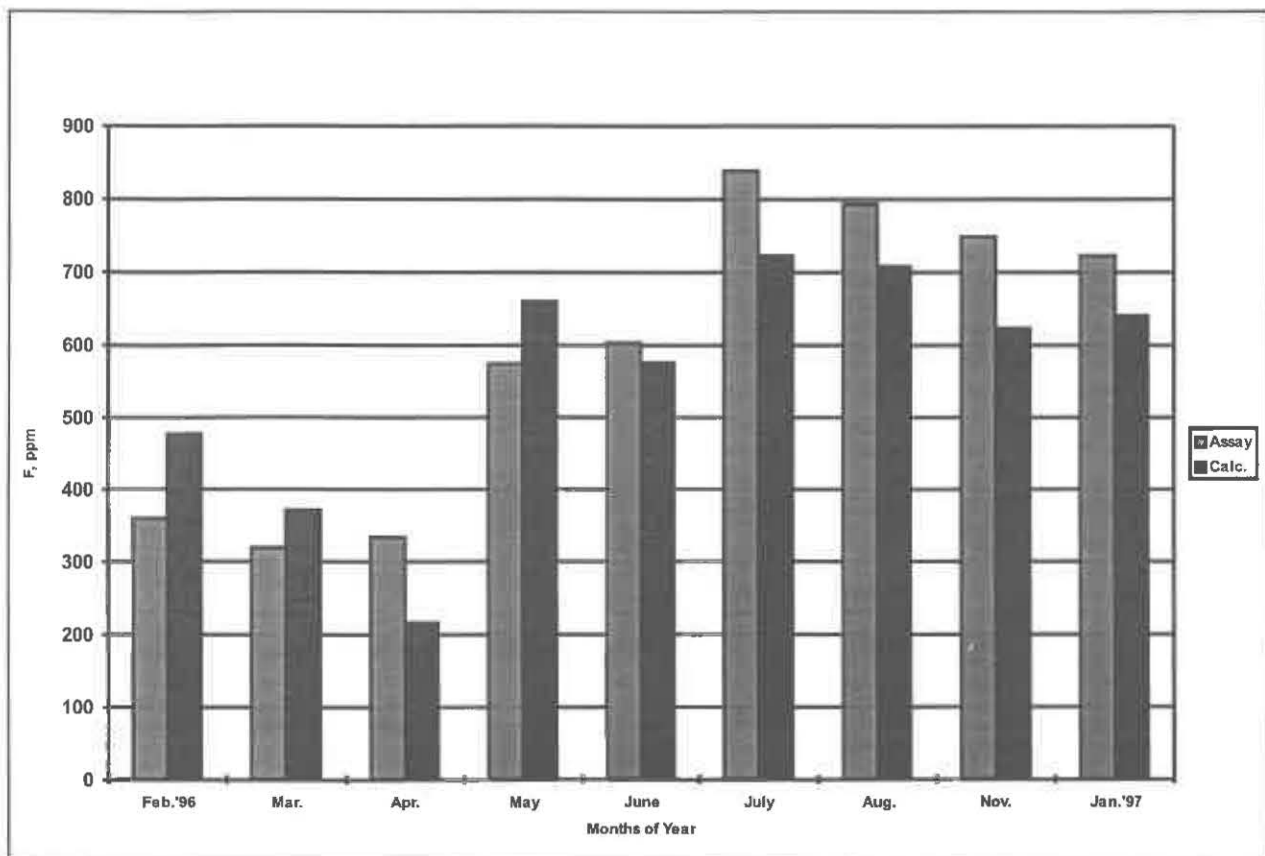

FIG 2 - Comparison of calculated and assay fluorine.

TABLE 3

Quantitive XRD data - mass % of phases present.

Stream	Feldspar	Quartz	Magnetite	Talc	Phlogopite	Illite	CuFeS₂	FeS₂	Cu1.8S	Other	Totals
New feed	63.4	11.6	11.7	2.0	6.5	1.1	1.1	2	0.3	0.3	100.0
Recyc feed	60.6	9.7	7.6	6.8	3.6	1.8	0.7	4.8	0.7	3.7	100.0
Cell 1- 3 Con	10.0	0.7	0.0	10.7	0.2	0.3	60.7	11.2	5.0	1.2	100.0
Cell 1 - 3 Tail	68.9	12.6	9.1	1.0	3.3	0.0	0.2	2.4	1.7	0.8	100.0
Cell 4 - 9 Con	34.7	6.3	7.4	7.2	3.1	0.0	20.9	12.4	6.5	1.5	100.0
Cell 4 - 9 Tail	67.9	11.7	8.6	4.2	2.5	1.3	0.2	1.4	1.7	0.5	100.0
Cell 10 - 15 Con	55.5	11.3	6.3	10.2	2.2	1.8	1.6	7.6	2.5	1.0	100.0
Cell 10 - 15 Tail	69.4	12.8	7.1	2.4	3.9	0.4	0.3	1.2	1.6	0.9	100.0
Clnr Tail	64.8	10.2	6.2	4.4	3.0	0.2	0.2	8.8	1.4	0.8	100.0
Final Conc	6.6	0.0	0.0	4.2	0.6	0.4	65	5.1	17	1.1	100.0
Mass % Rec	0.16	0.0	0.0	3.3	0.15	0.6	93.1	4.0	89.3	5.8	1.6

TABLE 4

Mineralogical composition of Ok Tedi copper concentrate (%).

Mineral	Siroquant	Lauder, 96	Pangum, 97
CuFeS₂	65	69.6	68.3
Digenite	16.1	17	15.9
Bornite	n/a	0.7	n/a
FeS₂	7	5.1	6.2
Silicates	12	7.6	9.6

Overall, the use of an XRD facility, combined with quantitative software for phase analysis can provide a quick and reliable method of providing talc and phlogopite (amongst other minerals) concentrations (mass per cent) in any batch of ore. This information can then be combined with other relevant available information, eg talc and phlogopite recoveries, respective fluorine contents, other fluorosilicate minerals behaviour during flotation, plant experience, etc to predict final concentrate fluorine content.

Metallurgical aspects

Briefly, the flotation properties of talc are well documented (Laskowski and Poling, 1995; Oliveira and Gomes, 1991; Fuerstenau *et al*, 1988 and Michot *et al*, 1994). It is essentially a naturally hydrophobic mineral. Apart from talc and other naturally hydrophobic minerals like graphite and sulphur, most silicate minerals, eg phlogopite are readily wetted by water and are therefore regarded as hydrophilic (Manser, 1967). Such minerals, therefore are recovered into, eg copper concentrates by entrainment (Smith and Warren, 1985).

Stage by stage recoveries of talc, phlogopite and fluorine are shown in Figure 3. It indicates an increasing trend down the flotation bank, with the maximum recoveries occuring in the scavenger banks. Fluorine recovery also appears to be sympathetic to talc recovery.

This suggests that fluorine recovery is a function of fluorosilicate mineral recovery. In fact, fluorine recovery is in direct proportion to talc recovery as depicted in Figure 4.

Cells 4 - 9 concentrate 80 per cent passing size (P80) is 180 microns whilst the P80's of cells 1 - 3 and 10 - 15 concentrates are 90 and 70 microns respectively. The anomaly in cells 4 - 9 could reflect a size effect in that coarser talc would float slower than finer talc.

The mass and material recoveries and circuit circulation loads are shown on Table 5. The stage recovery of fluorine varies between 1 to 1.5 per cent in the roughers to ten per cent in the scavengers, whilst the overall plant recovery is 0.6 per cent. This observation is in agreement with previous observations (Wawako, 1994). Water recovery in the roughers is 1.4 per cent indicating that fluorosilicate mineral flotation would be impacted more by natural flotation than entrainment.

TABLE 5

Mass and material recoveries and stream circulation loads.

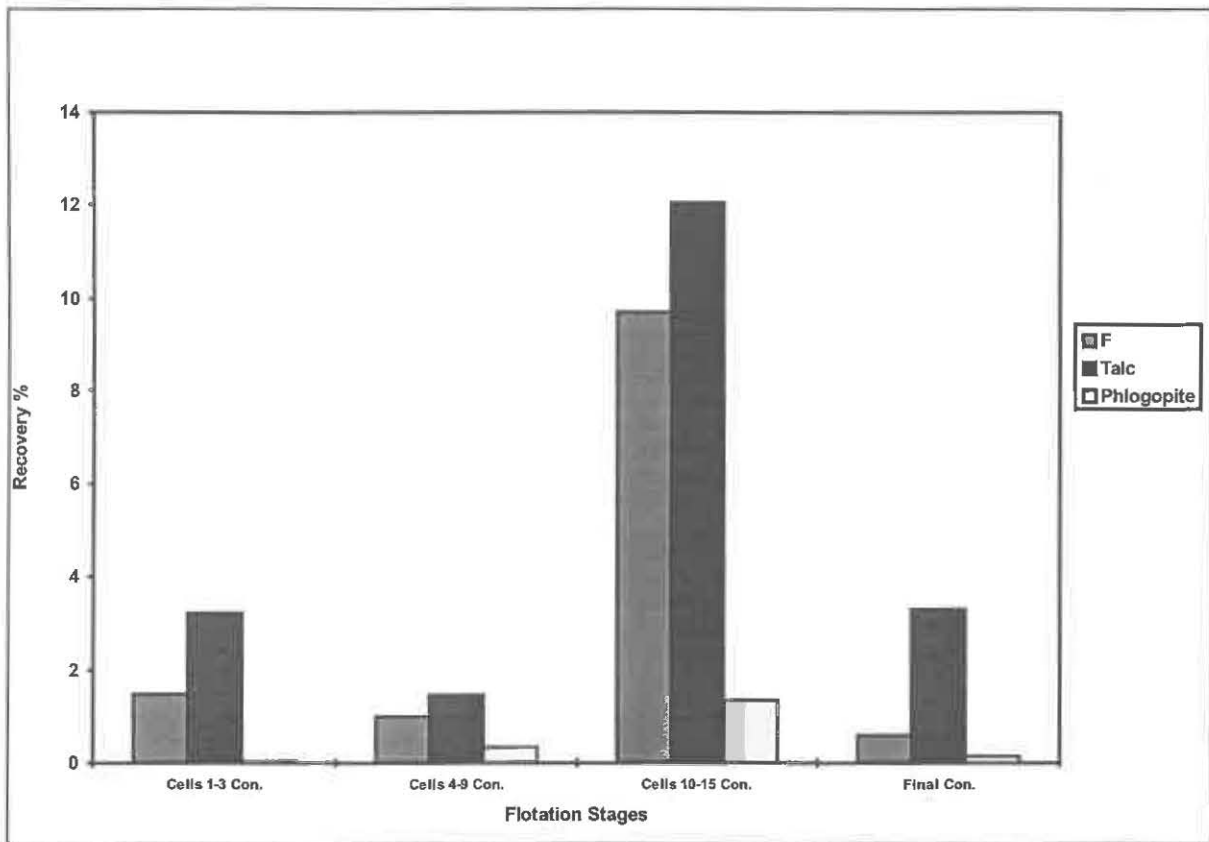|	New feed	Recycle feed	Comb feed	Cell 1 - 3 conc	Cell 1 - 3 tail	Cell 4 - 9 conc	Cell 4 - 9 tail	Cell 10 - 15 conc	Cell 10- 15 tail	Comb rough conc	Final conc	Cleaner tail
% Rec mass	100.0			1.6		1.1		6.4			1.6	45.4
% Rec Cu	100.0			57.7		15.3		12.9			83.6	0.8
% Rec Au	100.0			48.3		10.9		19.7			64.5	1.5
% Rec F	100.0			1.5		1.0		9.7			0.6	79.5
% C L* Cu	100.0	6.0						14.9				0.6
% C L* Au	100.0	6.5						21.1				0.9
% C L* F	100.0	10.8						10.8				2.2
% C L* mass	100.0	8.1						6.7				1.3

* C L = Circulating load

FIG 3 - Stage recovery of fluorine, talc and phlogopite.
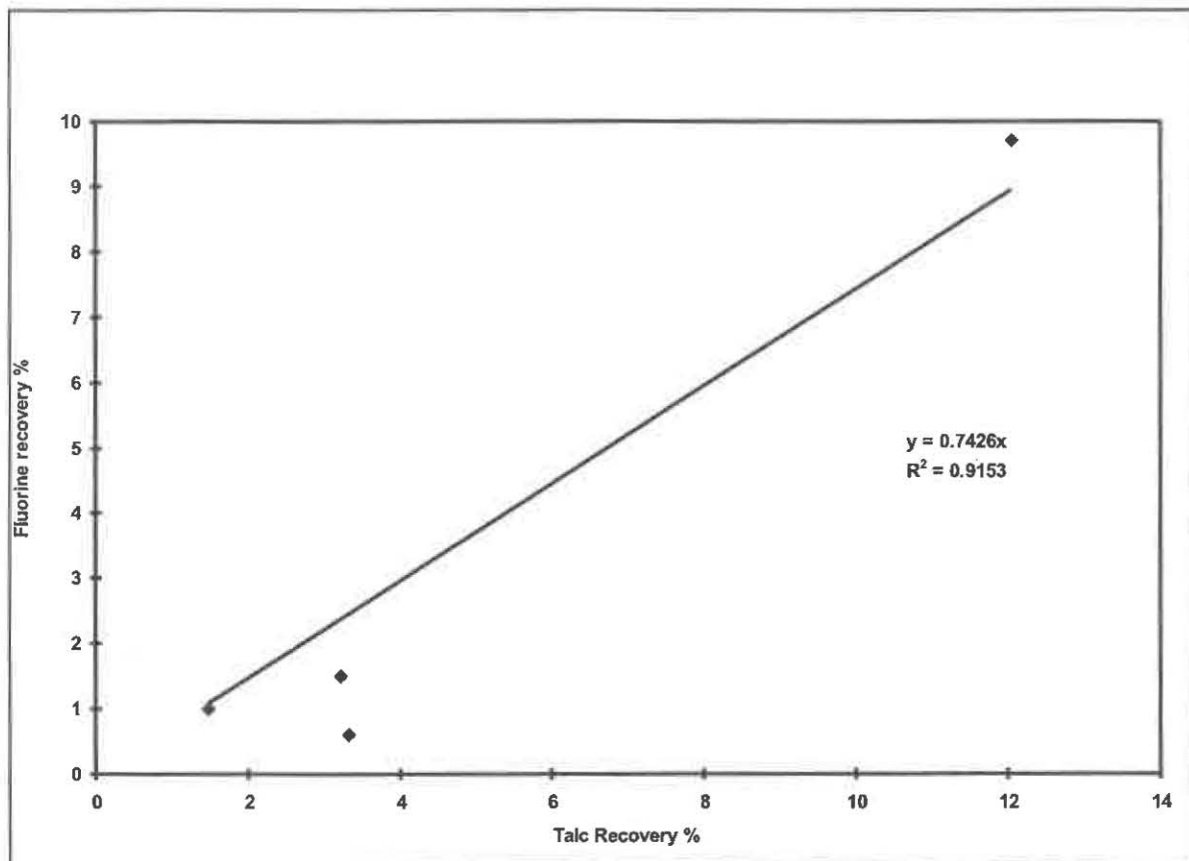

FIG 4 - Fluorine vs talc recovery.

The relatively higher scavenger recovery indicates that fluorine is contained in a slow floating species, ie talc and phlogopite. Such observations do not support the current plant practice of recycling the scavenger concentrate to the rougher heads because this introduces a fluorine recirculating load of 11 per cent. The new reconfigured circuit eliminates this recycle stream, by treating it in a separate scavenger - cleaner circuit (Cornish, 1997). At the time of this paper, the potential benefits of the new circuit, with respect to the fluorine problem, have not been fully assessed.

Out of all the streams surveyed, the streams of new feed, final concentrate and final tail have been chosen (for reasons of brevity) to study the distribution of mass and element (Cu and F) by size. That is, for a given stream, eg scavenger concentrate, the sum of percentage distribution of fluorine in each size fraction is 100. Figure 5 depicts fluorine distribution in all the major streams. These streams as they appear on the graph are: 10 - 15 conc (scavenger conc), 1 - 3 conc (rougher stage 1 conc), 4 - 9 conc (rougher stage 2 conc), new feed, final conc, cleaner tail and final tails.

It can be seen that all streams carry high fluorine in the -12.7 micron fraction. Scavenger concentrate in particular bears more than 40 per cent of the fluorine in this size fraction. Generally, most of the fluorine is deported in the -30 micron fraction. Minimising fines production would not only minimise fines fluorine flotation but would potentially reduce copper loss to tailings as is clearly evident below. Figure 6 depicts the distribution of mass and element (F and Cu) into each size fraction.

It indicates that up to 30 per cent of the copper loss occurs in the +75 μm fraction. This is probably unliberated copper whereas the 20 per cent loss in the -12 μm is most likely due to liberated copper minerals inhibited by hydroxide coating (Orwe et al, 1997; Senior and Trahar, 1991). The effect of slime coatings in depressing sulphide mineral flotation (Edwards et al, 1980) or increase hydrophilic mineral recovery (Smith and Warren, 1989) may be a possible cause of such copper loss.

Figure 7 depicts the size by size distribution of mass and element (Cu and F) in the final concentrate. Obviously, the largest proportion of fluorine (26 per cent) in the final concentrate is distributed to the -12.7 micron fraction. Silicate minerals, being softer than sulphides, tend to generate more fines during comminution. This supports earlier observations that minimum generation of fines in the milling circuit could potentially impact on the fluorine problem. Alternatively, if the fines fraction is 'screened off' from the flotation feed, the fluorine problem would be significantly reduced. A talc prefloat would be an option in this regard but the inclusion of a flash flotation unit in the current circuit to scavenge coarse liberated copper precludes such an option.

It is apparent in the foregoing discussions that much of the problem in terms of copper loss to tailings and fluorine deportment in final concentrate resides in the -12.7 micron fraction.

Figure 8 depicts mass and element distribution in the rougher feed. It indicates that only eight per cent of the copper in the flotation feed is contained in the -12.7 micron fraction which bears 20 per cent of the feed fluorine.

Finally, Figure 9 illustrates the relationship between water recovery and fluorine recovery.

Such relationships were also noted by Smith and Warren (1989) to describe recovery of water and hydrophobic particles. They noted that hydrophobic mineral flotation consists of two parts; namely inherent flotation and entrained flotation. In the present case, inherent flotation (zero water recovery) yields one per cent recovery of fluorine predominantly in talc. Levels above this would represent entrained talc and phlogopite. In view of the high water recovery in the scavengers, it can be said that entrainment is the predominant mechanism for fluorine flotation in the scavengers whilst natural flotation appears to be dominant in the roughers. High talc and hence fluorine recovery observed in Figure 3 could possibly be due to high levels of water recovery in the scavengers.
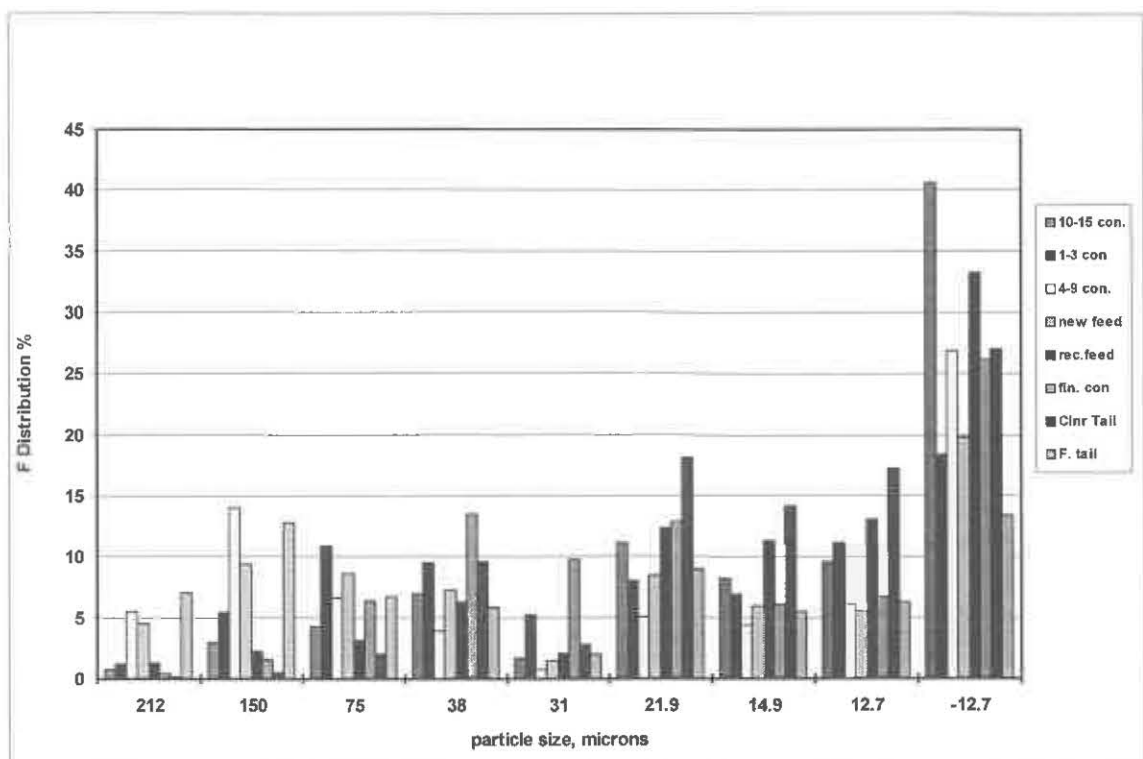

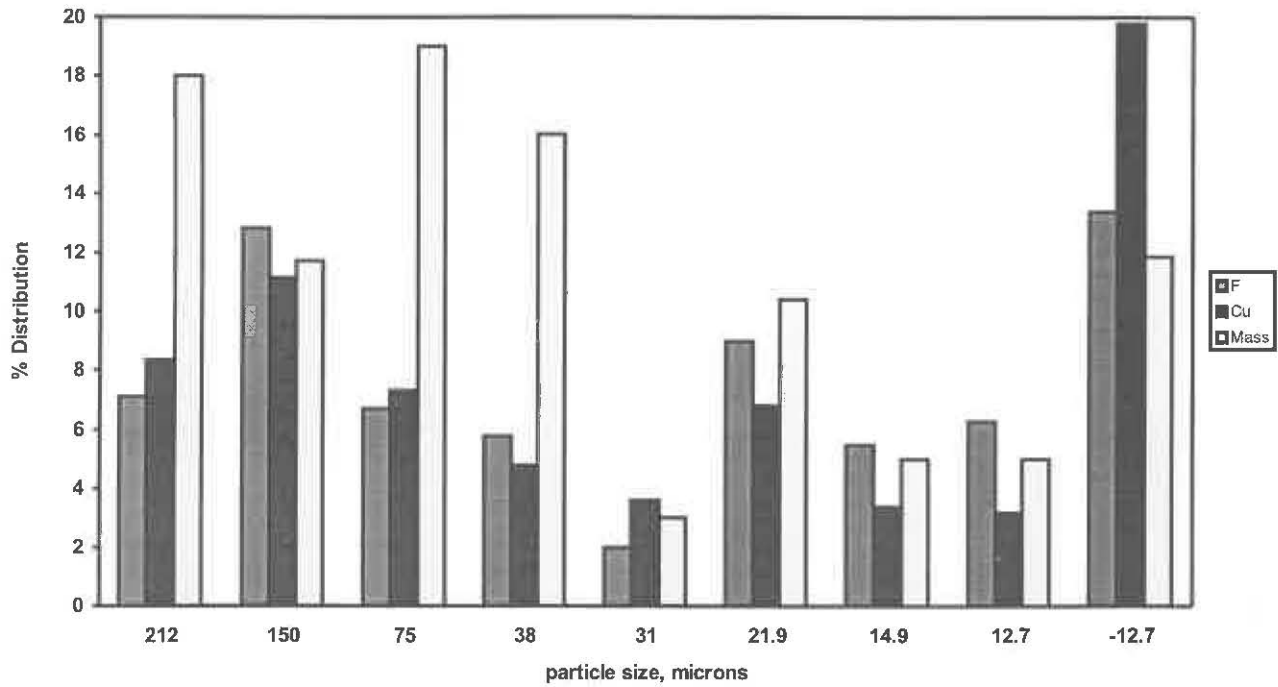FIG 5 - F distribution vs size in Ok Tedi float streams.

FIG 6 - Mass and material distribution by size - final tail.
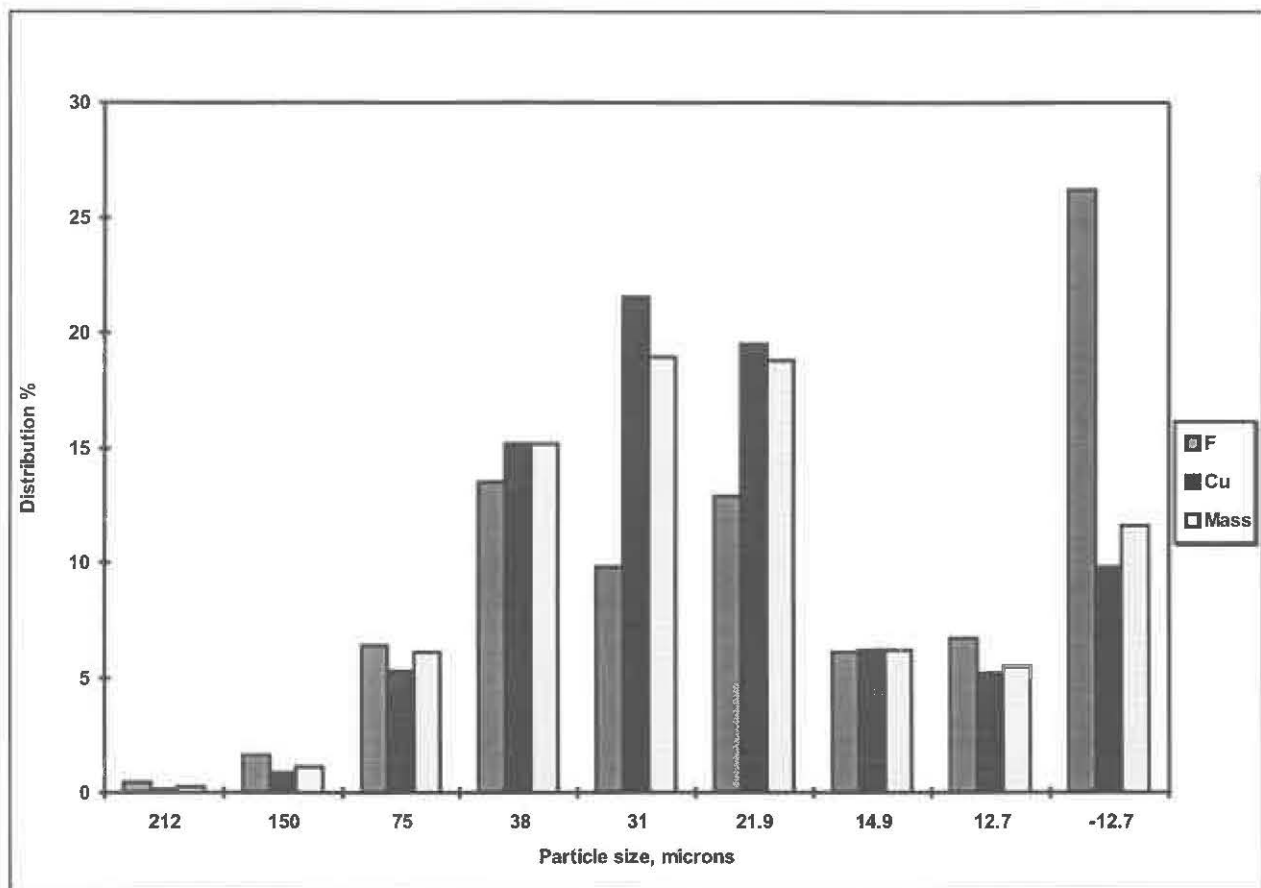

FIG 7 - Mass and material distribution by size final concentrate.
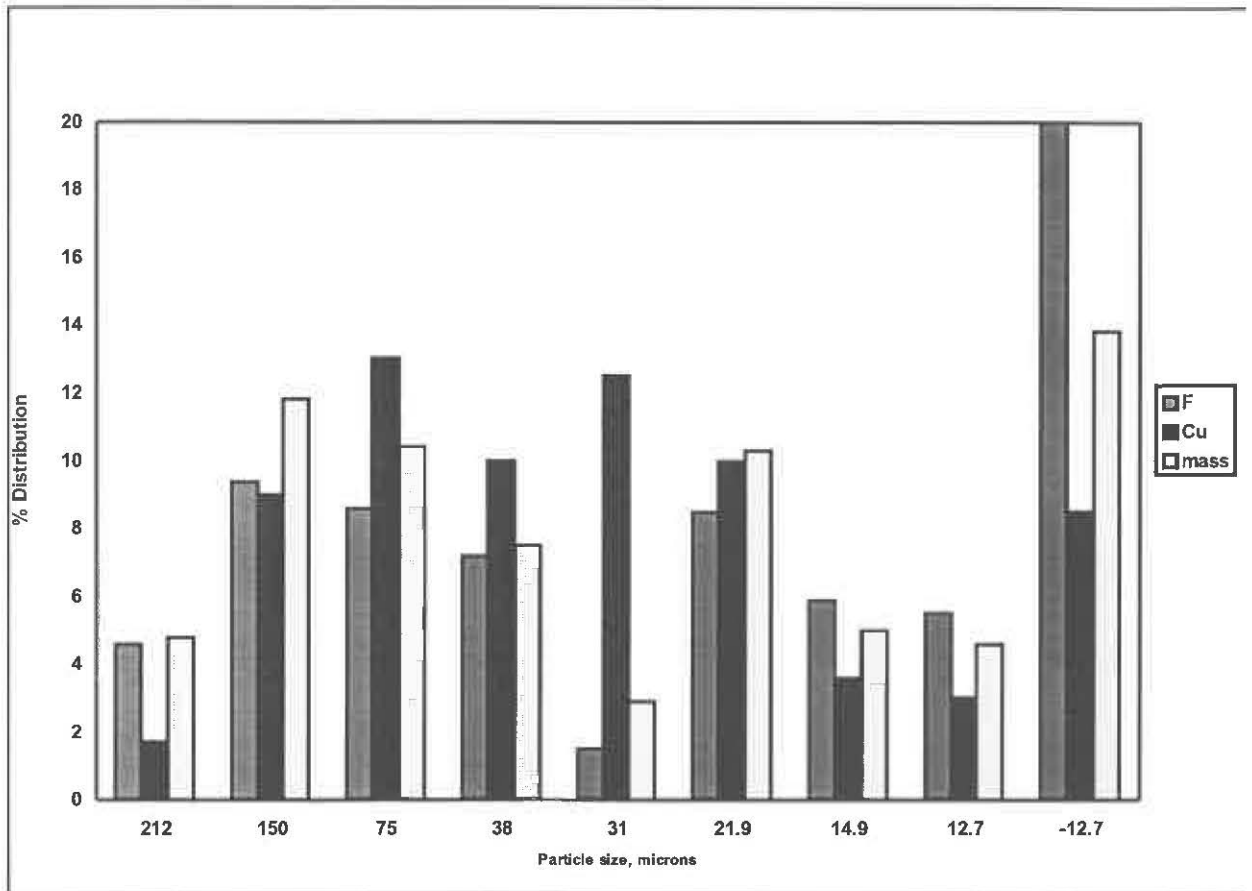
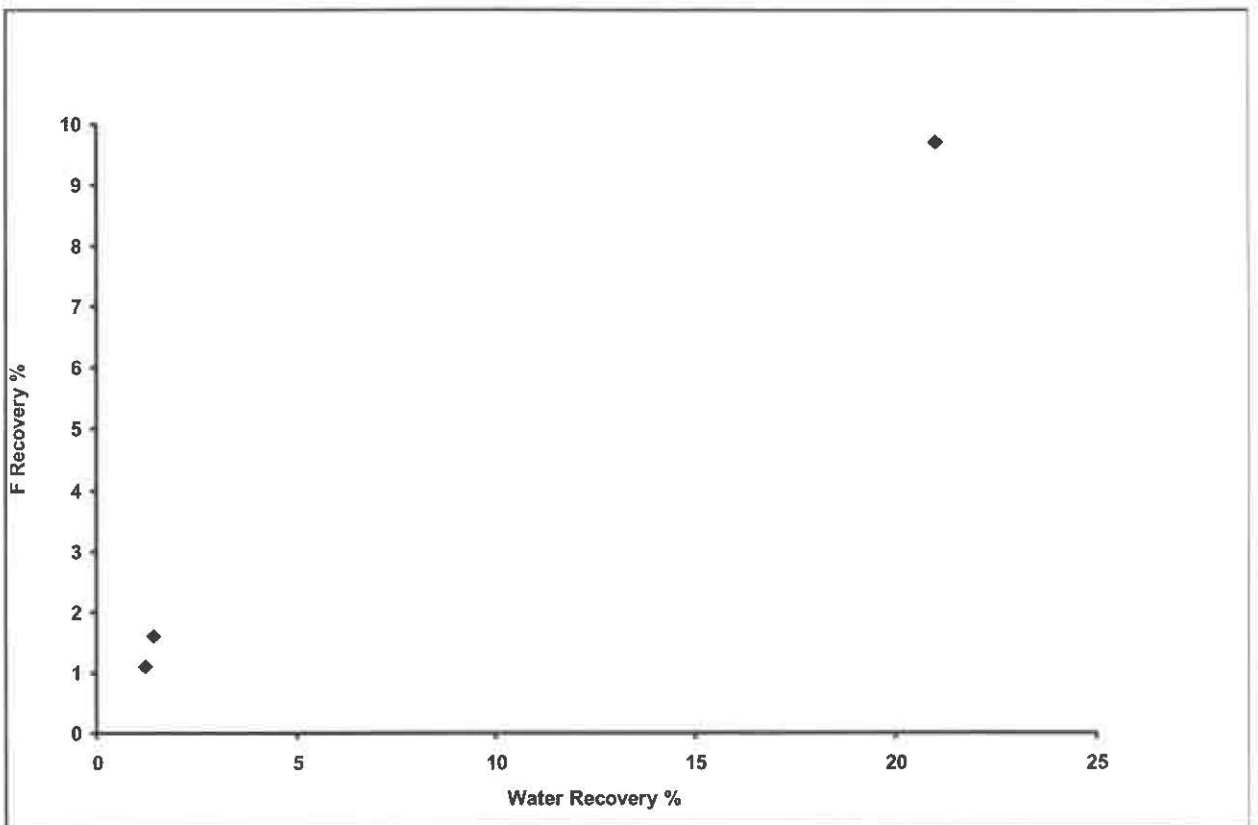FIG 8 - Mass and material distribution in new feed.

FIG 9 - Water recovery vs fluorine recovery.

CONCLUSIONS

With the aid of appropriate equipment, the process mineralogy of fluorosilicate minerals in the Ok Tedi copper flotation circuit have been examined. Of all the fluorosilicate minerals that have been identified in both the skarn and porphyry ore types, only talc and phlogopite have been shown to impact significantly on final concentrate fluorine with talc contributing up to 64 per cent of total fluorine. Fluorine recovery is dependent on talc and phlogopite recovery and much of the talc and phlogopite is recovered in the scavenger cells. Water recovery in the scavengers is up to 20 per cent implying that the observed mineral recovery could be due to a combination of entrainment and natural flotation. Overall plant talc and phlogopite recovery is 3.3 per cent and 0.15 per cent respectively whilst fluorine recovery is a trivial 0.6 per cent. Under steady state, a fluorine circulation load of 11 per cent can be expected in the current flotation circuit. Much of the fluorine resides in the -12.7 micron fraction in all the flotation streams.

Finally, the improved knowledge of the process mineralogy of fluorosilicate minerals have partly led to the reconfiguration of the flotation circuit to, amongst other benefits, reduce the circulation load of fluorine by eliminating the scavenger concentrate recycling, thereby reducing the fluorine loading on the circuit. The extra cleaning capacity offered by the reconfigured circuit, would also potentially assist in eliminating entrained fluorosilicates.

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