SECTOR DE PROCESSAMENTO DE MATÉRIAS-PRIMAS

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SCHEELITE RECOVERY BY FROTH FLOTATION FROM GRAVITY CONCENTRATION SLIMES OF "LOS SANTOS" ORE BODY

FINAL RESULTS – BENCH TESTS

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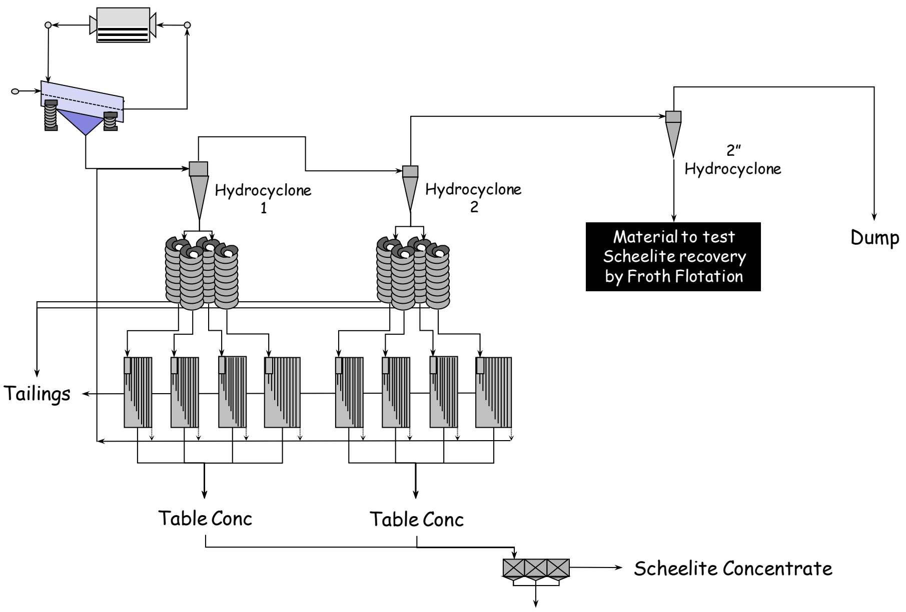
FINAL RESULTS – Bench Tests

Previously, Reports nº 01 and nº 02, were sent on 11th January and 24th April 2012, entitled INTERIM REPORT OF PRELIMINARY RESULTS and PRELIMINARY RESULTS, respectively.

This FINAL REPORT on Bench Tests was developed from those by adding a new paragraph, nº **7 - RESEARCH ON CALCITE AND FLUORITE DEPRESSION**, which substitutes the former one, **6.5 – Other families of Depressants.**

1 - PROBLEM PROPOSAL

Daytal Resources Spain S.L, the enterprise that is exploiting Los Santos Scheelite ore body, challenged LNEG Mineral Processing team to carry out an experimental testwork, aiming at studying the technical feasibility of Froth Flotation to recover the ultra-fine Scheelite present in the Overflow of the Hydrocyclone 2, which feeds the second floor of shaking tables, as shown in the Diagram below (given information).



2 - SAMPLE COLLECTION

As informed by DAYTAL staff, in order to reduce sample water content, the Hydrocyclone 2 Overflow, was passed through a 2" Hydrocyclone, of which the Underflow was recovered by filtering, before being collected in 10-12kg bags to be sent to LNEG for testwork. The first samples received in November 2011, were given a Laboratory Order Number - OL 380/2011. The amount of material was not enough, so, in order to carry on the study, more sample bags were sent in February 2012, which were given the Laboratory Order Number - OL 33/2012.

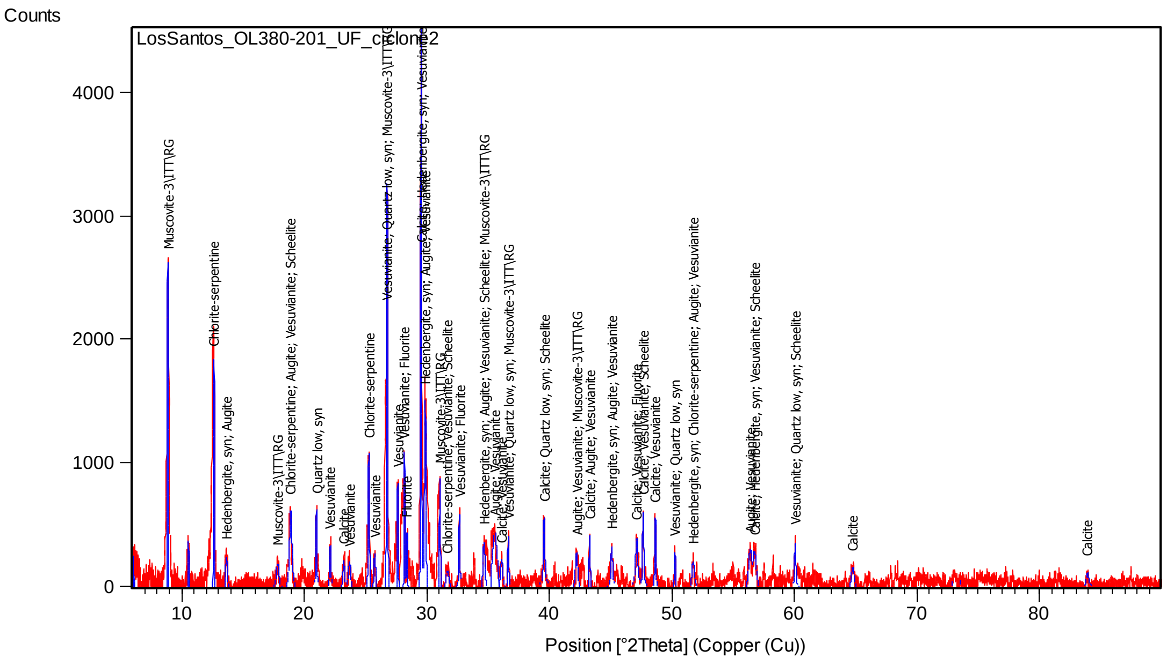
3 - MATERIAL CHARACTERIZATION

The samples were received with moisture contents corresponding to approximately 75% solids – OL 380/2011 and 80% solids – OL 33/2012 (determined/evaluated at the laboratory).
All tests were performed upon damp samples as delivered, preventing any kind of particle surface alteration, due to oxidization or dehydration phenomena.

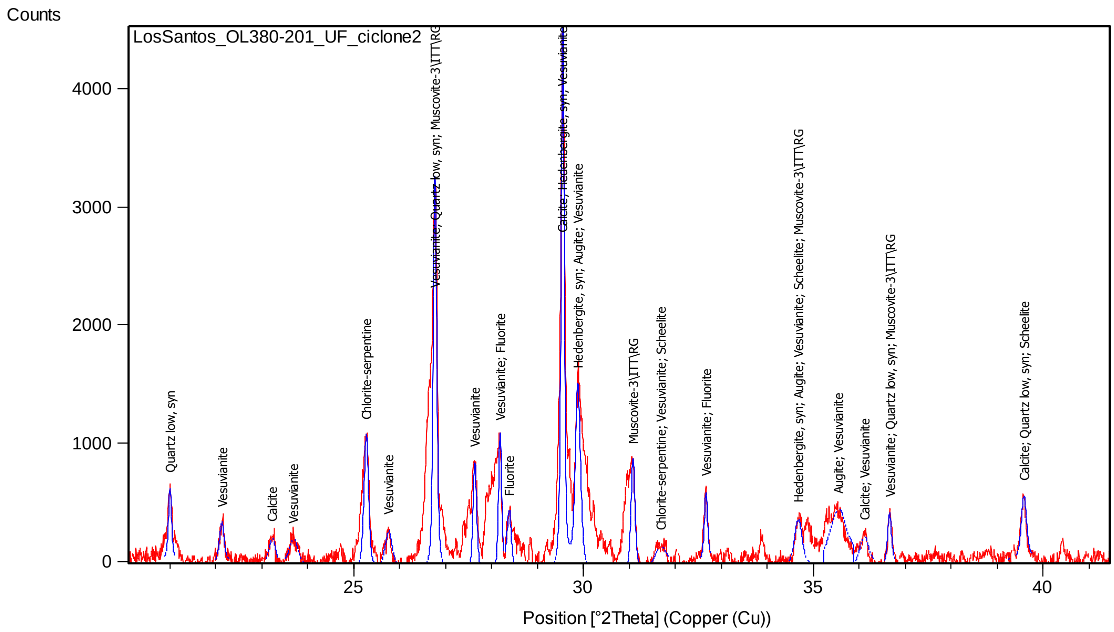
Before starting Batch Flotation tests, the sample was studied in order to obtain some relevant information as follows:

3.1 – Mineralogical Analysis

The 1st bulk sample, OL 380/2011, was analyzed by X-Ray Diffraction (XRD), the spectrum of which supports the mineralogy described below.



In next figure a zoom of the previous spectrum, showing reflections of Calcite, Scheelite and Fluorite, is presented:



Calcite	$Ca (C O_3)$	
Hedenbergite	$Ca (Fe , Mg) (Si O_3)_2$	
Chlorite-serpentine	$(Mg , Al)_6 (Si , Al)_4 O10 (O H)_8$	
Muscovite		
Augite	$Ca (Fe , Mg) Si_2 O_6$	30
Vesuvianite		
Quartz	$Si O_2$	
Scheelite	$Ca W O_4$	
Fluorite	$Ca F_2$	

This mineralogical analysis as well as spectra of a few concentrates where it is enhanced the presence of scheelite, fluorite and calcite, are included in Annex 1.

3.2 – Size Analysis

The size distribution of both bulk samples was determined in a Laser COULTER LS 130 Particle Size Analyzer as can be consulted in Annex 2.

The size analyses show that in the 1st sample OL 380/2011, the material is 90% below 40µm, 50% below 16µm and 25% below 9µm and in the 2nd sample OL 33/2012, 90% below 50µm, 50% below 19µm and 25% below 10µm, which means that, in what concerns the mineral processing technology we are dealing with a “very fine material” to treat.

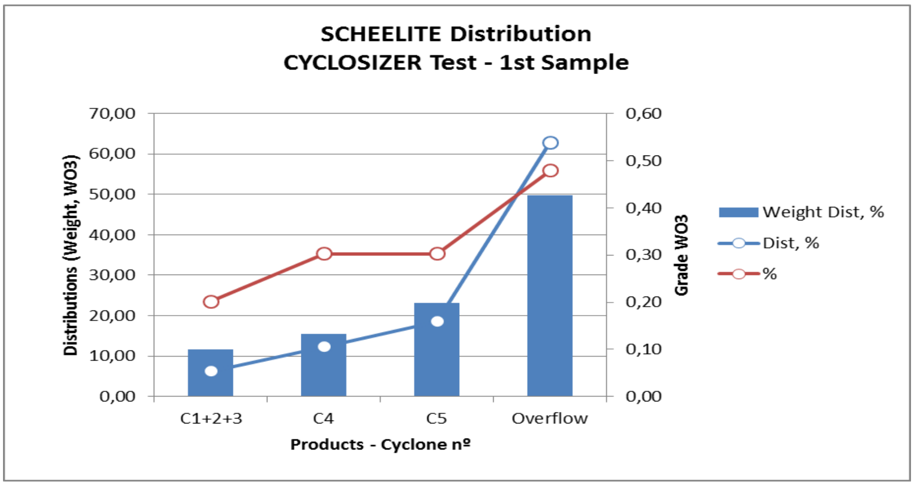
In order to appreciate the distribution of Scheelite, the valuable mineral, according to particle size, separations in a Cyclosizer were carried out.

Products collected in cyclones nºs 1, 2 and 3 were mixed together, as each of them had very low mass (especially in the 1st sample). Cyclones 4 and 5 collected enough products for analytical assays.

The feed material and the underflow products collected were analyzed to determine the WO_3 content. The overflow was estimated by mass balancing.

Results obtained for the 1st sample – OL 380/2011, are shown in the table and graph below:

Test	CYCLOSIZER - 1st Sample				
	DATA	20-12-2011		WO3	
	Weight, g	Weight Dist, %	%W	%	Dist, %
C1+2+3	4,67	11,68	0,16	0,20	6,23
C4	6,17	15,43	0,24	0,30	12,34
C5	9,29	23,23	0,24	0,30	18,58
Overflow	19,87	49,68	0,380	0,48	62,85
Total	40,00	100,00	0,30	0,38	100,00



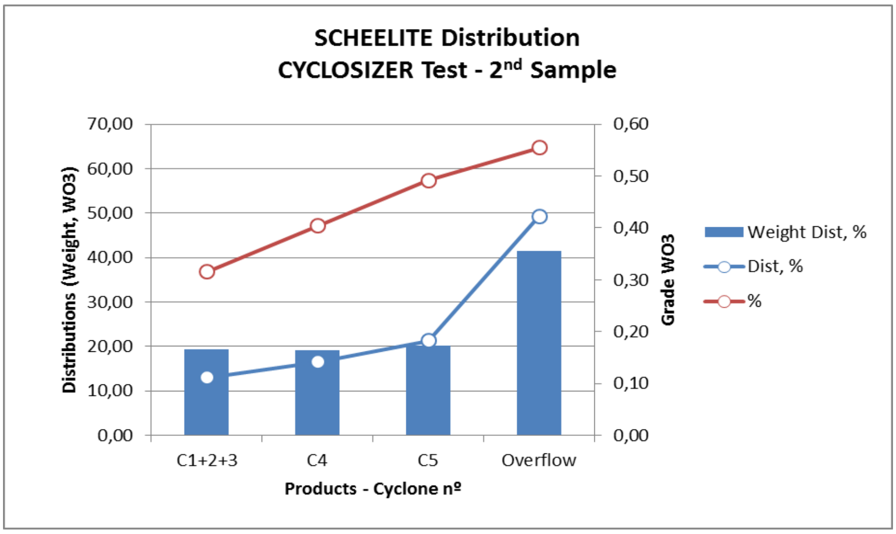
Some conclusions can be drawn:

- Almost 50% feed by weight is below 13-16µm (Cyclosizer is not calibrated)
- Scheelite clearly tends to concentrate in the fine range
- 55% WO_3 is in the ultra-fine range

Scheelite Distribution results for the 2nd Sample are shown in the table and graph below:

Ensaio	CYCLOSIZER - 2nd Sample				
	DATA			WO3	
	Weight, g	Weight Dist, %	%W	%	Dist, %
C1+2+3	9,65	19,30	0,32	0,40	13,02
C4	9,52	19,04	0,40	0,51	16,47
C5	10,07	20,14	0,49	0,62	21,22
Overflow	20,76	41,52	0,55	0,70	49,29
	50,00	100,00	0,47	0,59	100,00

Note:
WO_3 is obtained from W analytical values by stoichiometry calculations.



Although the 2nd sample seems to be slightly coarser, there is the same tendency for Scheelite to concentrate in the size range below 15µm.

These differences in Scheelite Distribution on the two samples should be taken into consideration when designing the dewatering process of the overflow of Hydrocyclones nº2 (2" Hydrocyclone) at the Plant.

Note:
W Analytical Assays were determined by XFR and are reported for all the testwork on a single Lab Bulletin, which is presented in Annex 3.

3.3 – Feed Samples WO_3 Average Grade

Samples were used as they arrived, i.e., not dried, and the average moisture contents determined in the Lab, as formerly referred to in this Chapter. Consequently no blending process was done.

As it will be seen, this Report is supported by 23 experimental tests. For each one of the products, bulk WO_3 grades were assayed and Feed Grade was back calculated.
For all those samples a Feed Average Grade of 0,449 % WO_3 was obtained, varying from 0,331 WO_3 to 0,573 WO_3. It can be seen that the 2nd sample is slightly richer.

4. BACTH FLOTATION –ROUGHER TESTS

In the late 80s LNEG team had conducted some research in Flotation of Scheelite ores from a Portuguese skarn deposit quite similar to Los Santos ore body.
Having in mind that former experience, some preliminary tests using traditional fatty acids, belonging to our own stock of reagents, as well as new (modern) reagents given by DAYTAL, were carried out.

Previous results of that first approach led to consider reagent AERO 726 from CYTEC, an anionic tall oil fatty acid-based promoter (containing surfactants and other chemical coupling agents that make them much more effective than straight tall oil fatty acids), as a very promising one.
However, we should stress that more experimental work is needed, to arrive at a more correct/accurate conclusion about the performance of other available reagents.

The factorial plan to test reagent AERO 726 is represented in the following table:

Test nº	NaOH	pH	Na_2SiO_4	$NaCO_3$	Quebracho	AERO 726	Obs
Nº 3	qb	9,3	4 kg/t	---	---	200 g/t +	Without Calcite Depressant
Nº 5	qb	9,3	4 kg/t	3 kg/t	---	200 g/t	NaCO3 for Calcite depression
Nº 6	qb	10,6	4 kg/t	5 kg/t	---	100 g/t	Idem, but less Collector
Nº 9	qb	10,6	4 kg/t	5 kg/t	12 g/t	100 g/t	+ Quebracho as Calcite depressant

In the next tables, all operational results and mass balancing computations from the above referred tests are presented.

Na2SiO4	4 kg/t	NaCO3	---	NaOH	qb
AERO 726	200 g/t +			pH	9,3

Time (min)	Products	Weight, g	Weight, %	%W	WO3 %	WO3 Dist, %	WO3 Cumulative %	WO3 Cumulative Dist, %
	Test nº 3	24-11-2011						
1	3C1	23	3,00	2,69	3,392	26,06	3,39	26,06
1	3C2	25	3,26	1,73	2,182	18,22	2,76	44,27
2	3C3	65	8,47	1,13	1,425	30,94	1,99	75,21
	3AF	654	85,27	0,09	0,113	24,79	0,390	100,00
	Total	767,00	100,00	0,310	0,390	100,00		

Na2SiO4	4 kg/t	NaCO3	3kg/t	NaOH	qb
AERO 726	200 g/t			pH	9,3

Time (min)	Products	Weight, g	Weight, %	%W	WO3 %	WO3 Dist, %	WO3 Cumulative %	WO3 Cumulative Dist, %
	Test nº 5	24-11-2011						
1	5C1	51,64	6,32	2,87	3,619	49,58	3,62	49,58
1	5C2	40,99	5,02	1,52	1,917	20,84	2,87	70,43
1	5C3	24,65	3,02	1,03	1,299	8,49	2,54	78,92
	5AF	700	85,65	0,09	0,113	21,08	0,461	100,00
	Total	817,28	100,00	0,366	0,461	100,00		

Na2SiO4	4 kg/t	NaCO3	5kg/t	NaOH	qb
AERO 726	100 g/t			pH	10,6

Time (min)	Products	Weight, g	Weight, %	%W	WO3 %	WO3 Dist, %	WO3 Cumulative %	WO3 Cumulative Dist, %	F %
	Test nº 6	13-12-2011							
1	6C1	67	8,46	3,32	4,187	72,08	4,19	72,08	3,6
1	6C2	38	4,80	1,3	1,639	16,01	3,26	88,09	
1	6C3	3,2	0,40	0,8	1,009	0,83	3,20	88,92	
	6AF	684	86,34	0,05	0,063	11,08	0,491	100,00	
	Total	792,20	100,00	0,390	0,491	100,00			

Na2SiO4	4 kg/t	NaCO3	5kg/t	NaOH	qb
AERO 726	100 g/t	Quebracho	12g/t	pH	10,6

Equivalent to Test nº 6 + Quebracho

Time (min)	Products	Weight, g	Weight, %	%W	WO3 %	WO3 Dist, %	WO3 Cumulative %	WO3 Cumulative Dist, %	F %
	Test nº 9	27-12-2011							
1	9C1	32	4,31	5,5	6,936	72,17	6,94	72,17	8,5
1	9C2	27	3,64	1,4	1,766	15,50	4,57	87,68	
	9AF	683	92,05	0,044	0,055	12,32	0,41	100,00	
	Total	742,00	100,00	0,329	0,414	100,00			

Notes:

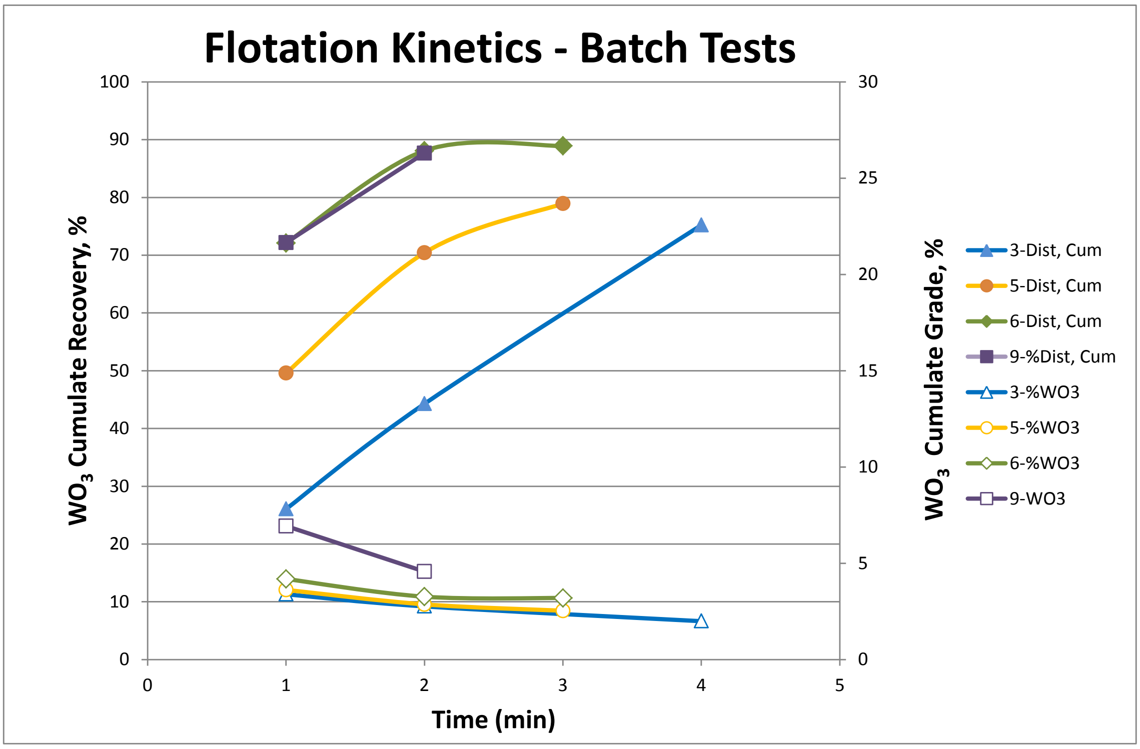
- Shaded cells refer to raw data, whilst calculated values are not shadowed
- WO_3 is obtained from W analytical values by stoichiometry calculations
- F was determined by Ion Selective Electrode and is reported in Annex 4, on a single Lab Bulletin.

The most expressive conclusions can be drawn as follows:

- pH should be maintained always above 10;
- Sodium Silicate (water glass) is useful as silicate depressant/dispersant;
- Sodium Carbonate plays an important role in the overall process selectivity, mainly for calcite depression;
- Collector dosage should be relatively low, according to the low WO_3 feed grade; this is appropriate to improve selectivity;
- Quebracho, although very sensitive, at very low dosages (~10g/t) seems to be important for calcite depression in order to improve WO_3 concentrate grade – Quebracho addition almost duplicates the 1st kinetics grade concentrate, in relation to the other tests, without penalizing WO_3 recovery;
- Quebracho addition leads to decreasing flotation time and froth instability. The mass recovered in the 2nd minute of flotation, was lower than that collected during the same period of time, in the absence of Quebracho;
- In what concerns mineral composition of the concentrates, for Tests nº 6 and 9 (the most relevant to date) was decided to determine the Fluor content (far right column). Stoichiometric Scheelite and Fluorite contents in those

concentrates are presented in next table and show that Fluorite is between 1,5 to 2 times the Scheelite content. In Annex 1, some XRD spectra of concentrate products show the presence of Fluorite.

	Scheelite %	Fluorite %
Test nº 6	5,2	7,40
Test nº 9	8,61	17,47



For the best test until now – **Test nº 9** – results can be resumed:

- 1 minute concentrate (Rougher 1st Conc) ≈ 6,9% WO_3, 72% WO_3 Recovery
- 2 minute concentrate (1st Conc + 2nd Conc) ≈ 4,6% WO_3, 88% WO_3 Recovery

Sulphide Removal

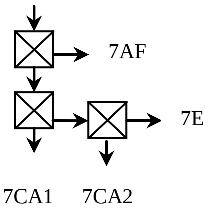
Until now, no sulphide removal has been tested. Although this problem could be looked at, there is some evidence that the responsibility for the low WO_3 grades achieved is due mainly to the presence of Calcite and Fluorite in the concentrates.

5. BATCH FLOTATION - CLEANING TESTS

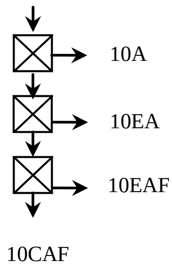
As the Recoveries obtained in the Rougher concentrates were reasonable, especially for such fine feed material, we tried to improve WO_3 Concentrate Grade by submitting the Rougher concentrate to one or two cleaning stages.

For that purpose, CLEANING flotation tests were carried out on Rougher concentrates (collected to completion) without any collector addition, as follows:
Test nº 7 with ONE CLEANING stage and **Test nº 10** with a 1st and a 2nd CLEANING stage

Test nº 7



Test nº 10



In the next tables, all operational results and mass balancing computations/ calculations from the above two tests are presented.

CLEANING

Na2SiO4	4 kg/t	NaCO3	5kg/t	NaOH	qb
AERO 726	100 g/t			pH	10,6

	Test nº 7	**15-12-2011**			WO3		WO3 Cumulative		
Time (min	Products	Weight, g	Weight, %	%W	%	Dist, %	%	Dist, %	F %
1	7CA1	54	3,35	6,52	8,222	69,08	8,22	69,08	9,6
1	7CA2	17	1,06	1,98	2,497	6,60	6,85	75,68	
1	7AE	83	5,16	0,09	0,113	1,47	3,22	77,15	
	7AF	1456	90,43	0,08	0,101	22,85	0,399	100,00	
	Total	1610,00	100,00	0,317	0,399	100,00			

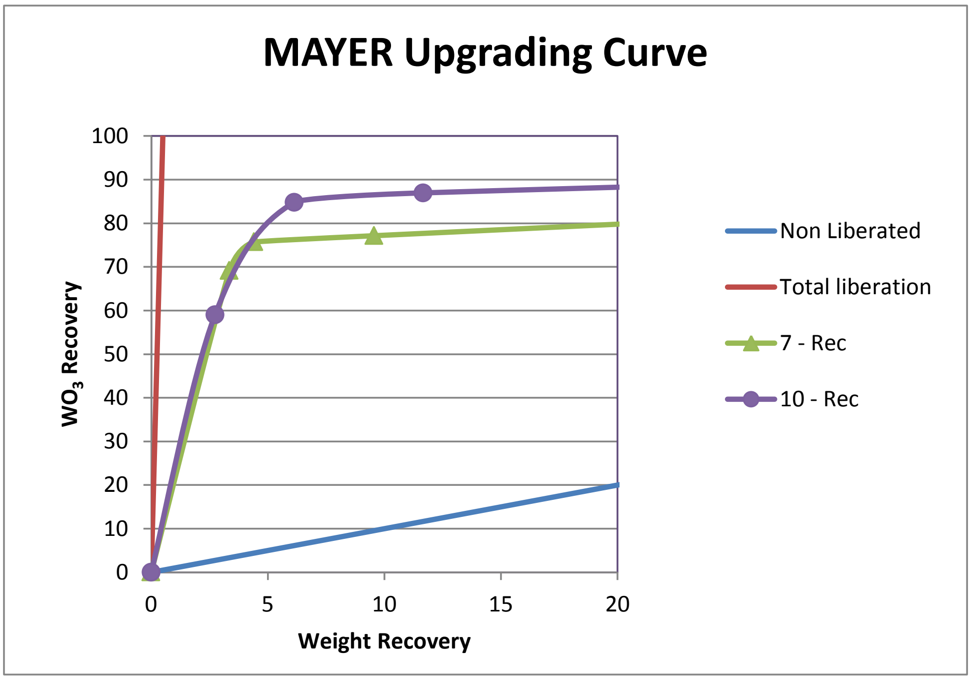
CLEANING

Na2SiO4	4 kg/t	NaCO3	5kg/t	NaOH	qb
AERO 726	100 g/t			pH	10,6

	Test nº 10	**27-12-2011**			WO3		WO3 Cumulative		
Time (min	Products	Weight, g	Weight, %	%W	%	Dist, %	%	Dist, %	F %
1	10CAF	41	2,73	7,17	9,042	59,01	9,04	59,01	7,8
1	10EAF	51	3,40	2,52	3,178	25,80	5,79	84,80	
1	10EA	83	5,53	0,13	0,164	2,17	3,12	86,97	
	10AF	1325	88,33	0,049	0,062	13,03	0,419	100,00	
	Total	1500,00	100,00	0,332	0,419	100,00			

For plotting the MAYER Upgrading Curves, the following figures, extracted from the above table, were used:

Test nº	2nd Cleaner Grade % WO_3	WO_3 Recovery %	1st Cleaner Grade % WO_3	WO_3 Recovery %	Rougher Grade % WO_3	WO_3 Recovery %
Nº 7	7CA1		7CA1+7CA2		7CA1+7CA2+7AE	
	8,2	69,1	6,9	75,7	3,2	77,2
Nº 10	10CAF		10CAF+10EAF		10CAF+10EAF+10EA	
	9,0	59,0	5,8	84,8	3,1	87,0



Conclusions on the CLEANING stage:

- The Grade obtained in the first Cleaning Concentrate is about 2 times greater than that of the Rougher Concentrate;
- Recovery did not decrease significantly in first the Cleaning stage;
- With the 2nd cleaning stage of flotation, a concentrate assaying 9% WO_3 was attained. However, Fluor assays show again that the ratio Flourite/Scheelite falls into the same interval]1,4 … 2[.

6. EXPLORATORY TESTS WITH OTHER COLLECTORS

Until this point, the best reagents configuration based on AERO 726, led to very interesting WO_3 recoveries, but concentrate grades above 10% WO_3, were never obtained.

As other available collectors were supplied by DAYTAL, some exploratory tests were performed in order to see if any other collector had better potential for obtaining higher concentrate grades.

All those exploratory tests were performed under conditions similar to those of Test nº 9, which will be used from now on as "standard" for comparison.

6.1 – FLOTINOR 2818

This test was performed trying the collector FLOTINOR 2818 (a sodium alkyl sulfate, or sulfated fatty alcohol, from CLARIANT), following recommendations from the reagent supplier, which included a reduction of Na_2SiO_3 to half when comparing to dosages usually added.

Na2SiO4	2 kg/t	NaCO3	5kg/t	NaOH	qb
FLOTINOR 2818	70 g/t	Montanol 8	25g/t	pH	10,6

	Ensaio 13	20-12-2011			WO3		WO3 Cumulate	
Time (min)	Products	Weight, g	Weight, %	%W	%	Dist, %	%	Dist, %
1	13C1	26	3,40	0,51	0,643	6,62	0,64	6,62
1	13C2	14	1,83	0,48	0,605	3,35	0,63	9,97
1	13C3	25	3,27	0,38	0,479	4,74	0,57	14,71
1	13SC	57	7,46	0,52	0,656	14,79	0,61	29,51
	13AF	642	84,03	0,22	0,277	70,49	0,33	100,00
	Total	764,00	100,00	0,262	0,331	100,00		

<u>Comments</u>: very poor Grades and Recovery

6.2 – FLOTINOR FS-2

FLOTINOR FS-2 was another collector tested, from the CLARIANT family of alkyl sulfates. As in this case there was no advice from the supplier, dosage conditions of the standard Test nº 9 were maintained varying only the collector.

			Na2SiO4	4 kg/t	NaCO3	5kg/t	NaOH	qb
			FLOTINOR FS-2	100 g/t + 100 + 100		Montanol 40g/ton	pH	10,6

	Ensaio 14	19-01-2012			WO3		WO3 Cumulate	
Time (min)	Products	Weight, g	Weight, %	%W	%	Dist, %	%	Dist, %
1	14C1	69	8,68	2,78	3,506	70,80	3,51	70,80
1	14C2	55	6,92	0,76	0,958	15,43	2,38	86,22
1	14SC	59	7,42	0,28	0,353	6,10	1,72	92,32
	14AF	612	76,98	0,034	0,043	7,68	0,43	100,00
	Total	795,00	100,00	0,341	0,430	100,00		

Comments: Recoveries similar to Test nº 9, but Grades are about half.
Although this configuration could be optimized, there are no significant signals indicating better selectivity.

6.3 – AERO 704

Collector AERO 704, a straight tall oil fatty acid promoter from CYTEC, was also tested.

			Na2SiO4	4 kg/t	NaCO3	5kg/t	NaOH	qb
			AERO 704	100g/t	Quebracho	10g/t	pH	10,6

	Ensaio 16	26-01-2012			WO3 Parcias		WO3 Cumulados	
parciais	Produtos	Peso, g	Peso, %	%W	%	Dist, %	%	Dist, %
1	16C1	49	6,16	3,03	3,821	55,60	3,82	55,60
1	16C2	23	2,89	1,86	2,346	16,02	3,35	71,62
1	16C3	40	5,03	0,87	1,097	13,03	2,55	84,65
	16AF	683	85,91	0,06	0,076	15,35	0,42	100,00
	Total	795,00	100,00	0,336	0,424	100,00		

Comments: Similar to the previous Test nº 14 with FLOTINOR FS-2.

6.4 – AERO 6493 (Alkyl Hydroxamate)

Following a CYTEC recommendation, based on an anionic alkyl hydroxamate-based collector and citric acid as depressor, four exploratory bulk rougher tests, nºs 25, 26, 27 and 28 were performed.
Results were very poor.
In the next table is shown the data referring to the best of the four, nº 27 (which is pretty bad!), performed at pH = 9,2.

Na_2SiO_4	4 kg/t	$NaCO_3$	---	NaOH	qb
AERO 6493	144g/t	Citric Acid	1kg/ton	pH	9,2

	Test nº 27	**17-04-2012**			WO3		WO3 Cumulative	
Time (min)	Products	Weight, g	Weight, %	%W	%	Dist, %	%	Dist, %
4	27C1	69	9,48	1,78	2,245	43,71	2,24	43,71
	27AF	659	90,52	0,24	0,303	56,29	0,487	100,00
	Total	728,00	100,00	0,386	0,487	100,00		

Comments: Concentrate Grade and Recovery are far below those obtained in standard Test nº 9 and there was no practical evidence of better selectivity.

7. RESEARCH ON CALCITE AND FLUORITE DEPRESSION

7.1 - Considerations about the point-of-zero-charge (PZC)

The pH value, at which the PZC occurs, varies from mineral to mineral. When the pulp pH is lower than that of PZC of a certain mineral, it will be positively charged and consequently attracts anions. If the pH is higher, that mineral surface will be negatively charged and attracts cations. These characteristics should be considered, in order to optimize mineral separation, envisaging better concentrate grades and recoveries.

Based on values referred to in the bibliography, Scheelite, Calcite and Fluorite PZCs occur, respectively, at pH values of 1,5, 9,5 and 10. So, for pH < 9,5, Calcite and Fluorite tend to attract anions from the depressants, whilst Scheelite, being negatively charged would not be depressed.

This expected behaviour was not confirmed at all during the test work, as it was at pH=10,5 that the best results were achieved.

7.2 – Most commonly used Depressants

Accordingly with traditional bibliography, the most commonly used depressants are:

- Sodium Carbonate – plays an important role in the overall process selectivity, apparently for calcite depression (improvement obtained from Test nº 3 to Test nº 5);
- Quebracho – although being a common depressant for semi-soluble salts minerals, acts mainly as a Calcite depressant (WO_3 concentrate grade improvement obtained from, Test nº 6 to Test nº 9);
- Na_2SiF_6 – as it adsorbs on the mineral surface, sodium oleate is desorbed. It is referred to be used on the cleaning stages;
- Phosphates – they form a hydrophilic phosphate calcium salt on mineral surfaces;
- $BaCl_2$ – is referred to as having a depressing effect on fluorite.

7.2.1 - Quebracho

When comparing Test nº 9 (kinetic, with Quebracho) with Test nº 7 (roughing + cleaning, without Quebracho), it can be seen that Quebracho depresses Calcite, but similar results can be achieved with a cleaning stage without its addition:

- Test nº 9 – C1 – 6,94% WO_3 ⇔ 80,0% Recovery
- Test nº7 (Cleaning) – CA1 – 8,23% WO_3 ⇔ 69,1% Recovery

Similar results where reproduced with the sample OL nº 33/2012 received later for continuing the test work program.

Na2SiO4	4 kg/t	NaCO3	5kg/t	NaOH	qb
AERO 726	100g/t	Quebracho	10g/t	pH	10,6

	Ensaio 17	06-02-2012			WO3		WO3 Cumulative	
Time (min)	Products	Weight, g	Weight, %	%W	%	Dist, %	%	Dist, %
1	17C1	73	10,15	3,58	4,515	79,98	4,51	79,98
0,5	17C2	7	0,97	2,10	2,648	4,50	4,35	84,48
1	17SC	21	2,92	0,65	0,820	4,18	3,62	88,65
	17AF	618	85,95	0,06	0,076	11,35	0,57	100,00
	Total	719,00	100,00	0,454	0,573	100,00		

CLEANING

Na2SiO4	4 kg/t	NaCO3	5kg/t	NaOH	qb
AERO 726	100g/t	Quebracho	---	pH	10,6

Test nº 18	29-02-2012			WO3		WO3 Cumulative	
Products	Weight, g	Weight, %	%W	%	Dist, %	%	Dist, %
18CA1	98	6,73	4,28	5,397	63,75	5,40	63,75
18CA2	42	2,88	2,1	2,648	13,40	4,57	77,15
18AE	75	5,15	0,35	0,441	3,99	3,13	81,14
18AF	1241	85,23	0,1	0,126	18,86	0,570	100,00
Total	1456,00	100,00	0,452	0,570	100,00		

In these cases should only be stressed that the Concentrate grades were a little bit lower than those obtained with first sample OL nº 380/2011:

- Test nº 17 – C1 – 4,51%WO_3 ⇔ 80,0% Recovery
- Test nº 18 (Cleaning) – CA1 – 5,40% WO_3 ⇔ 63,75% Recovery.

7.2.2 – Use of Depressants in the Cleaning stage – the cases Na_2SiF_6, PHOSPHATES and $BaCl_2$

Na_2SiF_6, Phosphates and $BaCl_2$, alone or combined are referred to as having influence on Calcite depression, when used on the Cleaning stages.

For this new series of tests, a new standard test was carried out at the same conditions as those of Test nº 6 (without Quebracho) for comparison.

CLEANING (standard)

Na2SiO4	4 kg/t	NaCO3	5kg/t	NaOH	qb
AERO 726	100 g/t			pH	10,6

Test nº 38	**22-06-2012**			WO3		WO3 Cumulative		
Products	Weight, g	Weight, %	%W	%	Dist, %	%	Dist, %	F %
38CA	56	4,08	6,36	8,020	64,66	8,02	64,66	6,3
38C2	41	2,99	1,59	2,005	11,84	5,48	76,50	
38EA	7	0,51	0,38	0,479	0,48	5,14	76,98	
38E	1268	92,42	0,10	0,126	23,02	0,506	100,00	
Total	1372,00	100,00	0,401	0,506	100,00			

Comments: results obtained fall between those formerly obtained in Tests nº 6 and 9 with the first sample received.

In Test nº 39 was tested the addition of Phosphate during the Cleaning stage.

CLEANING with PHOSPHATE

Na2SiO4	4 kg/t	NaCO3	5kg/t	NaOH	qb
AERO 726	100 g/t	Phosfate	150g/t	pH	10,6

Test nº 39	**26-06-2012**			WO3		WO3 Cumulative		
Products	Weight, g	Weight, %	%W	%	Dist, %	%	Dist, %	F %
39CA	20	1,34	10,6	13,367	44,14	13,37	44,14	7,3
39EA	78	5,22	1,83	2,308	29,72	4,56	73,86	
39E	1395	93,44	0,09	0,113	26,14	0,406	100,00	
Total	1493,00	100,00	0,322	0,406	100,00			

Comments: this was the BEST GRADE obtained in test work!

In the following Test nº 40, $BaCl_2$ was added in the Cleaning stages.

CLEANING with Ba Cl2	Na2SiO4	4 kg/t	NaCO3	5kg/t	NaOH	qb
	AERO 726	100 g/t	BaCl2	150g/t	pH	10,6

Test nº 40	26-06-2012			WO3		WO3 Cumulative		
Products	Weight, g	Weight, %	%W	%	Dist, %	%	Dist, %	F %
40CAF	115	7,95	4,35	5,486	83,67	5,49	83,67	3,2
40EAF	20	1,38	0,73	0,921	2,44	4,81	86,11	
40EA	40	2,77	0,17	0,214	1,14	3,76	87,25	
40E	1271	87,90	0,06	0,076	12,75	0,521	100,00	
Total	1446,00	100,00	0,413	0,521	100,00			

Comments: results not as good as the previous one in what concerns WO_3 Grade, however with much better Recovery.

When Phosphates and $BaCl_2$ depressants were added to the Cleaning stage, some improvements were achieved when comparing with the standard test:

- The best Grade 13,37% WO_3 was attained in the Concentrate 39CA with phosphate addition, although with a lower Recovery (44,14%);
- The use of $BaCl_2$ in Test nº 40, led to a Recovery of 83,7% in the 2nd Cleaning stage, although in detriment of the Grade obtained (5,49%WO_3);
- Results for Na_2SiF_6 are not reported because they were not suitable;
- Quebracho was not used as depressant in the Roughing stage, because this set of tests aimed at studying the performance other depressants.

Finally, an attempt was made when carrying out Test nº 41 in the same conditions as those of Test nº 39, except for the pulp pH, which was decreased to a value of 9,5, to avoid a further addition of NaOH as pH regulator.

CLEANING with PHOSPHATE	Na2SiO4	4 kg/t	NaCO3	5kg/t	NaOH	---
	AERO 726	100 g/t	Phosfate	150g/t	pH	9,5

Test nº 41	11-07-2012			WO3		WO3 Cumulative		
Products	Weight, g	Weight, %	%W	%	Dist, %	%	Dist, %	F %
41CAF	23	1,59	7,96	10,038	33,45	10,04	33,45	9,2
41EAF	77	5,31	3,26	4,111	45,87	5,47	79,32	
41EA	55	3,79	0,41	0,517	4,12	3,72	83,44	
41E	1295	89,31	0,07	0,088	16,56	0,476	100,00	
Total	1450,00	100,00	0,377	0,476	100,00			

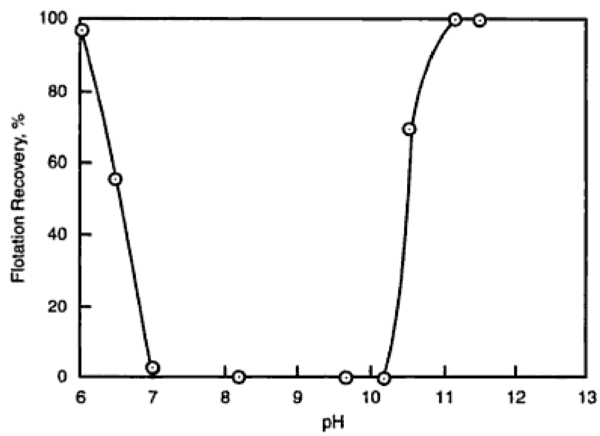
Comments: results showed a significant decrease in both Grade (10,04%WO_3), and Recovery (33,45%) when comparing with Test nº 39. Although Grade is greater than that obtained in standard Test nº 38 and Test nº 40, the Recovery was the worst.

Final remarks on this set of tests:

- The use of depressants in the Cleaning stage seems to be a good practice. Phosphates are more selective than $BaCl_2$;
- Some results point to that a pulp pH ≈ 10,5 may be crucial;
- In what concerns Fluorite Depression, the influence of the use of Phosphate or $BaCl_2$ in the Cleaning stages makes for a slight decrease of Fluorite / Scheelite ratio to values below 1 (see XRD spectra in Annex 1), whilst in Test nº 38, and Nºs 6 and 9 reported before, those ratios fall in the range [1,2 … 2];
- Meanwhile, results inscribed in the above tables, show that it was not possible to depress Fluorite effectively, even in the Cleaner Concentrate of Test 39, otherwise more Scheelite would be assayed in the concentrates;
- The influence on Calcite Depression is difficult to evaluate, but it is very little because Calcite is always the dominant mineral in the concentrates and so, the major responsible for the low WO_3 Concentrate Grade;

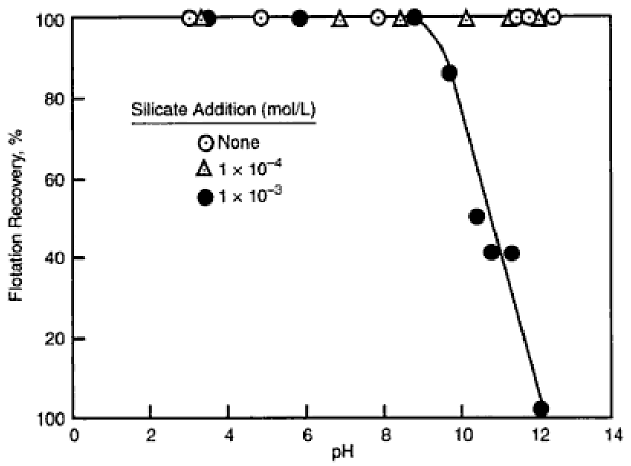
7.2.3 – Procedures proposed by FUERSTENAU

Based on Fuersteneau considerations, referred to in the text book “Principles of Mineral Processing”, there will be a Calcite/Fluorite selectivity band, in a pulp with $5x10^{-4}$ mol/L concentration of both Sodium silicate (dispersant/depressant) and sodium oleate (collector) as shown in the following two figures.



Source: Fuerstenau, Gutierrez, and Elgillani 1968.

FIGURE 8.43 Flotation recovery of calcite as a function of pH with 5×10^{-4} mol/L oleate and 5×10^{-4} mol/L sodium silicate (Type N)



Source: Fuerstenau, Gutierrez, and Elgillani 1968.

FIGURE 8.44 Flotation recovery of fluorite as a function of pH with 1×10^{-4} mol/L oleate in the absence and presence of sodium silicate (Type N)

Between pH=7 and pH=10, Calcite tends not to float, whereas the floatability of Fluorite only decreases for pH values > 9,5.

Tests were carried out in these conditions but the results achieved were far beyond expectations, with very poor grades being attained as it is shown by Test nº 46 - concentrate grade of 1,21%WO_3 ⇔ 88,63% Recovery.

Na2SiO4	$5x10^{-4}$ mol/L	NaCO3	---	NaOH	---
AERO 726	$5x10^{-4}$ mol/L	Quebracho	---	pH	8,0

	Test nº 46	31-07-2012			WO3		WO3 Cumulative	
Time (min)	Products	Weight, g	Weight, %	%W	%	Dist, %	%	Dist, %
3	46C1	226	32,75	0,96	1,211	88,63	1,21	88,63
	46AF	464	67,25	0,06	0,076	11,37	0,45	100,00
	Total	690,00	100,00	0,355	0,447	100,00		

Comments: consistent Froth, but very low selectivity.

As this preliminary result was not promising, it was decided to fit reagents dosages proposed by Fuersteneau to our former experience, i.e., to duplicate the sodium silicate dosage and to reduce to ¼ the collector dosage.

Envisaging a better behavior, this new test was planned with a Roughing stage at pH=8 (domain of Calcite Depression) followed by a Cleaning stage at pH=11 (domain of Fluorite Depression).

CLEANING	Na2SiO4	$1x10^{-3}$ ml/L	NaCO3	---	NaOH	qb
	AERO 726	$1,25x10^{-4}$ mol/L	Quebracho	---	pH	see below

Test nº 48	01-08-2012			WO3		WO3 Cumulative		
Products	Weight, g	Weight, %	%W	%	Dist, %	%	Dist, %	pH
48CA	78	10,82	1,52	1,917	47,32	1,92	47,32	11,00
48EA	55	7,63	1,01	1,274	22,17	1,65	69,49	
48E	588	81,55	0,13	0,164	30,51	0,438	100,00	8,00
Total	721,00	100,00	0,348	0,438	100,00			

Note
$1x10^{-3}$ $Na_2SiO_3 \approx 400$g/t
$1,25x10^{-4}$ mol/L $\approx$ 100g/t

Comments: even with the adjustment in reagent dosages, poor Concentrate Grades and Recoveries did not confirm the expectations based on the procedures proposed by Fuersteneau.

7.3 – Influence of Mine Water

In order to check the influence of Mine Water on the flotation results, it was decided to carry out two Rougher tests: Test nº 43 with Mine Water and Test nº 44 with Tap Water under the same conditions, varying only water origin/quality.

These tests were performed at pH=9,5, different from the value 10,5 defined as the most appropriate, because it was decided to avoid the use of any electrolyte as regulator.

MINE WATER	Na2SiO4	4 kg/t	NaCO3	5 kg/t	NaOH	---
	AERO 726	100g/t	Quebracho	---	pH	9,5

	Test nº 43	19-07-2012			WO3		WO3 Cumulative	
Time (min)	Products	Weight, g	Weight, %	%W	%	Dist, %	%	Dist, %
1	43C	23	1,56	5,00	6,305	26,43	6,31	26,43
	43E	1455	98,44	0,22	0,277	73,57	0,37	100,00
	Total	1478,00	100,00	0,294	0,371	100,00		

TAP WATER	Na2SiO4	4 kg/t	NaCO3	5 kg/t	NaOH	---
	AERO 726	100g/t	Quebracho	---	pH	9,5

	Test nº 44	20-07-2012			WO3		WO3 Cumulative	
Time (min)	Products	Weight, g	Weight, %	%W	%	Dist, %	%	Dist, %
1	44C	38	5,27	4,23	5,334	66,23	5,33	66,23
	44E	683	94,73	0,12	0,151	33,77	0,42	100,00
	Total	721,00	100,00	0,337	0,425	100,00		

Comments: it seems that for the same order of magnitude of Concentrate Grades, the Recovery dropped dramatically when Mine Water was used!
Although this result needs to be confirmed in further work, it should be taken into consideration.

7.4 – Froth stability in the presence of Sodium Carbonate

Since de beginning of this experimental research work, Sodium Silicate (water glass) and Sodium Carbonate were always used. The first acts as silicates depressant and dispersant and the second both as activator and Calcite depressant.

Flotation was always very fast, which led to the process instability and often the froth phase collapsed suddenly. As a result, it was difficult to reproduce some tests.

It should be underlined that Fuerstenau does not mention the addition of Sodium Carbonate.

Having in mind these considerations, tests were conducted in the absence of Sodium Carbonate or a much reduced addition. Broadly speaking, Froth stability increased significantly, but recovery decreases dramatically without a significant increase in Concentrate Grade, even in the Cleaning stages with Phosphate addition.

These results are shown for Test nº 52 in the table below.

	Na2SiO4	4 kg/t	NaCO3	2kg/t	NaOH+CaO	qb
CLEANING with PHOSPHATE	AERO 726	100 g/t	Phosfate	150g/t	pH	10,2

Test nº 52	**24-09-2012**			WO3		WO3 Cumulative	
Products	Weight, g	Weight, %	%W	%	Dist, %	%	Dist, %
52CAF	7	0,51	4,11	5,183	6,95	5,18	6,95
52EAF	27	1,95	2,32	2,926	15,13	3,39	22,08
52EA	92	6,64	0,77	0,971	17,11	1,62	39,19
52E	1259	90,90	0,20	0,252	60,81	0,377	100,00
Total	1385,00	100,00	0,299	0,377	100,00		

8. FINAL REMARKS

Before trying to delineate some relevant remarks about the results obtained in this research work, the LNEG team would like to express how challenging it was.

In fact, the proposed problem of recovering Scheelite from a very complex ore, with a mineralogical paragenesis in which the valuable mineral belongs to the same mineralogical groups (semi-soluble salts) as the main gangue minerals, occurring in tailings from a former treatment and in a very fine (ultra-fine) sizes with colloidal material, has revealed to be not trivial.

A huge amount of lab test work was done, as it can be appreciate in this Final Report.
However, some tests were not reported because they were used only to approach better lab conditions or the results showed that they were in the wrong way.

The complexity and diversity of the problem make difficult to synthetize very formal Final Conclusions.

However, the authors think that the following REMARKS could be presented:

- pH is very sensitive. The established value pH=10,5, although does not agree absolutely with bibliography, should be considered;
- Sodium Carbonate, although influencing Froth stability, its presence is fundamental to activate Scheelite, being difficult to evaluate its action as Calcite depressant;
- AERO 726 promoter proved to be the best collector tested;
- Phosphate addition during the Cleaning Stages showed to be a good practice;
- Concentrate Grades obtained, although below 15% WO_3 were in the same order of magnitude as those referred to in recent studies for the same type of Scheelite ores (Flotation Separation of Scheelite from Fluorite, Calcite and Quartz, by Watts, Griffis and McOuat Limited - "WGM");
- Finally, it should be said that all flotation tests were performed at summer temperature (because of its influence on oil type collector and on froth viscosity) which means that it was necessary to add more or less hot water when necessary.

9. ACKNOWLEDGMENTS

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31st October 2012

A. Botelho de Sousa M. Machado Leite

ANNEXES

- **Annex 1 – XRD Spectra of Feed Sample OL 380/2011 and some concentrate products**

- **Annex 2 - SIZE ANALYSES of Feed Samples OL 380/2011 and OL 33/2012 determined in a Laser COULTER LS130 Particle Size Analyser**

- **Annex 3 – W Assays by XRF**

- **Annex 4 - F Chemical ANALYSIS**

- **Annex 5 - Flotation Separation of Scheelite from Fluorite, Calcite and Quartz, by Watts, Griffis and McOuat Limited - "**WGM**"**

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XRD Spectra of Feed Sample OL 380/2011 and some concentrate products

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SIZE ANALYSES of Feed Samples OL 380/2011 and OL 33/2012, determined in a Laser Coulter LS 130 Particle Size Analyser

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ANNEX 5

Flotation Separation of Scheelite from Fluorite, Calcite and Quartz,
by Watts, Griffis and McOuat Limited – "WGM"