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## Characterization, classification, dry high intensity magnetic separation (DHIMS) and re-grinding techniques to improve the mineral performance of Sn-Ta-Nb mineral concentrates

Jennire Nava<sup>1,\*</sup>, Teresa Llorens<sup>1</sup>, and , Juan M. Menéndez-Aguado<sup>2</sup>

<sup>1</sup> Strategic Minerals Spain, Ctra/ OU-0901 Km 14, Penouta Mine, Viana do Bolo, 32558, Ourense, Galicia, Spain; [tllorens@strategicminerals.com](mailto:tllorens@strategicminerals.com);

<sup>2</sup> Escuela Politécnica de Mieres, Universidad de Oviedo.33600,Oviedo, Spain; [maguado@uniovi.es](mailto:maguado@uniovi.es).

\* Corresponding author: [jvanessanavar@gmail.com](mailto:jvanessanavar@gmail.com)



Universidad de Oviedo

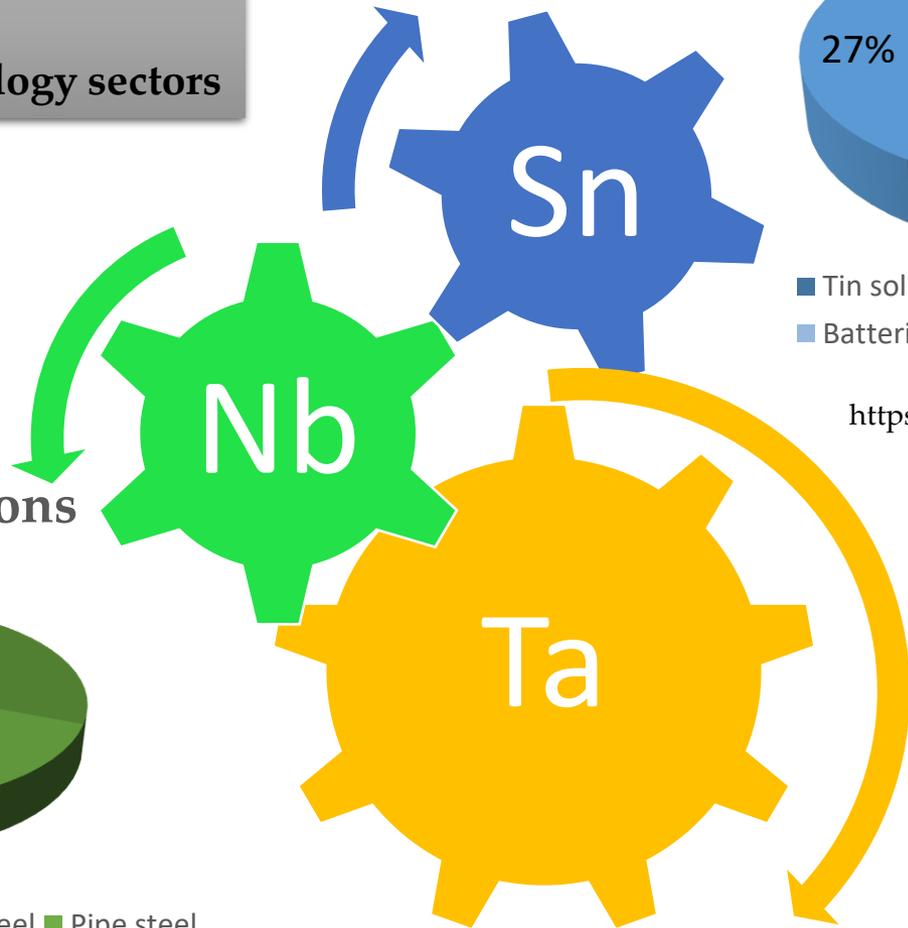


**Abstract:** Ta and Nb are considered critical raw materials; due to their properties and potential applications in wide sectors. This study deals with Sn-Ta-Nb minerals from the Penouta mine (Orense, Spain), the only active mine in Europe producing tantalum minerals. These are obtained from mining wastes accumulated during old mining jobs in tailing ponds. The industrial processing flowsheet is based on successive gravimetric stages followed by low intensity magnetic separation to reduce ferromagnetic contaminants. Sn-Ta-Nb concentrate, with grades between 35-45% Sn and 4-7% Ta<sub>2</sub>O<sub>5</sub> and Nb<sub>2</sub>O<sub>5</sub>, is obtained in this stage with plant recoveries around 60-70%, respectively. A chemical-mineralogical characterization by size fractions, XRF and XRD was carried out to implement a size classification stage using a circular vibrating screen in the processing plant. The finest fractions, containing higher grades of well liberated Sn, Ta, Nb minerals, were the feeding for dry high intensity magnetic separation (DHIMS) multifactorial tests, while, coarse fractions were re-grinded to maximize performance. The good results obtained in these tests demonstrate that two products with commercial quality could be obtained, a cassiterite concentrate with grades between 70-78% SnO<sub>2</sub> and a tantalite-columbite concentrate with grades ranging between 12 and 14% Ta<sub>2</sub>O<sub>5</sub> and Nb<sub>2</sub>O<sub>5</sub>, also increasing the overall recovery of the plant.

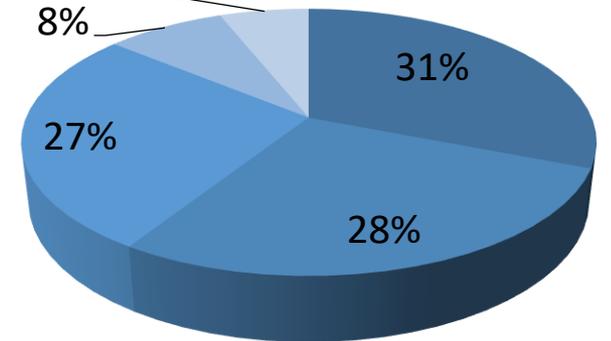
**Keywords:** high intensity magnetic separation; Sn-Ta-Nb; critical raw materials; Penouta mine.

# Introduction

Sn, Ta, Nb are strategic metals for the EU with important applications in technology sectors



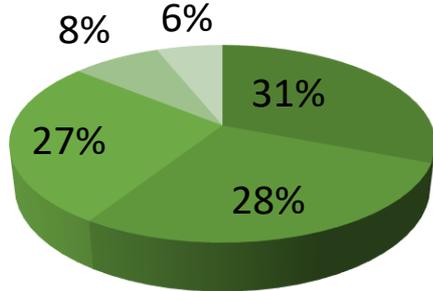
## Sn Applications



- Tin solder
- Chemical
- Tinplate
- Batteries
- Cu alloys

<https://roskill.com/market-report/tin/>  
(10/25/20)

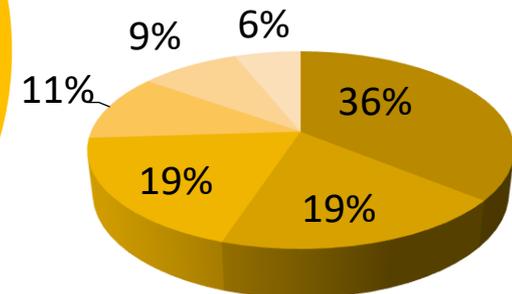
## Nb Applications



- Structural steel
- Automotive steel
- Pipe steel
- Superalloys
- Chemistry

Roskill (2016)

## Ta Applications



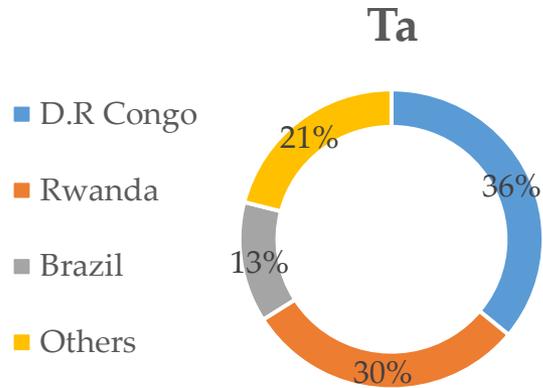
- Capacitor
- Chemical Industry
- Others
- Grinding Products
- Ingot
- Carbides

Consulting, C (2018)

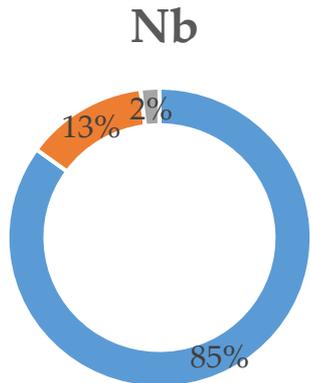
**Tantalite-columbite contains Ta and Nb**

# Introduction

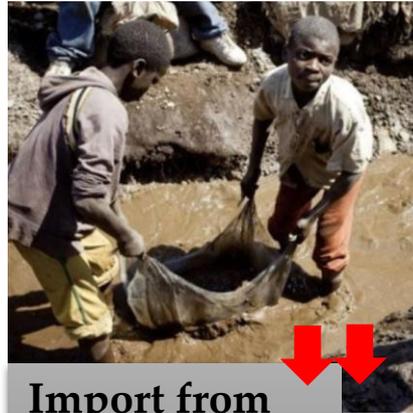
## Origin of Ta and Nb imports in the EU



To decrease import dependence



(EC, 2020)



Import from conflict zones



Investigation of CRM deposits and processes in Europe



Dodd-Frank Law



Regulation of the European Parliament



Grinding-Classification  
Circuit Stage

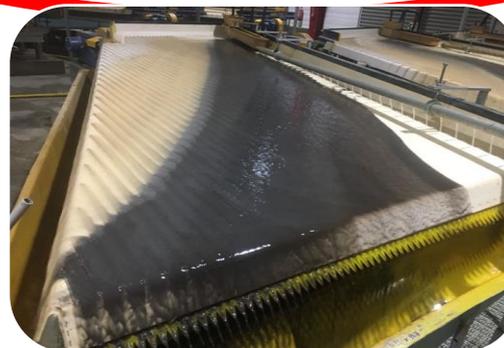
Wet Low Intensity  
Magnetic Separation Stage



The Penouta mine (Orense, Spain),  
the only tantalite active mine in  
Europe



35-45% Sn and 4-7% Ta<sub>2</sub>O<sub>5</sub>  
and Nb<sub>2</sub>O<sub>5</sub>



Gravimetric Concentration Stages



# Objective



35-45% Sn and 4-7% Ta<sub>2</sub>O<sub>5</sub>  
and Nb<sub>2</sub>O<sub>5</sub>



¿ How to obtain independent concentrates of Sn and Ta-Nb that could be more competitive at the commercial level and thus contribute to an increasing yields in the industrial plant of the Penouta mine?

Sn concentrate

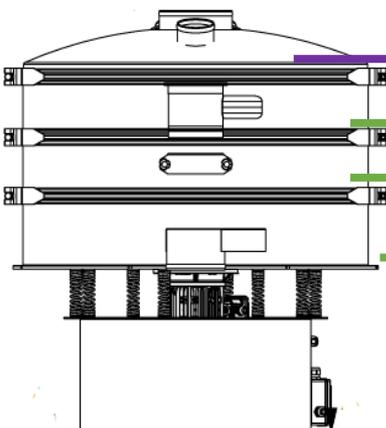


Nb-Ta concentrate

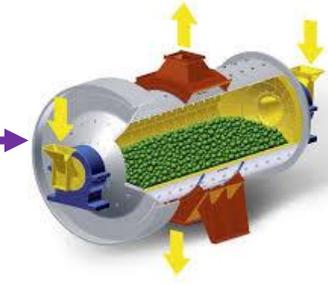


# Materials and Methods

Characterization



Classification and  
Assay by Size (XRF)



Re-grinding



DHIMS test work



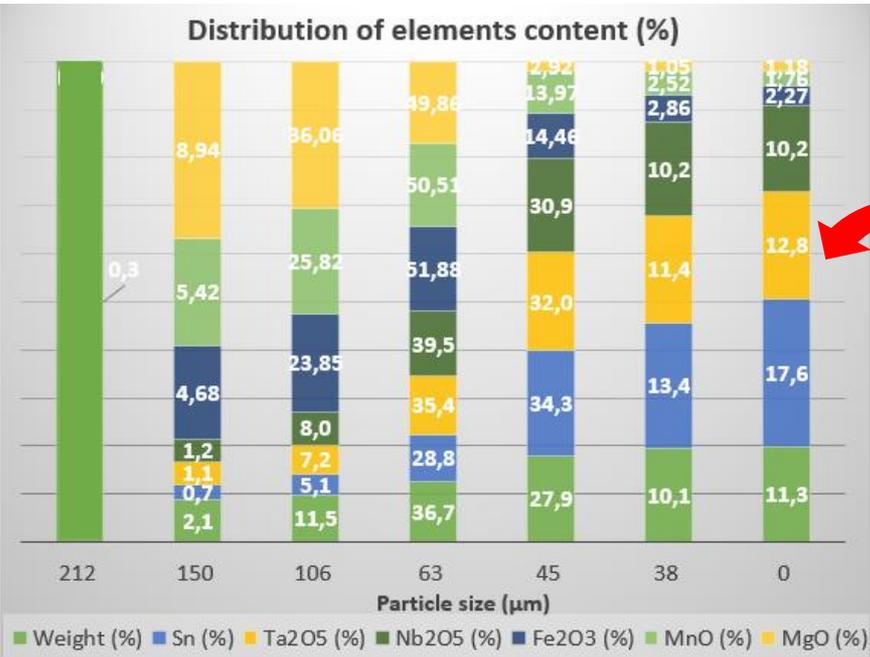
# Results and Discussion

Size ( $\mu\text{m}$ )	Retained weight		Accumulated weight (%)	Grades (%)						
	(g)	(%)		Sn	Ta <sub>2</sub> O <sub>5</sub>	Nb <sub>2</sub> O <sub>5</sub>	Fe <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
300	0.00	0.00	100.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
212	3.30	0.28	99.2	0.00	0.00	0.00	0.00	0.00	0.00	0.00
150	25.30	2.14	97.58	11.80	2.77	2.58	13.30	14.20	23.50	20.40
106	136.10	11.51	86.07	14.85	3.30	3.23	12.60	12.75	20.80	17.55
63	434.30	36.74	49.32	26.50	5.05	4.99	8.59	7.32	12.75	13.40
45	330.10	27.93	21.40	41.50	6.06	5.13	3.15	1.80	4.64	7.19
38	118.90	10.06	11.34	45.10	5.94	4.69	1.73	0.58	2.32	6.30
0	134.00	11.34	0.00	52.30	5.93	4.19	1.22	0.11	1.44	4.99
<b>Total:</b>	1182.00	100.00	<b>Calculated feed grade:</b>	33.75	5.25	4.64	6.08	5.04	9.28	10.59

*D<sub>50</sub>* ( $\mu\text{m}$ ): 99

**Feed:**  
Sn-Ta-Nb concentrate

33.75% Sn, 5.24% Ta, 4.64% Nb



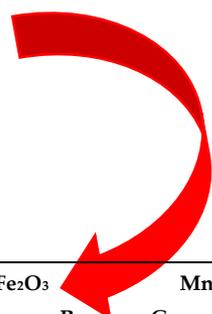
Increasing Sn, Ta and Nb contents with decreasing size particle. Highest concentrations below 106  $\mu\text{m}$ .

## DHIMS multifactorial assays (XRF)

Test Nº	Size fraction (µm)	Magnetic field intensity (A)	Roll speed (rpm)
1	150/0	Low	High
2	150/90	Low	High
3	150/90	High	High
4	150/90	High	Low
5	150/90	Low	Low
6	150/90	Changing split inclination	
7	90/0	Low	High
8	90/0	High	High
9	90/0	High	Low
10	90/0	Low	Low
11	90/0	Changing split inclination	

### DHIMS assays configuration.

(Due to SMS data protection, they are considered qualitatively)



Mineral	Ta <sub>2</sub> O <sub>5</sub>		SnO <sub>2</sub>		Nb <sub>2</sub> O <sub>5</sub>		SiO <sub>2</sub>		Fe <sub>2</sub> O <sub>3</sub>		MnO	
	Test nº	G (%)	R (%)	G (%)	R (%)	G (%)	R (%)	G (%)	R (%)	G (%)	R (%)	
1	12.20	57.62	7.10	2.95	12.65	76.76	8.97	30.87	8.74	65.87	12.71	79.18
2	10.33	68.18	3.34	2.09	11.10	89.39	13.44	52.81	12.01	83.44	17.36	96.14
3	10.15	68.09	3.33	2.11	11.02	88.59	13.83	55.02	12.13	83.38	17.51	95.22
4	8.80	69.82	26.99	19.18	8.79	85.61	9.41	49.87	7.62	78.34	10.84	89.55
5	8.79	68.96	28.09	19.82	8.78	85.97	9.31	50.78	7.50	78.38	10.66	90.97
6	11.71	70.11	7.29	3.87	12.21	91.88	10.13	42.86	9.47	80.89	13.86	97.86
7	13.66	67.78	10.09	4.23	13.21	89.42	6.30	25.49	6.83	71.40	10.04	92.31
8	13.47	67.80	10.77	4.61	13.61	90.95	6.15	24.76	6.65	70.24	9.75	91.11
9	8.89	76.07	39.01	28.59	8.23	91.63	5.47	36.89	4.37	76.75	6.01	94.01
10	9.03	76.34	37.93	26.69	8.42	94.08	5.38	35.81	4.38	77.93	6.03	95.78
11	13.13	68.24	13.65	5.93	13.24	91.15	5.36	22.51	5.89	69.52	8.69	90.58

### Paramagnetic Concentrate (Tantalite-columbite)

Test 6: 150/90 µm fraction. The best grades and recovery.

Test 7: <90 µm fraction. The best grades.

Test 10: <90 µm fraction. The best recovery.

- ✓ Tantalite has a well-liberated size below 100 µm.
- ✓ Granulometric size influencing both the increase of the grades and the yields of the species.
- ✓ Higher content of SiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub> and MnO in coarser fraction.

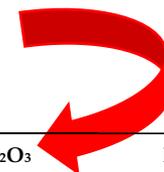
## DHIMS multifactorial assays (XRF)

Test Nº	Size fraction (µm)	Magnetic field intensity (A)	Roll speed (rpm)
1	150/0	Low	High
2	150/90	Low	High
3	150/90	High	High
4	150/90	High	Low
5	150/90	Low	Low
6	150/90	Changing split inclination	
7	90/0	Low	High
8	90/0	High	High
9	90/0	High	Low
10	90/0	Low	Low
11	90/0	Changing split inclination	

↑ Roller speed → greater recoveries

↓ Particle size → greater intensity is necessary

↑ The selectivity, when the feed is classified in two fractions



Mineral	Ta <sub>2</sub> O <sub>5</sub>		SnO <sub>2</sub>		Nb <sub>2</sub> O <sub>5</sub>		SiO <sub>2</sub>		Fe <sub>2</sub> O <sub>3</sub>		MnO	
	G (%)	R (%)	G (%)	R (%)	G (%)	R (%)	G (%)	R (%)	G (%)	R (%)	G (%)	R (%)
1	1.84	27.12	75.06	97.51	0.41	7.78	6.56	70.89	0.62	14.61	0.13	2.53
2	2.01	26.37	75.57	93.73	0.58	9.25	6.36	49.62	0.84	11.59	0.29	3.19
3	1.95	25.00	76.96	93.40	0.49	7.50	6.34	48.28	0.72	9.47	0.21	2.19
4	2.13	24.93	73.91	77.48	0.78	11.18	6.56	51.28	0.88	13.35	0.51	6.21
5	2.15	25.13	73.91	77.61	0.80	11.67	6.58	53.42	0.86	13.37	0.52	6.61
6	1.89	25.76	78.23	94.39	0.41	7.07	6.27	60.37	0.61	11.86	0.12	1.93
7	1.85	26.81	76.58	93.96	0.37	7.13	6.67	78.87	0.57	17.43	0.11	2.96
8	1.85	26.93	77.34	95.57	0.38	7.26	6.70	77.81	0.58	17.67	0.11	2.97
9	1.65	18.67	74.17	71.30	0.35	5.12	7.52	67.25	0.53	12.35	0.11	2.28
10	1.65	19.17	74.68	72.33	0.34	5.21	7.63	69.90	0.54	13.23	0.10	2.19
11	1.79	26.26	75.57	92.72	0.36	6.96	6.73	79.90	0.55	18.36	0.10	2.95

### No magnetic Concentrate (Cassiterite)

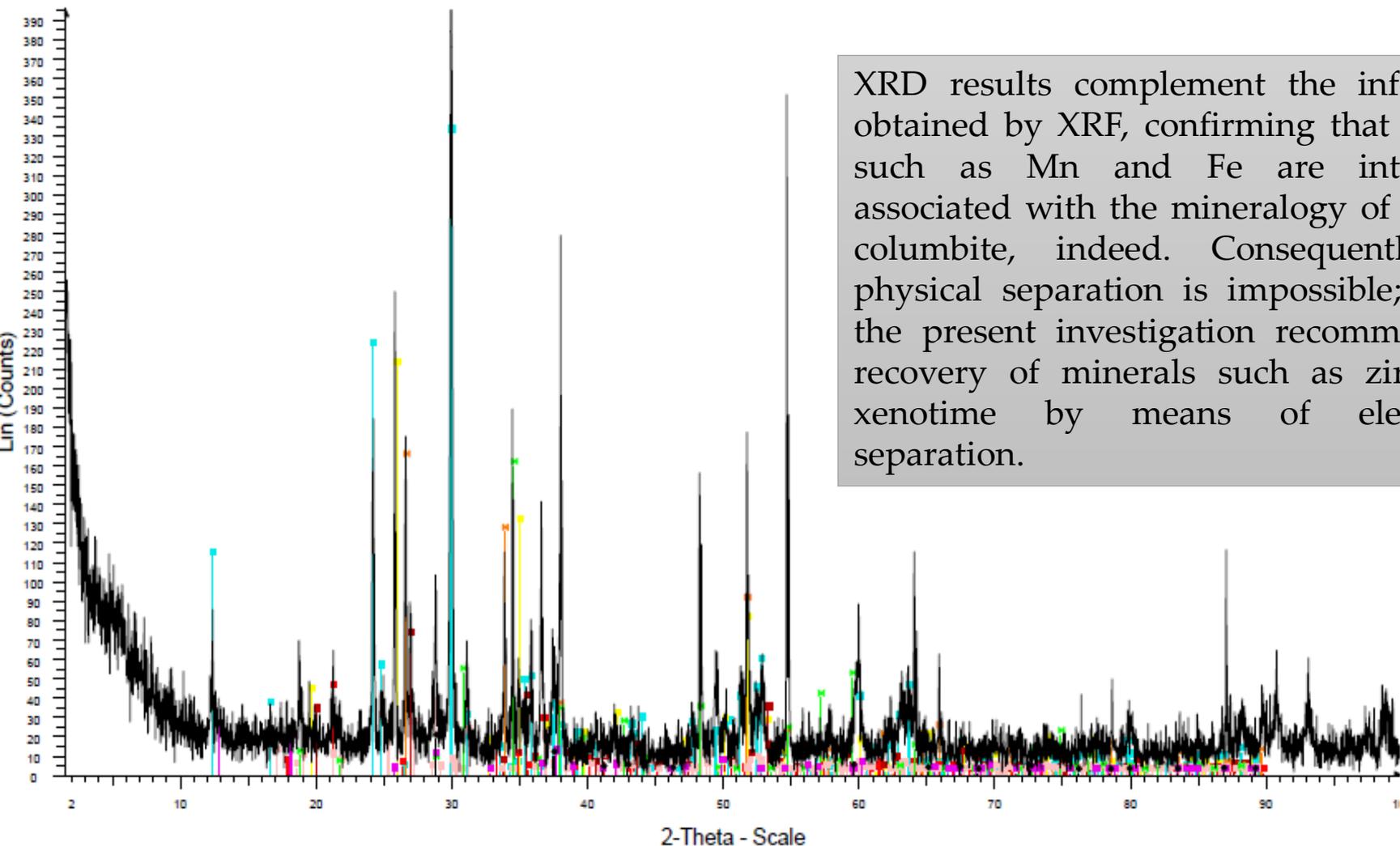
Test 6: 150/90 µm fraction. The best grades and recovery.

Test 8: <90 µm fraction. The best grades and recovery.

Finally, the **test 6** is the optimal configuration to treat the **150/90 µm** fraction, since it shows the best results for both grades and yields of SnO<sub>2</sub> and Ta<sub>2</sub>O<sub>5</sub>.  
The fraction <90 µm through the configuration of the **test 7**.

# Results and Discussion

## Tantalite-columbite concentrate <90 μm (XRD)



XRD results complement the information obtained by XRF, confirming that elements such as Mn and Fe are intrinsically associated with the mineralogy of tantalite-columbite, indeed. Consequently, their physical separation is impossible; despite, the present investigation recommends the recovery of minerals such as zircon and xenotime by means of electrostatic separation.

CT-90um - File: CT-90um final.raw - Type: 2Th/Th locked - Start: 1.500 ° - End: 100.000 ° - Step: 0.020 ° - Step Operations: Import

- 01-071-1807 (C) - Tantalite - S-Q 22.6 % - Mn.97(Ta.64Nb.36)2O6 - Y: 83.76 % - d x by: 1. - WL: 1.5406 - Orth
- 01-074-2429 (C) - Xenotime - S-Q 19.6 % - YPO4 - Y: 53.32 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 6.8780
- 01-077-0449 (C) - Cassiterite, syn - S-Q 7.7 % - SnO2 - Y: 41.27 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 4.7
- 01-074-2195 (C) - Goethite, syn - S-Q 7.5 % - FeOOD - Y: 11.09 % - d x by: 1. - WL: 1.5406 - Orthorhombic - a
- 01-080-1807 (C) - Zircon, syn - S-Q 6.5 % - Zr(SiO4) - Y: 17.88 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 6.62
- 01-085-1457 (C) - Monazite - from India, Kerala Beach Sands - S-Q 4.8 % - CePO4 - Y: 8.97 % - d x by: 1. - WL

- 01-076-0867 (C) - Spessartine - S-Q 26.5 % - Mn2.6Fe0.4Al2Si3O12 - Y: 40.25 % - d x by: 1. - WL: 1.5406 - Cu
- 01-072-1982 (C) - Cryptomelane - S-Q 3.0 % - MnO2 - Y: 5.77 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 9.81
- 01-072-1984 (C) - Pyrolusite - S-Q 2.0 % - MnO2 - Y: 4.47 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 4.38800 -

## Results and Discussion-Regrinding of >150 $\mu\text{m}$ fraction.

The assay by size of the resulting products passing the 100  $\mu\text{m}$  mesh, after regrinding of the >150  $\mu\text{m}$  fraction for each grinding time (0.5, 2.5, 5, 10 and 15 min)

Time (min)	Pass weight through	P <sub>80</sub> ( $\mu\text{m}$ )	Distribution of metal content (%)						
	100 $\mu\text{m}$ (%)		Sn	Ta <sub>2</sub> O <sub>5</sub>	Nb <sub>2</sub> O <sub>5</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
0.5	11.20	218	26.26	21.07	20.44	5.59	7.45	5.45	7.13
2.5	17.60	197	38.25	30.81	29.66	8.77	11.67	8.91	11.39
5.00	22.40	192	43.74	36.57	34.43	12.93	16.53	13.13	15.60
10.0	30.90	185	51.09	46.81	43.13	20.75	26.47	22.14	23.05
15.00	32.90	184	50.39	47.47	43.93	24.44	29.89	25.54	26.49

- ✓ As grinding time increase, the amount of mineral ground through the 100  $\mu\text{m}$  mesh increase.
- ✓ The distribution of the metal content in the ground product through the 100  $\mu\text{m}$  mesh after the milling time has elapsed, shows an increase, reaching a well-liberated of at least 50% in the species of interest such as Sn, Ta and Nb after 10 min to avoid regrinding of fines and loss of energy efficiency.

# Conclusions

- ✓ Implementing a size classification, better grades and recoveries of Sn, Ta and Nb are obtained when performing DHIMS instead of using a wide granulometric interval (150/0  $\mu\text{m}$ ), since selectivity increases during operation, and for this reason, the fraction  $>150 \mu\text{m}$  is also regrinded.
- ✓ The multifactorial DHIMS test works on the Sn, Ta and Nb primary concentrate generate two new products, a magnetic columbo-tantalite concentrate and a non-magnetic cassiterite concentrate with better grades and feasibility for the Penouta mine project.
- ✓ The XRF and XRD techniques combined with size fraction analysis were key for the characterization of both the feeds and the products obtained in all the test works carried out.
- ✓ The present work proposes to achieve electrostatic separation multifactorial tests to cut down on minerals such as zircon, monazite and xenotime and to purify the paramagnetic and non-magnetic concentrates obtained.

## Acknowledgments

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Universidad de Oviedo



thanks for your attention...

Jennire Vanessa Nava Rosario  
Metallurgical engineer  
Email: [jvanessanavar@gmail.com](mailto:jvanessanavar@gmail.com)