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Quantification of Nb, Fe, Si and Ti in columbite enriched ore samples by LIBS

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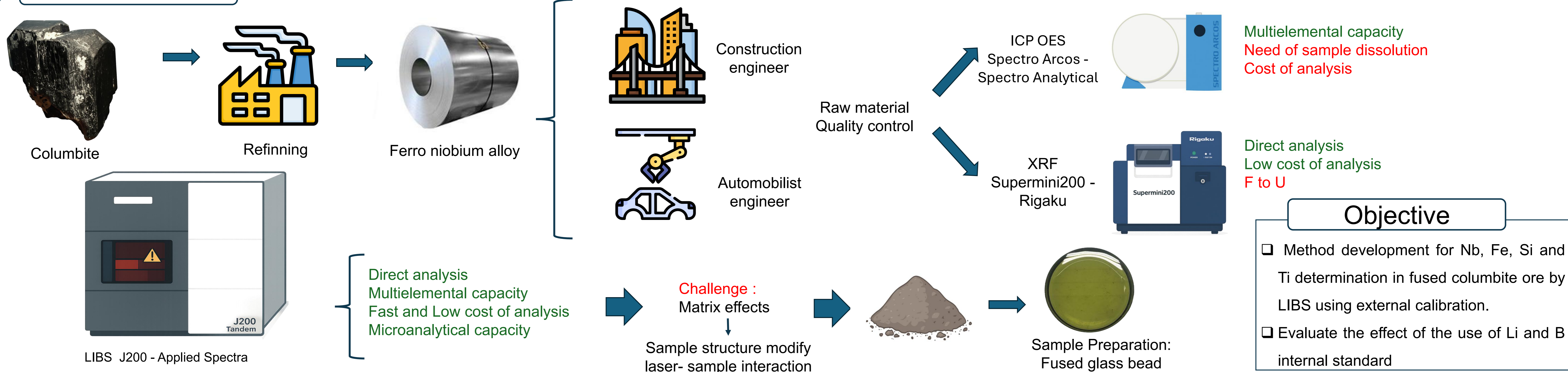
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Introduction



Objective

- Method development for Nb, Fe, Si and Ti determination in fused columbite ore by LIBS using external calibration.
- Evaluate the effect of the use of Li and B internal standard

Methodology

Sample	Nb	Fe	Si	Ti
1	28.7	4.35	6.04	0.423
2	24.3	6.17	4.96	*
3	24.5	5.71	4.81	0.437
4	29.6	6.87	3.29	0.565
5	26.5	8.28	3.73	0.482

*Value not reported

Instrumental conditions		
Temperature	Ramp time	Time
0-1000 °C	4 min	6 min
1000-1150 °C	0.5 min	10 min

Instrumental conditions	
Spot size	65 µm
Laser energy	20 mJ
Delay time	0.5 µs
Argon flow rate	1.0 L min ⁻¹
Pulses	500

Element	Wavelength (nm)
Nb	309.390
Fe	275.613
Si	230.306
Ti	308.731
Li (IS)	670.776
B (IS)	280.478

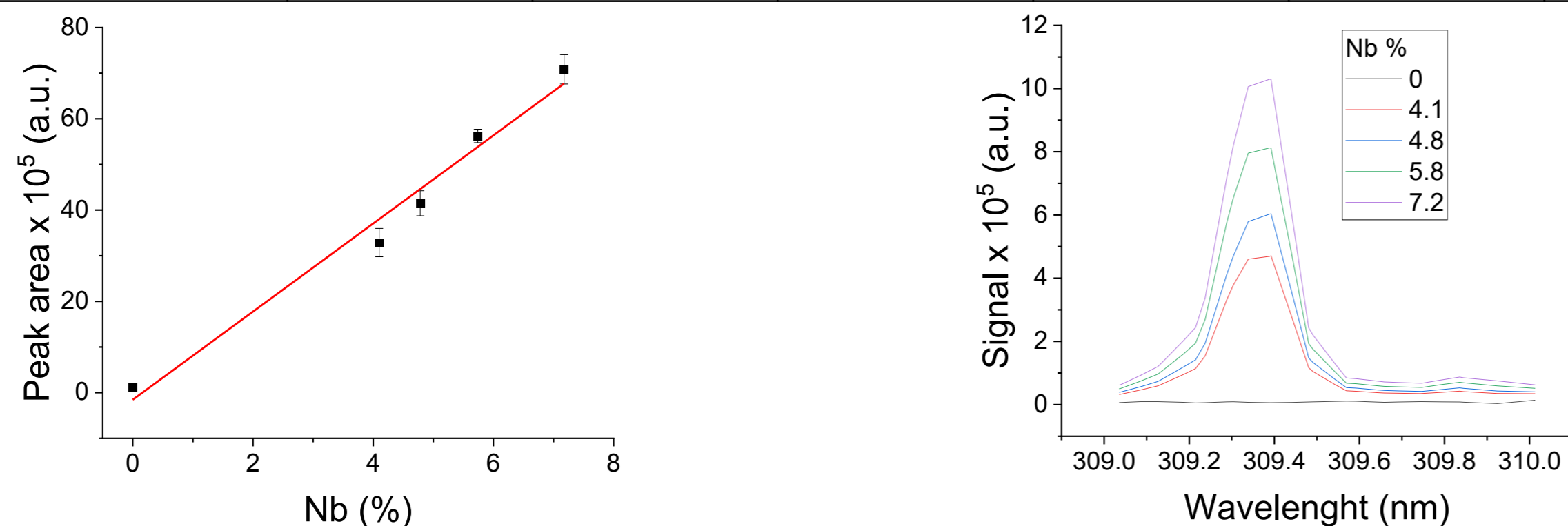
- Calibration curve proportions varying from 1:2 to 1:7 of standard: flux mixture
- Samples: prepared in proportion of 1:3 of sample: flux mixture

Results

Note: R.E. = relative error

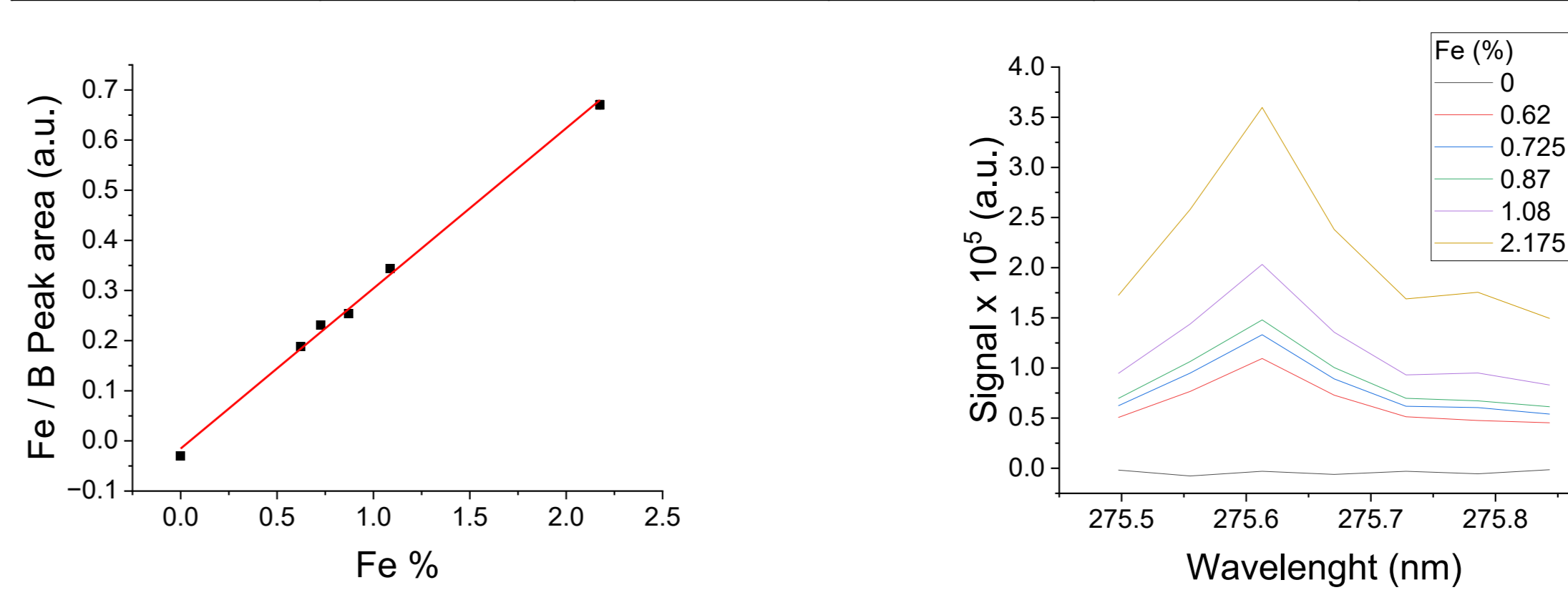
Nb

Sample	Mass fraction (%) w/w					
	Without IS		Li IS		B IS	
LOD	0.04	0.06	0.96			
LOQ	0.15	0.20	0.32			
Slope	9.65 x 10 ⁵	19.8 x 10 ⁻³	0.451			
Intercept	-1.53 x 10 ⁵	-6.89 x 10 ⁻³	0.031			
R ²	0.9785	0.9375	0.9769			
Sample	Mean ± SD	R.E. (%)	Mean ± SD	R.E. (%)	Mean ± SD	R.E. (%)
1	27 ± 2	6	26 ± 3	7	28 ± 2	1
2	28 ± 2	-16	30 ± 3	-26	28 ± 2	-16
3	28 ± 7	-16	24 ± 8	0	32 ± 8	-30
4	33 ± 1	-12	35 ± 1	-19	31.9 ± 0.6	-8
5	24 ± 2	10	22 ± 3	18	27 ± 2	-1



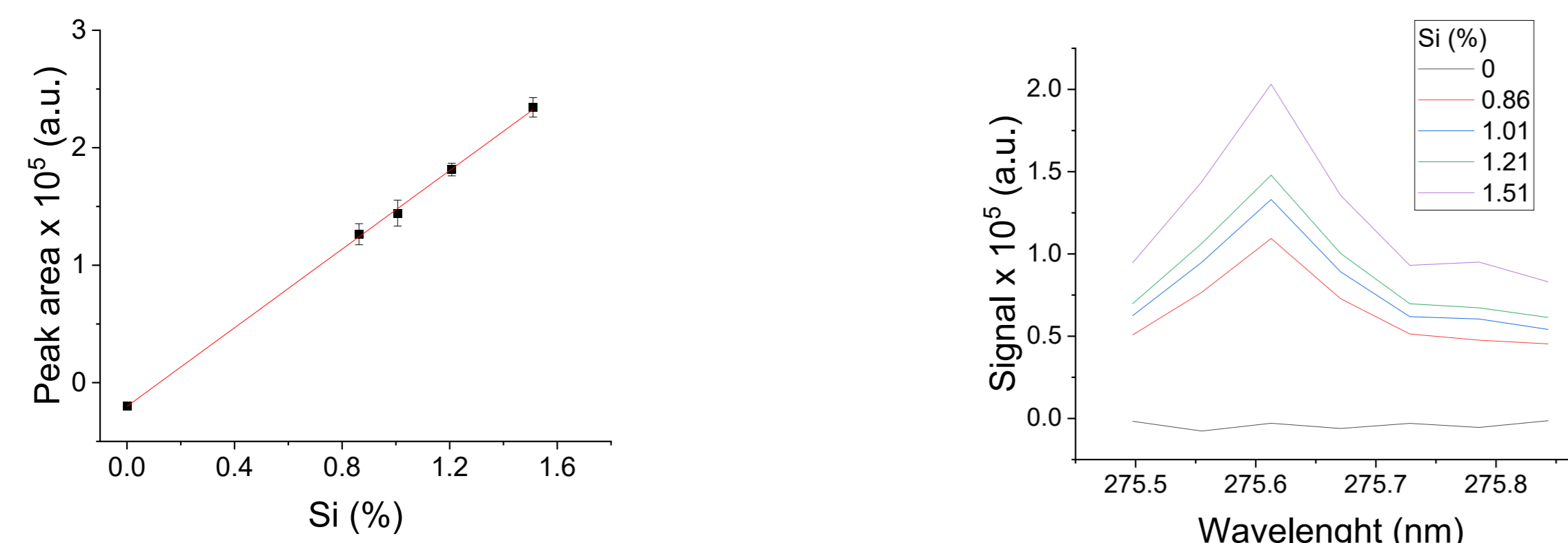
Fe

Sample	Mass fraction (%) w/w					
	Without IS		Li IS		B IS	
LOD	0.015	1.06	0.04			
LOQ	0.05	3.54	0.14			
Slope	0.163 x 10 ⁵	12.1 x 10 ⁻³	0.319			
Intercept	-5.85 x 10 ⁵	-0.208 x 10 ⁻³	-0.015			
R ²	0.9932	0.9888	0.9971			
Sample	Mean ± SD	R.E. (%)	Mean ± SD	R.E. (%)	Mean ± SD	R.E. (%)
1	4.7 ± 0.1	-8	4.3 ± 0.3	0	4.57 ± 0.08	-5
2	6.26 ± 0.06	-1	6.4 ± 0.3	-4	5.72 ± 0.05	7
3	4.9 ± 0.2	14	3.9 ± 0.6	32	5.09 ± 0.01	11
4	7.0 ± 0.1	-2	7.0 ± 0.2	-2	6.18 ± 0.07	10
5	6.4 ± 0.2	23	5.3 ± 0.5	35	6.6 ± 0.2	20



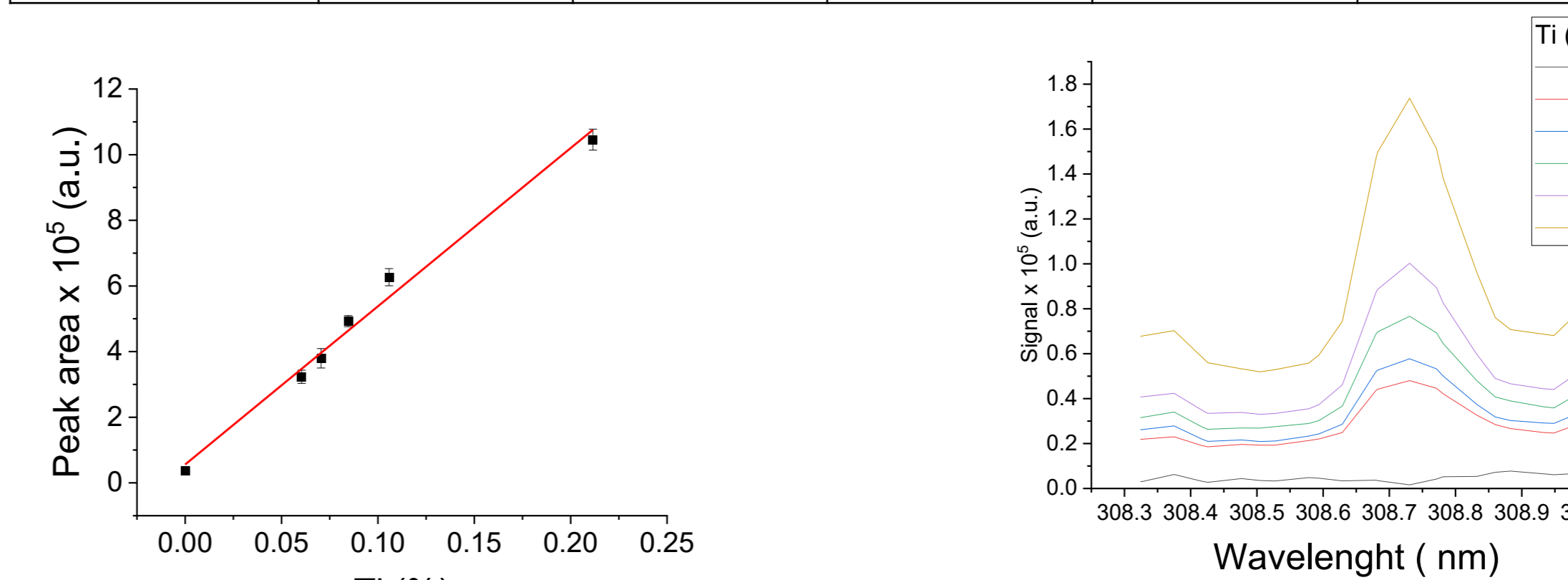
Si

Sample	Mass fraction (%) w/w					
	Without IS		Li IS		B IS	
LOD	0.05	0.08	0.16			
LOQ	0.16	0.27	0.55			
Slope	1.67 x 10 ⁵	2.89 x 10 ⁻³	0.078			
Intercept	-0.201 x 10 ⁵	-0.166 x 10 ⁻³	-0.012			
R ²	0.99932	0.9813	0.9856			
Sample	Mean ± SD	R.E. (%)	Mean ± SD	R.E. (%)	Mean ± SD	R.E. (%)
1	5.8 ± 0.1	5	6.3 ± 0.1	-4	6.26 ± 0.06	-4
2	4.78 ± 0.9	4	5.7 ± 0.1	-112	4.97 ± 0.06	0
3	4.3 ± 0.2	11	4.0 ± 0.6	-15	4.99 ± 0.09	-4
4	5.71 ± 0.6	-73	6.77 ± 0.01	16	5.74 ± 0.07	-74
5	4.23 ± 0.03	-14	4.2 ± 0.2	-13	4.94 ± 0.03	-33



Ti

Sample	Mass fraction (%) w/w					
	Without IS		Li IS		B IS	
LOD	0.003	0.04	1.1			
LOQ	0.01	0.13	3.5			
Slope	48.2 x 10 ⁵	9.158 x 10 ⁻³	0.239			
Intercept	-0.560 x 10 ⁵	0.731 x 10 ⁻³	0.031			
R ²	0.98835	0.9733	0.9925			
Sample	Mean ± SD	R.E. (%)	Mean ± SD	R.E. (%)	Mean ± SD	R.E. (%)
1	0.45 ± 0.03	-7	4.5 ± 0.4	-5	4.5 ± 0.2	-5
2	Value not reported		Value not reported		Value not reported	
3	0.56 ± 0.09	-28	5 ± 1	15	6.1 ± 0.9	-6
4	0.63 ± 0.01	-13	7.0 ± 0.2	-2	5.87 ± 0.08	15
5	0.49 ± 0.02	-3	4.5 ± 0.4	45	5.4 ± 0.2	35



Conclusion

- LIBS with fused glass beads allowed accurate quantification of Nb, Fe, Si, and Ti in columbite ore
- The use of Li as IS is highly unrecommended due to the decrease of accuracy
- The use of B as IS has showed effective only for Fe
- For other elements, the approach without IS is more recommended

References

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Acknowledgments

