Application note

EDXRF Analysis of Nickel Ore as Pressed Powders in an Air Environment

Nickel is produced from two very different ores, lateritic & sulfidic. Lateritic ore is found primarily in tropical countries such as Indonesia & mined from various depths beneath the surface while sulfidic ore is usually found in combination with copper ore and is mined underground.

Nickel production has seen increasing demand in recent times as nickel is a key component in lithium-ion batteries, used in electric vehicles. Hence, a fast, accurate & precise method is needed for quantification of these ores both in the mining and refining processes. X-ray fluorescence spectrometry (XRF) is a well-established analytical method to determine chemical composition in materials with high accuracy and minimum sample preparation therefore is a preferred technique in process and quality control across many industries.

Instrumentation

The Thermo Scientific™ ARL™ QUANT'X EDXRF Spectrometer is equipped with a Silicon Drift Detector (SDD) and a 50 Watt Rh or Ag target X-ray tube which is air cooled with a maximum excitation voltage of 50 kV. A set of nine primary beam filters is designed to optimize the peak-to-background signals for all elements from F to Am. The 10-position auto-sampler with spinner allows for unattended analysis of multiple samples. Its SDD remains the performance benchmark for all energy-dispersive detectors. The large active area of 30 mm^2 enables effective capture of characteristic element X-rays emitted by the sample.

Excitation Conditions

In EDXRF, sensitivity and precision are achieved by targeted excitation of the sample to fluoresce only the elements of interest. The ARL QUANT'X EDXRF Spectrometer offers a virtually unlimited combination of excitation voltages (4-50 kV) and multiple primary beam filters for optimal background control.

As shown in Table 1, spectra were collected on each nickel ore sample for a total live time of 5 minutes. Measurement time can be further fine-tuned according to specific applications. Analysis is conducted in air.

Condition	Voltage	Tube Filter	Medium	Live Time (s)	Elements
Low Za	4	No Filter	Air	180	Mg, Al, Si
Low Zb	8	C Thick	Air	60	S, Ca, K
Mid Za	16	Ag Thin	Air	60	Ti, Cr, Mn, Fe, Co, Ni, Cu, Zn

Table 1. Analytical conditions.

thermo scientific

Sample Preparation

Calibration was done using 17 nickel ore Certified Reference Materials (CRM) from OREAS®. Samples were pressed into pellets without binder at 20 tons. Table 2 shows the concentration ranges of the different oxides covered by the calibration as well as the R^2 and RMSE (root mean square error) values obtained for the different compounds.

Element	Min %	Max %	R^2	RMSE
MgO	0.7	27.3	0.9986	0.4
Al_2O_3	1.6	17.5	0.9985	0.2
SiO_2	22.8	48.0	0.9896	0.8
SO_3	0.03	0.19	0.9897	0.007
K_2O	0.069	0.228	0.9935	0.006
CaO	0.13	3.11	0.9995	0.019
TiO_2	0.02	1.36	0.9998	0.003
Cr_2O_3	0.17	1.75	0.9971	0.02
MnO	0.11	1.94	0.9997	0.008
Fe_2O_3	12.7	46.0	0.9986	0.4
Co	0.023	0.090	0.9839	0.003
Ni	0.05	2.94	0.9993	0.02
Cu	0.007	0.05	0.9999	0.00014
Zn	0.007	0.035	0.9968	0.0005

Table 2. Concentration ranges and calibration parameter values for the analysis of nickel ore.

Calibration

Calibration curves have been derived relating element characteristic X-ray intensity to oxide concentration. X-ray fluorescence measures elements, but the results can be related directly to the oxide forms of these elements when only one single form of oxide is present in the sample. Figures 1 to 14 show the calculated versus given concentration plots obtained for Ni, MgO, Al_2O_3, SiO_2, SO_3, K_2O, CaO, TiO_2, Cr_2O_3, MnO, Fe_2O_3, Co, Cu and Zn.

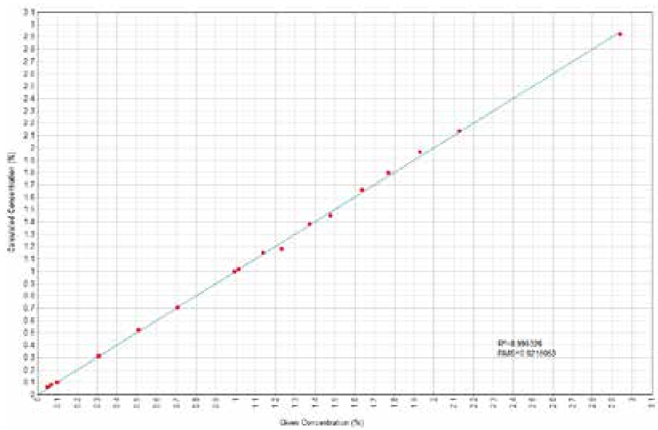

Figure 1. Ni Calculated versus Given Concentrations.

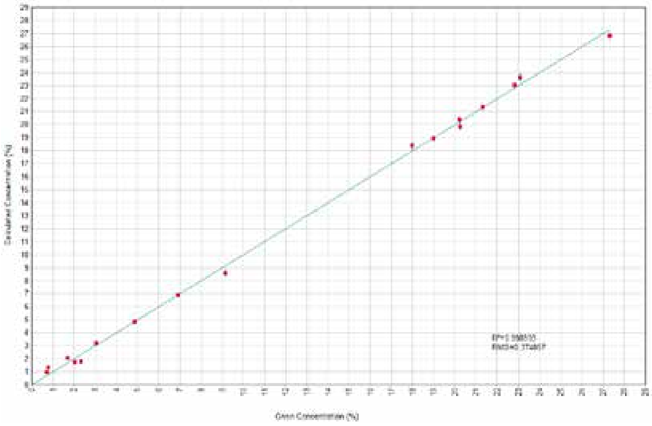

Figure 2. MgO Calculated versus Given Concentrations.

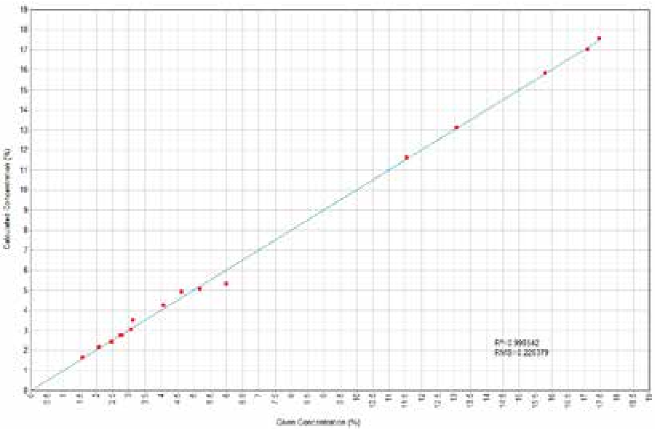

Figure 3. Al_2O_3 Calculated versus Given Concentrations.

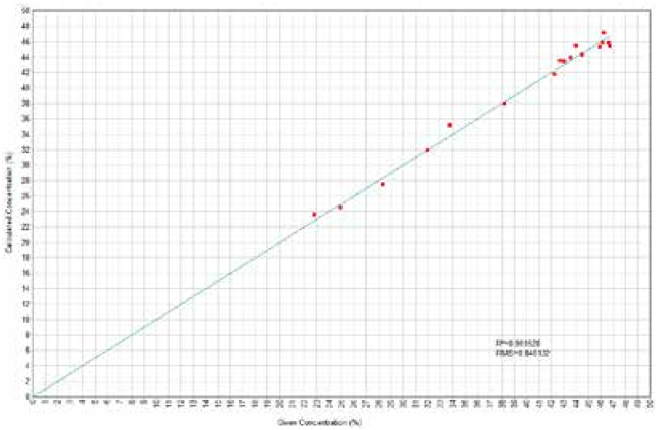

Figure 4. SiO_2 Calculated versus Given Concentrations.

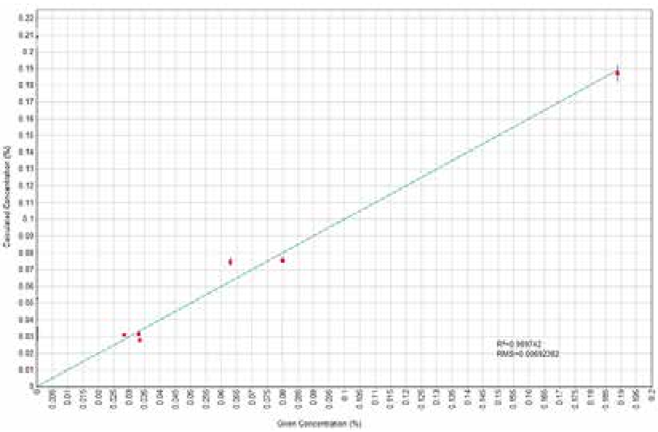

Figure 5. SO_3 Calculated versus Given Concentrations.

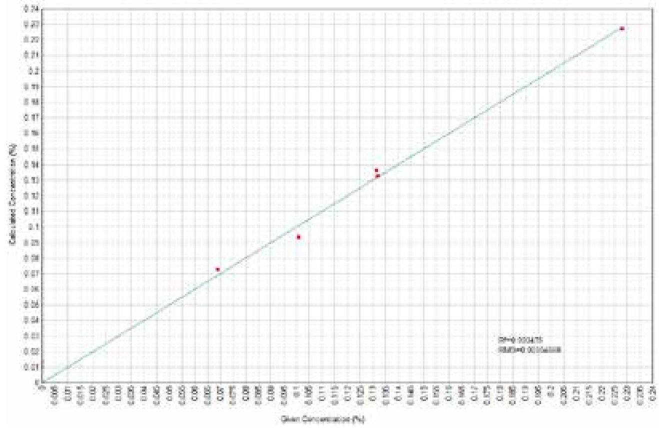

Figure 6. K_2O Calculated versus Given Concentrations.

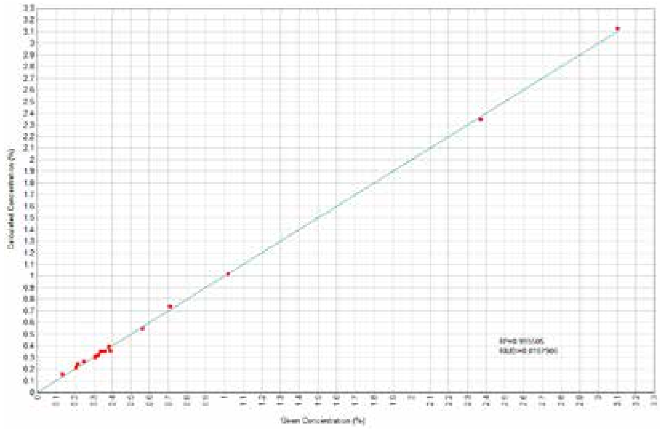

Figure 7. CaO Calculated versus Given Concentrations.

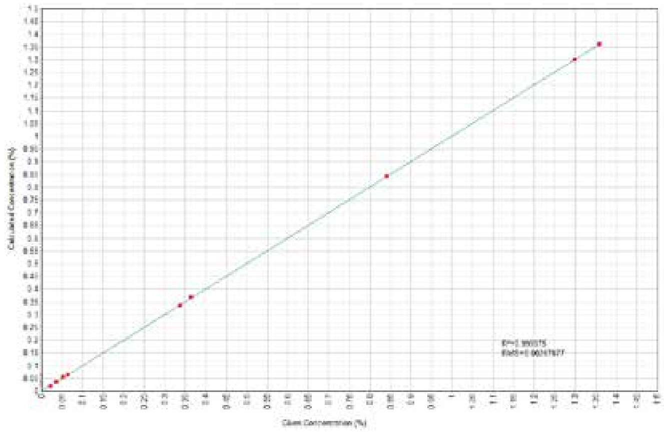

Figure 8. TiO_2 Calculated versus Given Concentrations.

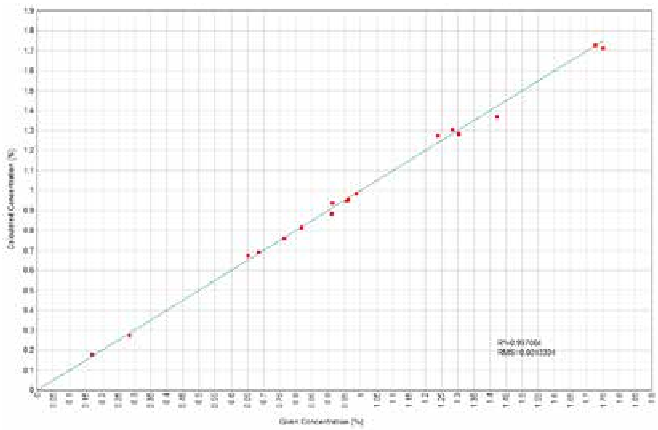

Figure 9. Cr_2O_3 Calculated versus Given Concentrations.

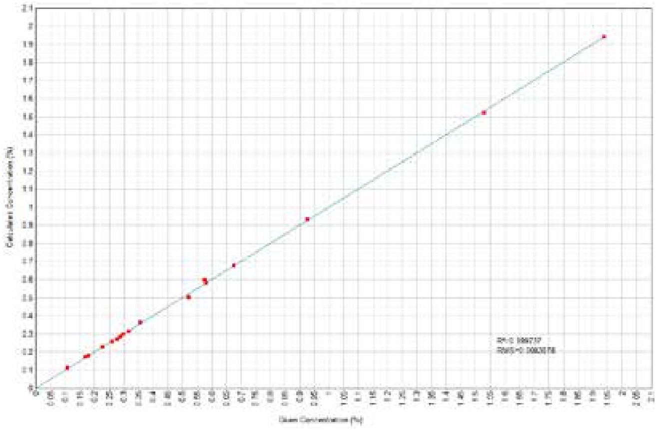

Figure 10. MnO Calculated versus Given Concentrations.

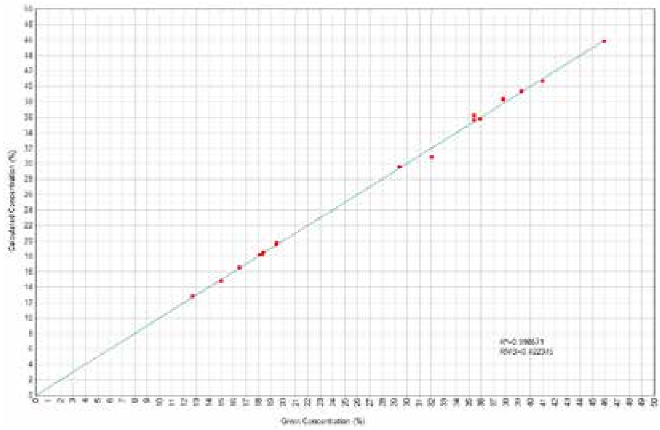

Figure 11. Fe_2O_3 Calculated versus Given Concentrations.

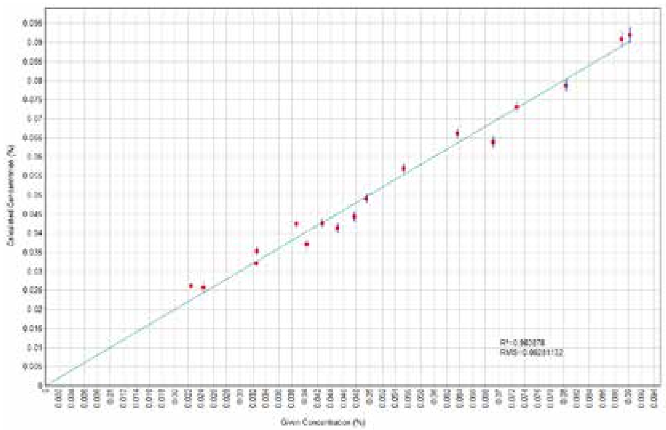

Figure 12. Co Calculated versus Given Concentrations.

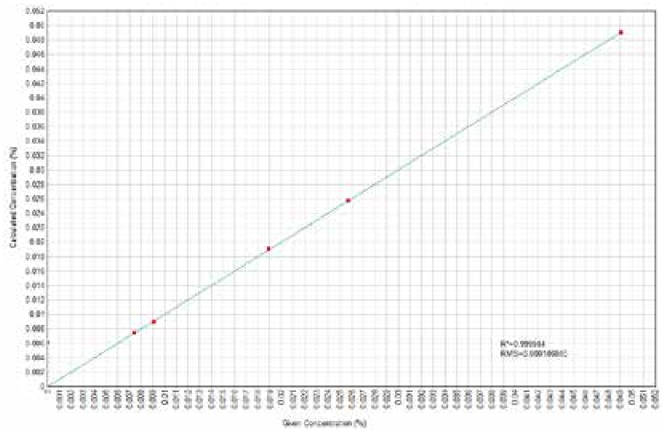

Figure 13. Cu Calculated versus Given Concentrations.

Validation

Two nickel ore reference materials (192 and 199) were used to validate the calibration. Table 3 shows the analysis results for these reference materials. CRM reference values are compared with the average of 10 replicate analysis of the two CRMs while Tables 4a and 4b show the repeatability results for each CRM.

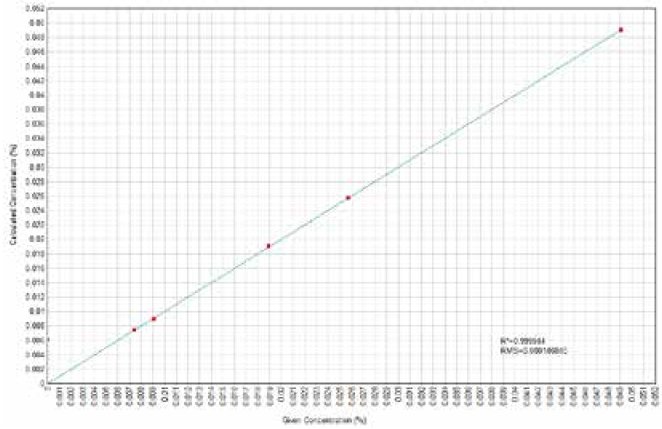

Figure 14. Zn Calculated versus Given Concentrations.

	MgO %	Al_2O_3 %	SiO_2 %	SO_3 %	K_2O %	CaO %	TiO_2 %	Cr_2O_3 %	MnO %	Fe_2O_3 %	Co %	Ni %	Cu %	Zn %
192 Reference	21.32	2.76	43.58	-	-	0.313	0.036	0.9129	0.277	18.1	0.0404	1.77	-	0.0176
192 Analysis	21.86	2.61	43.97	0.03	0.0104	0.308	0.034	0.8854	0.269	18.2	0.0376	1.80	0.0060	0.0164
199 Reference	0.742	17.47	24.93	0.08	0.069	0.208	0.842	0.686	1.94	41.01	0.0554	0.0995	0.0189	0.0198
199 Analysis	0.880	17.94	24.58	0.07	0.0724	0.213	0.840	0.695	1.95	40.68	0.0566	0.0984	0.0189	0.0206

Table 3. Accuracy of results for standards 192 and 199 using ARL QUANT'X Spectrometer.

	MgO %	Al_2O_3 %	SiO_2 %	SO_3 %	K_2O %	CaO %	TiO_2 %	Cr_2O_3 %	MnO %	Fe_2O_3 %	Co %	Ni %	Cu %	Zn %
Repeats 01	21.853	2.574	44.171	0.032	0.0104	0.307	0.034	0.885	0.269	18.187	0.036	1.803	0.0060	0.0164
Repeats 02	21.992	2.565	43.843	0.031	0.0104	0.309	0.032	0.882	0.266	18.175	0.038	1.803	0.0060	0.0158
Repeats 03	21.785	2.633	44.018	0.032	0.0104	0.307	0.034	0.886	0.268	18.174	0.039	1.802	0.0060	0.0164
Repeats 04	21.843	2.625	43.916	0.029	0.0104	0.308	0.033	0.886	0.270	18.202	0.037	1.800	0.0060	0.0164
Repeats 05	21.448	2.712	44.334	0.033	0.0104	0.308	0.038	0.886	0.270	18.209	0.036	1.807	0.0060	0.0167
Repeats 06	21.950	2.589	43.882	0.032	0.0104	0.307	0.034	0.888	0.268	18.188	0.037	1.800	0.0060	0.0162
Repeats 07	21.901	2.607	43.973	0.030	0.0104	0.307	0.030	0.886	0.270	18.173	0.039	1.804	0.0060	0.0167
Repeats 08	21.958	2.586	43.913	0.028	0.0104	0.306	0.034	0.886	0.270	18.199	0.038	1.803	0.0060	0.0165
Repeats 09	21.950	2.626	43.707	0.031	0.0104	0.308	0.038	0.888	0.269	18.185	0.038	1.802	0.0060	0.0163
Repeats 10	21.964	2.581	43.964	0.030	0.0104	0.310	0.035	0.882	0.271	18.198	0.040	1.804	0.0060	0.0166
Average	21.864	2.610	43.972	0.031	0.0104	0.308	0.034	0.885	0.269	18.189	0.038	1.803	0.0060	0.0164
1-Sigma	0.160	0.043	0.175	0.002	-	0.001	0.002	0.002	0.001	0.013	0.001	0.002	-	0.0003
% RSD	0.73	1.65	0.40	4.86	-	0.32	6.95	0.23	0.51	0.07	3.34	0.12	-	1.62
Minimum	21.448	2.565	43.707	0.028	0.0104	0.306	0.030	0.882	0.266	18.173	0.036	1.800	0.0060	0.0158
Maximum	21.992	2.712	44.334	0.033	0.0104	0.310	0.038	0.888	0.271	18.209	0.040	1.807	0.0060	0.0167

Table 4a. Repeatability results for standards 192 using ARL QUANT'X Spectrometer.

	MgO %	Al_2O_3 %	SiO_2 %	SO_3 %	K_2O %	CaO %	TiO_2 %	Cr_2O_3 %	MnO %	Fe_2O_3 %	Co %	Ni %	Cu %	Zn %
Repeats 01	1.009	17.750	24.609	0.069	0.074	0.213	0.845	0.696	1.943	40.653	0.056	0.098	0.0191	0.0212
Repeats 02	1.053	17.755	24.563	0.073	0.071	0.213	0.839	0.693	1.945	40.680	0.058	0.099	0.0192	0.0205
Repeats 03	0.981	17.827	24.559	0.077	0.073	0.215	0.847	0.694	1.942	40.698	0.056	0.098	0.0190	0.0209
Repeats 04	0.773	18.055	24.612	0.074	0.073	0.213	0.838	0.694	1.950	40.683	0.057	0.099	0.0186	0.0208
Repeats 05	0.887	17.903	24.685	0.074	0.073	0.214	0.841	0.690	1.950	40.651	0.058	0.098	0.0189	0.0208
Repeats 06	1.203	17.767	24.359	0.073	0.071	0.213	0.838	0.697	1.940	40.669	0.055	0.098	0.0188	0.0196
Repeats 07	1.024	17.885	24.488	0.078	0.074	0.211	0.848	0.690	1.939	40.656	0.057	0.098	0.0190	0.0209
Repeats 08	0.389	18.263	24.756	0.074	0.073	0.213	0.836	0.698	1.953	40.693	0.058	0.099	0.0188	0.0202
Repeats 09	0.746	18.160	24.516	0.076	0.072	0.215	0.835	0.699	1.941	40.687	0.055	0.099	0.0190	0.0210
Repeats 10	0.737	18.040	24.614	0.075	0.071	0.213	0.836	0.695	1.948	40.688	0.058	0.098	0.0186	0.0204
Average	0.880	17.941	24.576	0.074	0.072	0.213	0.840	0.695	1.945	40.676	0.057	0.098	0.0189	0.0206
1-Sigma	0.229	0.181	0.109	0.002	0.001	0.001	0.005	0.003	0.005	0.017	0.001	0.001	0.0002	0.0005
% RSD	26.03	1.01	0.44	3.28	1.56	0.49	0.56	0.43	0.25	0.04	2.29	0.62	1.06	2.27
Minimum	0.389	17.750	24.359	0.069	0.071	0.211	0.835	0.690	1.939	40.651	0.055	0.098	0.0186	0.0196
Maximum	1.203	18.263	24.756	0.078	0.074	0.215	0.848	0.699	1.953	40.698	0.058	0.099	0.0192	0.0212

Table 4b. Repeatability results for standards 199 using ARL QUANT'X Spectrometer.

Conclusion

This application note shows the suitability of the ARL QUANT'X EDXRF spectrometer for the analysis of nickel ores samples. This compact instrument allows for reliable and fast analysis of nickle ores. Accuracy and repeatability results show that satisfactory results are obtainable for an analysis of pressed powders in air. This is a significant advantage for nickel ore mines which often operate in remote areas.

Learn more at **thermofisher.com/quantx**

thermo scientific