Application note

Analysis of nickel ore with the ARL OPTIM'X WDXRF Spectrometer

Introduction

Nickel is produced from two very different ores: lateritic and sulfidic. Lateritic ore is found primarily in tropical countries such as Indonesia and is 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, and 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, it is a preferred technique in process and quality control across many industries.

Instrumentation

The Thermo Scientific™ ARL OPTIM'X™ WDXRF Spectrometer is designed for ease of use with minimal operation and maintenance costs. The instrument is fitted with a Thermo Scientific SmartGonio™ Goniometer covering elements from fluorine ($_{9}$F) to uranium ($_{92}$U). Two power versions exist, either 50 W or the new 200 W version. The 50 W version has been used for the tests shown in this report. The instrument does not require external or internal water cooling and has 10 times better spectral resolution than a conventional EDXRF instrument as well as superior precision and stability. It has good performance for sodium ($_{11}$Na), magnesium ($_{12}$Mg), and even for fluorine ($_{9}$F).

Analytical conditions

As shown in Table 1, spectra were collected from each nickel ore sample for a total analysis time of 10.4 minutes using the ARL OPTIM'X WDXRF Spectrometer at 50 W power. Measurement time can be further fine-tuned according to specific applications. All counting time could be decreased by a factor of 2.5 when using the 200 W version of the ARL OPTIM'X WDXRF Spectrometer while still allowing the same accuracy and precision. Hence, total analysis time would decrease to about 4 minutes.

Name	Component type	Analysis time(s)	kV	mA
Ni Ka 1,2	Gonio XRF	36	30	1.67
Zn Ka 1,2	Gonio XRF	60	30	1.67
Cr Ka 1,2	Gonio XRF	36	30	1.67
Ti Ka 1,2	Gonio XRF	36	30	1.67
Si Ka 1,2	Gonio XRF	36	30	1.67
S Ka 1,2	Gonio XRF	36	30	1.67
P Ka 1,2	Gonio XRF	60	30	1.67
Mg Ka 1,2	Gonio XRF	60	30	1.67
K Ka 1,2	Gonio XRF	60	30	1.67
Ca Ka 1,2	Gonio XRF	36	30	1.67
Al Ka 1,2	Gonio XRF	36	30	1.67
Mn Ka 1,2	Gonio XRF	36	30	1.67
Co Ka 1,2	Gonio XRF	60	30	1.67
Fe Ka 1,2	Gonio XRF	36	30	1.67

Table 1. Analytical conditions.

Sample preparation

Calibration was done using 18 nickel ore CRMs. Samples were fused into beads without ignition with a sample-to-flux ratio of 1:20. Ammonium nitrate oxidizer was added to the fusion mix. Table 2 shows the concentration ranges of the different oxides covered by the calibration. The R2 and SEE (standard error of estimate) values were obtained for the different compounds.

	Calibration				
Element	N	Min %	Max %	R2	SEE
Al_2O_3	17	1.599	17.470	0.9998	0.0813
CaO	18	0.133	3.106	0.9999	0.0096
Cr_2O_3	17	0.170	1.750	0.9980	0.0204
Fe_2O_3	15	12.728	45.990	0.9998	0.1521
K_2O	5	0.069	0.228	0.9831	0.0089
MgO	17	0.742	27.306	0.9998	0.1551
MnO	18	0.109	1.940	0.9996	0.0096
NiO	18	0.066	3.740	0.9996	0.0214
SiO_2	16	22.790	47.970	0.9993	0.2415
TiO_2	13	0.035	1.360	0.9998	0.0063
Co_3O_4	16	0.031	0.123	0.9921	0.0027
P_2O_5	7	0.005	0.173	0.9847	0.0099
SO_3	7	0.004	0.189	0.9763	0.0108
ZnO	18	0.009	0.044	0.9504	0.0023

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 intensities 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 1–10 show the calibration curves obtained for NiO, MgO, Al_2O_3, CaO, Cr_2O_3, Fe_2O_3, K_2O, MnO, SiO_2, and TiO_2.

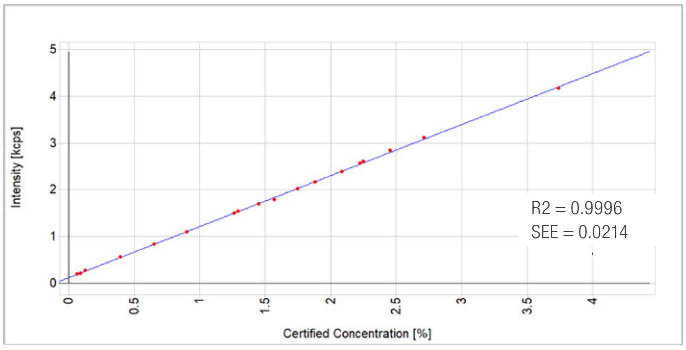

Figure 1. NiO calibration graph.

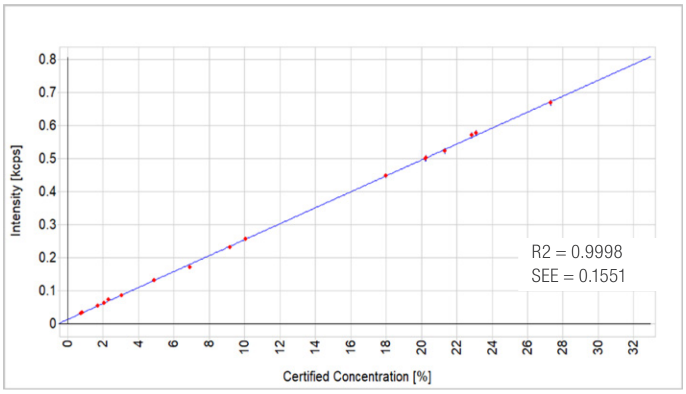

Figure 2. MgO calibration graph.

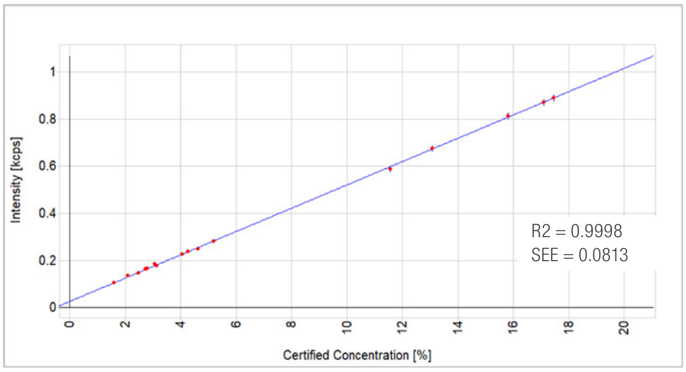

Figure 3. Al_2O_3 calibration graph.

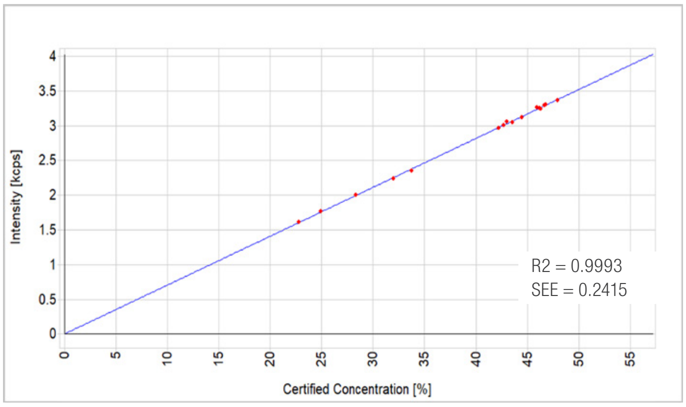

Figure 4. SiO_2 calibration graph.

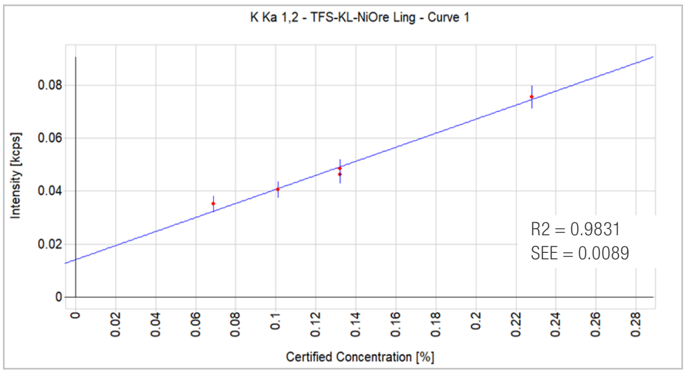

Figure 5. K_2O calibration graph.

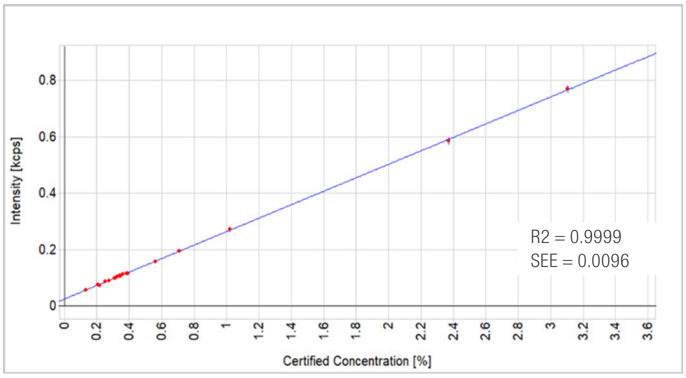

Figure 6. CaO calibration graph.

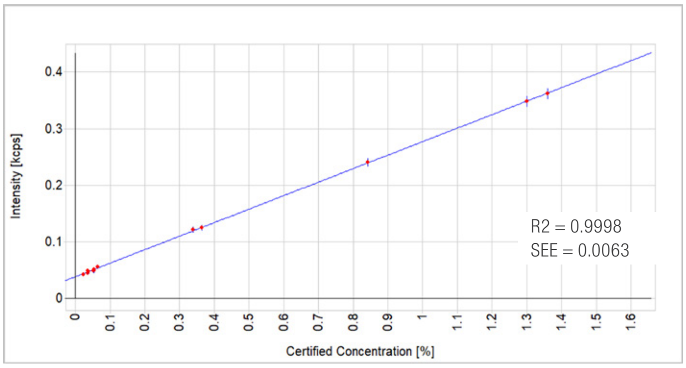

Figure 7. TiO_2 calibration graph.

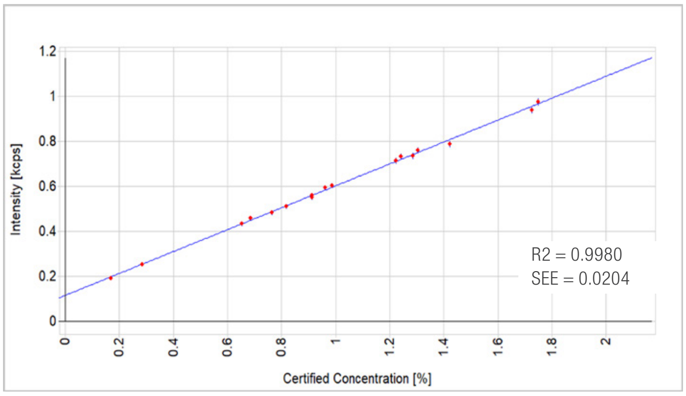

Figure 8. Cr_2O_3 calibration graph.

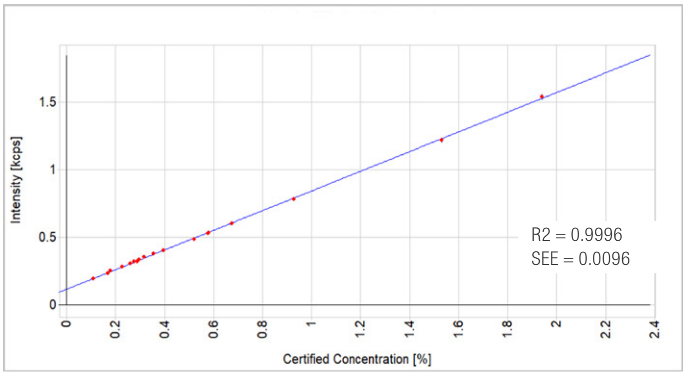

Figure 9. MnO calibration graph.

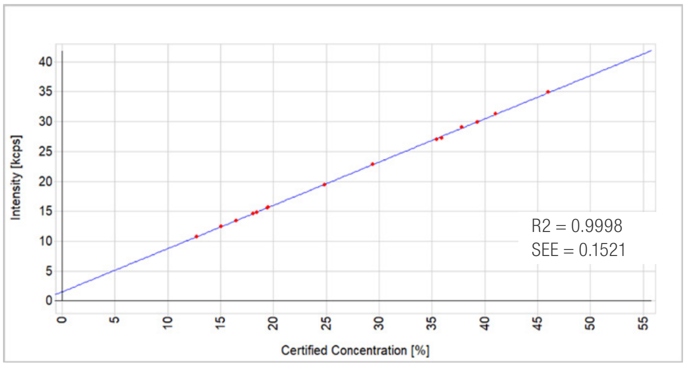

Figure 10. Fe_2O_3 calibration graph.

Validation

Three nickel ore reference materials (181, 184, and 198) were used to validate the calibration. Table 6 shows the analysis results for these reference materials. CRM reference values are compared with the average of 10 replicate analyses of the three CRMs, while Tables 3–5 show the repeatability of the 10 replicates for each CRM.

Sample 181: all values are %														
Elements	Al_2O_3	CaO	Co_3O_4	Cr_2O_3	Fe_2O_3	K_2O	MgO	MnO	NiO	P_2O_5	SO_3	SiO_2	TiO_2	ZnO
Rep 1	11.65	2.37	0.060	1.26	35.71	0.129	2.07	0.164	0.664	0.034	0.184	33.52	0.320	0.009
Rep 2	11.33	2.37	0.060	1.26	35.70	0.133	2.04	0.168	0.669	0.039	0.173	33.48	0.326	0.009
Rep 3	11.56	2.37	0.061	1.25	35.57	0.131	2.16	0.169	0.658	0.031	0.181	33.40	0.341	0.009
Rep 4	11.46	2.36	0.064	1.24	35.61	0.139	2.07	0.169	0.657	0.026	0.211	33.50	0.330	0.010
Rep 5	11.49	2.36	0.059	1.25	35.65	0.132	2.14	0.169	0.661	0.032	0.191	33.35	0.329	0.012
Rep 6	11.57	2.35	0.060	1.25	35.68	0.138	2.07	0.165	0.658	0.026	0.166	33.66	0.333	0.007
Rep 7	11.37	2.35	0.059	1.26	35.62	0.130	2.05	0.163	0.666	0.035	0.184	33.73	0.326	0.011
Rep 8	11.31	2.35	0.062	1.27	35.70	0.128	2.02	0.168	0.666	0.021	0.207	33.62	0.336	0.010
Rep 9	11.63	2.39	0.059	1.26	35.68	0.126	2.05	0.166	0.664	0.027	0.180	33.51	0.343	0.009
Rep 10	11.59	2.35	0.061	1.26	35.69	0.132	2.11	0.168	0.656	0.028	0.170	33.43	0.335	0.014
AVG	11.50	2.36	0.060	1.26	35.66	0.132	2.08	0.167	0.662	0.030	0.185	33.52	0.332	0.010
SD	0.124	0.013	0.002	0.008	0.047	0.004	0.045	0.002	0.005	0.005	0.015	0.119	0.007	0.002

Table 3. Repeatability results of nickel ore using the ARL OPTIM'X WDXRF Spectrometer for sample 181.

Sample 184: all values are %														
Elements	Al_2O_3	CaO	Co_3O_4	Cr_2O_3	Fe_2O_3	K_2O	MgO	MnO	NiO	P_2O_5	SO_3	SiO_2	TiO_2	ZnO
Rep 1	4.58	0.215	0.121	1.760	39.14	0.011	3.04	0.667	1.280	0.036	0.032	42.22	0.055	0.033
Rep 2	4.47	0.211	0.121	1.760	39.20	0.011	2.92	0.670	1.290	0.033	0.037	42.35	0.071	0.033
Rep 3	4.51	0.216	0.120	1.770	39.22	0.012	3.07	0.666	1.290	0.030	0.043	42.41	0.062	0.033
Rep 4	4.53	0.216	0.121	1.760	39.20	0.011	3.11	0.665	1.290	0.045	0.042	42.27	0.062	0.028
Rep 5	4.49	0.218	0.121	1.770	39.27	0.012	3.06	0.674	1.290	0.050	0.050	42.62	0.059	0.032
Rep 6	4.58	0.209	0.120	1.760	39.12	0.010	3.07	0.660	1.280	0.045	0.032	42.64	0.066	0.034
Rep 7	4.48	0.214	0.124	1.750	39.09	0.014	3.15	0.667	1.300	0.038	0.031	42.32	0.058	0.034
Rep 8	4.60	0.217	0.124	1.750	39.13	0.009	3.05	0.666	1.290	0.040	0.049	42.51	0.065	0.031
Rep 9	4.50	0.224	0.120	1.780	39.27	0.014	3.02	0.657	1.290	0.031	0.056	42.26	0.066	0.031
Rep 10	4.42	0.228	0.125	1.760	39.14	0.012	3.05	0.674	1.300	0.032	0.061	42.37	0.059	0.030
AVG	4.51	0.217	0.122	1.760	39.18	0.012	3.05	0.667	1.290	0.038	0.043	42.40	0.062	0.032
SD	0.057	0.006	0.002	0.009	0.063	0.001	0.060	0.005	0.007	0.007	0.010	0.148	0.005	0.002

Table 4. Repeatability results of nickel ore using the ARL OPTIM'X WDXRF Spectrometer for sample 184.

Sample 198: all values are %														
Elements	Al_2O_3	CaO	Co_3O_4	Cr_2O_3	Fe_2O_3	K_2O	MgO	MnO	NiO	P_2O_5	SO_3	SiO_2	TiO_2	ZnO
Rep 1	15.75	0.350	0.106	0.285	46.18	0.100	0.807	1.53	0.085	0.167	0.105	22.83	1.35	0.022
Rep 2	15.74	0.345	0.106	0.281	46.15	0.099	0.841	1.51	0.088	0.170	0.115	23.04	1.36	0.022
Rep 3	15.83	0.348	0.109	0.281	46.08	0.098	0.808	1.52	0.081	0.179	0.124	22.84	1.37	0.024
Rep 4	15.79	0.357	0.106	0.269	46.09	0.103	0.820	1.51	0.090	0.179	0.119	22.91	1.36	0.021
Rep 5	15.89	0.368	0.105	0.271	46.07	0.106	0.882	1.52	0.086	0.179	0.132	23.05	1.36	0.020
Rep 6	15.72	0.349	0.108	0.273	46.12	0.100	0.829	1.52	0.091	0.170	0.132	22.89	1.36	0.024
Rep 7	15.67	0.357	0.112	0.279	46.18	0.101	0.797	1.51	0.092	0.179	0.128	22.85	1.36	0.023
Rep 8	15.88	0.342	0.109	0.281	46.01	0.099	0.870	1.51	0.088	0.171	0.119	22.90	1.38	0.024
Rep 9	15.90	0.355	0.111	0.283	46.04	0.107	0.861	1.51	0.089	0.186	0.117	23.01	1.36	0.026
Rep 10	15.65	0.345	0.112	0.275	46.22	0.106	0.817	1.51	0.089	0.172	0.126	23.13	1.37	0.024
AVG	15.78	0.352	0.108	0.278	46.11	0.102	0.833	1.51	0.088	0.175	0.122	22.95	1.36	0.023
SD	0.091	0.008	0.003	0.005	0.068	0.003	0.029	0.007	0.003	0.006	0.008	0.104	0.008	0.002

Table 5. Repeatability results of nickel ore using the ARL OPTIM'X WDXRF Spectrometer for sample 198.

	Sample 181			Sample 184			Sample 198		
Element	CRM	AVG	Difference	CRM	AVG	Difference	CRM	AVG	Difference
Al_2O_3	11.55	11.50	0.050	4.622	4.510	0.112	15.81	15.78	0.030
CaO	2.372	2.360	0.012	0.216	0.217	-0.001	0.346	0.352	-0.006
Cr_2O_3	0.061	0.060	0.001	0.123	0.122	0.001	0.110	0.108	0.002
Fe_2O_3	1.241	1.260	-0.019	1.75	1.76	-0.010	0.285	0.278	0.007
K_2O	35.944	35.66	0.284	39.298	39.180	0.118	45.99	46.11	-0.120
MgO	0.132	0.132	0.000	NA	NA	NA	0.101	0.102	-0.001
MnO	2.054	2.08	-0.026	3.045	3.050	-0.005	0.798	0.833	-0.035
NiO	0.168	0.167	0.001	0.676	0.667	0.009	1.53	1.51	0.020
SiO_2	0.652	0.662	-0.010	1.295	1.290	0.005	0.088	0.088	0.000
TiO_2	0.017	0.030	-0.013	0.017	0.038	-0.021	0.161	0.175	-0.014
Co_3O_4	0.189	0.185	0.004	0.060	0.043	0.017	NA	NA	NA
P_2O_5	33.785	33.52	0.265	42.252	42.400	-0.148	22.79	22.95	-0.160
SO_3	0.339	0.332	0.007	NA	NA	NA	1.36	1.36	0.000
ZnO	0.012	0.010	0.002	0.035	0.032	0.003	0.026	0.023	0.003

Table 6. Analysis results of nickel ore using the ARL OPTIM'X WDXRF Spectrometer.

Conclusion

This application note has shown the suitability of the ARL OPTIM'X WDXRF Spectrometer for the analysis of nickel ore samples. This compact instrument allows for reliable and fast analysis results combined with excellent repeatability. A total analysis time of 10.4 minutes was used with the ARL OPTIM'X WDXRF Spectrometer at 50 W power. Measurement time can be further fine-tuned according to specific applications. All counting time could be decreased by a factor of 2.5 when using the 200 W version of the ARL OPTIM'X WDXRF Spectrometer while still allowing the same accuracy and precision. Hence, total analysis time would decrease to about 4 minutes.

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