

Iron ore analysis with the ARL OPTIM'X XRF Spectrometer

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Figure 1. Thermo Scientific ARL OPTIM'X XRF Spectrometer with its 13-position sample loader.

Introduction

The production of commercial iron relies on the refinement of iron ore, a massive global undertaking; in 2021, 2.6 billion metric tons of iron ore were mined, which translates to ~1.6 billion metric tons of iron content.* 90% of extracted iron ore goes toward the production of steel, while the rest is used for a wide variety of other applications. This includes a number of battery materials, where iron is used due to its ready availability and ease of oxidation. Whether in traditional nickel-iron batteries or burgeoning technologies such as iron-air batteries and lithium iron phosphate (LFP) cathodes, iron plays a critical role in battery manufacturing and research.

Knowing the exact composition of iron ore can aid in refinement and subsequent purity determination of the final iron metal, which is vital for highly technical applications such as batteries, where purity can have a significant impact on performance. In this application note, a wavelength dispersive X-ray fluorescence (WDXRF) spectroscopy technique for iron ore analysis is described that minimizes analysis time. Given the massive quantities of ore that are extracted and processed annually, it is vital that characterization techniques are as efficient as possible, as even a minute of additional time can multiply exponentially at the quantities being discussed.

*U.S. Geological Survey, Mineral Commodity Summaries – Iron Ore, January 2022

Instrumentation

The Thermo Scientific™ ARL OPTIM'X™ XRF Spectrometer is a WDXRF instrument designed for ease of use with minimal operation and maintenance costs. It is fitted with the innovative Thermo Scientific™ SmartGonio™ Goniometer, which allows the ARL OPTIM'X Spectrometer to cover the elemental range from fluorine (${}_{9}\text{F}$) to americium (${}_{95}\text{Am}$). Two power versions of the spectrometer exist; a 50 and 200 W version. Overall, the 200 W version typically offers 2.5x faster data acquisition than the 50 W version. For this application note, the 50 W version was used. The ARL OPTIM'X Spectrometer does not require external or internal water cooling and has ~10x better spectral resolution than conventional energy-dispersive XRF (EDXRF) instruments, as well as superior precision and stability. It has reliable performance for critical elements such as sodium (${}_{11}\text{Na}$), magnesium (${}_{12}\text{Mg}$), and even fluorine (${}_{9}\text{F}$).

Analytical conditions

Using the 50 W ARL OPTIM'X Spectrometer, data for 12 elements (Al, Ca, Cr, Fe, K, Mg, Mn, Si, Ti, P, S, and V) was collected from each iron ore sample at 30 kV and 1.67 mA, taking 36 seconds of analysis time each (with the exception of Mg, which required 60 seconds) for a total analysis time of 7.6 minutes. Measurement time can be further fine-tuned according to specific applications. Total counting time can also be decreased by a factor of 2.5 with the 200 W instrument without any reduction in accuracy and precision. Total analysis time would, therefore, decrease to ~3 minutes in case the 200 W ARL OPTIM'X Spectrometer is used.

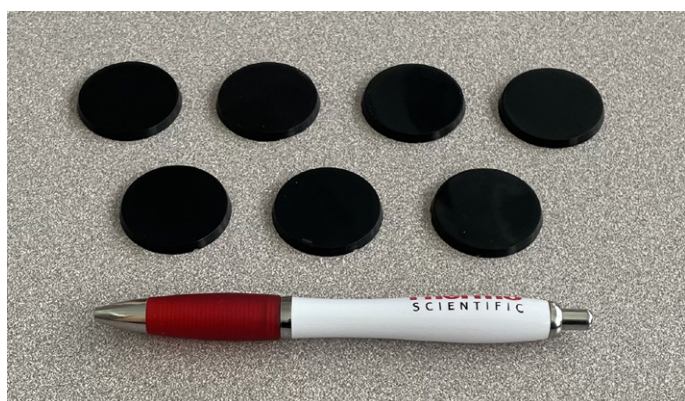


Figure 2. Seven fused beads are used for the calibration.

Sample preparation

Calibration was performed using 7 iron ore certified reference materials (CRMs). Samples were fused into beads, without ignition, with a sample to flux ratio of 1:10. Ammonium nitrate oxidizer was added to the fusion mix. Table 1 shows the concentration ranges of the different oxides covered by the calibration. R^2 and standard error of estimates (SEE) values were obtained for each compound.

The sample preparation as fused beads removes any grain size effect or mineralogical effects that may spoil the X-ray fluorescence analysis. Therefore an excellent analysis accuracy is obtained especially for major and minor elements/oxides. Trace elements determination is more difficult due to the dilution of the sample where trace levels are ten times lower in the actual fused bead compared to the original sample. Hence longer counting time may be used for trace elements determination when needed. Alternatively samples are prepared as pressed pellets when best trace elements determination is required.

Calibration					
Element	N	Min %	Max %	R^2	SEE (%)
Al_2O_3	7	0.1300	2.9700	0.9984	0.0445
CaO	7	0.0150	0.1960	0.9969	0.0039
Cr_2O_3	6	0.0149	0.0181	0.5476	0.0050
Fe_2O_3	7	65.24	95.39	0.9993	0.3376
K_2O	6	0.0100	0.0200	0.8356	0.0022
MgO	4	0.0170	0.0770	0.9552	0.0071
MnO	3	0.0200	0.0390	0.9975	0.0007
P_2O_5	7	4.64	24.88	0.9995	0.1866
SO_3	7	0.0470	0.3850	0.9986	0.0052
SiO_2	7	0.0090	0.3460	0.9863	0.0138
TiO_2	4	0.0550	0.0800	0.9206	0.0037
V_2O_5	7	0.0020	0.0075	0.1384	0.0021

Table 1. Concentration ranges and calibration parameter values for iron ore components. N = number of CRMs; R^2 = coefficient of determination; SEE = standard error of estimate.

Calibration

Calibration curves were created to relate elemental characteristic X-ray intensities to oxide concentrations (Figure 3).

X-ray fluorescence can measure individual elements, and the results can also be related directly to the oxide forms when only one single form of each oxide is present in the sample.

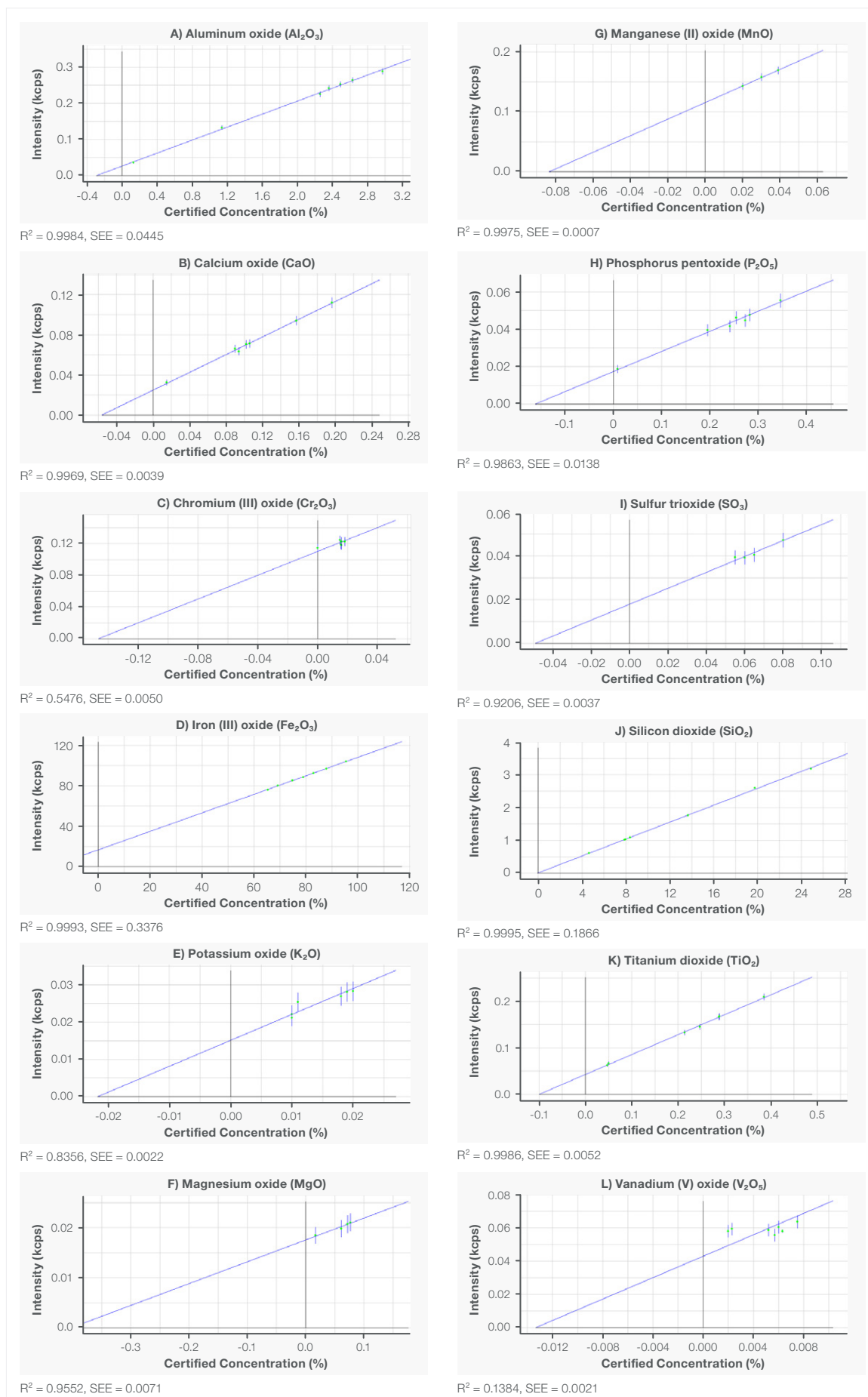


Figure 3. Calibration curves for a selection of oxides found in iron ore.

Validation

An iron ore reference material (405) was used to validate the calibration (Table 2). CRM reference values are compared with the first run of 10 replicate analyses. Table 3 shows the repeatability of the 10 replicates for this CRM.

Conclusion

This application note has shown the suitability of the ARL OPTIM'X Spectrometer for the analysis of iron ore samples. This compact instrument allows for fast and reliable analysis combined with excellent repeatability. A total analysis time of 7.6 minutes was required with the 50 W ARL OPTIM'X Spectrometer. Obviously the sample preparation as fused beads dilutes the sample 10 times which means that the trace elements levels are very low in the actual fused beads. This explain the poor precision for traces, notably for Cr_2O_3 and for V_2O_5 . Measurement time can be further fine-tuned according to specific applications, e.g., increasing counting time to improve precision for trace elements.

All counting time could be decreased by a factor of 2.5 by using the 200 W version of the ARL OPTIM'X Spectrometer, while still achieving the same accuracy and precision. In such case, total analysis time would decrease to about 3 minutes.

Sample ID		405		
Element	Unit	CRM	First run	Difference
Al_2O_3	%	2.26	2.26	0.00
CaO	%	0.196	0.191	-0.005
Cr_2O_3	ppm	149	148	-1
Fe_2O_3	%	82.96	83.00	0.04
K_2O	ppm	200	206	6
MgO	%	N/A	0.089	N/A
MnO	ppm	300	298	-2
P_2O_5	%	0.254	0.286	0.032
SO_3	%	0.045	0.082	0.037
SiO_2	%	8.37	8.38	0.01
TiO_2	%	0.214	0.213	-0.001
V_2O_5	ppm	52	56	4

Table 2. Reference iron ore (405) analysis with the ARL OPTIM'X Spectrometer.

Sample 405												
Element	Al_2O_3	CaO	Cr_2O_3	Fe_2O_3	K_2O	MgO	MnO	P_2O_5	SO_3	SiO_2	TiO_2	V_2O_5
Counting time	36 s	36 s	36 s	36 s	36 s	60 s	36 s	36 s	36 s	36 s	36 s	36 s
Unit	%	%	ppm	%	ppm	%	ppm	%	%	%	%	ppm
Run 1	2.26	0.191	148	83.00	206	0.089	298	0.286	0.082	8.38	0.213	56
Run 2	2.25	0.197	178	83.07	184	0.103	329	0.279	0.085	8.31	0.212	50
Run 3	2.25	0.195	167	83.06	207	0.089	312	0.294	0.083	8.39	0.210	44
Run 4	2.21	0.205	138	82.95	199	0.080	311	0.269	0.077	8.38	0.223	50
Run 5	2.29	0.196	160	82.90	177	0.046	326	0.287	0.086	8.31	0.204	56
Run 6	2.23	0.203	188	82.92	197	0.079	325	0.281	0.079	8.43	0.211	55
Run 7	2.24	0.198	162	82.95	209	0.088	324	0.275	0.078	8.41	0.214	39
Run 8	2.27	0.203	136	82.89	193	0.088	315	0.269	0.087	8.42	0.208	48
Run 9	2.21	0.202	140	82.95	192	0.066	305	0.272	0.081	8.30	0.213	50
Run 10	2.24	0.189	179	82.86	190	0.054	302	0.287	0.076	8.37	0.212	49
Average	2.24	0.198	160	82.96	195	0.078	315	0.280	0.081	8.37	0.212	50
SD	0.026	0.006	19	0.069	10	0.018	11	0.009	0.004	0.047	0.005	5

Table 3. Repeatability of reference iron ore (405) analysis using the ARL OPTIM'X Spectrometer.

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