Iron ore analysis with the ARL PERFORM'X XRF Spectrometer

Background

The production of commercial iron relies on the refinement of iron ore, a massive global undertaking; in 2024, 2.5 billion metric tons of iron ore were mined, which translates to ~1.6 billion metric tons of iron content.*

The iron is usually found in the form of hematite (Fe_2O_3), magnetite (Fe_3O_4), goethite (FeO(OH)), limonite (FeO(OH)·n(H_2O), or siderite ($FeCO_3$,).

90% of extracted iron ore goes toward the production of steel, while the rest is used for a wide variety of other applications.

Knowing the exact composition of iron ores 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 with a minimized time of analysis. Given the massive quantities of ore that are worldwide extracted and processed annually, it is vital that characterization including quantification techniques are as efficient as possible, as even a minute of additional time can multiply exponentially at the quantities being discussed.

Instrumentation

A Thermo Scientific™ ARL PERFORM'X™ XRF Spectrometer (cf Figure 1) is fitted with a Rh anode X-Ray tube, type 5GN+. This X-ray tube brings unequalled stability over the very long term thanks to the LoVap technology of its filament: lower temperature avoids slow filament evaporation and W coating of the inside of the tube window.

The geometry of the instrument is optimized to provide the highest sensitivity. The ARL PERFORM'X spectrometer is equipped with the unique F54 Moiré fringe goniometer. Speed, flexibility and reliability of analysis are guaranteed thanks to the ingenious friction-free positioning system. Up to nine crystals can be fitted with a standard four collimators, five primary beam filters and two detectors for custom conditions and parameters in the elemental analysis from Be to U.

Figure 1. ARL PERFORM'X XRF Spectrometer.

Ease of operation is obtained through the innovative Thermo Scientific™ OXSAS™ Software running under the current version of Windows®.

*U.S. Geological Survey, Mineral Commodity Summaries – Iron Ore, January 2025

Analytical conditions

Using the 4.2 kW ARL PERFORM'X XRF Spectrometer and 29 mm aperture, data for 12 elements (Al, Ca, Cr, Cu, Fe, K, Mg, Mn, Ni, Si, Ti, V) were collected under vacuum from each iron ore sample at 40 kV and 70 mA, taking 20 seconds of analysis time each (with the exception of Mg, which required 40 seconds) for a total analysis time of 5 minutes (loading-unloading included).

Table 1: Analytical conditions and performances

Element/Line	kV	mA	Counting time (s)	Compound	Min conc. (%)	Max conc. (%)	SEE (%)	LOD (ppm)	LOQ (ppm)
Fe Ka 1	40	70	20	Fe	LoQ	69.930	1.177	10	30
Si Ka 1, 2	40	70	20	SiO2	LoQ	39.980	0.111	30	90
Al Ka 1, 2	40	70	20	Al	LoQ	7.270	0.045	30	90
Ca Ka 1, 2	40	70	20	CaO	LoQ	28.050	0.090	12	36
Mg Ka 1, 2	40	70	40	MgO	LoQ	20.080	0.070	52	156
Mn Ka 1, 2	40	70	20	Mn	LoQ	2.590	0.0098	6	18
Ti Ka 1, 2	40	70	20	Ti	LoQ	0.430	0.0046	5	15
K Ka 1, 2	40	70	20	K2O	LoQ	0.728	0.0111	12	36
Ni Ka 1, 2	40	70	20	Ni	LoQ	1.480	0.0001	6	18
Cr Ka 1, 2	40	70	20	C	LoQ	1.840	0.0030	5	15
Cu Ka 1, 2	40	70	20	Cu	LoQ	3.370	0.0016	5	15

Sample preparation

Calibration was performed using 17 iron ore certified reference materials (CRMs). Samples were fused into beads, with ignition, with a sample to flux ratio of 1 to 11. An ammonium nitrate oxidizer was added to the fusion mix.

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

Table 1 shows the concentration ranges of the different compounds covered by the calibration. Standard error of estimates (SEE), LoD (limits of detection), LoQ (limits of quantification) values were obtained for each compound.

Calibration

Calibration curves were created to relate elemental characteristic X-ray intensities to compound concentrations. 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.

Validation

Table 2 shows the repeatability of 10 successive replicates for selected CRMs.

Table 3 shows the longer-term stability over 5 successive days.

Conclusion

This application note demonstrates the suitability of the ARL PERFORM'X WDXRF Spectrometer for the analysis of iron ore samples, prepared as standardized and repeatable fused beads.

With a total analysis time of 5 minutes, the ARL PERFORM'X XRF allows for fast and reliable analysis combined with excellent repeatability, over a wide range of concentration from few dozen of ppm to several dozen of percents.

Table 2: Results of 10 successive replicates for selected CRMs

SARM 11

Line	Fe Ka 1	Si Ka 1,2	Al Ka 1,2	Ca Ka 1,2	Mg Ka 1,2	Mn Ka 1,2	Ti Ka 1,2	K Ka 1,2	Ni Ka 1,2
Compound	**Fe**	**SiO_2**	**Al_2O_3**	**CaO**	**MgO**	**Mn**	**Ti**	**K_2O**	**Ni**
Run 1	69.002	3.222	1.433	0.0328	0.1186	0.0242	0.0434	0.1178	0.0029
Run 2	69.008	3.228	1.426	0.0332	0.1204	0.0249	0.0425	0.1163	0.0026
Run 3	68.975	3.228	1.418	0.0322	0.1154	0.0246	0.0424	0.1189	0.0029
Run 4	68.943	3.219	1.416	0.0332	0.1191	0.0246	0.0427	0.1181	0.0026
Run 5	69.099	3.226	1.418	0.0321	0.1210	0.0247	0.0428	0.1170	0.0030
Run 6	69.079	3.223	1.435	0.0326	0.1159	0.0245	0.0428	0.1164	0.0029
Run 7	68.924	3.207	1.422	0.0330	0.1199	0.0244	0.0428	0.1183	0.0026
Run 8	69.037	3.215	1.428	0.0325	0.1176	0.0245	0.0426	0.1165	0.0031
Run 9	69.024	3.224	1.432	0.0330	0.1190	0.0247	0.0426	0.1168	0.0034
Run 10	69.044	3.243	1.422	0.0318	0.1160	0.0245	0.0428	0.1179	0.0027
Average	**69.013**	**3.223**	**1.425**	**0.0326**	**0.1183**	**0.0246**	**0.0427**	**0.1174**	**0.0029**
SD	0.056	0.009	0.007	0.0005	0.0020	0.0002	0.0003	0.0009	0.0003

610-1

Line	Fe Ka 1	Si Ka 1,2	Al Ka 1,2	Ca Ka 1,2	Mg Ka 1,2	Mn Ka 1,2	Ti Ka 1,2	Ni Ka 1,2	Cr Ka 1,2	Cu Ka 1,2
Compound	**Fe**	**SiO_2**	**Al_2O_3**	**CaO**	**MgO**	**Mn**	**Ti**	**Ni**	**Cr**	**Cu**
Run 1	46.948	6.783	3.653	0.1468	3.115	0.5708	0.0166	1.480	1.889	0.0190
Run 2	46.966	6.816	3.647	0.1469	3.099	0.5701	0.0168	1.478	1.888	0.0182
Run 3	46.898	6.803	3.631	0.1459	3.101	0.5709	0.0163	1.480	1.897	0.0185
Run 4	46.914	6.812	3.632	0.1470	3.105	0.5701	0.0169	1.477	1.893	0.0190
Run 5	46.938	6.801	3.628	0.1451	3.111	0.5697	0.0169	1.479	1.890	0.0184
Run 6	46.941	6.807	3.634	0.1458	3.106	0.5715	0.0169	1.479	1.891	0.0181
Run 7	46.938	6.803	3.651	0.1469	3.110	0.5706	0.0167	1.479	1.891	0.0186
Run 8	46.966	6.821	3.652	0.1474	3.087	0.5694	0.0167	1.478	1.887	0.0180
Run 9	46.939	6.787	3.629	0.1471	3.099	0.5702	0.0165	1.481	1.892	0.0188
Run 10	46.890	6.809	3.628	0.1461	3.102	0.5720	0.0168	1.477	1.897	0.0188
Average	**46.934**	**6.804**	**3.638**	**0.1465**	**3.104**	**0.5705**	**0.0167**	**1.479**	**1.891**	**0.0185**
SD	0.026	0.012	0.011	0.0007	0.008	0.0008	0.0002	0.001	0.004	0.0004

677-1

Line	Fe Ka 1	Si Ka 1,2	Al Ka 1,2	Ca Ka 1,2	Mg Ka 1,2	Mn Ka 1,2	Ti Ka 1,2	Ni Ka 1,2	Cr Ka 1,2
Compound	**Fe**	**SiO_2**	**Al_2O_3**	**CaO**	**MgO**	**Mn**	**Ti**	**Ni**	**Cr**
Run 1	51.097	25.134	0.5978	0.0472	0.0898	0.0208	0.0140	0.0019	0.0025
Run 2	51.107	25.149	0.5939	0.0480	0.0887	0.0209	0.0141	0.0019	0.0026
Run 3	51.162	25.192	0.6076	0.0478	0.0844	0.0204	0.0140	0.0017	0.0024
Run 4	51.111	25.156	0.5939	0.0475	0.0872	0.0205	0.0142	0.0018	0.0025
Run 5	51.180	25.200	0.5985	0.0481	0.0921	0.0208	0.0141	0.0019	0.0025
Run 6	51.144	25.172	0.5983	0.0489	0.0908	0.0209	0.0142	0.0017	0.0025
Run 7	51.166	25.145	0.6062	0.0467	0.0892	0.0208	0.0138	0.0019	0.0025
Run 8	51.111	25.134	0.6008	0.0486	0.0904	0.0208	0.0142	0.0020	0.0025
Run 9	51.220	25.185	0.6075	0.0483	0.0905	0.0209	0.0141	0.0019	0.0024
Run 10	51.179	25.218	0.6048	0.0480	0.0896	0.0206	0.0139	0.0019	0.0024
Average	**51.148**	**25.168**	**0.6009**	**0.0479**	**0.0893**	**0.0207**	**0.0141**	**0.0019**	**0.0025**
SD	0.040	0.029	0.0053	0.0007	0.0022	0.0002	0.0001	0.0001	0.0001
RSD %	0.08%	0.12%	0.88%	1.36%	2.42%	0.86%	0.96%	5.19%	2.55%

Table 3 : Results of longer-term stability of 5 days

SARM-11	Line	Fe Ka 1	Si Ka 1,2	Al Ka 1,2	Ca Ka 1,2	Mg Ka 1,2	Mn Ka 1,2	Ti Ka 1,2	K Ka 1,2	Ni Ka 1,2
	Compound	**Fe**	**SiO_2**	**Al_2O_3**	**CaO**	**MgO**	**Mn**	**Ti**	**K_2O**	**Ni**
5 Days	Average	68.957	3.226	1.425	0.0324	0.1184	0.0246	0.0429	0.1174	0.0028
5 Days	SD	0.070	0.009	0.006	0.0005	0.0026	0.0002	0.0003	0.0007	0.0002

610-1	Line	Fe Ka 1	Si Ka 1,2	Al Ka 1,2	Ca Ka 1,2	Mg Ka 1,2	Mn Ka 1,2	Ti Ka 1,2	Ni Ka 1,2	Cr Ka 1,2	Cu Ka 1,2
	Compound	**Fe**	**SiO_2**	**Al_2O_3**	**CaO**	**MgO**	**Mn**	**Ti**	**Ni**	**Cr**	**Cu**
5 Days	Average	46.993	6.822	3.647	0.1471	3.110	0.5726	0.0168	1.4791	1.9030	0.0184
5 Days	SD	0.067	0.016	0.014	0.0010	0.009	0.0014	0.0002	0.0016	0.0093	0.0003

677-1	Line	Fe Ka 1	Si Ka 1,2	Al Ka 1,2	Ca Ka 1,2	Mg Ka 1,2	Mn Ka 1,2	Ti Ka 1,2	Ni Ka 1,2	Cr Ka 1,2
	Compound	**Fe**	**SiO_2**	**Al_2O_3**	**CaO**	**MgO**	**Mn**	**Ti**	**Ni**	**Cr**
5 Days	Average	51.153	25.193	0.6019	0.0475	0.0886	0.0208	0.0140	0.0018	0.0025
5 Days	SD	0.037	0.026	0.0048	0.0008	0.0022	0.0002	0.0002	0.0001	0.0001