

Iron Ore is mined and transported to many parts of the world and from the initial mining operation to end user, at all stages in the production and transportation process analysis is required to confirm product specification.

X-ray Fluorescence analysis (XRF) is a well respected analytical technique for the analysis of Iron Ore and the **X-Supreme** takes this analysis to another level. The **X-Supreme** offers simple, rapid, accurate, multi-element analysis with the instrument operated by either laboratory or production staff, operating on a 24/7 basis leading to high quality analysis with potential cost savings. To demonstrate the performance of the **X-Supreme8000** for the analysis of Iron Ore, samples of known elemental concentration, covering a wide concentration range, e.g. 12 to 68% Fe were used to derive an empirical calibration. From this calibration samples were measured to validate the calibration, and demonstrate instrument repeatability, limit of detection etc.

In comparison when only a few standards are initially available, the alternative XRF technique of "Standardless" (Fundamental Parameter) analysis can be used. This technique is available on the **X-Supreme** as an option, and iron ore samples were measured to demonstrate instrument repeatability, etc using this alternative technique.



Sample Preparation and Presentation

In order to ensure the high accuracy required for cost optimised process control, the sample was first ground in a swing mill (with the simple addition of an additive to increase sample adhesion) and the resulting powder pressed into a strong pellet (40mm diameter) using an automatic hydraulic press set to 20 tons.

The pellet was then placed into a standard Oxford Instruments' sample holder P/No Q59 and the holder placed onto the **X-Supreme** sample carousel. Note: Up to 10 samples can be loaded at a time allowing unattended operation leading to maximised efficiency.



The Business of Science®

Performance and Results

Qualitative Analysis

The **X-Supreme8000** uses the analytical technique of Energy Dispersive X-ray Fluorescence (EDXRF) and this offers the ability to simply and rapidly observe a sample's X-ray elemental spectrum. For the determination of Al, Si, P, Mn and Fe in Iron ore, two optimised "instrument conditions" are used. The first instrument condition is for the simultaneous measurement of Al, Si and P, and a typical X-ray spectrum is shown below.

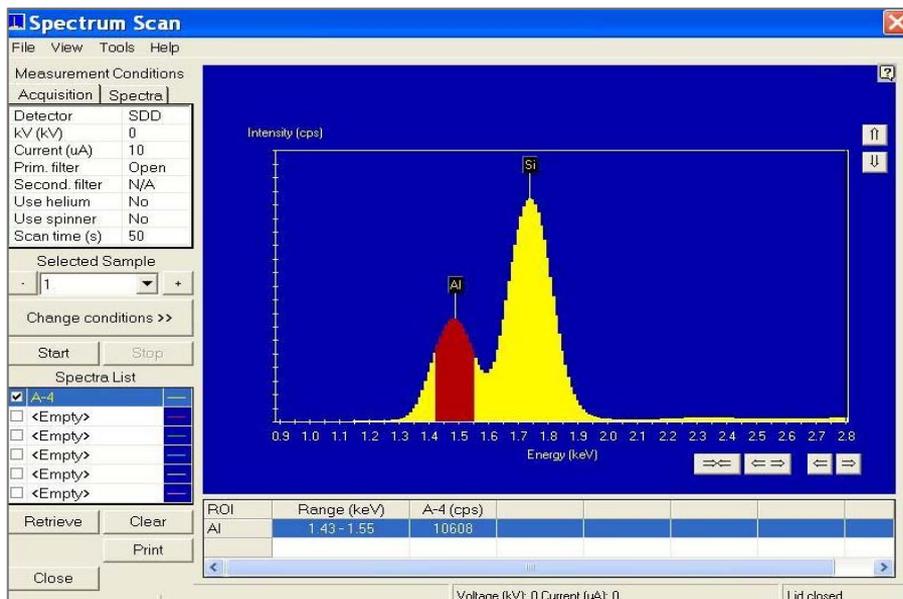


Figure 1: X-ray spectrum of iron ore standard containing 52% SiO₂ and 12% Al₂O₃
Note: High sensitivity of both Al and Si using the W target X-ray tube.

For the determination of these low atomic number elements, i.e. Al, Si and P, the **X-Supreme** uses the *combined* power of the Tungsten target X-ray tube, and high resolution of the Silicon Drift Detector.

The Tungsten target X-ray tube provides excellent elemental excitation, for example measuring the lowest atomic number of Al₂O₃ (leading to high sensitivity), while the high elemental resolution of the SDD allows the determination of the element P at a low concentration of approx 250 parts per million (0.025%) even in the presence of high concentration of SiO₂ (50%).

Second instrument condition to simultaneously measure Mn and Fe

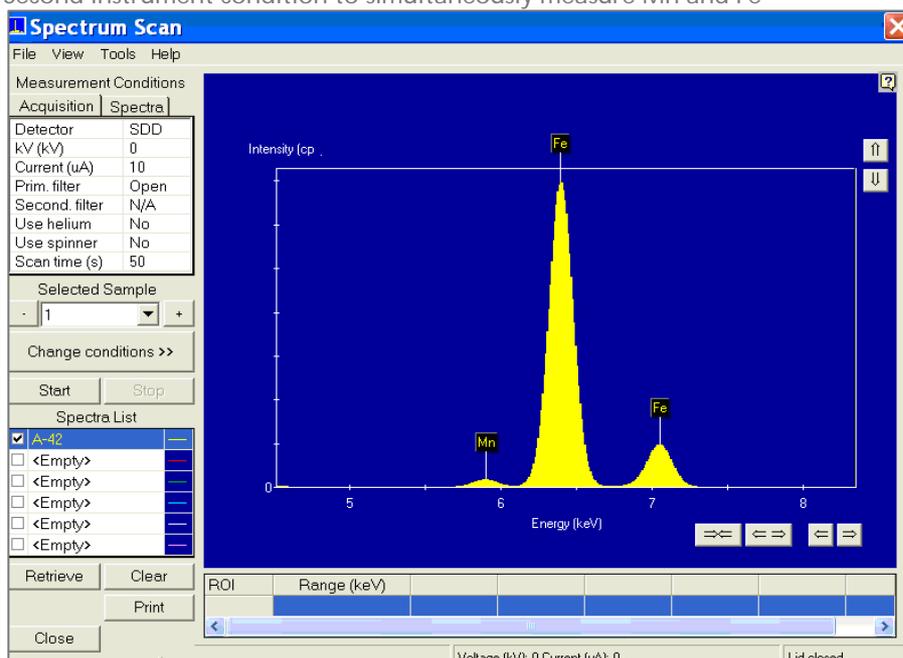


Figure2: Simultaneous measurement of Mn and Fe As for condition number 1, the high resolution SDD allows the determination of Mn (at approx. 1.5%) in the presence of high concentration of Fe 50%.
Note: Spectrum shows Mn alpha, Fe K alpha and Fe K beta X-ray emission lines.

Quantitative Analysis (Empirical calibrations)

After measuring the twenty six iron ore standards, a calibration (regression) was derived and good correlation was obtained. Typical examples are shown:

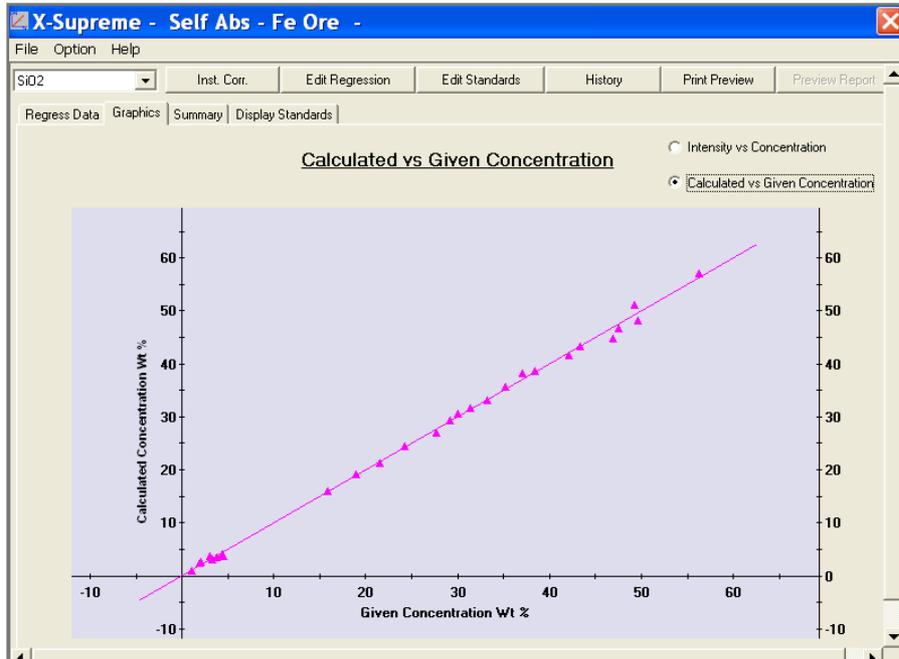


Figure 4: Calibration for SiO₂ in Iron Ore, concentration range 1-56%.



Figure 5: Calibration for Fe in Iron Ore, concentration range 12.4-62.3%.

Using the above calibration curves an iron ore sample was measured five times, i.e. leaving the sample in the instrument and taking five repeat readings, to demonstrate instrument repeatability (precision):

	Al ₂ O ₃ %	SiO ₂ %	P %	Mn %	Fe %
#1	8.32	36.28	0.0277	3.03	32.15
#2	8.34	36.27	0.0278	3.02	32.12
#3	8.31	36.28	0.0278	3.02	32.17
#4	8.33	36.3	0.027	3.01	32.08
#5	8.35	36.43	0.0281	3.02	32.11
STD Dev	0.016	0.07	0.0004	0.01	0.04

Table 1

From the above data *Table 2* below shows the performance and results for the multi-element analysis of Iron Ore using an empirical calibration.

Analyte	Range % m/m	Standard error % m/m	Theoretical limit of detection % m/m	Guaranteed limit of detection % m/m	Precision at mid range 95% Confidence (%)	Total measurement time seconds
Al ₂ O ₃	1.6 - 11.7	0.41	0.01	0.02	0.03	200
SiO ₂	1 - 56	0.83	0.05	0.10	0.14	
P	0.018 - 0.076	0.005	0.0020	0.004	0.008	
Mn	0.6 - 4.8	0.10	0.003	0.005	0.02	
Fe	12.4 - 62.3	0.96	n/a*	n/a	0.10	

Table 2

Note: A detection limit is not applicable when measuring at high percent concentrations analysis using the above empirical calibration approach allows traceability of results

Semi-Quantitative (Fundamental Parameters) Analysis

Analysis of Iron Ore can also be accomplished using the Fundamental Parameter approach. In this case one of the Oxford Instrument's standard FP templates called "FP-Minerals" was used. This template was modified to report just the elements of interest, i.e. Aluminium, Silica, Phosphorus, Manganese and Iron, sum these elements to 100%, and a post analytical calculation used to report the element compounds in the customer specified units of Al₂O₃, SiO₂, P, Mn, and Fe. In addition one iron ore standard was measured as a "type standard" (pressed pellet) to set the elemental sensitivities for an Iron Ore matrix, and whose concentration is similar to the "unknown" samples to be measured.

Using this Iron-Ore FP method, a sample was measured as an "unknown", using a total measurement time of 200 seconds, and the values obtained as follows:

Sample X2	Al ₂ O ₃ %	SiO ₂ %	P %	Mn %	Fe %
Given concentration	6.9	30.1	0.022	3.83	54.9
Measured concentration	7.64	32.3	0.013	4.08	55.85
Difference (%)	0.74	2.23	0.009	0.25	0.95
% Relative difference	11%	8%		7%	2%

As can be seen from the above data the use of the Fundamental Parameters approach allows a good semi-quantitative analysis to be achieved using just a few known elemental content samples. Results are typically within 5-20% relative, except at very low concentrations.

X-Supreme8000 instrument configuration

- a) **X-Supreme** fitted with W tube and high performance SDD
- b) Sample spinner (to reduce effects of sample inhomogeneity)
- c) Three Setting Up Samples (SUS's) P/No SUSI99D, SUFE15R, SUMI54R, for long term instrument restandardisation
- d) Helium gas purge for "Condition 1" Parameters, i.e. accurate determination of Al, Si and P.
- e) One to ten sample cells P/No Q59 for loading of samples onto the sample carousel for unattended multi-sample operation
- f) Instrument parameters: 5kV, 600uA, 100s, He (for Al, Si, P) and 15kV, 46uA, A6, 50s, Air (for Mn, Fe).
- g) Optional Fundamental Parameter software P/No XSP-FPCAL

Conclusion

The above data shows the **X-Supreme8000** offers simple, rapid, routine quality control analysis of Iron ore. If required the **X-Supreme** can be operated by production staff offering 24/7 operation giving consistent product quality and optimisation of cost.