

**Excerpt from a poster presented at the
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**PROCESS INTEGRATION OF MODERN X-RAY SPECTROMETERS:
FUTURE PERSPECTIVES IN THE STEEL AND METALS INDUSTRIES**

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3.1.2 Analysis of Fe²⁺ in sinters:

Analysis of Fe²⁺ (also commonly expressed as FeO) in sinters can be made much faster and on-line with the help of the integrated XRD system of the ARL 9900 instrument, typically in less than 100s with very good precision. A series of sinter samples were measured to establish the calibration curve using concentrations determined by wet chemistry.

A typical range of FeO may be between 4 to 9%. This means that very high sensitivities are necessary to achieve good precision. Thanks to the closely coupled optics, the integrated XRD system offers the necessary peak to background intensities to achieve satisfactory performance.

Figure 5 shows a calibration curve obtained for a series of sinter samples. Excellent correlation is obtained between the XRD intensities and the chemical concentrations of FeO. Table 1 provides the corresponding regression results. A typical standard error of estimate (accuracy) of around 0.2% is achieved.

Sinter Nr.	Intensity K.cps	Conc. of FeO (%)		Diff absol. (%)
		Nom	Calc.	
1	3.5583	5.0	5.41	0.41
2	3.5384	5.3	5.38	0.08
3	2.9626	4.4	4.27	-0.13
4	3.5743	5.4	5.44	0.04
5	3.2299	4.8	4.79	-0.01
6	3.7489	5.6	5.78	0.18
7	3.5981	5.5	5.49	-0.01
8	3.4294	5.2	5.17	-0.03
9	3.9115	6.4	6.09	-0.31
10	3.8248	6.2	5.93	-0.27
11	3.7422	5.6	5.77	0.17
12	3.7274	5.8	5.74	-0.06
13	3.0838	4.6	4.51	-0.09
Standard Error of Estimate:				0.20

Table 1: XRD calibration results of FeO in sinters

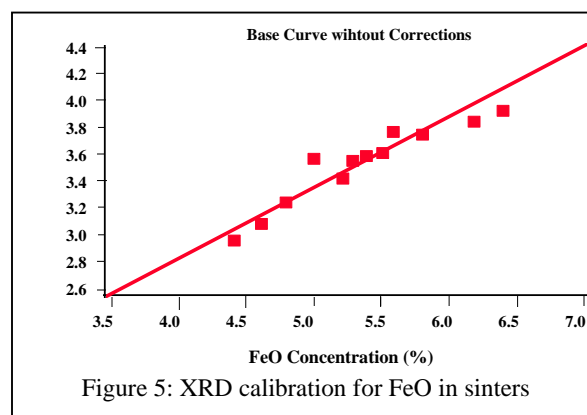


Table 2 shows short term repeatability results on a sinter sample. Since the instrument is capable of analysing all the elements or oxides by XRF together with the FeO concentration by XRD in the same sample, one can effectively combine the results from the two methods

and exploit the synergy between total elemental concentration and the specific phase content of the same element.

Run	FeO	Fe Tot.	SiO ₂	Al ₂ O ₃	CaO	MgO	TiO ₂	Mn	S	P	V
1>	8.32	56.25	5.81	2.14	10.21	1.78	0.22	0.32	0.004	0.098	0.029
2>	8.31	56.24	5.82	2.13	10.21	1.79	0.22	0.32	0.004	0.098	0.028
3>	8.30	56.25	5.81	2.13	10.20	1.79	0.22	0.32	0.004	0.098	0.029
4>	8.32	56.255	5.82	2.14	10.21	1.79	0.22	0.32	0.004	0.097	0.029
5>	8.30	56.26	5.83	2.14	10.20	1.79	0.22	0.32	0.004	0.099	0.030
6>	8.32	56.27	5.82	2.13	10.21	1.79	0.22	0.32	0.004	0.097	0.029
7>	8.30	56.27	5.82	2.13	10.20	1.79	0.22	0.32	0.004	0.098	0.030
8>	8.30	56.27	5.83	2.13	10.20	1.79	0.22	0.32	0.004	0.099	0.028
9>	8.29	56.27	5.82	2.14	10.21	1.78	0.22	0.32	0.004	0.099	0.030
10>	8.31	56.27	5.83	2.14	10.20	1.79	0.22	0.32	0.004	0.097	0.029
11>	8.32	56.28	5.82	2.14	10.20	1.79	0.22	0.32	0.004	0.099	0.030
Avg	8.31	56.26	5.82	2.14	10.20	1.79	0.22	0.32	0.004	0.098	2.43
Sd	0.01	0.01	0.01	0.00	0.00	0.00	0.00	0.00	0.000	0.001	0.00
Sd%	0.13	0.02	0.10	0.23	0.04	0.17	0.39	0.39	1.748	2.130	0.11

Table 2: Short term repeatability results on a sinter sample using the integrated XRF-XRD spectrometer

The standard deviation shown in Table 2 for most of the constituents does confirm the suitability of the instrument in a typical process control environment.

5. CONCLUSIONS

The analysis of Fe²⁺ in sinters can be done with good accuracy and precision using the integrated XRD system of the ARL 9900 instrument. A counting time of 40s to 100s is necessary depending on the precision required. This is to be compared with the typical time of 45 minutes to get the same analysis by wet chemistry.

In conclusion, the ARL 9900 X-ray spectrometer offers the state-of-the-art instrumentation to meet the present and the future analytical requirements of the metals industry in general. Thanks to its built-in modularity, the instrument can be configured and optimised with various analytical modules to handle routine elemental and phase analysis together with analysis of non-routine samples. The following figure summarizes the analytical applications in iron and steel industries using this instrument.

