

# EDXRF Analysis Report

Thermo Electron – Scientific Instruments Division

## INTRODUCTION

A set of six slag samples was submitted to Thermo Electron for composition analysis of the eight elements Mg, Al, Si, P, Ca, Ti, Mn and Fe. The samples had been prepared by rapidly cooling molten slag on a flat surface, resulting in flat, black, glassy pieces about 3 mm thick. Elemental analysis was performed with the QuanX-EC EDXRF system using the Intensity Correction quantitative algorithm.

## PROCEDURE

### Instrumentation

The samples were analyzed with the QuanX-EC system, which consists of:

- ⊕ 50 kV Rhodium target X-ray tube
- ⊕ <150 eV resolution Si(Li) Peltier-cooled detector
- ⊕ Eight-position X-ray filter wheel
- ⊕ Variety of sample-positioning options, including the 10-position automatic tray used for this analysis.



The figure on the right illustrates the chamber environment. A PC controls all setup, automation and data analysis through the Wintrace software.

### Sample Preparation

Sample preparation is critical to accurate and reproducible XRF analysis. Since x-rays effectively probe only the first 10 microns of the sample surface, any surface non-uniformities, contamination and variations in particle size could skew the analytical results. However, the very point of this evaluation was to determine if accurate quantitative results could be obtained *without* the effort and delay of sample preparation, which currently involves grinding samples and pressing the powders into pellets. Thus, no sample preparation was performed at all. A piece of each sample was placed into a standard xrf plastic sample cup and analyzed as submitted.

### Excitation Conditions

To optimize the sensitivity for the elements of interest, each sample was measured at the three different excitation conditions shown in a program screen-capture on the next page. The color-coded chart indicates which elements are optimized at the given settings (in green green) and which aren't (in yellow, red). In this case, the color code applies to the 10 kV excitation setting.

The total analysis time per sample was 320 seconds, of which over 50% was spent for the sole purpose of measuring P. As P is not critical to the analysis, this time could be cut in half or more without a noticeable effect on the quality – that is, reproducibility and accuracy – of the reported results.

H																	He
Li	Be											B	C	N	O	F	Ne
Na	Mg											Al	Si	P	S	Cl	Ar
K	Ca	Sc	Ti	Y	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
Cs	Ba	L	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn
Fr	Ra	A															
		L	La	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
		A	Ac	Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr
Condition	Filter	Voltage	Current	Atmosphere	Time	Analytes											
Mid Zc	Pd Thick	40 kV	Auto	Air	20 sec	Cr, Mn, Fe, Sr, Y, Zr, Nb, Ru											
Low Za	No Filter	4 kV	Auto	Vacuum	120 sec	Mg, Al, Si, P											
Low Zb	Cellulose	10 kV	Auto	Air	20 sec	Ca, Ti, V, Cr, Mn, Fe											

### Peak Deconvolution and Integration

Peak intensities were extracted using the XML peak-fitting method. The XML routine corrects for peak overlaps using a least-squares algorithm that fits stored reference spectra of pure analyte elements to the sample spectrum. Spectra are digitally filtered to correct for the background before the actual fitting calculation is performed.

### Quantitative Analysis

The concentration results reported below were calculated using the Intensity Correction algorithm, which empirically accounts for matrix (inter-element) effects in the range of analyte concentrations.

## RESULTS

When evaluating the outcome of an XRF analysis, there are two criteria of interest:

- ✓ [Repeatability](#) of the analysis, which depends wholly on the instrument, the analysis time, and the user's skill in selecting the right settings for the elements of interest.
- ✓ [Accuracy](#) of the analysis, which depends in large part on the accuracy of the standards, but is also affected by peak extraction and the technique used to calculate the calibration curve.

To evaluate the repeatability of the results, sample F45406 was measured 25 times with the following statistics (double-click this table to open the embedded Excel document):

F45406	Concentrations in weight percent (%)							
	MgO	Al2O3	SiO2	P2O5	CaO	TiO2	MnO	FeO
1	7.9	29.5	11.55	0.37	38.0	1.11	3.44	7.96
2	7.9	29.3	11.64	0.42	38.0	1.11	3.42	8.00
3	8.0	29.8	11.43	0.30	37.7	1.10	3.59	8.04
4	8.0	29.5	11.57	0.32	37.8	1.12	3.55	8.06
5	7.9	29.4	11.58	0.32	37.8	1.12	3.54	8.16
6	8.2	30.1	11.47	0.26	37.4	1.11	3.54	7.87
7	8.0	29.5	11.60	0.38	37.6	1.14	3.40	8.05
8	8.1	30.0	11.47	0.30	37.2	1.14	3.66	7.98
9	7.7	30.6	11.35	0.26	36.4	1.16	3.66	7.80
10	8.0	29.8	11.49	0.32	37.5	1.13	3.62	8.06
11	8.1	29.9	11.48	0.25	37.4	1.10	3.56	7.95
12	8.0	29.9	11.47	0.30	37.4	1.10	3.47	8.03
13	8.4	28.8	11.79	0.33	38.5	1.11	3.42	8.09
14	8.0	30.2	11.45	0.24	37.1	1.12	3.58	7.88
15	8.1	30.0	11.48	0.32	37.2	1.15	3.58	8.02
16	8.0	29.7	11.54	0.26	37.6	1.15	3.54	7.99
17	8.1	29.6	11.54	0.32	37.6	1.14	3.54	8.08
18	8.1	29.7	11.53	0.27	37.5	1.13	3.50	8.03
19	8.1	29.7	11.59	0.33	37.5	1.11	3.49	8.00
20	8.1	30.4	11.43	0.31	36.9	1.14	3.57	7.91
21	8.0	29.9	11.53	0.35	37.2	1.15	3.50	8.06
22	8.0	29.8	11.58	0.30	37.5	1.14	3.54	7.93
23	8.1	29.8	11.56	0.26	37.4	1.14	3.60	8.00
24	8.1	29.2	11.60	0.37	38.6	1.08	3.54	7.92
25	8.2	29.9	11.59	0.37	37.0	1.16	3.55	7.94
<b>1-Sigma</b>	0.1	0.4	0.09	0.05	0.5	0.02	0.07	0.08
<b>% RSD</b>	<b>1.6</b>	<b>1.3</b>	<b>0.8</b>	<b>15.2</b>	<b>1.2</b>	<b>1.8</b>	<b>1.9</b>	<b>1.0</b>
<b>Average</b>	<b>8.0</b>	<b>29.8</b>	<b>11.53</b>	<b>0.31</b>	<b>37.5</b>	<b>1.13</b>	<b>3.54</b>	<b>7.99</b>
<b>Given</b>	<b>8.0</b>	<b>29.9</b>	<b>11.57</b>	<b>0.31</b>	<b>37.5</b>	<b>1.09</b>	<b>3.54</b>	<b>7.90</b>

With the exception of Phosphorus – a very small peak next to a large Si peak – the repeatability of all elements was under 2.0% rsd.

The Average vs. [Given](#) comparisons for this particular standard F45406 looks very good. However, the comparisons do vary between standards, because, as mentioned above, analysis accuracy is affected by a variety of factors, including the accuracy of the standards themselves. A convenient way to evaluate analysis accuracy across the whole standard set is to review the calibration curves, which are presented for each analyte in [Appendix A](#). In an ideal world, all points would fall on the same line. However, such agreement rarely occurs in any analytical technique and even less so in XRF, because most standards and samples are not perfectly uniform – compositionally or physically – in the first 10 microns of the material surface. By the XRF yardstick, the curves in Appendix A are very good.

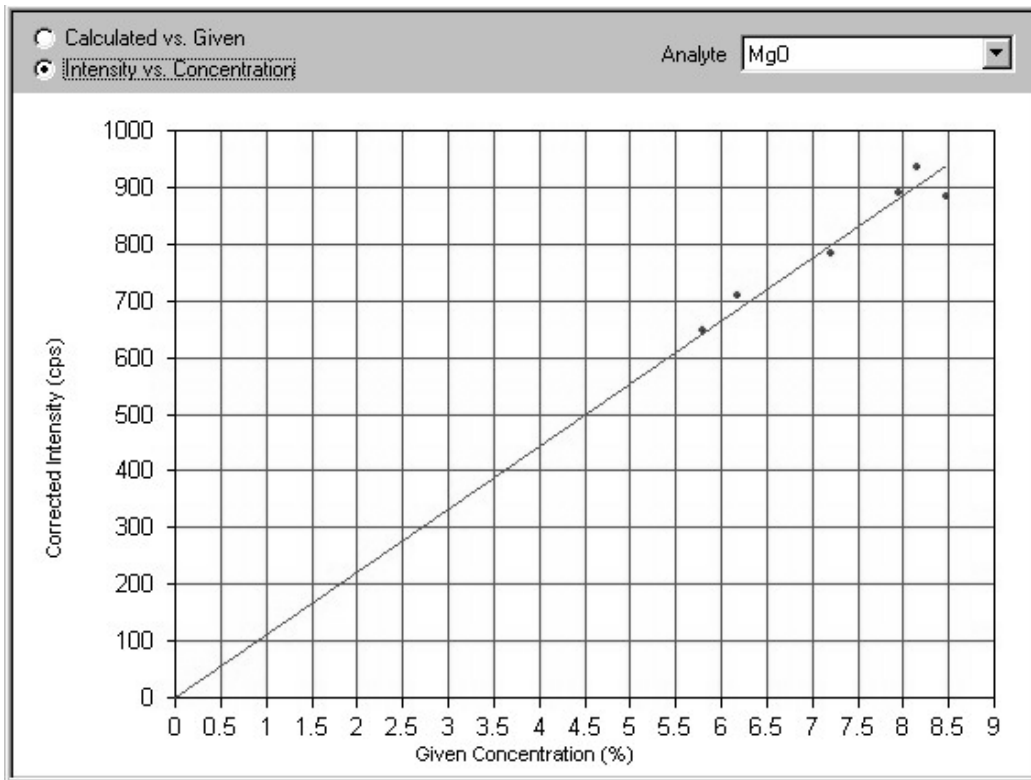
Of course, the ultimate test for analysis accuracy would be to analyze an “unknown” slag sample using this method and compare the results to expected values. This test could be done easily during your visit to our facility for a demonstration of the QuanX-EC.

Finally, since a picture is worth a thousand words, overlaid spectra of standards F45407 and R46997 are presented in [Appendix B](#).

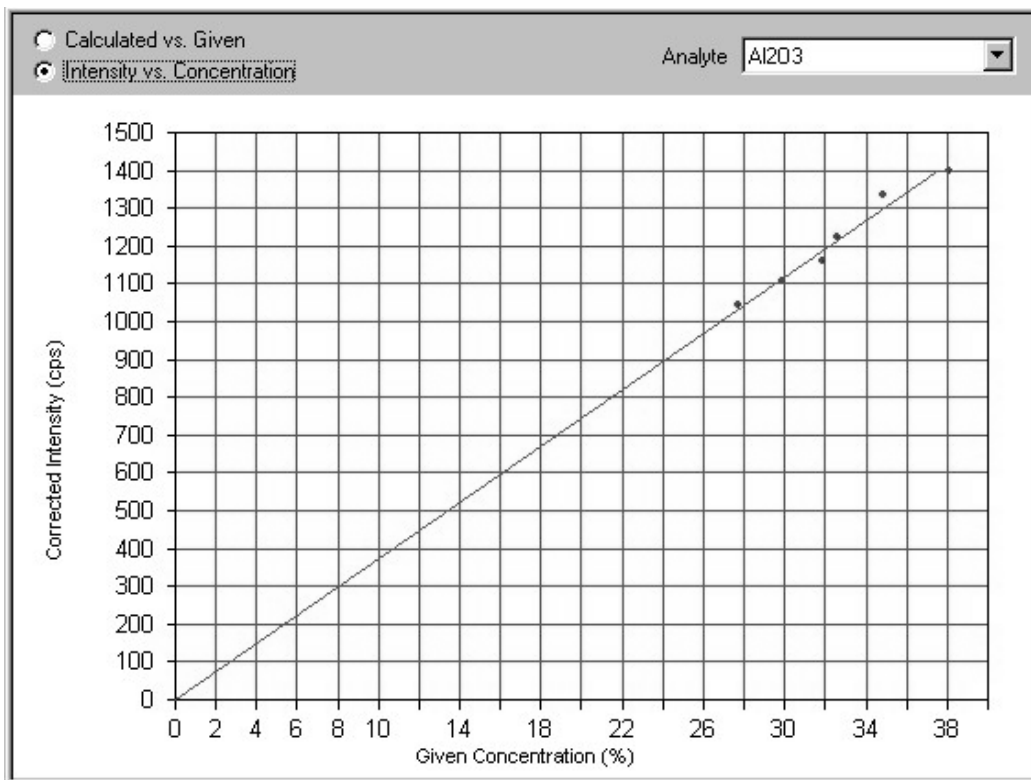
#### CONCLUSION

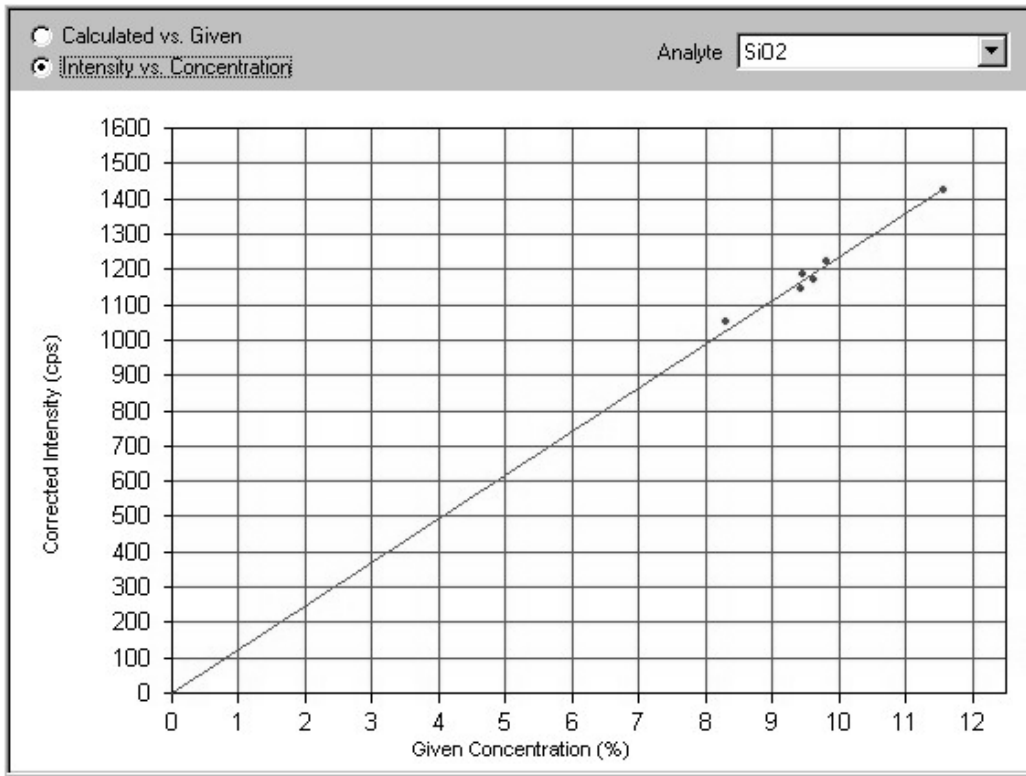
The results presented in this report demonstrate that the analysis of rapidly-cooled molten slag with the QuanX-EC EDXRF system could save the analyst hours of time in sample preparation without compromising analytical accuracy or repeatability.

APPENDIX A: CALIBRATION CURVES

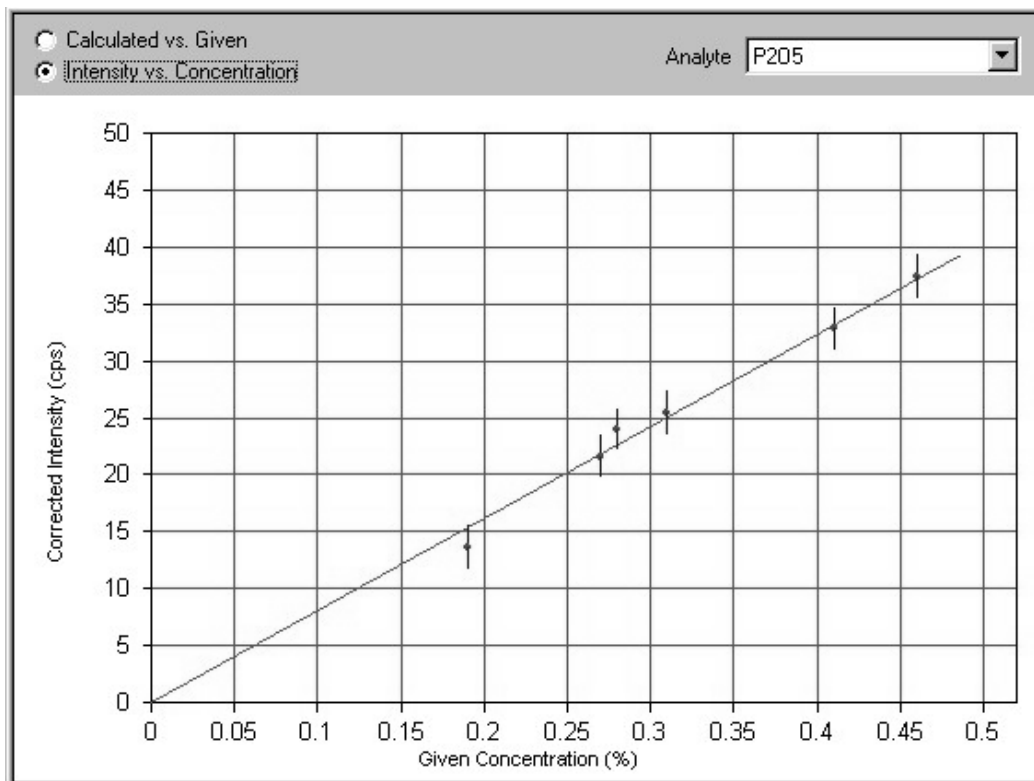


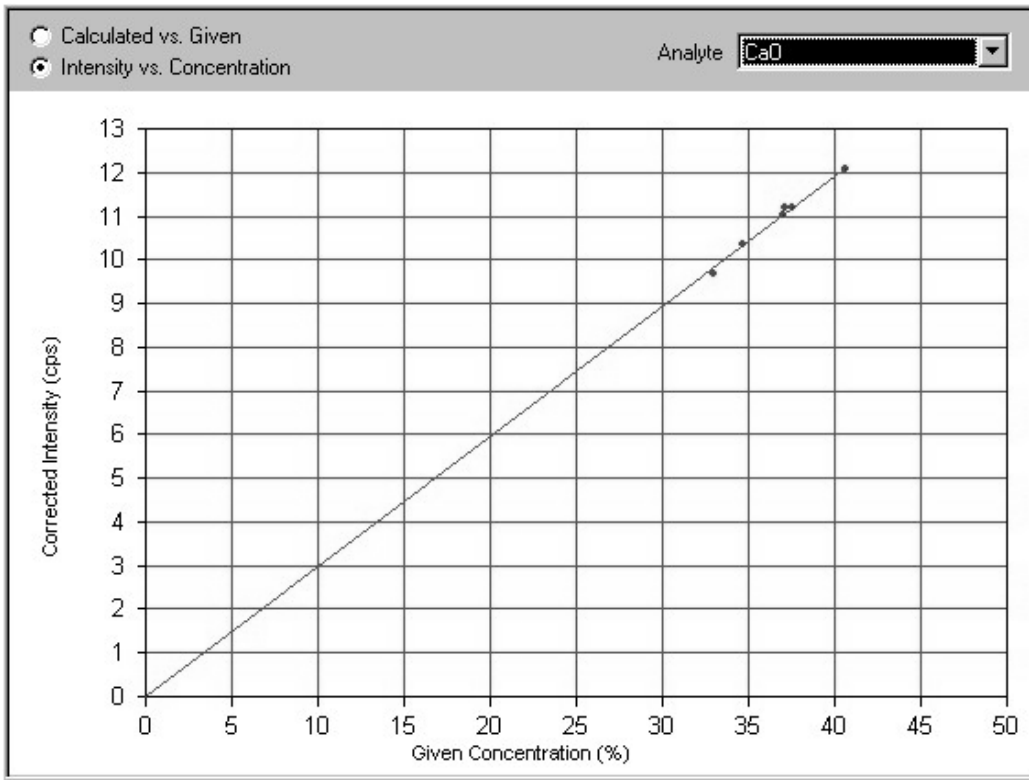
Calibration curves for MgO (above) and Al<sub>2</sub>O<sub>3</sub> (below)



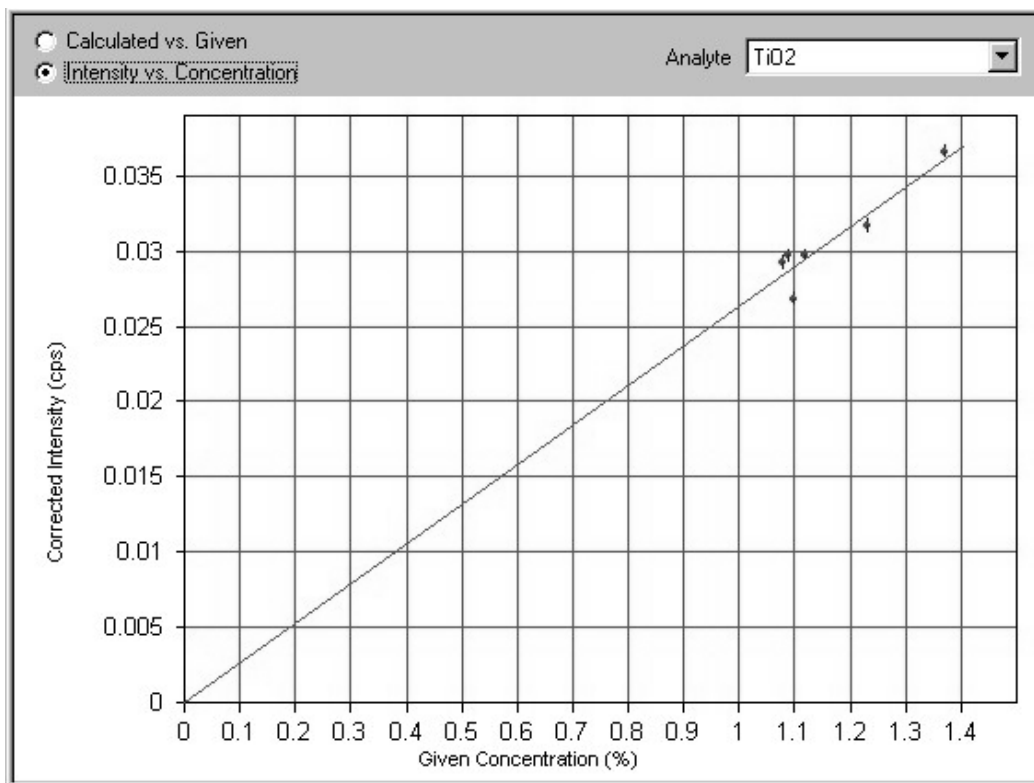


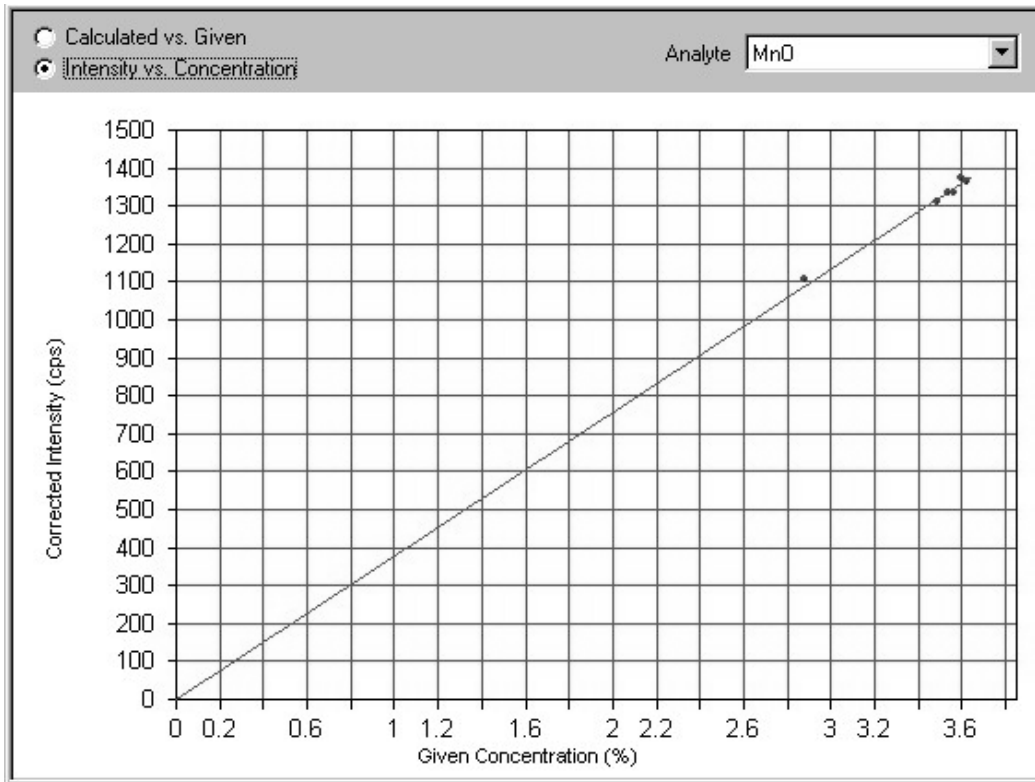
Calibration curves for SiO<sub>2</sub> (above) and P<sub>2</sub>O<sub>5</sub> (below)



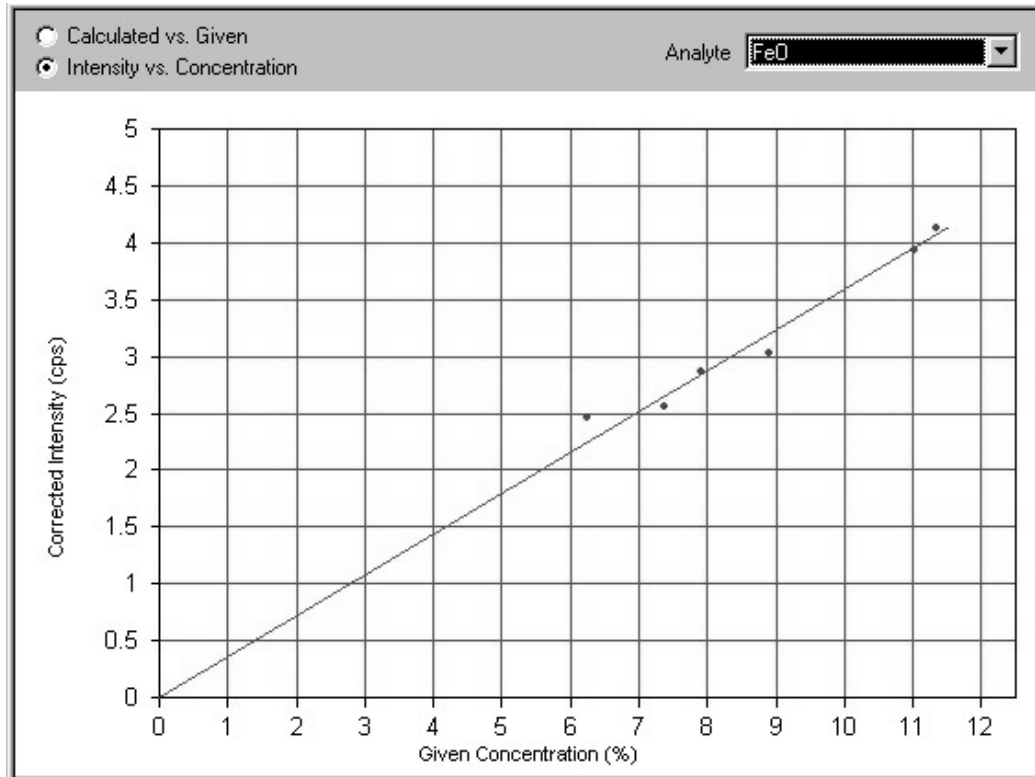


Calibration curves for CaO (above) and TiO<sub>2</sub> (below)



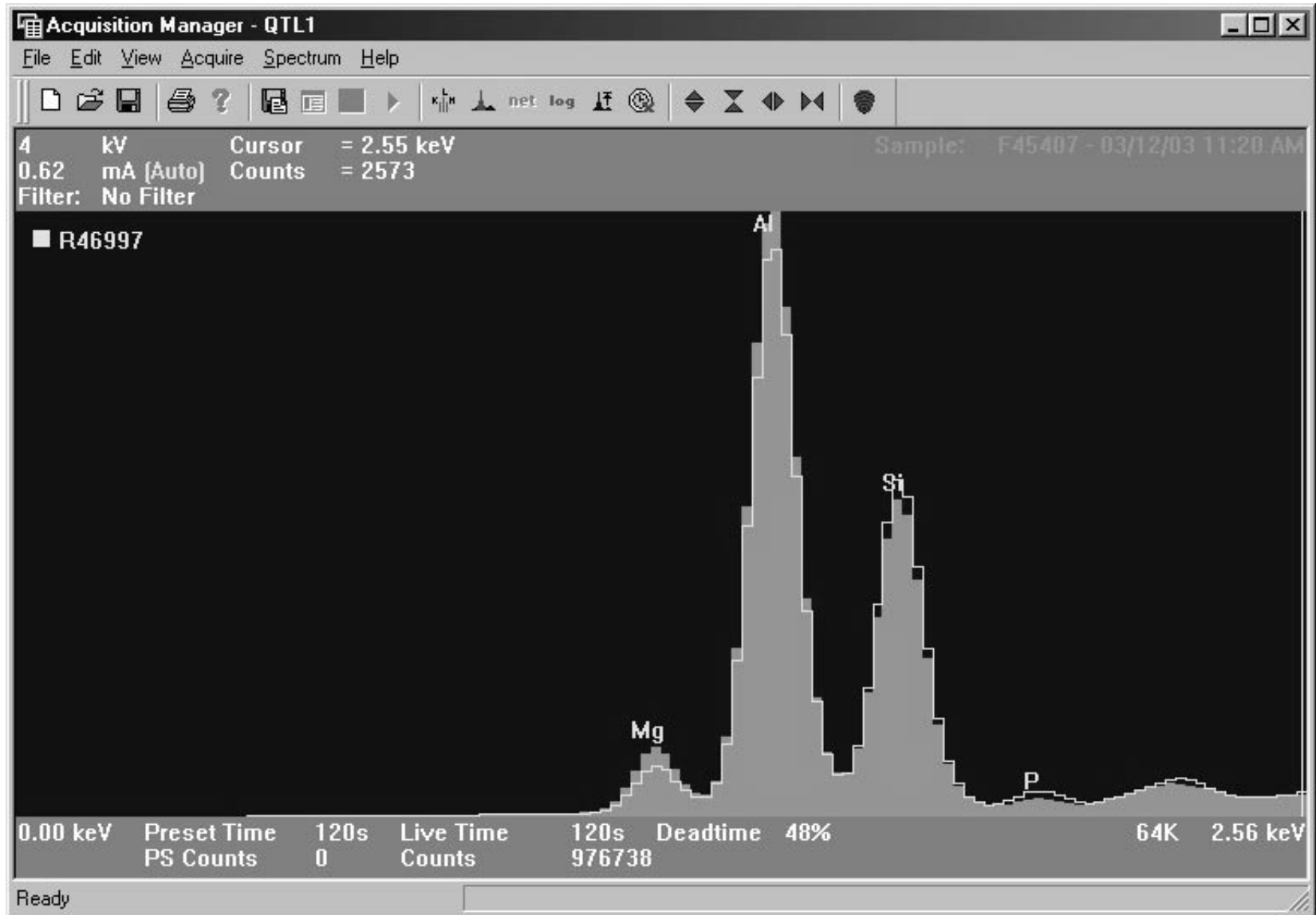


Calibration curves for MnO (above) and FeO (below)

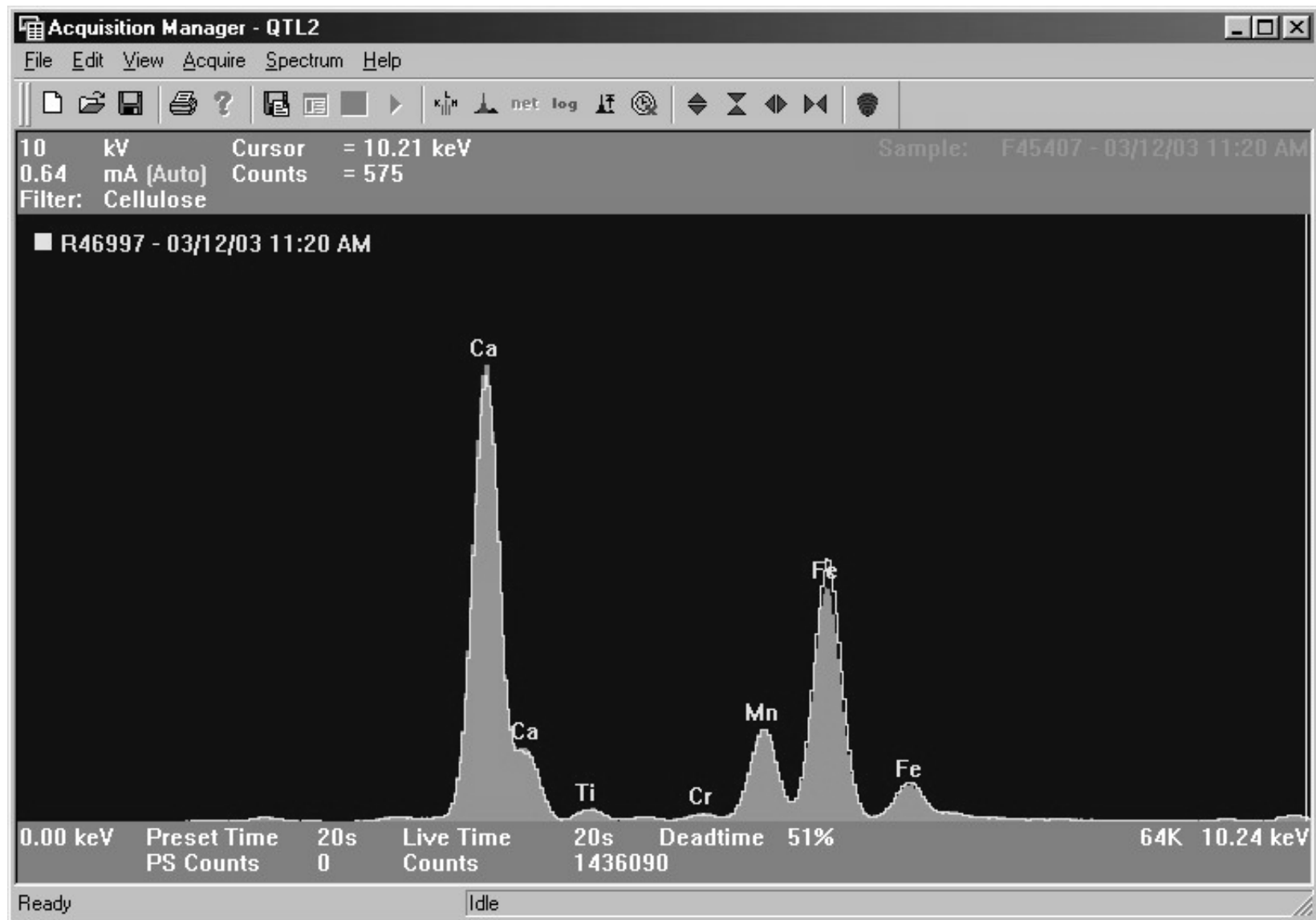




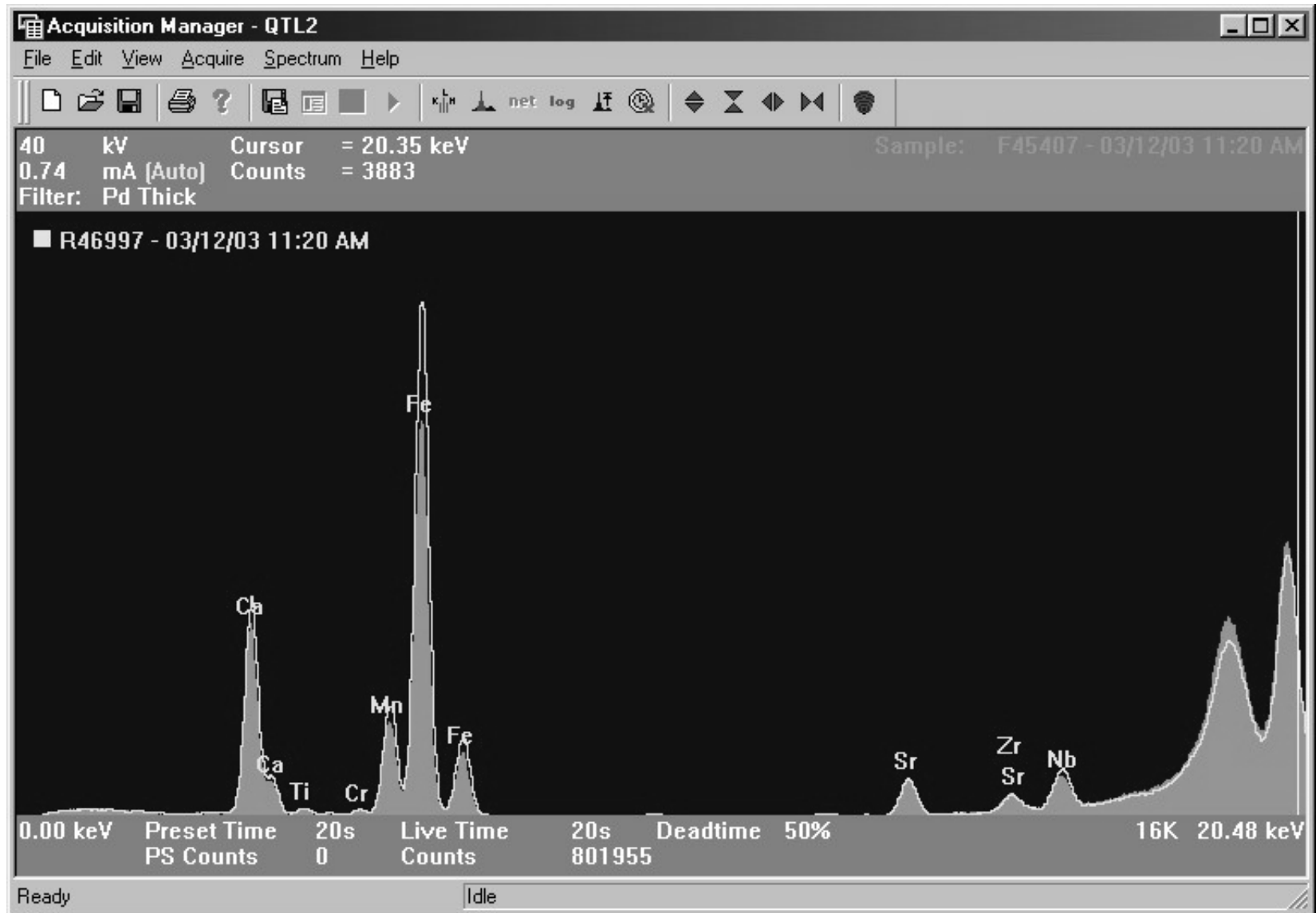
APPENDIX B: SAMPLE SPECTRA



**Figure B1** Spectra of samples F45407 (in blue) and R46997 (in yellow) acquired under conditions optimized for Mg, Al, Si and P.



**Figure B2** Spectra of samples F45407 and R46997 (in yellow) acquired under conditions optimized for Ca and Ti. The element Cr was detected in the slags but was not quantified.



**Figure B3** Spectra of samples F45407 and R46997 (in yellow) acquired under conditions optimized for Mn and Fe, as well as Sr, Zr and Nb, which were detected but not quantified.

