

# Application Note

## Portland Cement Analysis

#### Introduction:

X-ray fluorescence (XRF) is an effective and widely used method for determining the composition of cements. In recent years, manufacturers and state DOTs have both been required to pass the C114 requirements to be accredited. Routine characterization needs to be completed quickly with a minimum of operator involvement. The Rigaku **ZSXmini II** benchtop XRF spectrometer easily handles all of the process control needs for cement applications, and provides the user with exceptional resolution for separating elements that are neighbors on the periodic table. The qualitative scans shown in Figure 1 demonstrate the outstanding peak-to-background ratios of the **ZSXmini II**.

#### Sample preparation and analysis:

This test was run using a suite of NIST Portland cement standard reference materials: 1880A, 1880, 1881, 1882, 1883, 1884, 1885, 1888 and 1889. The samples were ground to the optimum particle size (< 250  $\mu$ ) for analysis by XRF. Ten (10) grams of the specimens were combined with one (1) gram boric acid H<sub>3</sub>B<sub>2</sub>O<sub>3</sub> as a binder and grinding aid. This mixture was placed in a tungsten rotary swing mill for three (3) minutes. Using a hydrolic press at twelve (12) tons for thirty (30) seconds, five (5) grams of the mixture were pressed into a thirty-one (31) mm diameter disk, which was mounted on a sample holder for analysis using the **ZSXmini II**.





### Regression statistics:

A quantitative group employing the Portland cement standard samples was established. These standards cover the entire range of values needed to control a cement-manufacturing environment. An example of the calibration curve is shown in Figure 2.

### Precision and stability test:

The Rigaku **ZSXmini II** benchtop WDXRF spectrometer is ideal for process control of cement production. It is a compact unit with a low power, 50 W X-ray tube. The system needs only 110 V power and does not require water for cooling. The user only needs to supply P-10 gas for the flow proportional detector. The Microsoft Windows<sup>®</sup> software for the **ZSXmini II** allows both novice and expert users to simply follow a flow bar through an application from start to finish. It will prompt the user at every step of the way with recommendations such as sample preparation techniques or matrix concerns. With the mere click of a mouse button, this powerful but easy-to-use software, combined with the performance of the **ZSXmini II**, delivers accurate results in a compact and cost-effective system.



#### Figure 2. Sample calibration curve

Table I. Typical elemental ranges									
Component	Quant. range (mass%)	Accuracy (mass%)							
CaO	58.67 - 67.43	0.22							
SiO <sub>2</sub>	19.98 - 23.19	0.21							
Al <sub>2</sub> O <sub>3</sub>	3.31 - 5.61	0.23							
Fe <sub>2</sub> O <sub>3</sub>	0.31 - 4.68	0.032							
SO3	1.67 - 4.61	0.018							
MgO	0.71 - 4.02	0.048							
K <sub>2</sub> O	0.16 - 1.27	0.012							
Na <sub>2</sub> O	0.02 - 0.38	0.032							
TiO <sub>2</sub>	0.16 - 0.29	0.005							
P <sub>2</sub> O <sub>5</sub>	0.025 - 0.15	0.008							

Table II. Precision and stability test												
	CaO	SiO <sub>2</sub>	$AI_2O_3$	Fe <sub>2</sub> O <sub>3</sub>	SO3	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	TiO <sub>2</sub>	$P_2O_5$		
n=1	65.08	21.03	5.33	2.98	2.05	0.90	0.52	0.19	0.26	0.112		
2	65.11	21.21	5.34	3.00	2.03	0.89	0.51	0.20	0.27	0.108		
3	65.04	21.14	5.34	2.99	2.05	0.91	0.51	0.22	0.25	0.111		
4	64.90	21.15	5.34	2.96	2.07	0.90	0.52	0.18	0.26	0.108		
5	64.91	21.22	5.33	2.98	2.07	0.90	0.52	0.22	0.25	0.108		
6	64.97	21.11	5.34	2.98	2.05	0.90	0.52	0.23	0.25	0.110		
7	64.95	21.12	5.33	2.99	2.06	0.91	0.52	0.17	0.25	0.108		
8	64.96	21.12	5.31	2.96	2.05	0.86	0.52	0.17	0.25	0.117		
9	65.02	21.27	5.31	2.97	2.05	0.89	0.52	0.21	0.26	0.112		
10	64.99	21.11	5.37	2.99	2.06	0.93	0.52	0.20	0.25	0.107		
Average	64.99	21.15	5.33	2.98	2.05	0.90	0.52	0.20	0.26	0.110		
SD	0.07	0.07	0.02	0.01	0.01	0.02	0.004	0.021	0.007	0.003		
Chem. value	65.20	21.40	5.40	3.00	2.00	0.90	0.51	0.26	0.26	0.110		

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