



LAB-X5000 for the rapid analysis of cement and cement making materials

INTRODUCTION

Cement is manufactured by combining different raw materials (e.g. limestone, clay, bauxite, sand) to give the finished product its desired chemical composition and properties: the raw materials are finely ground, blended together, then heated at very high temperature in a kiln to generate cement clinker. Gypsum is added to the clinker to produce the finished cement.

To ensure quality, it is critical to control the contents of key compounds during the manufacturing process: while the silica (SiO_2) content influences the cement's strength and alumina (Al_2O_3) affects the setting time, the lime (CaO) content impacts both; the iron oxide (Fe_2O_3) mostly imparts colour to the cement.

X-ray fluorescence (XRF) spectrometry is one of the simplest instrumental techniques for analysis in minerals extraction facilities and cement works because the sample preparation is simple, and the analysis is fast. For many years, benchtop energy-dispersive XRF (EDXRF) spectrometers have been used successfully as a quality control tool in quarries and cement plants to control raw material as well as production samples. Hitachi High-Tech have a long and highly respected reputation within the minerals and cement industry with instruments such as the Lab-X3000 series, Twin-X and X-Supreme providing easy to use, accurate, cost-effective and dependable 24/7 analysis.

CEMENT ANALYSIS MADE EASY

With the Hitachi High-Tech LAB-X5000 EDXRF analyser, the analysis of minerals and cement couldn't be easier. Routine analysis is carried out by placing a pressed pellet into a sample holder, placing the holder in the LAB-X's analysis port, and pressing a button to start the measurement. Results are available within minutes on the large, industrial LCD touch screen, showing the contents for all key oxides for example Al_2O_3 , SiO_2 , CaO and Fe_2O_3 in cement and process calculations such as lime saturation factor (LSF), silica ratio (SR) and alumina ratio (AR). Note: these calculations can be switched off if not needed.



The combination of a high-resolution detector and optimised calibration parameters ensure that you get results you can trust. For this application, the LAB-X is equipped with a sample spinner to compensate for residual sample heterogeneity and deliver repeatable results. For most analyses, built-in atmospheric compensation delivers reliable analysis without the need for helium or vacuum purge, minimising the cost per analysis while retaining optimum stability. Helium is used only when strictly necessary, for example when measuring low energy elements such as Na and Mg.

Users can set-up a QC routine with concentration targets and lower and upper limits for each control element; the displayed control graphs clearly show if the test is in control.

With up to 100,000 results stored on the analyser itself, operators can view new and old results easily, print them on the integrated printer for a hard-copy record, download them on a USB memory device, and even upload them to our ExTOPE Connect cloud service to manage them remotely. With the LAB-X connected to WiFi, the results are uploaded to your cloud account automatically, so you do not need to be near the analyser to access your data!

SAMPLE PREPARATION

For precise and accurate multi-element analysis, it is essential to prepare the samples to meet the high standards of production control that are the norm in the minerals and cement industry. This means grinding a powder sample in a swing mill with a grinding additive to prevent the sample clogging the mill and to help it bind into a pellet.

The best type of additive is one available as tablets of precise weight so that only the sample needs weighing. Hitachi High-Tech supply one that has been used in the cement industry for many years (Part Number CM0039). The resultant fine mixture is formed into a strong pellet (usually 40 mm diameter) by compression in a die using a hydraulic press. Operators then fit the pellet into a pellet holder (Part Number Q59), place it in the LAB-X's analysis port, and press the Start button on the analyser.

PERFORMANCE AND RESULTS

The data shown in this section highlights the typical performance that the LAB-X delivers. A simple empirical calibration was created by measuring a series of reference materials for each type of mineral and for cement to establish the relationship between the oxides' contents and their X-ray signal. The calibrations used the pre-defined parameters included in the LAB-X5000 Cement Maxi package (pre-loaded in the analyser). The detailed measurement parameters can be found in the LAB-X method sheets included in the package.

The limits of detection were calculated from the results of 10 repeat measurements of standards containing the lowest concentration of the element and takes into account the effect of the other elements.

Table 1: Typical calibration performance for routine cement analysis (air path)

Analyte	Calibration range (% m/m)	Standard error of calibration (% m/m)	Limit of detection (3σ) (% m/m)	Limit of quantification (10σ) (% m/m)	Precision (95% confidence) (% m/m)	Total analysis time (seconds)
Al ₂ O ₃	3.9-7.1	0.1	n/a	n/a	0.08	160
SiO ₂	18.6-22.4	0.3	n/a	n/a	0.09	
CaO	57.6-67.9	0.5	n/a	n/a	0.09	
Fe ₂ O ₃	0.2-3.1	0.05	0.005	0.015	0.01	

Note: the precision was calculated from the results of 10 repeat measurements of a standard containing 6.2% Al₂O₃, 18.6% SiO₂, 60.9% CaO and 2.9% Fe₂O₃.

Element	Concentration	Unit
Al ₂ O ₃	3.78	%
SiO ₂	20.64	%
CaO	65.39	%
Fe ₂ O ₃	1.92	%
%LSF	103	%
SR	3.63	

Clear results display



Pressed pellet in pellet holder



Starting the analysis

Table 2: Typical calibration performance for routine cement analysis (Helium path)

Analyte	Calibration range (% m/m)	Standard error of calibration (% m/m)	Guaranteed limit of detection (3σ) (% m/m)	Limit of quantification (10σ) (% m/m)	Precision (95% confidence) (% m/m)	Total analysis time (seconds)
Al ₂ O ₃	3.9 - 7.1	0.08	n/a	n/a	0.03	160
SiO ₂	18.6 - 22.4	0.3	n/a	n/a	0.04	
CaO	57.6 - 67.9	0.5	n/a	n/a	0.13	
Fe ₂ O ₃	0.2 - 3.1	0.04	0.005	0.012	0.011	

Note: the sample used to determine the precision contained 6.2% Al₂O₃, 18.6% SiO₂, 60.9% CaO and 2.9% Fe₂O₃.

Table 3: Typical calibration performance for extended finished cement analysis

Analyte	Calibration range (% m/m)	Standard error of calibration (% m/m)	Guaranteed limit of detection (3σ) (% m/m)	Limit of quantification (10σ) (% m/m)	Precision (95% confidence) (% m/m)	Total analysis time (seconds)
Na ₂ O	0.02 - 1.1	0.04	0.06	0.13	0.04	300
MgO	0.81 - 4.5	0.08	0.03	0.06	0.03	
Al ₂ O ₃	3.9 - 7.1	0.1	n/a	n/a	0.03	
SiO ₂	18.6 - 22.4	0.3	n/a	n/a	0.04	
P ₂ O ₅	0.02 - 0.31	0.013	0.004	0.009	0.003	
SO ₃	2.1 - 4.6	0.09	0.02	0.05	0.01	
K ₂ O	0.09 - 1.2	0.03	0.006	0.013	0.005	
CaO	57.6 - 67.7	0.5	n/a	n/a	0.2	
TiO ₂	0.08 - 0.37	0.005	0.007	0.015	0.004	
Cr ₂ O ₃	0.002 - 0.06	0.002	0.001	0.002	0.001	
Mn ₂ O ₃	0.007 - 0.26	0.005	0.001	0.002	0.002	
Fe ₂ O ₃	0.15 - 3.1	0.04	0.004	0.009	0.005	
ZnO	0.001 - 0.11	0.001	0.0006	0.001	0.003	
SrO	0.02 - 0.64	0.005	0.002	0.005	0.002	

Note: the precision was calculated from the results of 10 repeat measurements of a standard containing elements concentrations at around the mid-range of the calibration.

Table 4: Typical calibration performance for high-calcium limestone analysis

Analyte	Calibration range (% m/m)	Standard error of calibration (% m/m)	Guaranteed limit of detection (3σ) (% m/m)	Limit of quantification (10σ) (% m/m)	Precision (95% confidence) (% m/m)	Total analysis time (seconds)
MgO	1.0 - 4.1	0.05	0.02	0.07	0.02	180
Al ₂ O ₃	0.6 - 3.3	0.03	0.02	0.06	0.02	
SiO ₂	2.3 - 10.4	0.11	n/a	n/a	0.02	
K ₂ O	0.1 - 0.8	0.012	0.005	0.016	0.003	
CaO	42.3 - 52.6	0.4	n/a	n/a	0.08	
Fe ₂ O ₃	0.4 - 1.3	0.005	0.004	0.015	0.005	

Note: the sample used to determine the precision contained 1.3% MgO, 2.2% Al₂O₃, 6.3% SiO₂, 0.5% K₂O, 48.2% CaO and 0.9% Fe₂O₃.

Table 5: Typical calibration performance for dolomitic limestone analysis

Analyte	Calibration range (% m/m)	Standard error of calibration (% m/m)	Guaranteed limit of detection (3σ) (% m/m)	Limit of quantification (10σ) (% m/m)	Precision (95% confidence) (% m/m)	Total analysis time (seconds)
MgO	18.8 - 21.2	0.04	n/a	n/a	0.09	180
Al ₂ O ₃	0.2 - 0.4	0.02	0.010	0.033	0.010	
SiO ₂	0.3 - 2.2	0.11	0.013	0.044	0.013	
CaO	30.0 - 32.8	0.26	n/a	n/a	0.07	
Fe ₂ O ₃	0.1 - 0.6	0.02	0.002	0.007	0.002	

Note: the sample used to determine the precision contained 20.3% MgO, 0.3% Al₂O₃, 1.2% SiO₂, 31.3% CaO and 0.2% Fe₂O₃.

SUMMARY

Once calibrated, Hitachi High-Tech's LAB-X5000 provides reliable minerals and cement analysis, enabling operators to make process decisions fast. Its ease of use and ruggedness make it an ideal tool at the mine, quarry and cement plant, delivering results within minutes for maximum productivity.

Visit www.hitachi-hightech.com/hha for more information.



ORDERING INFORMATION

The minimum needed for this application is:

LAB-X5000 Cement package, which includes: the analyser with sample spinner, user manual (on USB memory device), pre-loaded method parameters and method sheet (calibration instructions), setting up samples, pressed pellet holders and a consumables pack

LAB-X5000 Cement Maxi package; includes all the above, plus a helium purge for enhanced performance, and additional pre-loaded method parameters and method sheets for extended cement analysis, and limestone analysis.

Optional:

Grinding/binding pellets, bag of approximately 5000, Part Number CM0039

40 mm-diameter aluminium backing cups, box of 600, Part Number QX1015/600



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