Hitachi X-Supreme8000 for cost-effective quality control analysis of china clay (kaolin), ball clay and silica sand

Instrument Package: 10011621

INTRODUCTION

In the production and use of minerals and ceramics, quality control includes the chemical analysis of raw materials and finished goods. For this the available techniques are: wet chemical analysis, atomic absorption spectrophotometry (AAS), inductively coupled plasma spectrometry (ICP) and X-ray fluorescence (XRF) spectrometry. Wet chemical analysis is slow and needs skilled chemists to be reliable. AAS and ICP both involve a lengthy chemical preparation stage that demands a high degree of skill. Traditional wavelength dispersive X-ray fluorescence spectrometry (WDXRF) and the new generation of benchtop energy dispersive XRF (EDXRF) offer simplicity of sample preparation, and speed of measurement.

Hitachi have a long and highly respected reputation within the minerals industry with instruments such as the LAB-X and Twin-X providing simple to use, accurate, cost-effective and dependable 24/7 analysis.

Hitachi's X-Supreme is a high-performance EDXRF spectrometer that successfully performs a wide range of elemental analyses required in the minerals industry. The X-Supreme is the perfect analyzer for the rapid multi-element determination of a range of mineral and ceramic materials. It includes sodium determination as well as the standard elements such as Al, Si, K, Ca, Fe etc., and retains the ability for routine use by production staff. This new level of performance has been achieved using Hitachi's unique Focus SD technology.

Silica sand is an abundant industrial material with many varied applications. High purity silica sand is the largest component in glass making and less pure sand is one of the raw materials for the manufacture of ceramics. The foundry industry uses silica sand for making molds and in construction it is a component of asphalt. It also has uses as a media for water filtration.

China clay (kaolin) and ball clay are two of the raw materials that go into many ceramics. Ball clay has good plasticity and strong bonding power, whereas china clay, due to its great purity, is white burning and the most refractory of all clays.

In all cases routine quality control, often carried out by production staff is an increasing requirement.

INSTRUMENTAL

Hitachi have focused on the particular requirements of the mineral and ceramic industries to produce a dedicated analyzer optimized for the analysis of china clay, ball clay, silica sand, etc. whilst retaining many of the critically important features for the minerals industry such as minimal dust ingress, high reliability and stability, and "operation by anyone" principles.

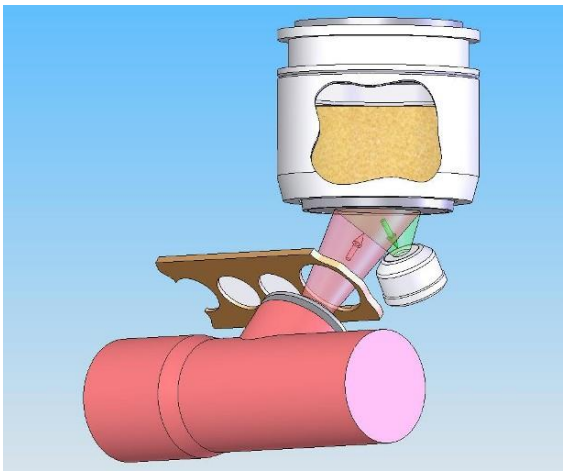

FIGURE 1: HITACHI FOCUS SD TECHNOLOGY

Hitachi's Focus SD technology obtains the highest level of performance from low atomic number elements such as sodium to high atomic number elements such as strontium. It combines a field-proven tungsten-target X-ray tube and associated Silicon Drift Detector (SDD) which provides high spectral resolution. Focus SD allows optimum speed of analysis and low detection limits.
This delivers supreme performance for all elements of interest, including sodium.

All control of the instrument is through the X-Supreme's integrated PC and software which provides sophisticated calibration models that handle a wide variability of samples, while remaining easy to use. The software features easy data manipulation and storage, a report writing facility and data export.

With an enviable reputation for attention to detail on the cement industries' principal requirement of "minimal dust ingress", the X-Supreme was designed with this in mind. A unique "wind tunnel" arrangement draws external air into a section of the X-Supreme which provides a heat transfer. This section is completely isolated from the main components thereby preventing dust ingress ensuring long term reliability (see figure below).

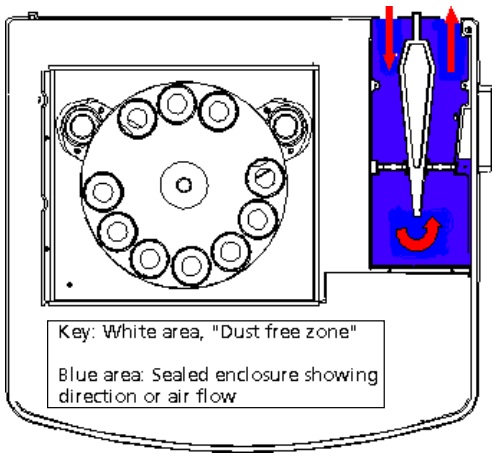

FIGURE 2: X-SUPREME: CUTAWAY PLAN VIEW SHOWING THE EXTERNAL AIR FLOW

The X-Supreme's compactness and robustness make it ideal for location either in laboratories or in production sites for twenty-four-hour operation. The X-Supreme includes a ten-position autosampler to enable simple and unattended multi-sample analysis.

SAMPLE PREPARATION AND PRESENTATION

Sample preparation is exactly the same as for traditional WDXRF in order to give the high accuracy necessary for production control. For ball clays and silica sand which contain relatively large particles of silica, this means grinding the dry sample in a swing mill with an additive to increase adhesion (such an additive is available from Hitachi as an option). The resultant fine powder forms into a strong pellet by compression in a die using a hydraulic press. Pellets then fit into a Hitachi pellet holder. China clays simply need drying and reducing to a fine powder in a small analytical mill or blender ("coffee mill" type) and pressed into a pellet.

After placing the samples on the instrument tray, entering their identification and selecting their tray position and method, measurement can be started by simply pressing the Start key on the integrated keypad. Preliminary results are available after only a few seconds (Simultaneous mode) or after the first condition (Sequential mode) and are updated until the end of the measurement. Results can be displayed in user-defined order and format, printed on an external USB printer, as well as electronically transmitted using USB or Ethernet as required.

CALIBRATION

Each X-Supreme comes pre-programmed with operating parameters that are optimized for each application included in this package. It is then simply a matter of following the relevant Hitachi method sheet and running at least six standards with known analyte concentrations.

Note: An optional Standardless (Fundamental para meter) method is available on the X-Supreme and this allows for elemental measurements when no standards are available.

QUALITY CONTROL AND INSTRUMENT CORRECTION

Setting-up-samples (SUSs) are provided by Hitachi and are measured at the time of calibration. They act as long term reference for the sensitivity of each element's X-rays.

From time to time, the instrument needs restandardizing by measuring the setting-up-samples. Capitalizing on the excellent stability of the X-Supreme, the best strategy is regular measurement of a quality control (QC) sample and only to restandardize when a result exceeds control limits. This is an easy process on the X-Supreme when using the QC check sample routine. This routine displays the QC check sample's results over time, in both graphical and numerical format, allowing a rapid assessment to be made. If the results are inside customer specified tolerances then routine analysis can proceed but if outside then restandardization is necessary.

TYPICAL PERFORMANCE AND RESULTS

The optimized Focus SD technology in this package enables unprecedented determination of sodium. **Figure 3** illustrates a typical sodium calibration for finished cement.

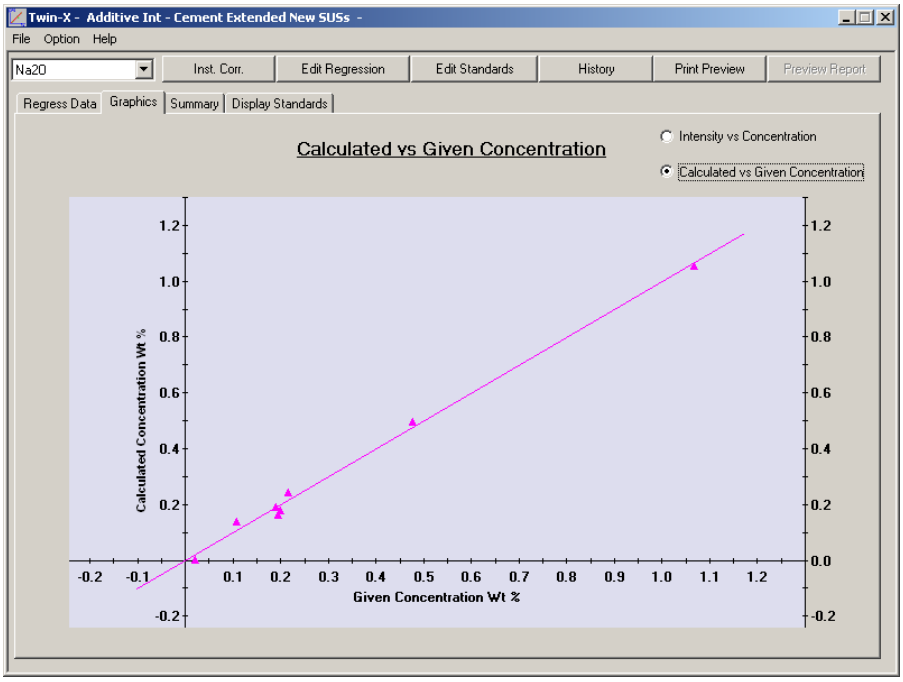

FIGURE 3: Na_2O CALIBRATION GRAPH FOR CEMENT

Tables 1 to **4** show typical calibration performance that illustrates how the X-Supreme can accurately and precisely analyses china clay, ball clay and silica sand.

TABLE 1: TYPICAL CALIBRATION PERFORMANCE FOR CHINA CLAY ANALYSIS XSMET-04D.v1

Analyte	Range (%$^m/_m$)	Standard error of calibration (%$^m/_m$)	Guaranteed limit of detection (3σ) (%$^m/_m$)	Precision (95% confidence) (%$^m/_m$)	Measurement time (minutes)
MgO	0.14 - 0.38	0.008	0.010	0.003	~ 3
Al_2O_3	35 - 38	0.2	n/a	0.02	
SiO_2	47 - 50	0.2	n/a	0.03	
K_2O	0.82 - 2.80	0.037	0.010	0.01	
TiO_2	0.02 - 0.09	0.002	0.002	0.001	
Fe_2O_3	0.26 - 1.39	0.021	0.003	0.003	

The precision was calculated from 10 repeat measurements of a standard containing 0.28% m/m MgO, 36.5% m/m Al_2O_3, 48.3% m/m SiO_2, 2% m/m K_2O, 0.06% m/m TiO_2, and 0.9% m/m Fe_2O_3.

Note: Precision can be improved by increasing the counting time for the analytes.

TABLE 2: TYPICAL CALIBRATION PERFORMANCE FOR BALL CLAY ANALYSIS XSMET-04C.v1

Analyte	Range (%$^{m}/_{m}$)	Standard error of calibration (%$^{m}/_{m}$)	Guaranteed limit of detection (3σ) (%$^{m}/_{m}$)	Precision (95% confidence) (%$^{m}/_{m}$)	Measurement time (minutes)
Na_2O	0.25 - 0.44	0.04	0.030	0.004	~ 4
MgO	0.33 - 0.68	0.01	0.013	0.008	
Al_2O_3	13.1 - 26.1	0.4	n/a	0.11	
SiO_2	55.2 - 78.7	1.2	n/a	0.25	
K_2O	1.37 - 2.38	0.04	0.008	0.005	
CaO	0.05 - 0.31	0.008	0.005	0.003	
TiO_2	1.17 - 1.89	0.04	0.010	0.006	
Fe_2O_3	0.75 - 1.37	0.01	0.004	0.003	

The precision was calculated from ten repeat measurements of a standard containing 0.36% m/m Na_2O, 0.51%m/m MgO, 22.3% m/m Al_2O_3, 65.8% m/m SiO_2, 2.11% m/m K_2O, 0.11%m/m CaO, 1.54% m/m TiO_2 and 0.92% m/m Fe_2O_3.

Note: Precision can be improved by increasing the counting time for the analytes.

TABLE 3: TYPICAL CALIBRATION PERFORMANCE FOR SILICA SAND XSMET-04B.V1

Analyte	Range (mg.kg^{-1})	Standard error of calibration (mg.kg^{-1})	Guaranteed limit of detection (3σ) (mg.kg^{-1})	Precision (95% confidence) (mg.kg^{-1})	Measurement time (minutes)
Al_2O_3	310 - 6290	19	100	45	< 7
SiO_2	98.8 - 99.9 % $^{m}/_{m}$	0.3% $^{m}/_{m}$	n/a	0.1% $^{m}/_{m}$	
K_2O	35 - 4640	137	12	6	
CaO	45 - 150	14	9	4	
TiO_2	135 - 500	24	10	5	
Fe_2O_3	70 - 725	22	6	3	

The precision was calculated from 10 repeat measurements of a standard containing 550 mg.kg^{-1} Al_2O_3, 80 mg.kg^{-1} K_20, 110 mg.kg^{-1} CaO, 180 mg.kg^{-1} TiO_2 and 110 mg.kg^{-1} Fe_2O_3.

TABLE 4: TYPICAL CALIBRATION PERFORMANCE FOR LOW FE IN SILICA SAND XSMET-04A.V1

Analyte	Range (mg.kg^{-1})	Standard error of calibration (mg.kg^{-1})	Guaranteed limit of detection (3σ) (mg.kg^{-1})	Precision (95% confidence) (mg.kg^{-1})	Measurement time (seconds)
Fe_2O_3	2 - 900	8	< 3	1.4	300
Fe_2O_3	2 - 900	8	7	3.4	50

The precision was calculated from 10 repeat measurements of a standard containing 36mg.kg^{-1} Fe_2O_3.

ROUTINE ANALYSIS

Pressed pellets are placed in Hitachi pellet holders, and placed on the X-Supreme sample tray. Their labels are entered on the routine analysis screen, and the analysis run is started.

The X-Supreme “Live result update” function displays preliminary results after only a few seconds (Simultaneous mode) for each sample. This offers the potential to spot production or sample issues very quickly and respond adequately.

INSTRUMENT SPECIFICATION

The X-Supreme instrument package for the analysis of mineral and ceramic materials is 10011621. This includes the pre-loaded analytical methods, associated method sheets, setting-up samples and other accessories, e.g. sample holders etc. required for the complete operation.