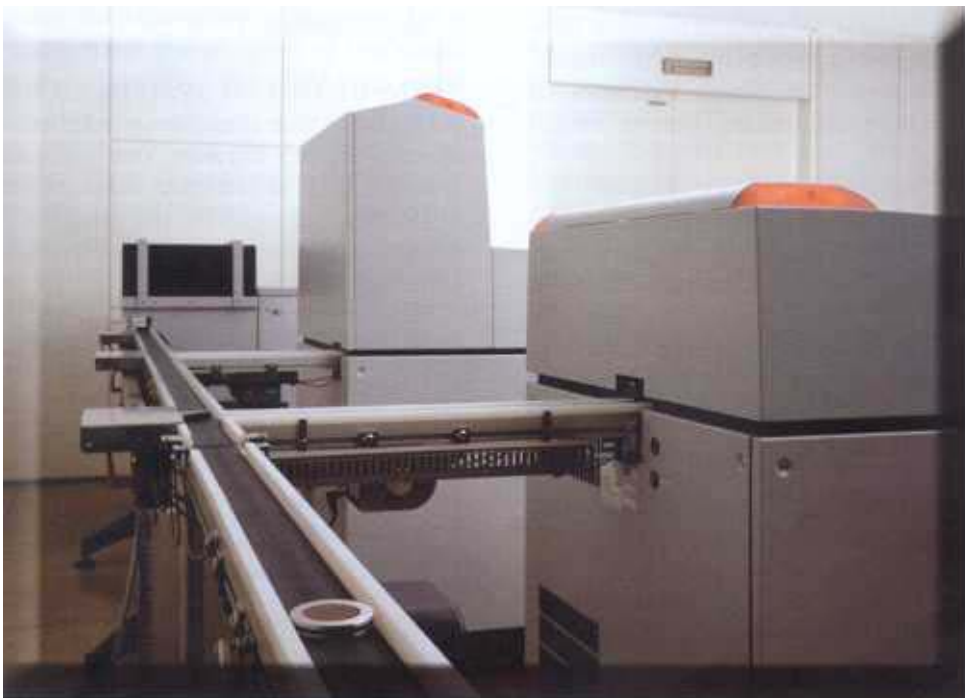


Laboratory Automation



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Introduction

Process optimisation in cement production involves control of the material stream through the system. Information from laboratory analysis of material samples taken periodically at various points in the manufacturing process provides for timely feedback to adjust set points for the plant and equipment control systems.

The degree of automation varies between full scope, completely automated sampling of the material and laboratory analysis including adjustment of the process parameters in a closed loop, and partial automation on the laboratory level.

During recent years there has been an increasing demand for automated laboratories equipped for efficient collection of quality relevant information from raw materials, fuels, process and final products. The motivation for this investment has been improvement of the analytical accuracy and reproducibility, followed by increased laboratory efficiency.

Modern Bruker AXS X-ray instruments are designed for seamless integration into total automation environments (Figure 1), and have proven successful in many installations managed by all major engineering companies in the field of process automation.

AXSLab: web-based laboratory automation

AXSLab is a new software package for fully automated laboratory analysis of material samples. It was developed to provide a complete solution for integrated operation of X-ray analysis and sample preparation. It facilitates functions such as set up and start of jobs for series of samples, status monitoring of the system components, data storage and retrieval, and sample tracking through the individual machines' respective instruments.

The major design criteria for the development were as follows:

- Integration of standard components for both instrumentation as well as software, while maintaining the high analytical performance.
- Safe and reliable control of all components.
- All communication based on standard TCP/IP.
- Flexible sample handling.

- Easy to use operation from any workstation in the plant network.
- Simple interfacing to LIMS and other third party applications.

State-of-the-art system architecture

AXSLab is a true client/server application using TCP/IP communication between the individual system components such as standard measurement control and evaluation packages for the X-ray instruments on one side, and preparation and sample transport control units on the other side.

The kernel of the system consists of the AXSLab server application which controls all instruments,

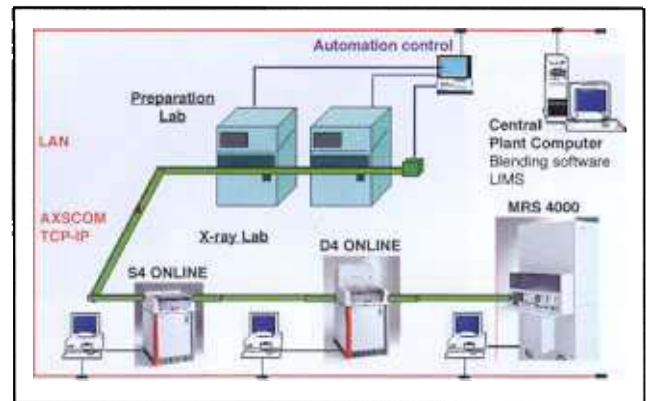


Figure 1. AXS X-ray instruments integrated into a total automation environment.

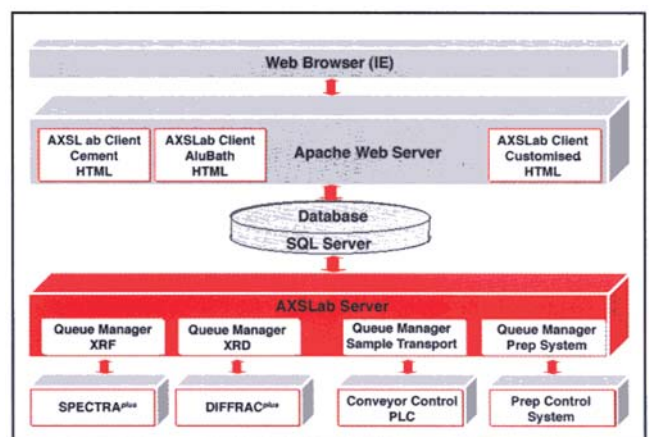


Figure 2. The system architecture.

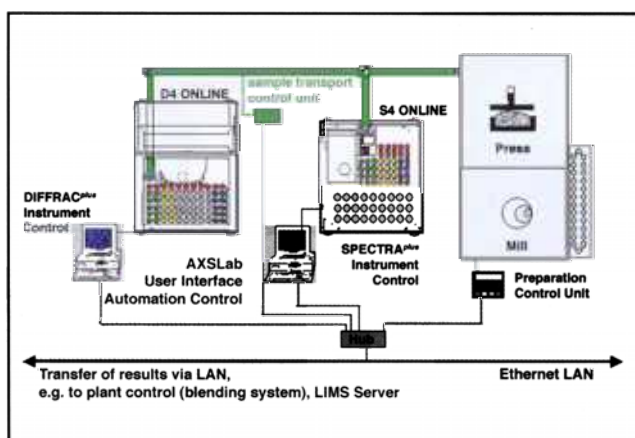


Figure 3. The typical configuration of an automated laboratory including sample preparation and X-ray analysis.



Figure 4. An X-ray spectrometer S4 Online used for chemical analysis and a diffractometer D4 Online used for quantitative phase analysis.

preparation machines and sample transport, and a relational database driven by a SQL server (Figure 2).

The structure of the database logically reflects the real configuration and status of the system, with all components and their specific properties. An important aspect is the dynamic and asynchronous operation of the system requiring fast and safe access to the database in order to guarantee a consistent context of all information at any time.

The database contains an hierarchical structure for all measurement and preparation methods and procedures for various analytical tasks. Based on these definitions, a set of predefined jobs for specific

routine analysis sequences may be stored, thus providing an easy way to launch even extensive series of samples by a single command, a typical requirement for routine laboratories.

Besides the management of the dynamic information of the system, the database serves the more classical purpose of long term storage of results.

An outstanding feature of this automation solution is the web-based user interface. It follows the same principles as any web site: the various pages that are the typical menus, lists, messages of the human interface, are generated by the web server and displayed by a standard web browser on the client PC. For the AXSLab package the Apache web server was chosen: this is one of the most common and well proven web servers in the world wide web.

The Microsoft internet explorer is the final link in the communication chain between the operator and the database server. The restriction on this browser is for practical reasons only: the need to use browser specific functions in the implementation and the fact that this browser is part of the underlying Windows™ platform.

The great advantage of this concept is obvious: it allows access to the system from virtually any PC in the plant or even a corporate wide network. That means instant information about the status of the equipment, progress of the analysis or retrieval of results, simply by mouse clicks wherever needed. Without the installation of special software on the client PC, the only requirement is web-based access.

Of course this immediately raises the question of security of data and safety of the equipment. This aspect has been taken care of by protecting the laboratory system against unauthorised access by requiring a user to login with a password.

Multi-instrument laboratory automation

Figure 3 shows a typical configuration of an automated laboratory including sample preparation and X-ray analysis.

Sample preparation is performed using high quality machines from the two major vendors of such equipment, ranging from manual to fully automated operation. The equipment has to meet the requirements for contamination free, reliable and highly reproducible preparation of the material to be analysed by X-ray instruments. The preparation system is equipped with its own control unit.

The samples are prepared in two steps: firstly the material is ground in a swing mill to the desired particle size and mixed with a grinding aid such as a wax binder. Then a pelletising press is used to press the powder into a steel ring suitable for handling in the X-ray instruments. The operating parameters for both mill and press, such as grinding time, amount of wax binder and pressure have to be optimised for different materials on one hand and for the analytical methods applied for the analysis on the other.



Figure 5. Management of process automation sample magazine.

Chemical analysis is performed by X-ray fluorescence (XRF) using a sequential wavelength-dispersive X-ray spectrometer S4 Online. An X-ray diffractometer D4 Online is used for the quantitative phase analysis by X-ray diffraction (XRD) (Figure 4).

The X-ray instruments are controlled by individual PCs using standard software packages for measurement and evaluation SPECTRA^{plus} and DIFFRAC^{plus}.

The advantages of this dual-instrument configuration are firstly the independent XRF and XRD operation without any restrictions concerning analytical versatility and functionality, and secondly the simultaneous execution of all analytical tasks with highest sample throughput.

The preparation system and X-ray instruments are connected via a conveyor belt system for fully automated transport of the samples. A standard industry PLC with TCP/IP communication processor controls all component functions of the transport system, such as optical sensors, diverters and belt drive motors. Moreover, the integrated modem allows remote diagnostic and maintenance, i.e. changing operating parameters or downloading control software updates.

Control units and instrument PCs as well as the AXSLab server system are linked through a hub. For communication with other plant computers, such as plant control, LIMS server, PCs in the offices, the hub is connected to the plant network.

Thus the automation system is in principle not affected by potential network problems, which means the internal communication between AXSLab server and components should be operational even in case of breakdown of the company's network.

Flexible sample handling

Modern Bruker AXS X-ray instruments fit the requirement for convenient integration into

automated systems. For laboratory automation, the S4 Online spectrometer is equipped with a process automation sample magazine divided in two areas: one area for powder samples pressed in steel rings and the second to handle normal sample holders for other types of samples, i.e. liquids prepared in disposable liquid cups. The first area includes the connection to the conveyor belt for transfer of samples from the automation system.

However, flexible sample handling in the sense of AXSLab means more than just this division into physically different sample areas. In addition there is a logical level to define groups of positions which serve different purposes.

The number and size of these groups can be adapted to suit the customer's needs.

These groups are marked with colours to illustrate their role in the management of the magazine, such as one area for the temporary buffering of process samples transferred via conveyor belt (red), one area for manually loaded samples (green), one area for completed samples (yellow), and one output area for buffering of samples for special treatment (blue) (Figure 5).

The principle outlined above is not restricted to the S4 Online, but also applies to the D4 Online even offering the possibility of distributing the groups on several magazines.

It has to be emphasised that this kind of magazine management is optional and by no means enforced. The purpose is rather to assist in establishing certain rules in the production laboratory for easy and clear handling of samples in daily routine work.

Easy to use

The sample material delivered to the production laboratory is manually filled into cups and loaded into the preparation input magazine. The operator registers the samples in the job list. The scheme for identification of samples and their allocated analytical procedures can be tailored according to the practice in the individual laboratories. As an example 'RM1' could identify the sample collected at the raw mill number 1: for this sampling point respective material type all parameters for both preparation and analysis are defined in the database, the key 'RM1' together with the time stamp uniquely identifies the sample in the system.

In this case selecting the sample key from a list is sufficient to initiate the entire analytical process:

Firstly the material is ground to a defined particle

size, then the powder is pressed into a steel ring. As soon as the conveyor system is free the sample is transported to the appropriate X-ray instrument for analysis. After completion of the measurement, the sample is either transported to the next instrument or directly returned to the press, where the steel ring is cleaned for re-use. The results are stored in the database and additionally transmitted to the plant computer for incorporation into a blending control system for optimisation of the production parameters.

The overall status of the complete system is displayed online indicating the status (idle, busy, alarm, warning) of each machine or instrument and the current location of each sample of the job list. The operator may even leave the laboratory unattended for a while and can easily track the progress of the batch of samples from any location in the plant with web-access (Figure 6).

At any time, longer routine measurements can easily be interrupted for priority samples for immediate process control. Another useful feature is the scheduler which allows launching of jobs at a given time or in regular time intervals, for example to perform an automatic drift correction or to run a monitor sample to verify the instrument performance at the beginning of each shift.

X-ray analysis with highest analytical performance

Chemical analysis forms the basis of cement plant control by providing information for proportioning raw materials, adjusting kiln and burning conditions, as well as cement mill feed proportioning.

Knowledge of the absolute phase abundance in cement clinkers is necessary for both research and quality control. The composition of the raw meal, the resulting clinker phases, as well as the phases arising from the hydration process, have a large influence on the physical and chemical properties of the cement.

Chemical analysis

Sequential wavelength-dispersive X-ray spectrometers are the best choice to cover all analytical tasks for chemical analysis in the cement industry. All the different materials can be analysed with one single instrument: besides the traditional routine analysis of raw meal, clinker and cement it provides for control of natural and waste recovery raw materials, S or Cl in alternative liquid or solid fuels, filters, plants or sewage, and is therefore of benefit for the environment.

Recent publications have detailed the superb analytical performance of the S4 Online for the measurement of routine cement samples.

To further improve the speed of analysis, a measurement method has been developed with the S4 Online which enables the determination of nine analytes in less than 3 mins. This time is inclusive of sample loading and unloading, changing of instrumental parameters, actual analysis time and reporting of the result. Extreme care was taken to ensure that the improvements in speed did not compromise the quality of the analytical data produced.

The range of analytical cement routines is easily extended by the 'standard less' XRF approach which allows analysis of nearly all elements of the periodic table in any totally unknown sample which can enter an XRF process and quality control laboratory in the cement industry (Figure 7). Any sample, metal pieces, minerals, rocks, slurries, filter samples or even alternative liquid fuels, or plastic cuttings, can be analysed with this uniform, pre-calibrated programme.

Another analytical advantage of the S4 Online is the optimised performance for safe measurement of liquid samples. When measuring liquid fuels (for example S or Cl in waste oils) in helium or nitrogen atmosphere the unique vacuum seal of the spectrometer closes the spectrometer chamber with the optics and only the small volume of the sample chamber is flushed. This drastically reduces the consumption of Helium or Nitrogen and contributes to highest stability and reliability because the flow counter remains under vacuum.

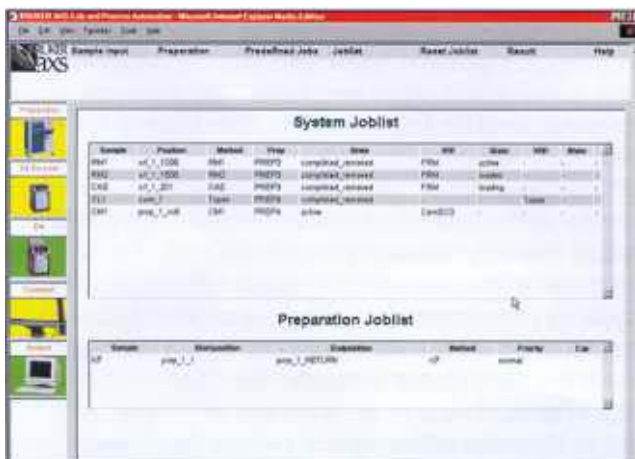


Figure 6. Tracking the progress of the batch of samples from any location in the plant with web access.

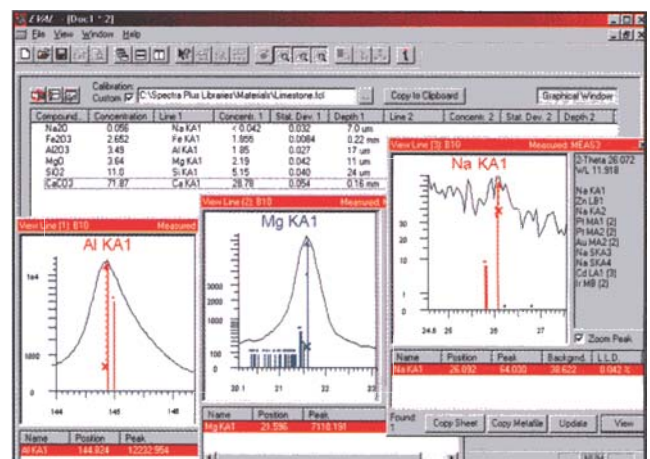


Figure 7. The range of analytical cement routines extended easily by the 'standard less' XRF approach.

Quantitative phase analysis by Rietveld method

Traditionally the absorption-diffraction method ('regression method') is applied for determination of free-lime (CaO) by means of conventional single line analysis. In general, when using carefully prepared standards, the phase composition of unknown samples can be determined with high accuracy. However this method is not suitable for full clinker analysis. As the chemical and physical properties of the product are governed by the mineralogy, and not by the elemental composition, quantitative phase analysis is a necessity for quality control. The most widely spread methods to date are theoretical calculation from chemical analysis using the Bogue method and optical microscopy. The deficiencies of these methods are well known.

More recently, X-ray powder diffraction (XRD) in combination with quantitative Rietveld analysis has been shown to offer new insights into the field of cement clinkers, cements and raw materials.

With XRD and Rietveld analysis, a full quantitative analysis of cement clinkers can be performed providing detailed mineralogical information about the product, as has been shown in the recent years (Füllmann (2001), Füllmann (2001), Möller (1998), Neubauer (1997)).

The latest developments of online XRD instrumentation, as well as dedicated Rietveld software for quantitative phase analysis (TOPAS), now make a decisive breakthrough possible. TOPAS is a completely new Rietveld programme system, which has been developed with particular emphasis on improving speed, stability, and convergence, thus eliminating the major deficiencies of existing software.

TOPAS not only allows the analysis of extremely complex phase mixtures in the shortest time possible, but also a fully automated on-line phase analysis for production control and quality management, without any user input. In addition, using the latest PSD (position sensitive detector) detector technology in a new process diffractometer, measurement and evaluation times can be reduced to minutes, enabling real time control of the cement clinker mineralogy (Figure 8).

As a result, a fully automatic quantitative analysis from XRD data is possible without any user input, and it can be used for unattended control of clinker kiln operation for the first time.

Conclusion

Minimal effort in sample preparation, short measuring times, extreme analytical flexibility, high accuracy and precision, as well as full integration in automation systems, are important advantages of modern analytical X-ray instruments for process and quality control.

By developing and optimising the instrumental and analytical parameters, it is possible to determine nine analyte concentrations for finished cement samples in less than 3 mins with uncompromised analytical quality using the sequential X-ray spectrometer S4 Online.

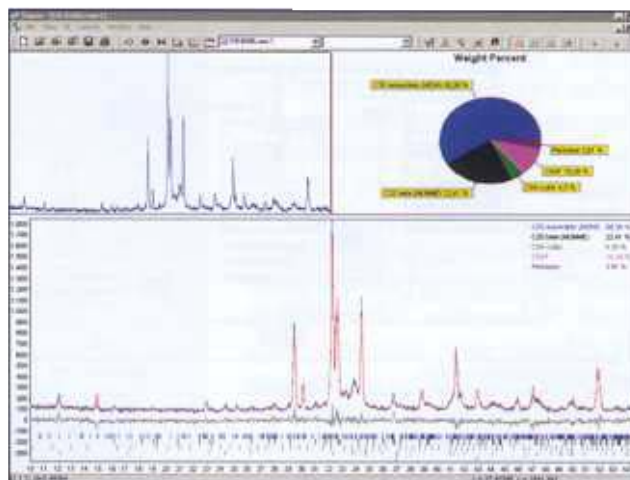


Figure 8. TOPAS quantification of cement clinker.

A breakthrough in the application of quantitative analysis by XRD is the latest development of the dedicated Rietveld software TOPAS allowing fully automated phase analysis of even extremely complex phase mixtures in the shortest time possible without any user input. Using the latest PSD detector technology in the new process diffractometer D4 Online enables real time control of the cement clinker mineralogy thus closing the gap between the conventional quantification of free-lime (CaO) and the total cement solution.

The new web-based laboratory automation solution AXSLab integrates well proven and reliable sample preparation technique together with modern state-of-the-art X-ray analysis systems forming the base for highly reproducible and economical control of the material stream through the process.

The compact new Bruker AXS X-ray instruments with small foot print and low requirements for installation allow containerised at-line laboratories to be a reality. Laboratory automation has eliminated the human factor resulting in far more reproducible preparation and analysis, and even improved laboratory efficiency. It thus perfectly fits the needs of typical production laboratories in the cement industry.

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