Polysius

polab® System

Cutting production costs with polab® quality control systems.

thyssenkrupp

polab®
– sampler, pneumatic delivery system and laboratory equipment

polab® Linea *Quality Control System*

The new

modular

Linearity

Plant Side

Screw sampler for raw meal or cement. Installation in a chute.

Hot meal sampler. Water cooled screw sampler made of special steel. Installation in the hot meal chute.

Clinker sampler. Installation at the discharge end of the clinker conveyor belt.

Alternative: sampling clinker on the cooler grate.

Jaw crusher

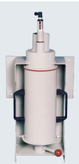
Trough-type sampler for raw meal or cement. Installation in a pneumatic trough conveyor.

Screw sampler for coal dust. ATEX-certified.

Sample dispatch station with sample mixer for collective samples and spot samples with pneumatic material return system.

Alternative: sample dispatch station without sample mixer.

Container for manual sample conveyance.



Pneumatic delivery tube diverter for connecting two pneumatic delivery lines.

Pneumatic tube: pneumatic delivery network made of precision steel pipe.

Pneumatic delivery capsule: 350 cm³ volume.

Forwarding air blower

Laboratory

Semi-automatic laboratory

Semi-automatic pneumatic delivery receiving station with magazines for received and empty capsules.

Alternative: manual pneumatic delivery receiving station.

Fully automatic laboratory

Laboratory for 8–10 samples per hour

polab® APM sample preparation module with vibratory disc mill and tablet press.

Options: sample input magazine, sample output magazine, connection of XRF and XRD by tablet conveyor belt.

polab® ACT automatic laboratory unit (for 8 to 10 samples/hour) with pneumatic delivery receiving station and sample preparation system (integral polab® APM). Connection of XRF and XRD by tablet conveyor belt.

Option: integral laser granulometer.

Laboratory for up to 14 samples per hour

polab AMT automatic laboratory unit for a high sample throughput rate of up to 14 samples per hour, with capsule receiver, sample distribution via integral industrial robot, sample preparation by means of 1 to 3 integral polab® APMs, and connection of XRF and XRD by tablet belt conveyor.

Optional: integral laser granulometer, colorimeter.

Individually configurable laboratory

The POLAB® Shuttle is the new laboratory automation system with unique flexibility.

Sample conveyance by mobile robot provides previously unachieved freedom regarding system configuration, upgradability and room layout.

polab® AQCnet Software

- Client – Server structure
- .net technology / Web interface
- SQL database
- Adaptive mixture control

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Polysius

polab® APM

The automatic sample preparation module for XRF and XRD – compact, flexible and upgradable.

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polab® APM – Sample Preparation for XRF and XRD

Grinding unit and tablet press integrated into one compact housing.

Compact, flexibly configurable and upgrade-capable. **polab®** APM integrates into a single machine all the components needed for preparing samples of mineral materials.

polab® APM basis unit
The basis unit for sample preparation consists of the following components:
- Sample loader for blind sample and main sample
- Grinding aid dosing unit
- Grinding unit (patented)
- Tablet press
- Optional: volumetric or gravimetric dosing device

The **polab®** APM basis unit can be upgraded into polab® APMplus by simply changing certain components.

Benefits of polab® APM

- Patented grinding unit for efficient and gentle grinding
- Optimised sample preparation for the different requirements of XRF and XRD
- Excellent reproducibility
- High sample throughput rate and less routine work to be carried out by the laboratory personnel
- Predefined and user-specific sample preparation routines
- Operator panel for simple and intuitive operation
- Sequential control by PLC
- Grinding and pressing functions can be selected in a combined process or as single processes

Technical data

Weight:	443 kg (APM), 500 kg (APMplus)
Dimensions (W x H x D):	600 x 1230 x 735 mm (APM) 600 x 1230 x 900 mm (APMplus)
Power consumption:	2.0 kVA
Power supply:	1-phase
Compressed-air supply:	7-9 bar
Compressed-air consumption per sample:	160 dm³

Sample material

Properties:	mineral materials
Grain size:	0-7 mm
Residues on 1 mm screen (D 50 = 1 mm):	< 50 %
Moisture content:	< 1 %

Control system and control panel

Siemens
Optional: Rockwell

Sample holders: steel rings	**Standard**	**Optional**
Outside diameter:	51.5 mm	40.0 mm
Inner diameter:	35.0 mm	35.0 mm
Height:	8.6 mm	14.0 mm

Tablet press

Pressing power:	47 ... 93 kN
Pressure holding time:	adjustable

Grinding unit: vibratory disc mill

Grinding bowl and grinding elements:	tungsten carbide
Effective volume:	30 cm³

polab® APMplus Upgrading stages

polab® APMplus with output magazine

The expanded configuration for the automatic processing of sample series consists of:

polab® APM basis unit
+ Input magazine (with up to 20 input positions)
+ Volumetric dosing device
+ Tablet and ring cleaning device
+ Magazine for steel rings
+ Output chute
+ Optional: output magazine

polab® APMplus with connection for tablet conveyor belt

The expanded configuration for the automatic transport of samples to the analysers consists of:

polab® APM basis unit
+ Input magazine (with up to 20 input positions)
+ Volumetric dosing device
+ Tablet and ring cleaning device
+ Magazine for steel rings
+ Connection for a tablet conveyor belt feeding the analysers

polab® APMplus for integration

The automatic sample preparation unit for integration into robot-based laboratories, such as polab® AMT, polab® Shuttle or polab® ACT, consists of:

polab® APM basis unit
+ Volumetric dosing device
+ Tablet and ring cleaning device
+ Magazine for steel rings
+ Connection for a tablet conveyor belt feeding the sample output or the analysers
+ Optional: gravimetric dosing device

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Verification of suitability according to ASTM C114

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Preparation of cement samples using the polab® APM

Introduction

The ASTM standard C114 describes test methods for the chemical analysis of cement samples. The reference methods it describes are generally accepted classical analysis processes based mainly on wet-chemical methods. The standard also describes a procedure for verifying the suitability or qualification of alternative test procedures (performance requirements for rapid test methods).

X-ray fluorescence analysis (XRF) is a rapid, precise, long-established analysis technique for the quantitative determination of cement samples as well as raw materials and intermediates used in the cement manufacturing process. Alongside a qualified test technique, sample preparation is of vital importance in XRF analysis. Analysis samples are generally prepared in the form of fused tablets[1] or pressed tablets. Fused tablets have the advantage of eliminating mineralogical and grain size effects as well as significantly reducing matrix effects. However, the disadvantage is that their production is complex, time-consuming, costly and difficult to automate. As long as the samples to be analyzed come from the same cement plant and thus have a similar matrix and granulometric distribution, it makes sense to prepare the samples in the form of pressed tablets. Moreover, this is easy to automate and ideal when a large number of samples need to be prepared – for instance for product and process control in the cement manufacturing process.

The sample preparation module polab® APM is part of the labora-tory automation system from thyssenkrupp Polysius and it is specially tailored to the preparation of samples, inter alia from the cement industry. The fully automated APM grinds the samples to a uni-form granulometric distribution and presses them into a standardized steel ring to form tablets. The pressed tablets thus prepared are then ideal for x-ray fluorescence analysis as well as for x-ray diffractometry.

Below you will see how preparing cement samples using the polab® APM in combination with XRF analysis meets the requirements of the ASTM standard C114 and is thus qualified as an alternative test method for the chemical analysis of cement samples.

[1] by fusion, e.g. using lithium tetraborate

Tests and results

To verify the qualification of APM sample preparation in combination with XRF analysis as an alternative test method according to ASTM C114, in total seven certified reference samples[2] were prepared on a single day using a polab® APMplus. The reference samples were prepared gravimetrically in the APMplus using 12.0 g sample material and adding three polab¶ grinding aid tablets. Adding a grinding aid

[2] Portland cements from the National Institute of Standards & Technology as well as from the Cement and Concrete Reference Laboratory

Verification of suitability according to ASTM C114

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ASTM C114 qualification in terms of precision				
Element oxide	Concentration range [%]	Maximum difference between duplicates (day 1 - day 2) [%]	Limit value ASTM [%]	Compliance?
SiO_2	19.3 - 21.2	0,08	0.16	Yes
Al_2O_3	3.7 - 5.9	0.02	0.20	Yes
TiO_2	0.2 - 0.3	< 0.01	0.02	Yes
Fe_2O_3	2.1 - 4.5	0.01	0.10	Yes
Mn_2O_3	0.1 - 0.2	< 0.01	0.03	Yes
CaO	61.6 - 64.2	0.12	0.20	Yes
MgO	1.2 - 4.8	0.02	0.16	Yes
SO_3	2.2 - 4.4	0.04	0.10	Yes
P_2O_5	0.1 - 0.2	< 0.01	0.03	Yes
Na_2O	0.1 - 0.4	< 0.01	0.03	Yes
K_2O	0.4 - 1.2	0.01	0.03	Yes
Cl	0.00 - 0.02	0.001	0.003	Yes

Table 1

ASTM C114 qualification in terms of accuracy				
Element oxide	Concentration range [%]	Difference between average of duplicates & certificate values [%]	Limit value ASTM [%]	Compliance?
SiO_2	19.3 - 21.2	0.2	0.2	Yes
Al_2O_3	3.7 - 5.9	0.1	0.2	Yes
TiO_2	0.2 - 0.3	0.01	0.03	Yes
Fe_2O_3	2.1 - 4.5	0.04	0.1	Yes
Mn_2O_3	0.1 - 0.2	< 0.01	0.03	Yes
CaO	61.6 - 64.2	0.2	0.3	Yes
MgO	1.2 - 4.8	0.1	0.2	Yes
SO_3	2.2 - 4.4	0.1	0.1	Yes
P_2O_5	0.1 - 0.2	< 0.01	0.03	Yes

Table 2

makes for more efficient particle size reduction as well as vacuum-resistant pressed tablets, and prevents the grinding bowl from becoming caked in the mill feed material. Each sample was ground for a period of 150 s at the lowest selectable speed.

Then, the pressed tablets were analyzed using an Axios Advanced XRF spectrometer from the company PANalytical. The spectrometer was calibrated using valid curve-fitting methods. The element oxides SiO_2, Al_2O_3, TiO_2, Fe_2O_3, Mn_2O_3, CaO, MgO, SO_3, P_2O_5, Na_2O and K_2O were measured as well as chlorine (Cl).

On a non-consecutive day, a new set of reference samples was prepared in the polab® APMplus and then analyzed. The difference between the analysis values obtained on day 1 and day 2 was determined for each of the element oxides measured, as was the mean value of the two measurements in each case. While the differences in the analysis values obtained on day 1 and day 2 are an indicator of the precision of the measuring method, comparing the mean values with the certified concentration values provides an indicator of the accuracy.

The results have been summarized in Table 1 (precision) and Table 2 (accuracy) along with the concentration ranges covered by the reference materials. The maximum variation determined for each element oxide in the seven reference materials is shown, and pursuant to ASTM C114 this must not exceed the respective limit value shown.

It can be seen from Table 1 that in the case of all element oxides the measured values are, with regard to precision, much lower than the limit values specified by the ASTM. With regard to accuracy (see Table 2), the measured values are also within the limit values specified by the standard.

Conclusion

X-ray fluorescence analysis is a long-established analysis technique, inter alia in the cement industry. The qualification of this method as an alternative test procedure in accordance with the ASTM standard C114 has already been verified in the past by manufacturers of such analytical instruments. Preparing cement samples as well as raw materials and intermediates used in the cement manufacturing process in the form of pressed tablets using the polab® APM has also a long proven success. Samples prepared in this manner are ideal for both x-ray fluorescence and x-ray diffraction analysis.

The present tests show that preparing cement samples using a polab® APM in combination with XRF analysis meets the performance requirements of ASTM C114 for alternative test procedures (performance requirements for rapid test methods). For all seven certified reference materials tested, the variations both in terms of precision (see Table 1) and in terms of accuracy (see Table 2) are lower or equal to the limit value specified in the standard. APM sample preparation in combination with XRF analysis thus qualifies as a rapid test method in accordance with ASTM.

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Services for polab®
Laboratory Automation

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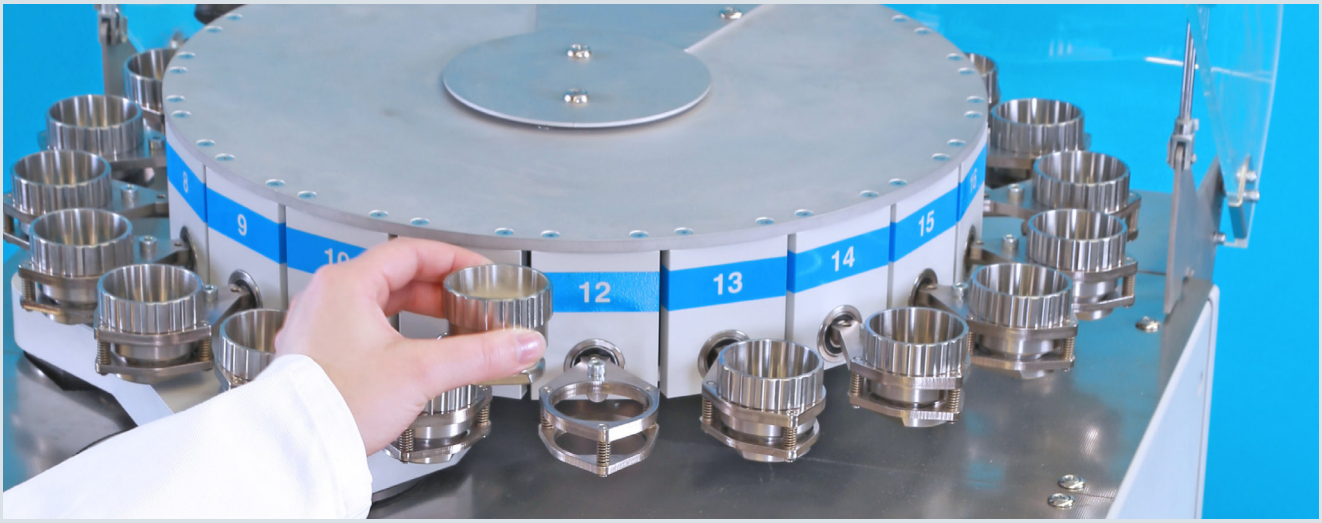

polab® APMplus (Automatic Preparation Module)

Safeguarding uninterrupted automation availability

If your production monitoring system fails, the result is often production losses. That is why it is so important to have your polab® system checked. Ongoing system maintenance and servicing are the recipe for success in ensuring the optimum availability of your quality assurance system. What we offer is specially tailored to your automation system, with the service conducted by our specialists at the agreed intervals.

Our services ensure the high reliability and cost effectiveness of your laboratory automation system. The core components of what we offer are continuous system maintenance and servicing, rapid supply of spare parts, up-to-date information, as well as comprehensive training measures.

During the assignment, we check your polab® configuration for functional reliability. This includes checking and, if necessary, replacing all sensitive-to-wear parts. On request, we will also integrate any new advanced developments into your system. As a result, up-to-date experience and state-of-the-art technology can be automatically integrated into your installation.

For every system we offer a customized package of spare parts for on-site storage. This allows you to carry out repairs quickly and without problems. If a particular spare part should still be missing, we arrange for delivery as quickly as possible.

Our newsletter will also inform you about ongoing developments and optimization potential – so you stay up to date and can draw up long-term, forward-looking investment plans.

We would be pleased to train your staff in all aspects of application, maintenance and servicing. One of our specialties are on-site seminars tailored to your specific applications.

Your service advantages

- Continuous system maintenance and servicing
- Integration of new and further developments
- Safeguarding your polab® system's availability
- Rapid provision of spare parts
- Up-to-date information
- Comprehensive training courses

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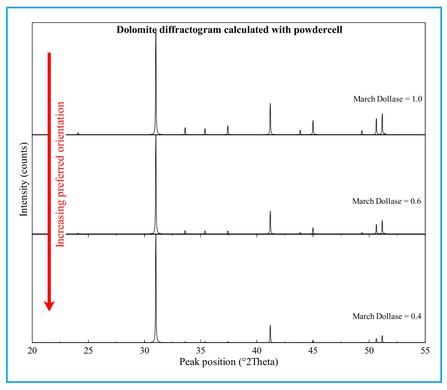
Automated sample preparation

Does preferred orientation matter?

Preferred orientation is a structural property of certain minerals with needle or plate like crystal habit. The minerals orient themselves while pressing to the cleavage planes. Besides changing the intensity of the XRD pattern also the quantitative analyses will be incorrectly influenced.

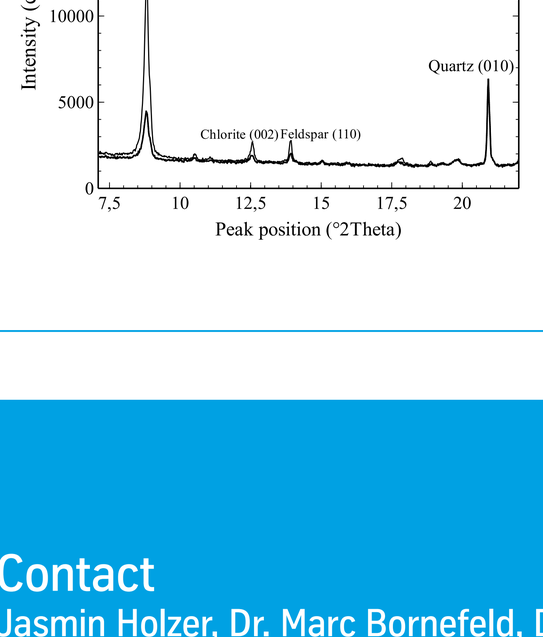


Experimental

A granitic rock with minerals showing a preferred orientation has been selected as reference material for sample preparation for X-ray diffraction with the polab® APM. The effect of the various grinding and pressing parameters of the APM on the resulting diffractogram have been investigated. The subsequent quantification has been carried out using TOPAS V.4.2.

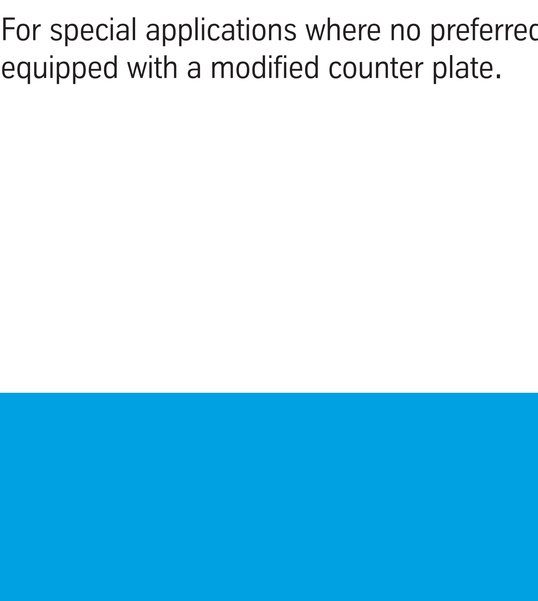
Does preferred orientation matter for quantitative X-ray diffraction?

Effect of the grinding time on the preferred orientation



With increasing grinding time the intensity of the (001) Biotite peak is reduced. The shift of the March Dollase correction to higher values shows a positive effect on the preferred orientation, nevertheless the quantification is not influenced.

Effect of the pressing force on the preferred orientation



If the pressing force increases the (001) peak of Biotite is shifted to higher 2theta angles. This effect is a result of an increased sample displacement when higher pressing forces are applied. Effects on the quantification cannot be detected.

Effect of a modified surface on the preferred orientation

Samples were pressed using a counter pressure plate with a modified surface. Therefore the surface of the pressed sample was not even but slightly rough as it can be seen on the image. This has a positive effect on the preferred orientation of micas and feldspars but in contrast minerals without preferred orientation were not affected.

Conclusion

Samples prepared with the polab® APM demonstrate that preferred orientation of minerals has no effect on the quantification with the Rietveld method. Nevertheless preferred orientation of crucia l minerals like micas and feldspars can be detected in the diffraction patterns. However, by choosing the correct grinding parameters the preferred orientation can be reduced.

For special applications where no preferred orientation is desired the APM will be equipped with a modified counter plate.

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laboratory automation – 50 years!

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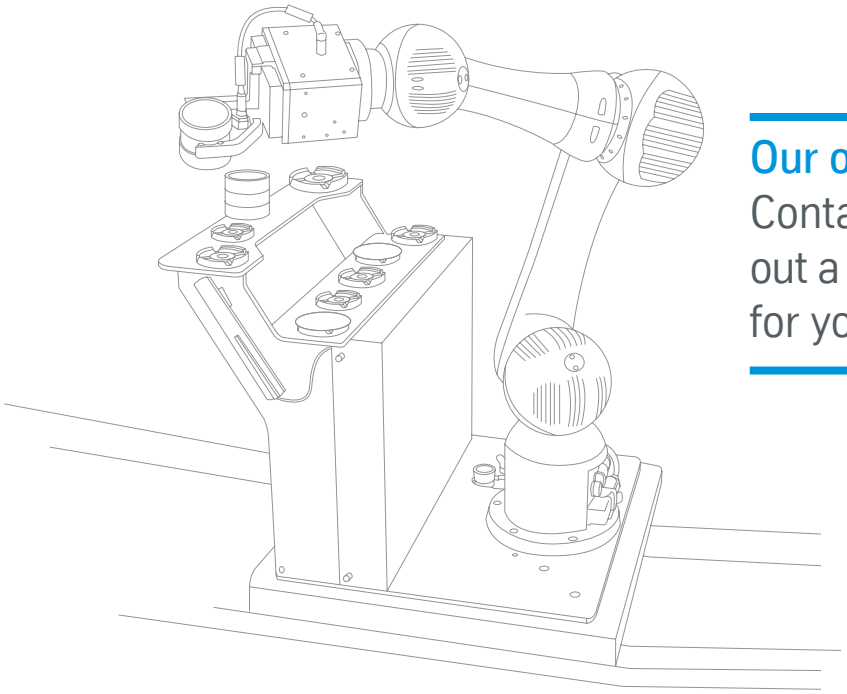
Quality control during cement production is a must! polab® is therefore essential.

Why use polab® laboratory automation?

High product quality – with maximum profit. Payback times of less than 2 years can be achieved.

Reliability for you!

50 years of laboratory automation know-how, proven in more than 500 installations worldwide.



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