# Automated sample preparation for Rietveld analysis of potash and salt minerals


## Abstract

**Rietveld analysis is a commonly used method in various industrial applications in the mining sector. Herzog provides special designed sample preparation equipment for the automated analysis of potash and salts utilizing the back-loading method.**



## Keywords

**XRD • Rietveld • automation • sample preparation • grinding • pressing • back loading • Potash • salts**


## XRD in mining applications

XRD as a commonly used analytical technique is applied to solve various problems of mining interests, ranging mainly from exploration aspects to process control. Phase determination and quantification with the Rietveld method is essential for a broad range of analytical demands in [Villiers, 1999]:

- pyrometallurgical processes
- hydrometallurgical processes
- quantitative analysis of limestone based products
- and material charaterization.

With its possibilities XRD analysis combined with Rietveld quantification not even ensures high quality production, its also helps to reduce energy consumption and CO2 emission for modern plant operation [König et al., 2010].

This application shortly summarizes the realization of an automated laboratory solution for XRD Rietveld analysis for potash and salt products.

## Laboratory Layout

The laboratory solution provided by Herzog offers generally four main functions:

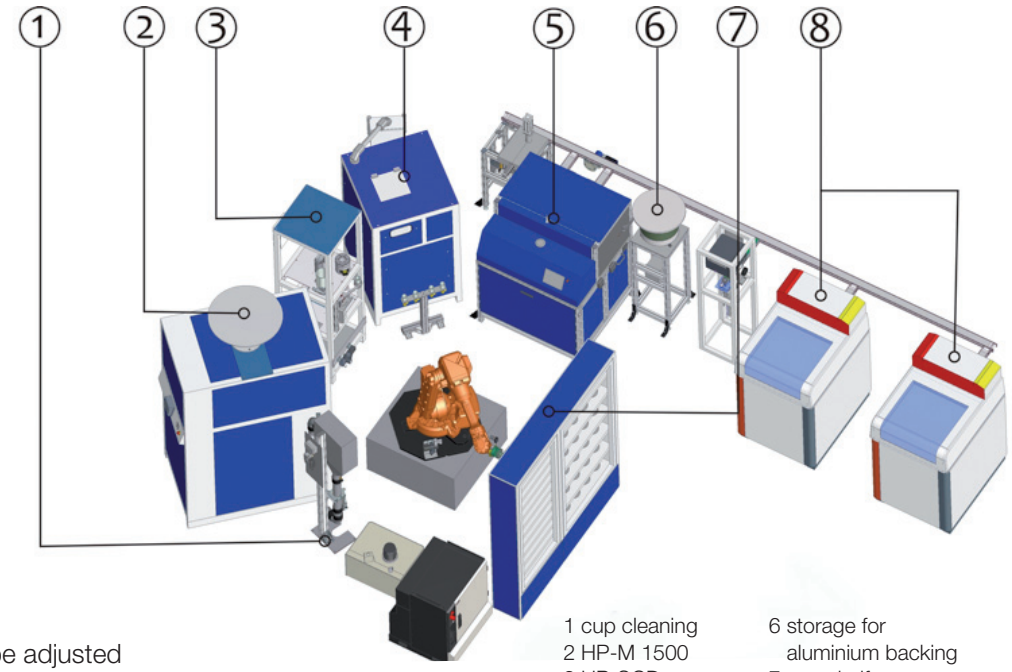
- grinding
- sub-sampling
- fine grinding
- pressing utilizing back loading technique.

All machines implemented in the automation can be used as stand-alone devices in service mode operation. Because of it's modularly setup the system can be adjusted according to specific demands on sample preparation and to cope the analytical requirements. All machines are designed to resist the corrosive and abrasive conditions of processing salt based raw materials.

The laboratory is composed of two vibrating mills (HP-M 1500 & HP-MS), the sub-sampler HP-SCD and the HP-PD6 for realizing material transfer into a XRD sample holder. Additionally the system is equipped with all necessary facilities to afford fully automatic operation. Sample handling between all individual components is done by an industrial robot or via conveyor belts to the spectrometer. Sample preparation is controlled by the PREPMASTER software, supplied by Herzog with the automation.

## Potash and salt minerals

Samples can be introduced into the system via a sample cup shelf. The modular setup allows defining a specific sample routing for each sample type. Grinding bigger samples amounts with a volume up to 1.5 l can be done by the HP-M 1500. With this mill it is possible to obtain the requested grain size of 95 % < 200 µm. The reduced particle size allows taking a sub-sample for further sample preparations steps. This is done by the HP-SCD, which extracts a suitable sample size for fine grinding with the HP-MS. To fulfill analytical requirements the sample is comminuted down to 40 µm (95 %) in the HP-MS. The grinding speed of both mills can be adjusted infinitely for each sample type in order to preserve crystalline structures, prevent strain or re-agglomeration of material during grinding. Both mills are equipped with sand storage on top, which provides the possibility to interpose a sand cleaning between each sample. This is necessary to avoid contamination caused by residual material in the grinding vessel, which mainly can be attributed to water bearing salt crystals (e.g. carnallite & kieserite). After pulverization the material is pressed in the HP-PD6 into a steel ring via back-loading technique. To stabilize the material an aluminum backing is additionally introduced into the steel ring. Compared to normal sample procedures of pressed pellets for XRF analysis in this case only very small pressures are applied, to minimize preferred orientation of particles, which is the mostly the reason for unwanted intensity distortion (Jenkins & Snyder, 1996).




1 cup cleaning
2 HP-M 1500
3 HP-SCD
4 HP-MS
5 HP-PD6
6 storage for aluminium backing
7 cup shelf
8 XRD spectrometer

Fig.1: Laboratory layout for XRD analysis allowing grinding, sub-sampling, fine grinding and pressing via back-loading into steel rings.

## Summary

The modular system with its versatile parameterization provides the requested flexibility requested for a XRD sample preparation solution. Various sample types of potash and salt minerals can be processed in fully automatic mode.

## References

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