

Report

X-ray analysis of Zirconium oxide



Introduction

The **planet** is the new, high-resolution portable X-ray powder diffractometer from xplorex. By using the para-focusing Seemann-Bohlin geometry in reflection mode, we were able to design a very compact, light weight diffractometer with a performance comparable to current standard laboratory-based diffractometers.

Because the **planet** uses reflection mode rather than transmission, it is especially suited for the identification of heavy minerals. In this note the identification of ZrO_2 nicely demonstrates the capability of the **planet** to analyze heavier minerals.

Experimental

About 1g of the as-received powder was mixed with approximately 1mg of Hoechst wax and subsequently pressed into the sample holder (so-called front loading). The prepared specimen was mounted in the diffractometer and measured. The measurement parameters are summarized in Table 1.

Parameter	Value
Range	
Start Angle ($^{\circ}2\theta$)	15.70
End Angle ($^{\circ}2\theta$)	102.24
Step size	Variable; The planet has predefined step sizes
Integration time	60s / data point (the measurement comprises 17 times 640 data points)
Total Measurement time	20 minutes
Diffractometer settings	
High Tension	30 kV
Emission Current	0.65 mA
Tube anode	Cu
Focus dimensions	40 μ m diameter
Take off angle ($^{\circ}$)	8
Beam divergence ($^{\circ}$)	1.25
Specimen dimensions	7mm diameter; 2mm thickness
Spinning frequency	0.5 Hz
Optical path	Seemann – Bohlin based
Focusing circle radius	160mm
Detector	Dectris' Mythen 1D solid state linear detector
Identification software	Match! From Crystal Impact
Reference database	Crystallographic Open Database

Table 1 Measurement parameters for ALS sample 31814 / Zirconium dioxide

The diffractometer settings are constant for the **planet**. The operator can choose the measurement range and the integration time for optimal results and ease-of-use. The first 1280 datapoints were measured with an angle of incidence of 7.00°; the next 640 datapoints at 11.54° and the rest of the datapoints at 14.29°.

Results and Discussion

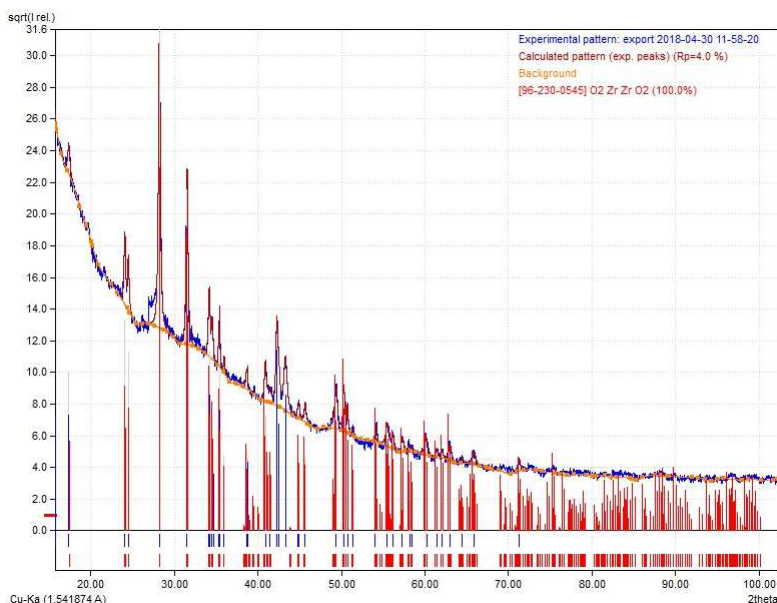


Figure 1 Diffraction pattern of Specimen 31814/Zirconium Dioxide

The measured diffraction pattern was imported in the Match! Software from Crystal Impact. Subsequently we determined the peak positions (shown as blue sticks in Figure 1). The peaks at 42.27° and 43.28° stem from the brass sample holder and can be ignored.

Next, we compared the peak positions and intensities to those from all reference patterns in the Crystallographic Open Database (COD). No restrictions were applied. Of the list of candidates, the top 17 reference patterns all corresponded to compounds that are iso-structural with monoclinic Zirconia (baddeleyite). Accepting one of those candidates and recalculating the scores, gave no indication for another compound than Baddeleyite. Hence, the received sample comprises only ZrO_2 .

Figure 1 shows the measured diffraction pattern we recorded from sample 31814 together with a reference pattern for ZrO_2 . Clearly all peaks that don't correspond to the sample holder are explained by the reference pattern.

Conclusion

With the planet you can measure a complete diffraction pattern in 20 minutes or less. The resulting pattern is of good quality and therefore enables reliable phase identifications. Because the planet uses reflection mode, the quality of the pattern is not affected by the presence of heavy elements (i.e. strong absorbers).