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Report
Identification of $MnCO_3$

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Introduction

X-ray diffraction is a well-established technique to identify the presence of -polycrystalline- compounds (also referred to as "phases") in specimens. Therefore, it comes to no surprise that X-ray diffraction is also used to complete the files for REACH registration of substances.

Xplorex, develops and manufactures the **planet**; a high resolution portable x-ray diffractometer specifically aimed at phase identification.

The **planet** uses para focussing reflection geometry, which yields hitherto unsurpassed resolution for portable XRD. We chose reflection geometry so that the sample thickness is not critical for both heavily absorbing materials and lighter matrices.

Experimental

About 1g of the as-received powder was mixed with a 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 (°2θ)	20
End Angle (°2θ)	90
Step size	Variable; The **planet** has predefined step sizes
Integration time	180s/data point (the measurement comprises 14 times 640 data points)
Total Measurement time	45 minutes
Diffractometer settings	
High Tension	30 kV
Emission Current	0.65 mA
Tube anode	Cu
Focus dimensions	40µm diameter
Take off angle (°)	8
Beam divergence (°)	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 $MnCO_3$ sample

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.

Results

Figure 1 shows the recorded diffraction pattern together with the result of the phase identification. Rhodochrosite (mineral name for $MnCO_3$) was fifth in the candidate list with the same score as the four candidates above. These four other candidates could be rejected, because their reference patterns contained strong lines at positions where we didn't measure any intensity above the background. The single unidentified peak at 42.6 °2θ stems from the sample holder (Brass).

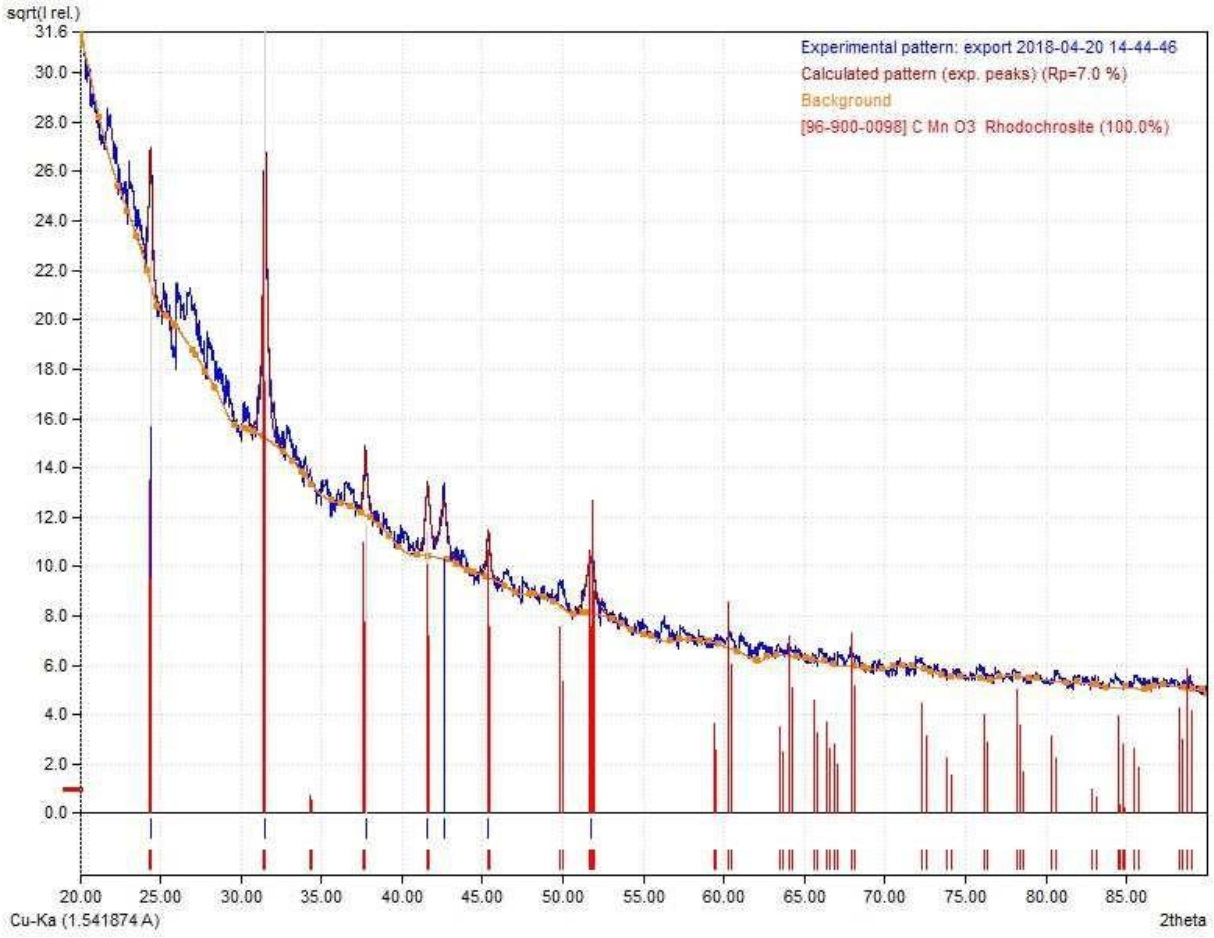

Figure 1 Diffraction pattern of $MnCO_3$ sample; The red lines correspond to the reference pattern for Rhodochrosite.

Conclusion

The X-ray diffraction measurement we performed with the **planet** clearly identifies the diffraction pattern of Rhodochrosite. No other compounds were detected. Therefore, the sample consists of $MnCO_3$, with possible other compounds below the limit of detection (of about 0.5 to 1 wt%)

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