

**Volume 56 (2023)**

**Supporting information for article:**

**Remote and Automated High Throughput Powder Diffraction Measurements Enabled by a Robotic Sample Changer at SSRL Beamline 2-1**

**Kevin H. Stone, Monty Cosby, Nicholas A. Strange, Vivek Thampy, Richard C. Walroth and Charles Troxel Jr**

**Table S1****Table S2** List of example data sets measured using the automated data collection strategy and the robotic sample changer:

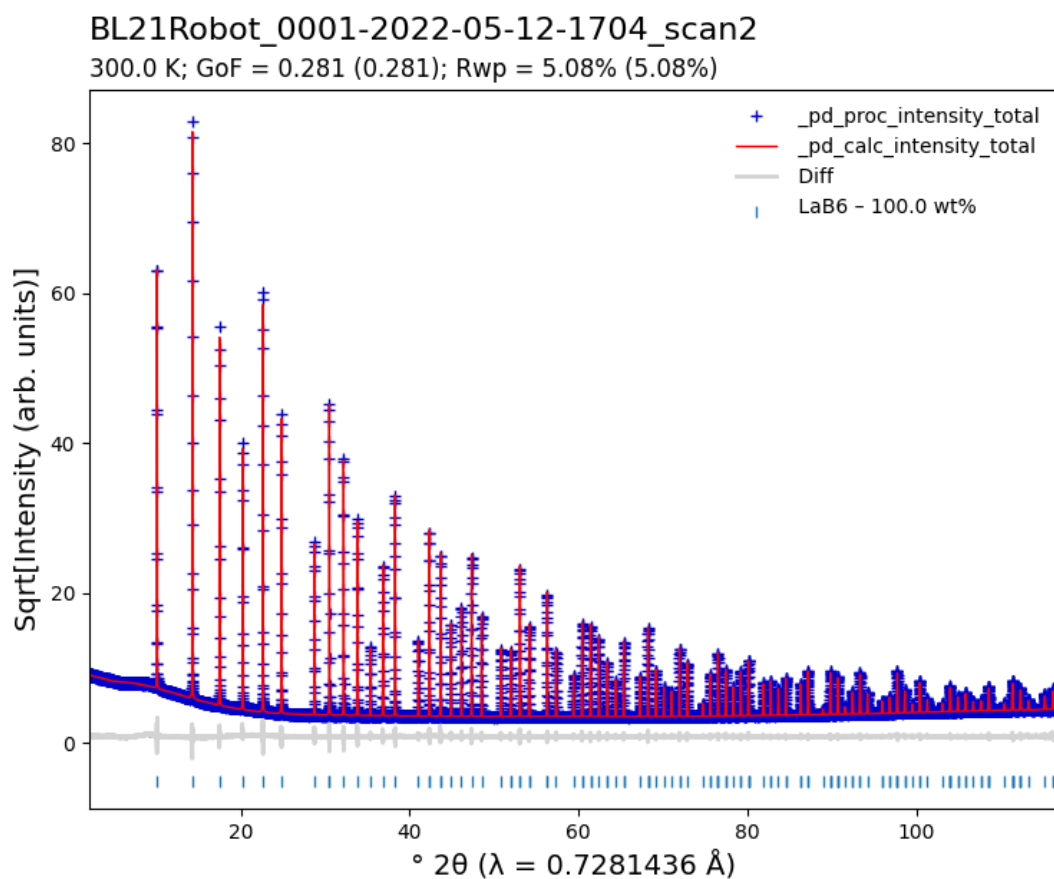
Sample ID	Sample Composition	Sample Notes	Refinement Included	Refinement Notes
0001	LaB <sub>6</sub>	NIST SRM 660c, lanthanum hexaboride	TA, GSASII	Includes instrument parameter file for GSAS-II created from Pawley refinement.
0002	SiO <sub>2</sub>	NIST SRM 1878b, alpha-quartz (respirable)	TA	Includes Pawley fit in TOPAS-Academic.
0003	ZnS	Zinc Sulfide phosphor material	TA	Includes individual peak fit, indexing, and Pawley fit in TOPAS-Academic.
0047	Pt(O <sub>2</sub> C <sub>3</sub> H <sub>7</sub> ) <sub>2</sub>	Platinum(II) bis(acetylacetonate) diluted with diamond powder	TA	Includes individual peak fit, indexing, and Pawley fit in TOPAS-Academic.
0093	Re	Rhenium metal diluted with diamond powder	TA	Includes individual peak fit, indexing, and Pawley fit in TOPAS-Academic. Cylindrical absorption correction included to keep thermal parameters physical.
0188	Er <sub>2</sub> O <sub>3</sub>	Erbium (III) Oxide mixed with diamond powder to dilute	TA	
0911	Li <sub>2</sub> CO <sub>3</sub>	Lithium Carbonate - Sigma Aldrich item 255823-100G	TA	Includes individual peak fit, indexing, and Pawley fit in TOPAS-Academic.
0912	MnO	Manganese(II) Oxide 60 mesh - Sigma Aldrich item 377201-500G	TA	Includes individual peak fit, indexing, and Pawley fit in TOPAS-Academic. Correct indexing is 14 <sup>th</sup> best solution found. Cylindrical absorption correction included to keep thermal parameters physical.

---

0913	Mn <sub>2</sub> O <sub>3</sub>	Manganese (III) Oxide - Sigma Aldrich item 463701- 25G	TA	Rietveld refinement suggests minor impurities of Mn <sub>3</sub> O <sub>4</sub> and β-MnO <sub>2</sub> .
0916	Fe <sub>2</sub> O <sub>3</sub>	Iron(III) Oxide - Sigma Aldrich item 529311-5G	TA	Rietveld refinement suggests minor impurity of FeO(OH).
0919	Sb <sub>2</sub> O <sub>3</sub>	Antimony (III) Oxide - Sigma Aldrich item 379255- 50G	TA	Includes individual peak fit, indexing, and Pawley fit in TOPAS-Academic.
0927	TiO <sub>2</sub>	TiO <sub>2</sub> less than 100nm particle size, mixture of Anatase and Rutile - Sigma Aldrich	TA	Would likely benefit from a more advanced size and strain model for peak broadening.
0933	Cr <sub>2</sub> O <sub>3</sub>	Chromium (III) Oxide - Sigma Aldrich item 393703- 100G, sample is hygroscopic and bottle was freshly opened just prior to loading the sample into the capillary and measuring on July 10, 2022	TA	

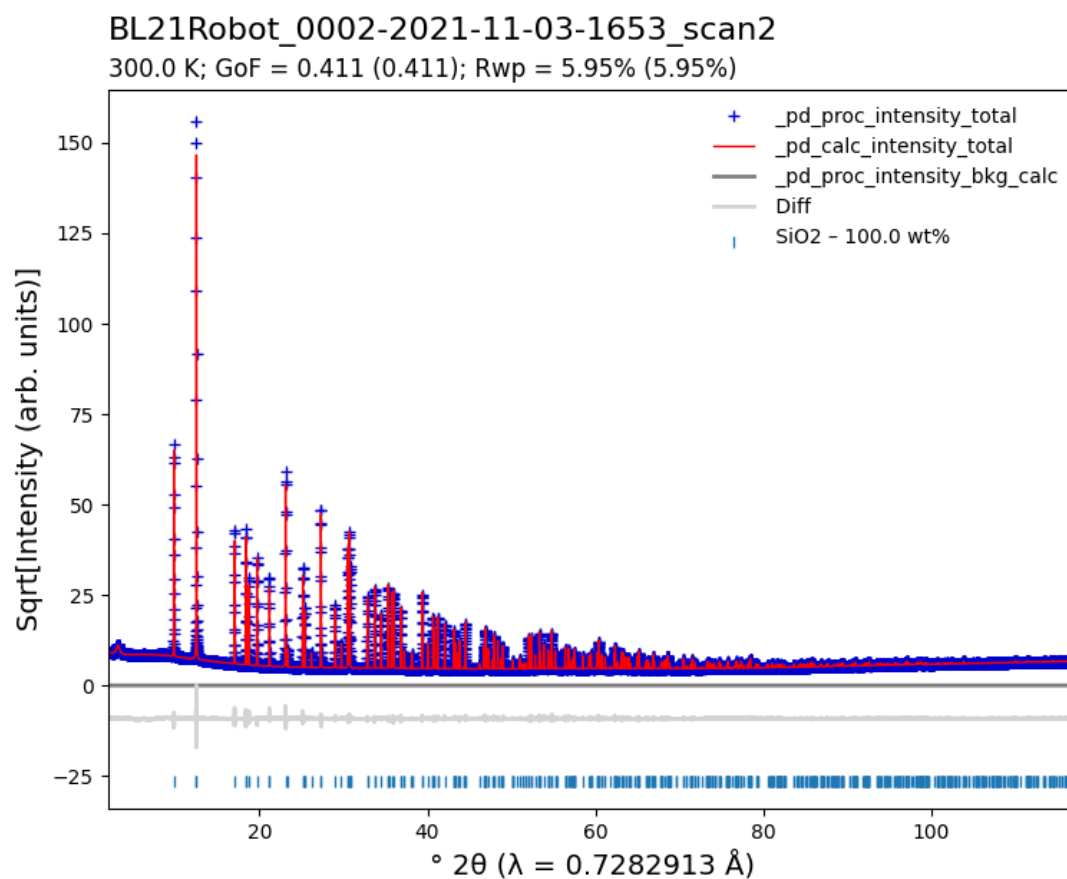
---

Rietveld refinements were done in TOPAS-Academic v6 (unless otherwise noted) and output generated using the pdCIF macros developed by Matthew Rowles<sup>19</sup>. Refinement data are plotted using the pdCIFplotter program<sup>20</sup>. The included refinements are not meant to be considered as final, these have been refined to basic structural models to give an overall impression of the data quality. Further improvements to the Rietveld refinement models are very possible. Similarly, the CIF output is not intended to be comprehensive, but of sufficient detail to enable easy plotting of the refinement result and the resulting structures. Plots of the Rietveld refinements performed in TOPAS-Academic are shown in Figures S1-S13.

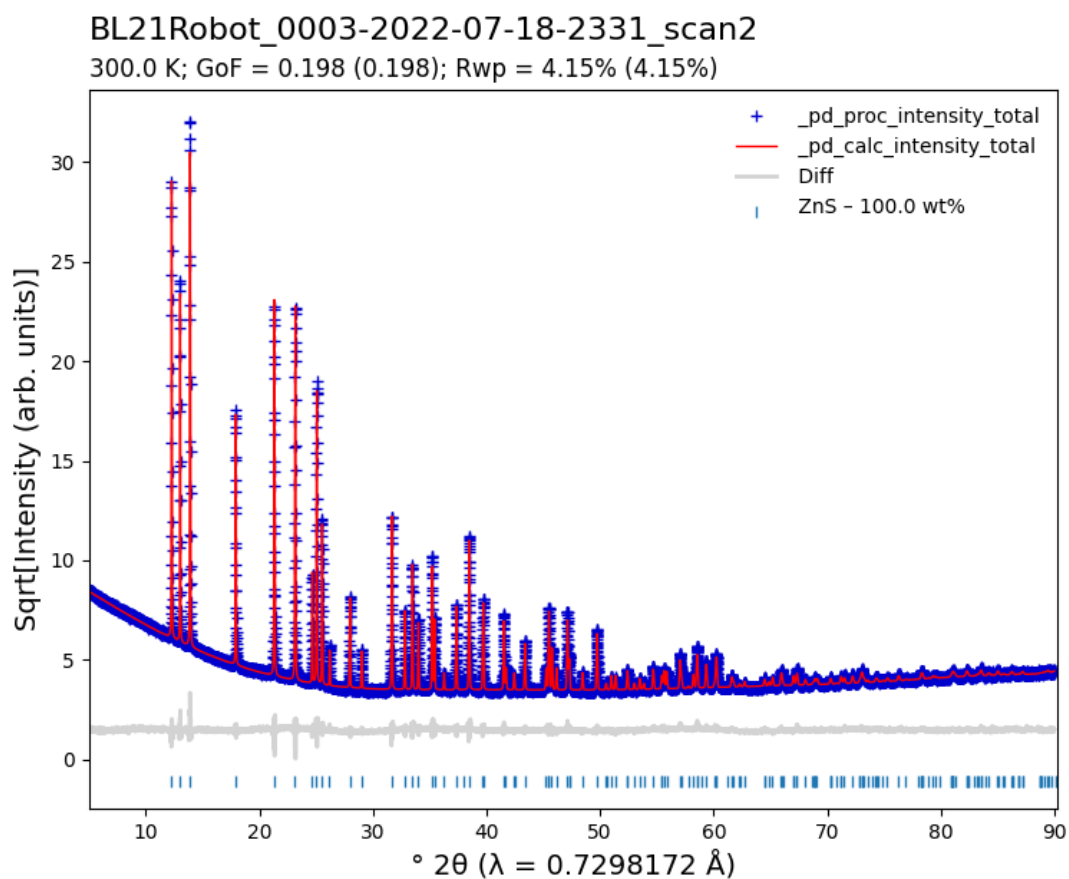


**Figure S1** Rietveld refined data for sample BL21Robot\_0001, lanthanum hexaboride powder.

Measured data are shown as blue crosses, calculated pattern in red with the difference shown below in grey. Tick marks indicate allowed peak positions.

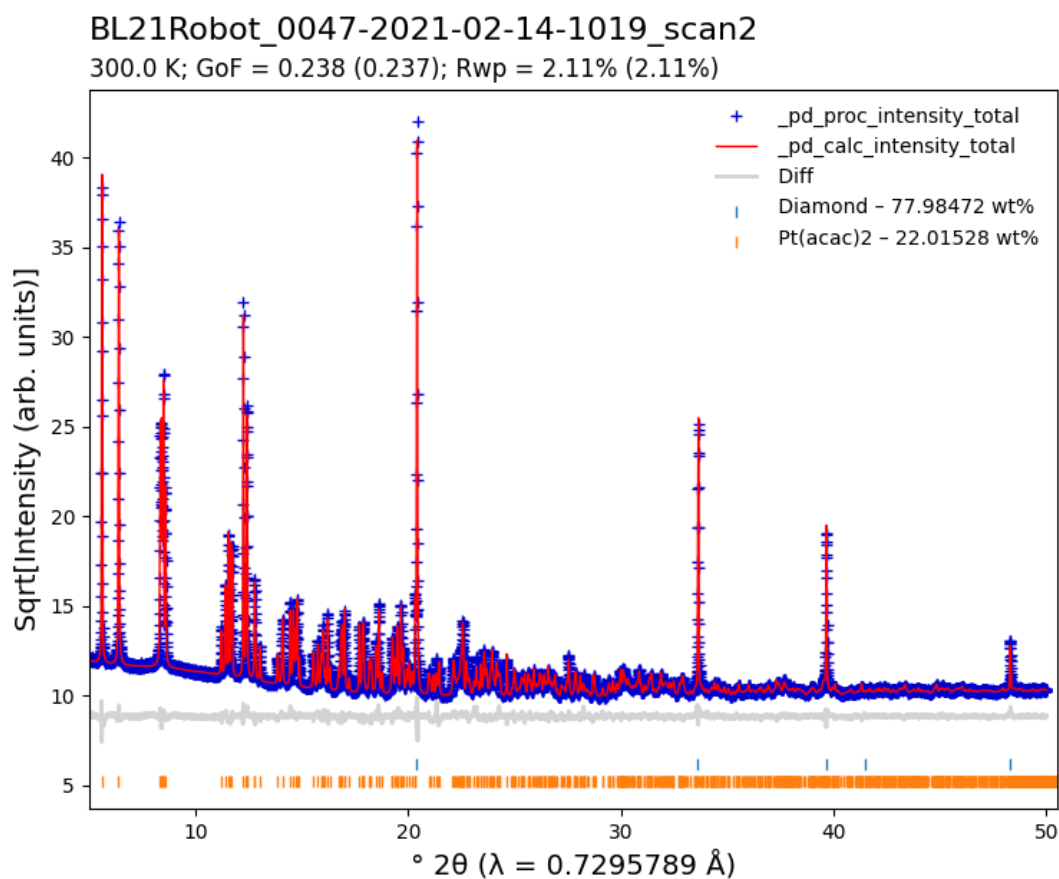


**Figure S2** Rietveld refined data for sample BL21Robot\_0002,  $\alpha$ -quartz. Measured data are shown as blue crosses, calculated pattern in red with the difference shown below in grey. Tick marks indicate allowed peak positions.

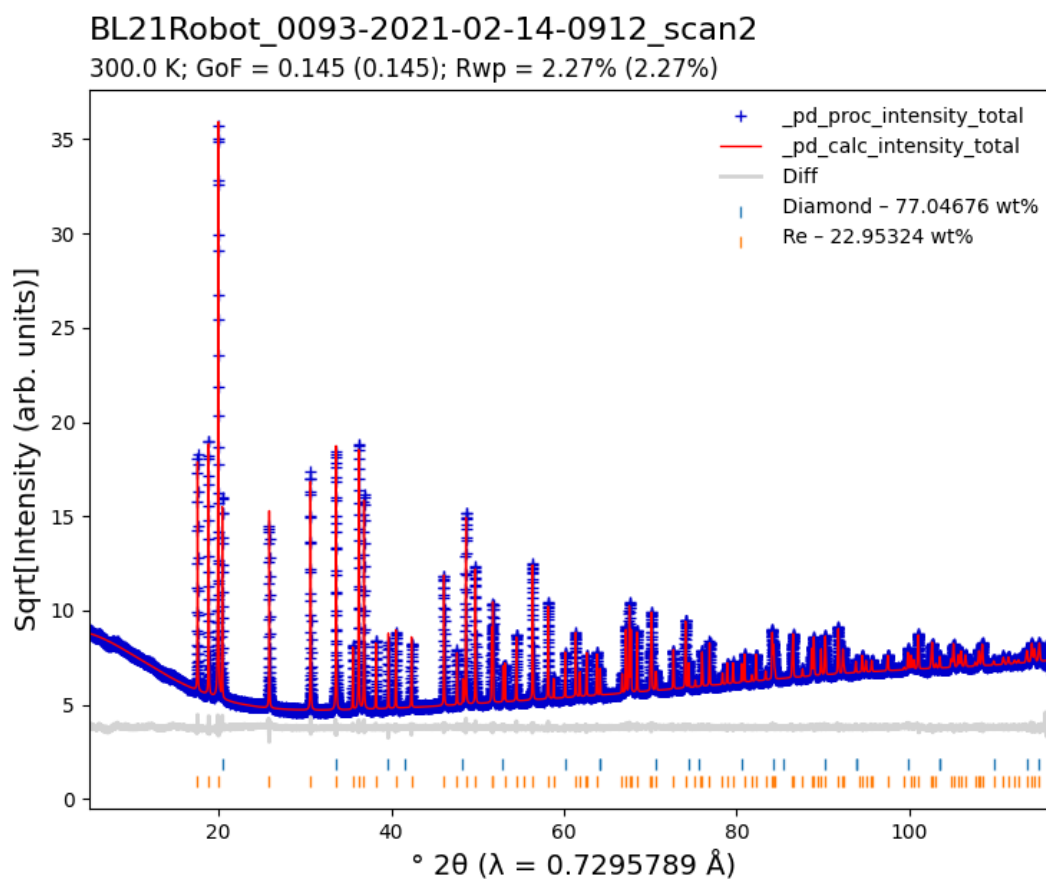


**Figure S3** Rietveld refined data for sample BL21Robot\_0003, zinc sulfide phosphor powder.

Measured data are shown as blue crosses, calculated pattern in red with the difference shown below in grey. Tick marks indicate allowed peak positions.

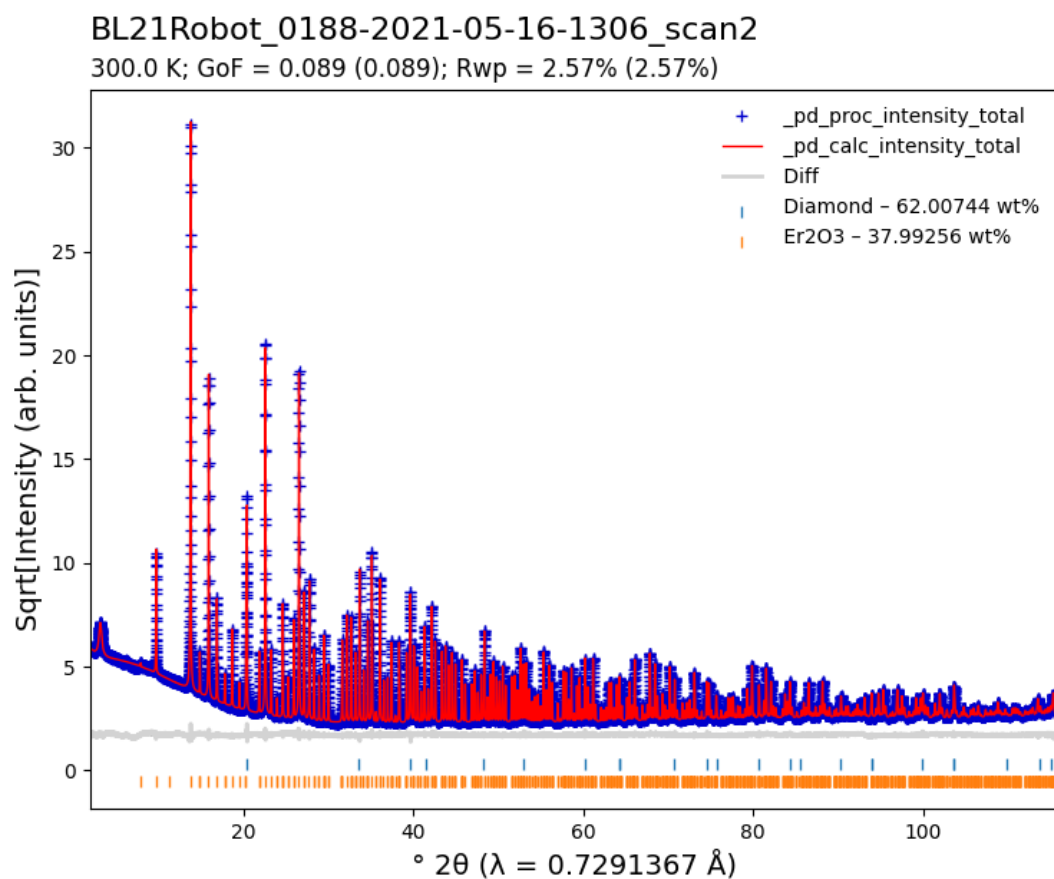


**Figure S4** Rietveld refined data for sample BL21Robot\_0047, platinum (II) bis(acetylacetonate) diluted with diamond powder. Measured data are shown as blue crosses, calculated pattern in red with the difference shown below in grey. Tick marks indicate allowed peak positions.

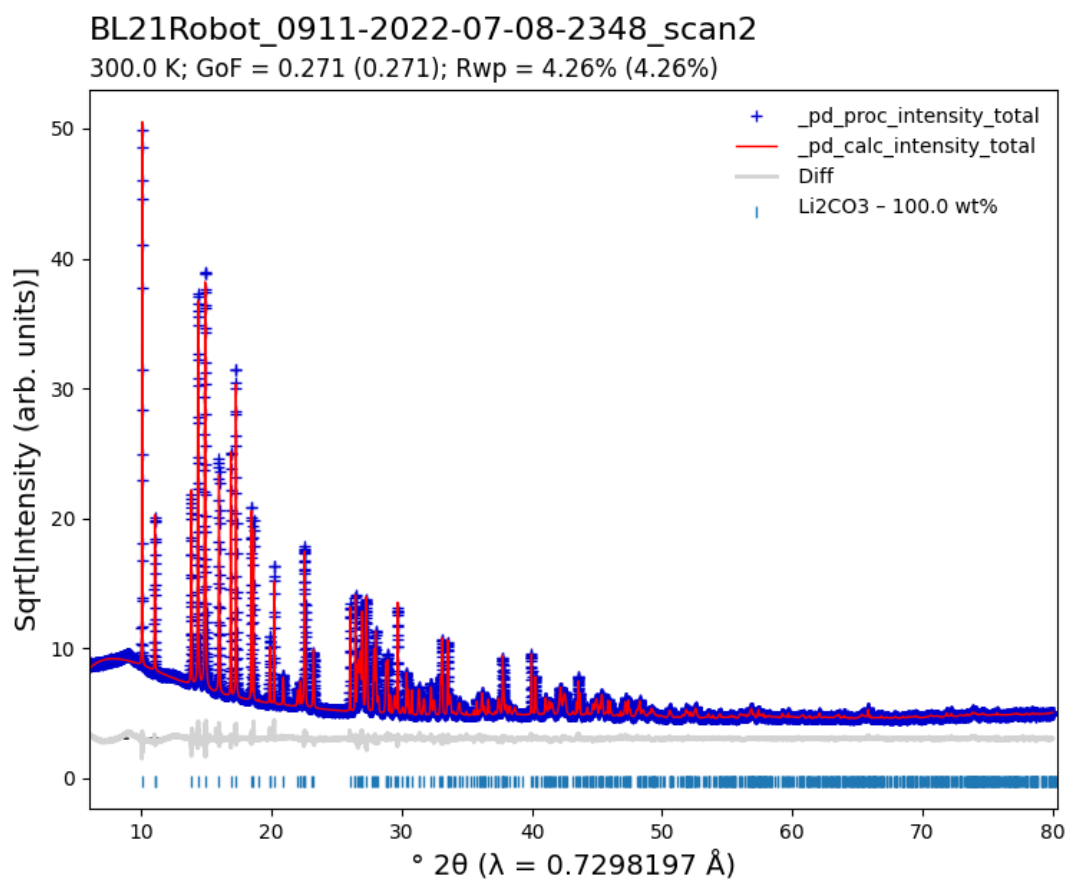


**Figure S5** Rietveld refined data for sample BL21Robot\_0093, rhenium metal diluted with diamond powder. Measured data are shown as blue crosses, calculated pattern in red with the difference shown below in grey. Tick marks indicate allowed peak positions.

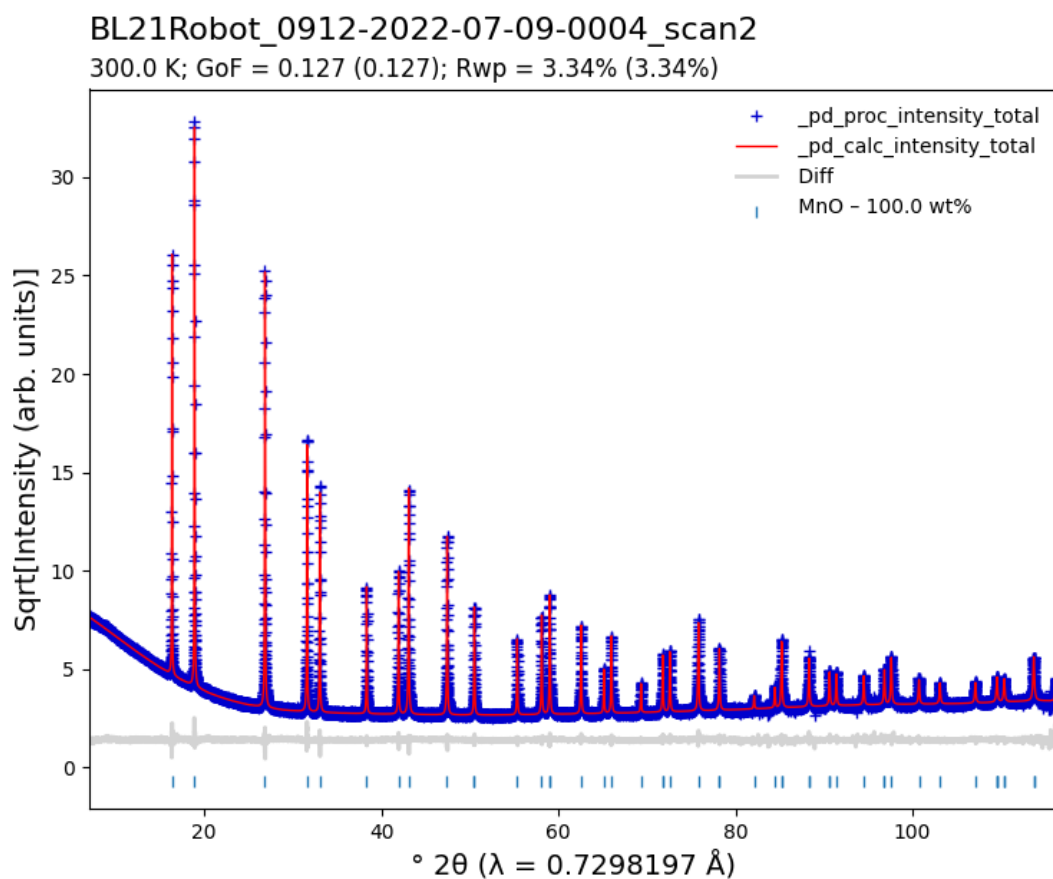




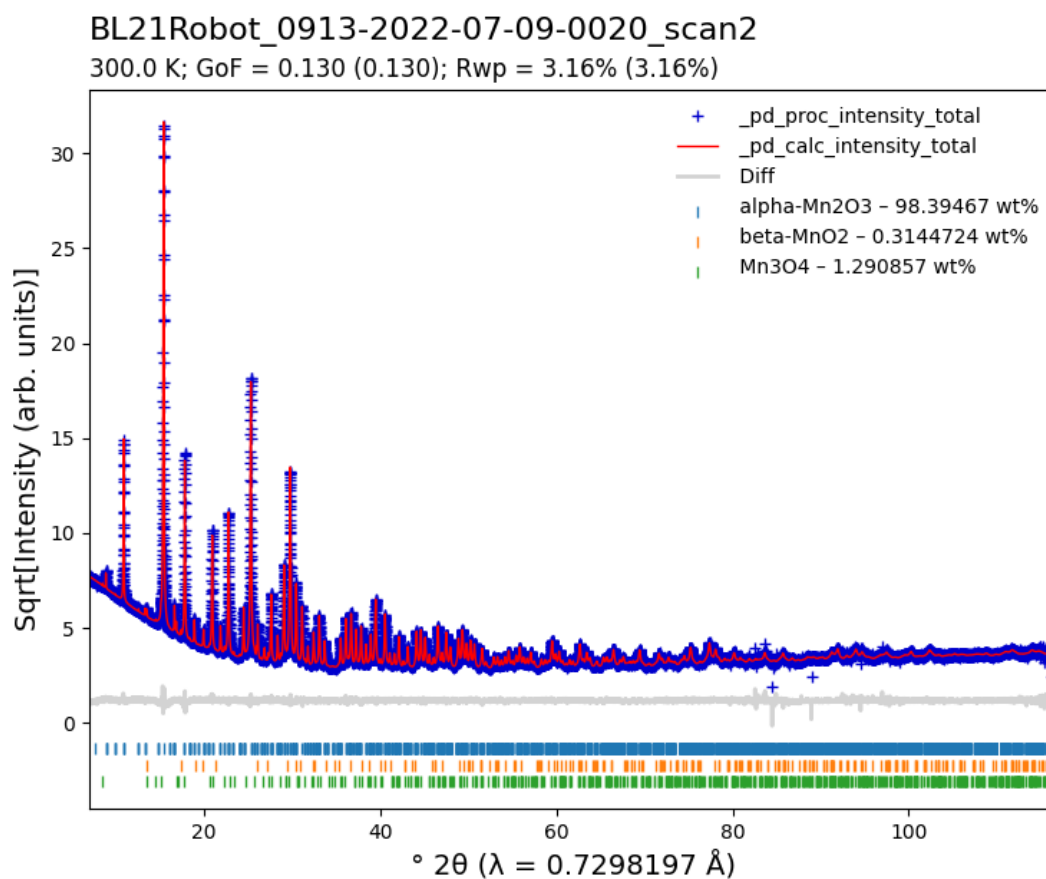
**Figure S6** Rietveld refined data for sample BL21Robot\_0188, erbium (III) oxide diluted with diamond powder. Measured data are shown as blue crosses, calculated pattern in red with the difference shown below in grey. Tick marks indicate allowed peak positions.



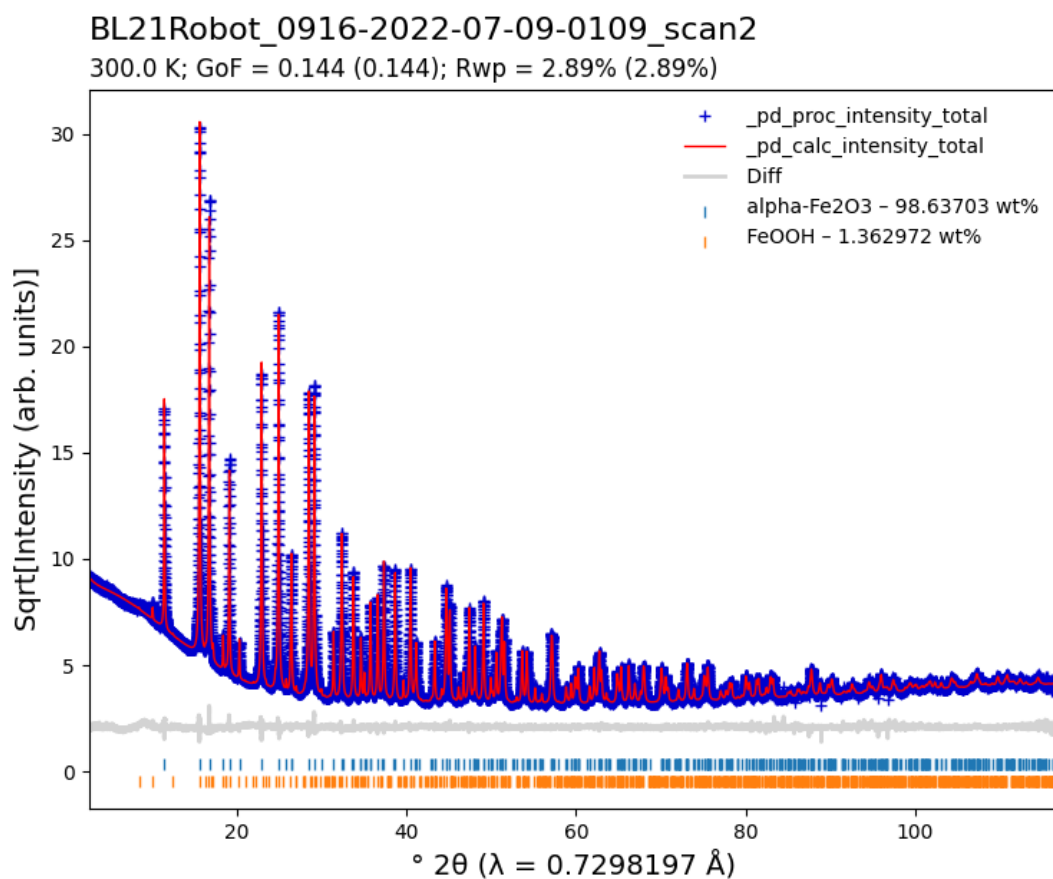
**Figure S7** Rietveld refined data for sample BL21Robot\_0911, lithium carbonate powder. Measured data are shown as blue crosses, calculated pattern in red with the difference shown below in grey. Tick marks indicate allowed peak positions.



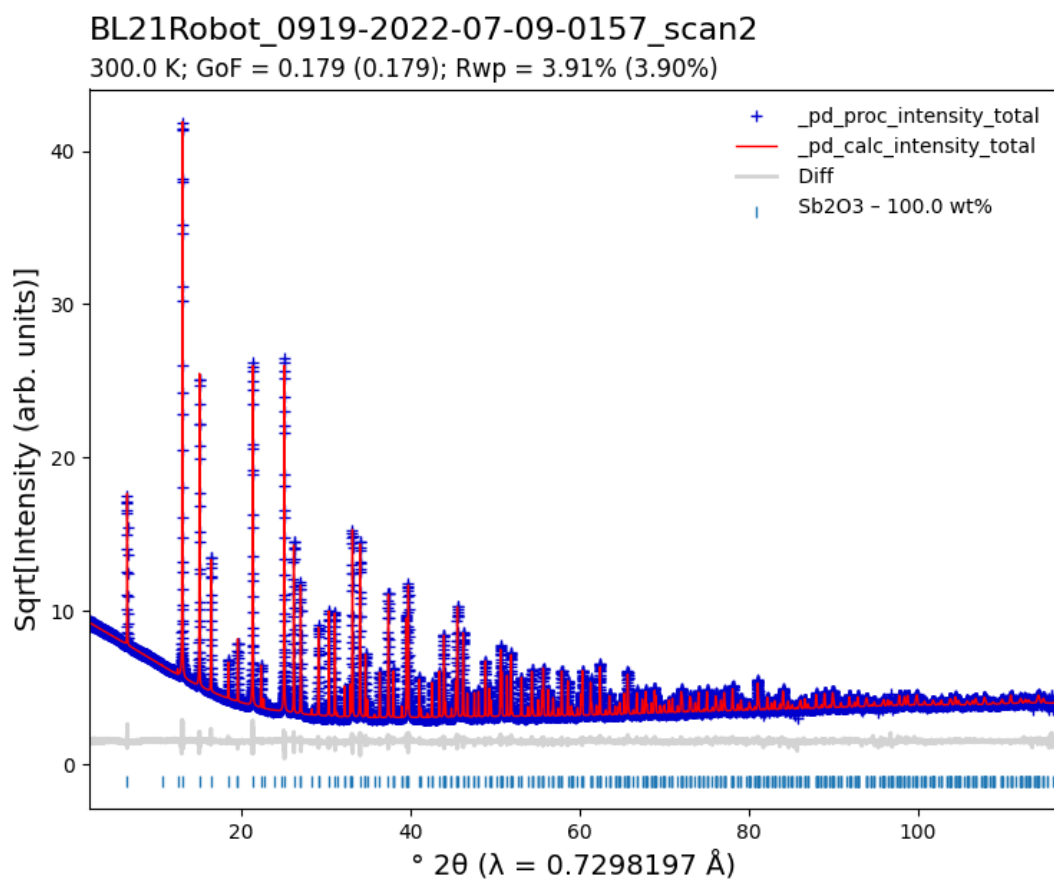
**Figure S8** Rietveld refined data for sample BL21Robot\_0912, manganese (II) oxide. Measured data are shown as blue crosses, calculated pattern in red with the difference shown below in grey. Tick marks indicate allowed peak positions.



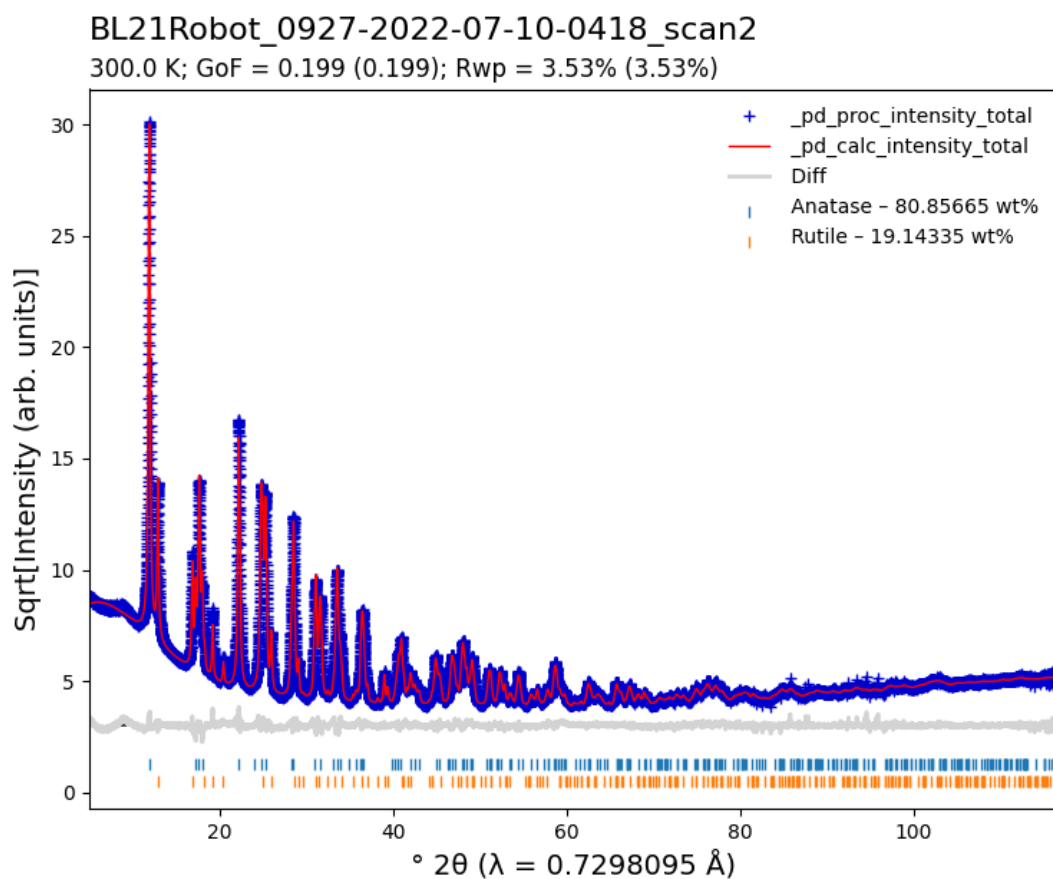
**Figure S9** Rietveld refined data for sample BL21Robot\_0913, manganese (III) oxide with minor impurities of  $\beta$ -MnO<sub>2</sub> and Mn<sub>3</sub>O<sub>4</sub>. Measured data are shown as blue crosses, calculated pattern in red with the difference shown below in grey. Tick marks indicate allowed peak positions.



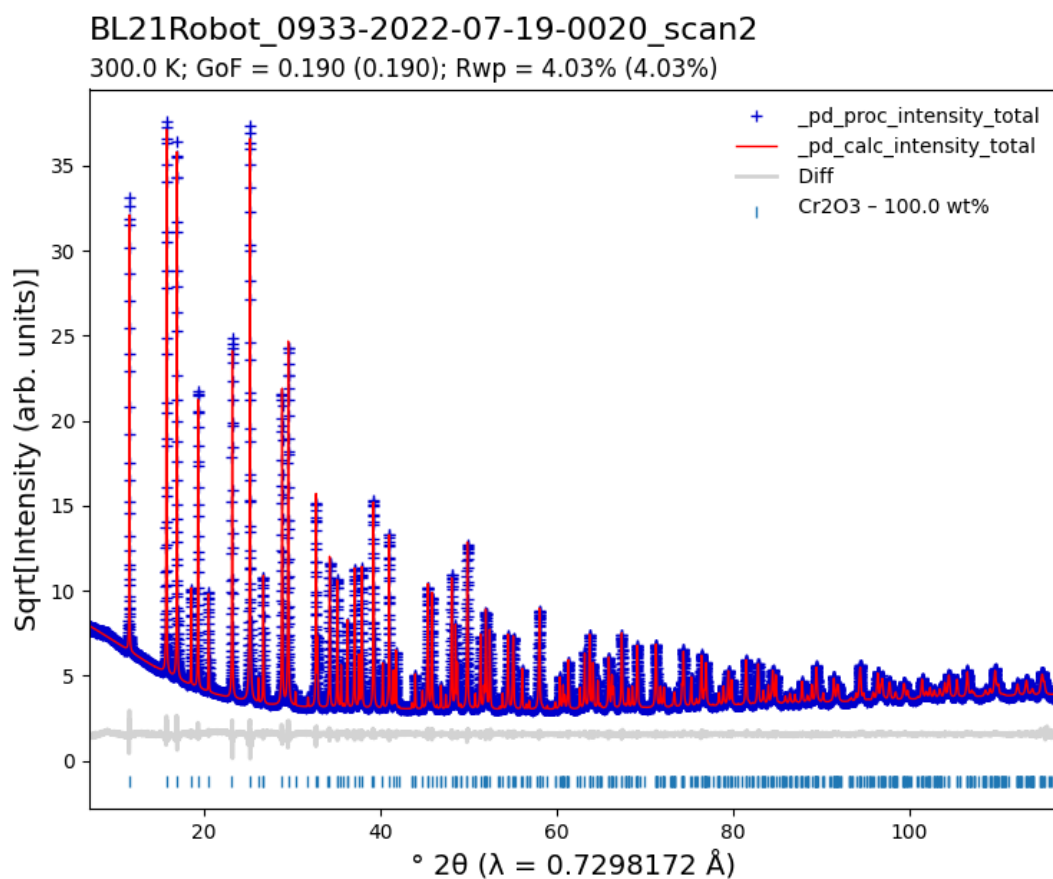
**Figure S10** Rietveld refined data for sample BL21Robot\_0916, iron (III) oxide with minor impurity of iron oxide-hydroxide. Measured data are shown as blue crosses, calculated pattern in red with the difference shown below in grey. Tick marks indicate allowed peak positions.



**Figure S11** Rietveld refined data for sample BL21Robot\_0919, antimony (III) oxide. Measured data are shown as blue crosses, calculated pattern in red with the difference shown below in grey. Tick marks indicate allowed peak positions.



**Figure S12** Rietveld refined data for sample BL21Robot\_0927, titanium dioxide in both rutile and anatase phases. Measured data are shown as blue crosses, calculated pattern in red with the difference shown below in grey. Tick marks indicate allowed peak positions.



**Figure S13** Rietveld refined data for sample BL21Robot\_0933, chromium (III) oxide. Measured data are shown as blue crosses, calculated pattern in red with the difference shown below in grey. Tick marks indicate allowed peak positions.