

X-RAY FACILITIES GROUP

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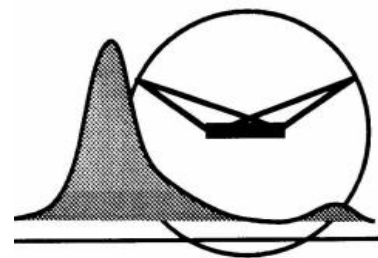
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XRD identification and semi-quantification of battery powders

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Researcher : Joep van de Ven, MPRR
Research question : Phase identification and semi-quantification

Samples

The samples are battery oxide powders.

Specimen

A thin layer of powder was deposited on a Si510 zero-background wafer in a standard PMMA holder.

Experimental

Instrument: Bruker D8 Advance diffractometer Bragg-Brentano geometry and Lynxeye position sensitive detector. Cu K α radiation. Divergence slit V12, scatter screen height 5 mm, 45 kV 40 mA. Detector settings: "LL 0.19 W 0.06".

Measurements

Coupled θ - 2θ scan 5° - 135° , step size 0.03° 2θ , counting time per step 2 s.

Data evaluation

Bruker software DiffracSuite.EVA vs 7.1, Profex BGMN 5.2.

Results

Figures 1 and 2 show the measured XRD patterns, after background subtraction and displacement correction. The "precipitate" sample is clearly amorphous. For the "residue" samples, the coloured sticks give the peak positions and intensities of the possibly present phases, using the ICDD pdf4 database.

Rietveld refinement was used in the semi-quantification. In table 1 the results are listed. The values in wt% are truncated to integer values. Figure 3 shows the Profex fitting plot.

<i>sample</i>	<i>compound</i>	<i>Wt%</i>
LA exp7 precipitate	amorphous	
LA exp7 residue	Heterosite	FePO ₄ 83 ± 1
	Lithium Manganese Oxide	Li _{0.57} Mn _{0.86} O ₂ 16 ± 1
	Graphite	C 1 ± 1

Table 1

*If the analysis is a significant part of a publication, a co-authorship is preferred.
In any case, it is useful to involve us in the preparation of any presentation to ensure optimum and correct use of the analysis results!*

*Whenever used in a publication, an acknowledgement will be appreciated, e.g.:
"personX at the Department of Materials Science and Engineering of the Delft University of Technology is acknowledged for the X-ray analysis".*

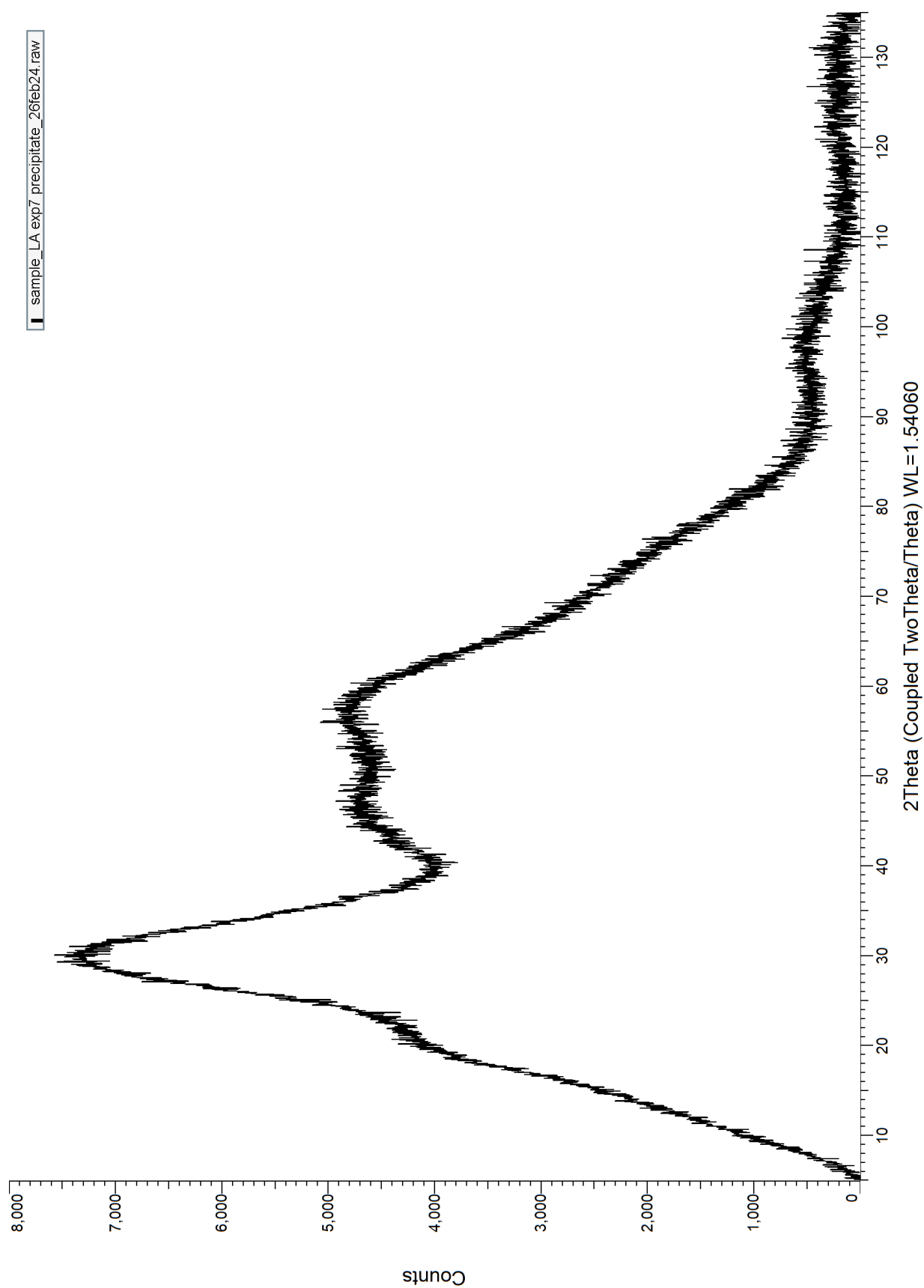


Figure 1 XRD pattern sample " LA exp7 precipitate"

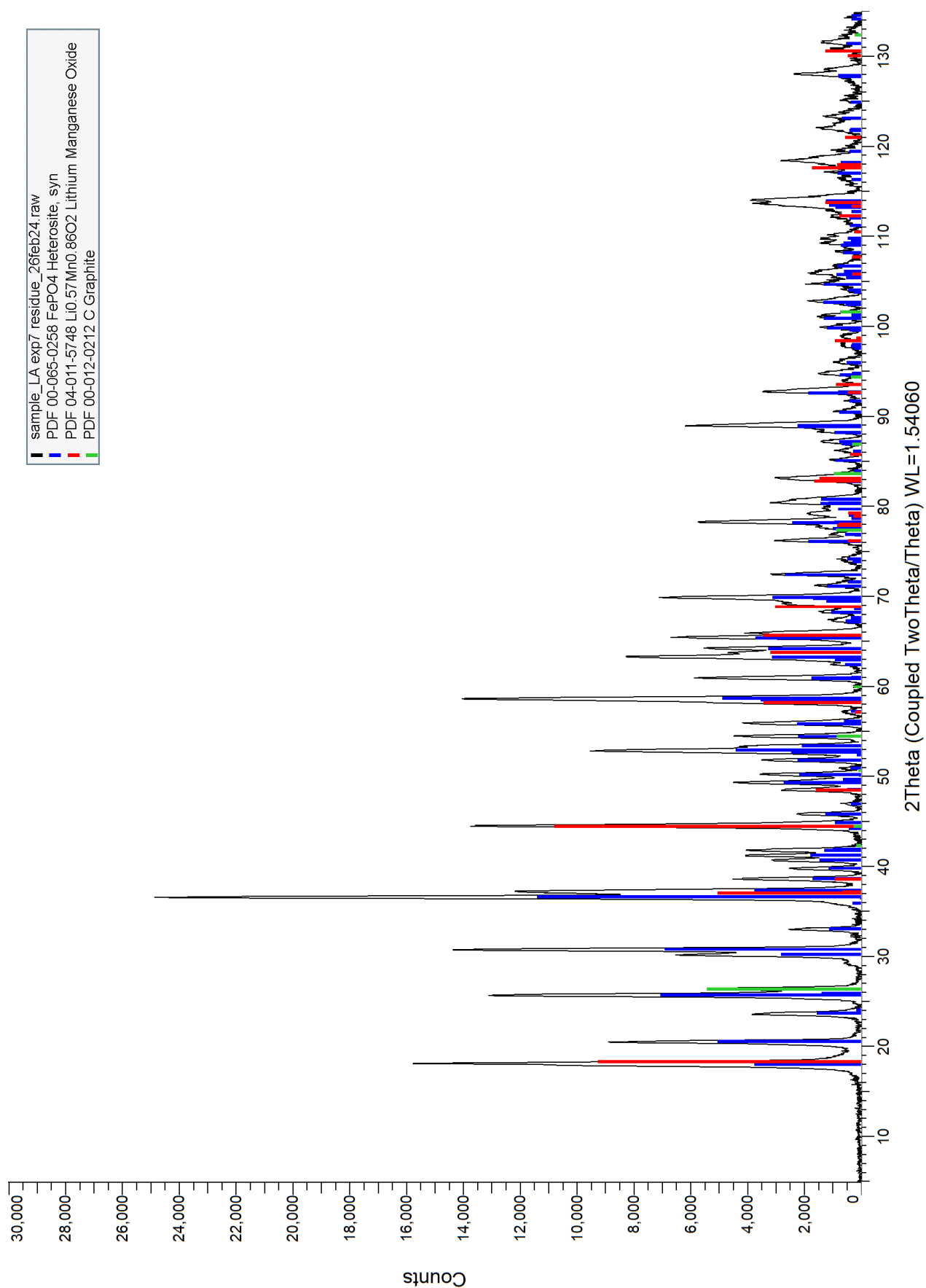


Figure 2 XRD pattern sample "LA exp7 residue"

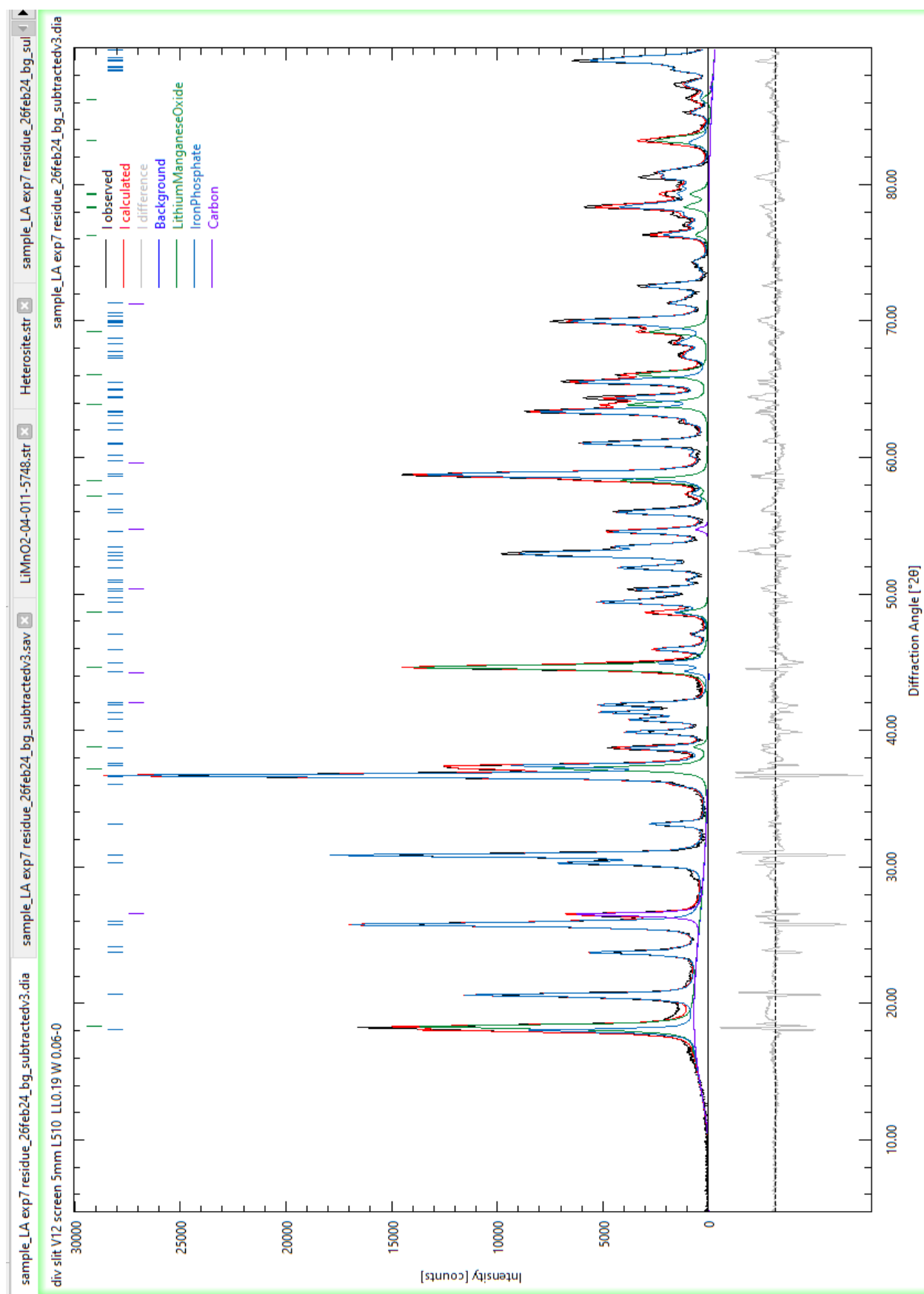


Figure 3 Profex fit sample " LA exp7 residue"