

X-RAY FACILITIES GROUP

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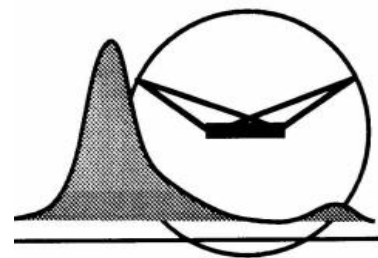
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XRD identification and semi-quantification of battery powders

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Date : 08 feb 2024
Researcher : Joep van de Ven, MPRR
Research question : Phase identification

Samples

The samples are battery oxide powders.

Specimen

A thick layer of powder (1mm) was deposited in the 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 $0.04^\circ 2\theta$, 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 exp6 precipitate	amorphous		
LA exp6 residue	Graphite-2H	C	74 ± 1
	Bunsenite	NiO	7 ± 1
	Heterosite	Fe(PO ₄)	13 ± 1
	Lithium Cobalt Iron Phosphate	LiFe _{0.75} Co _{0.25} (PO ₄)	5 ± 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".*

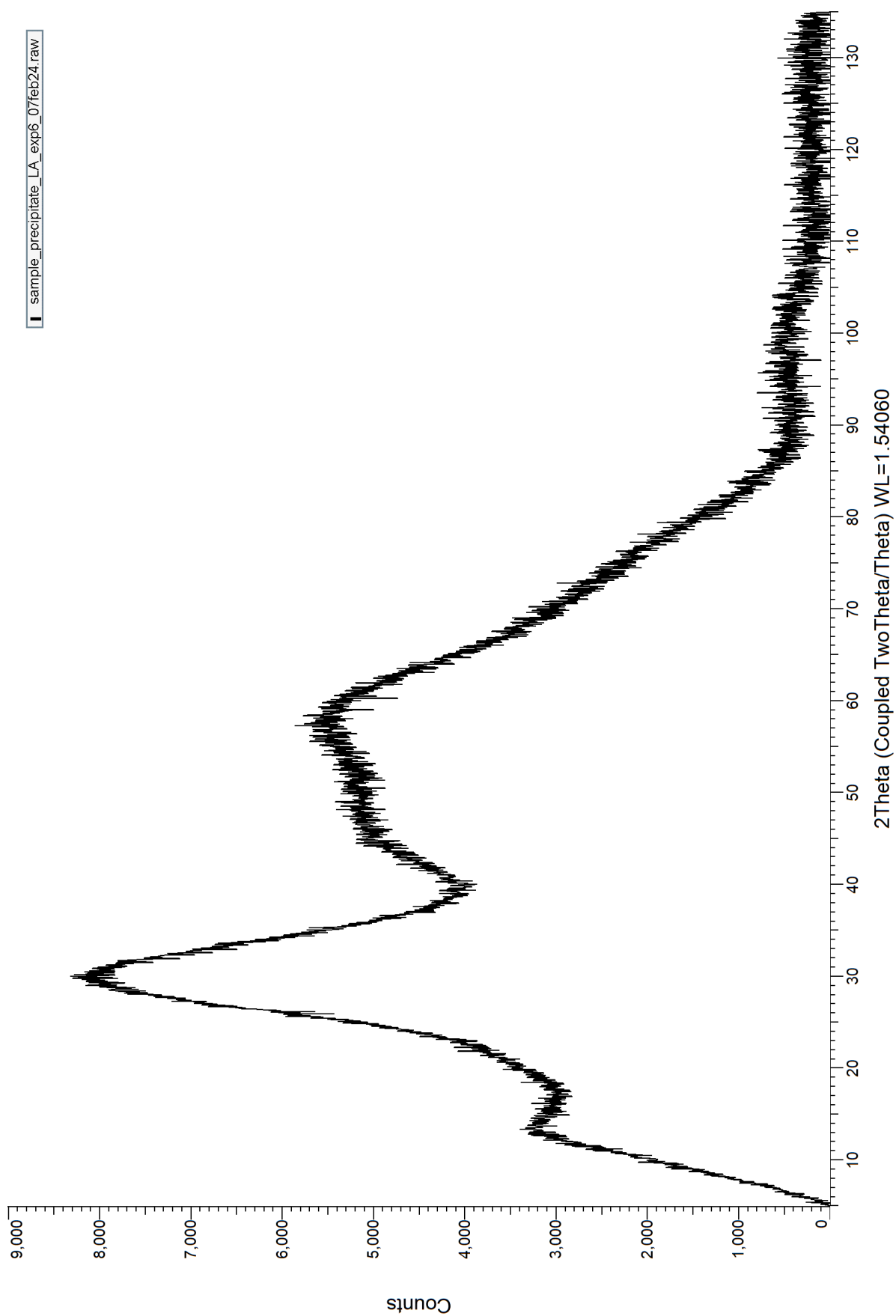


Figure 1 XRD pattern sample " LA exp6 precipitate"

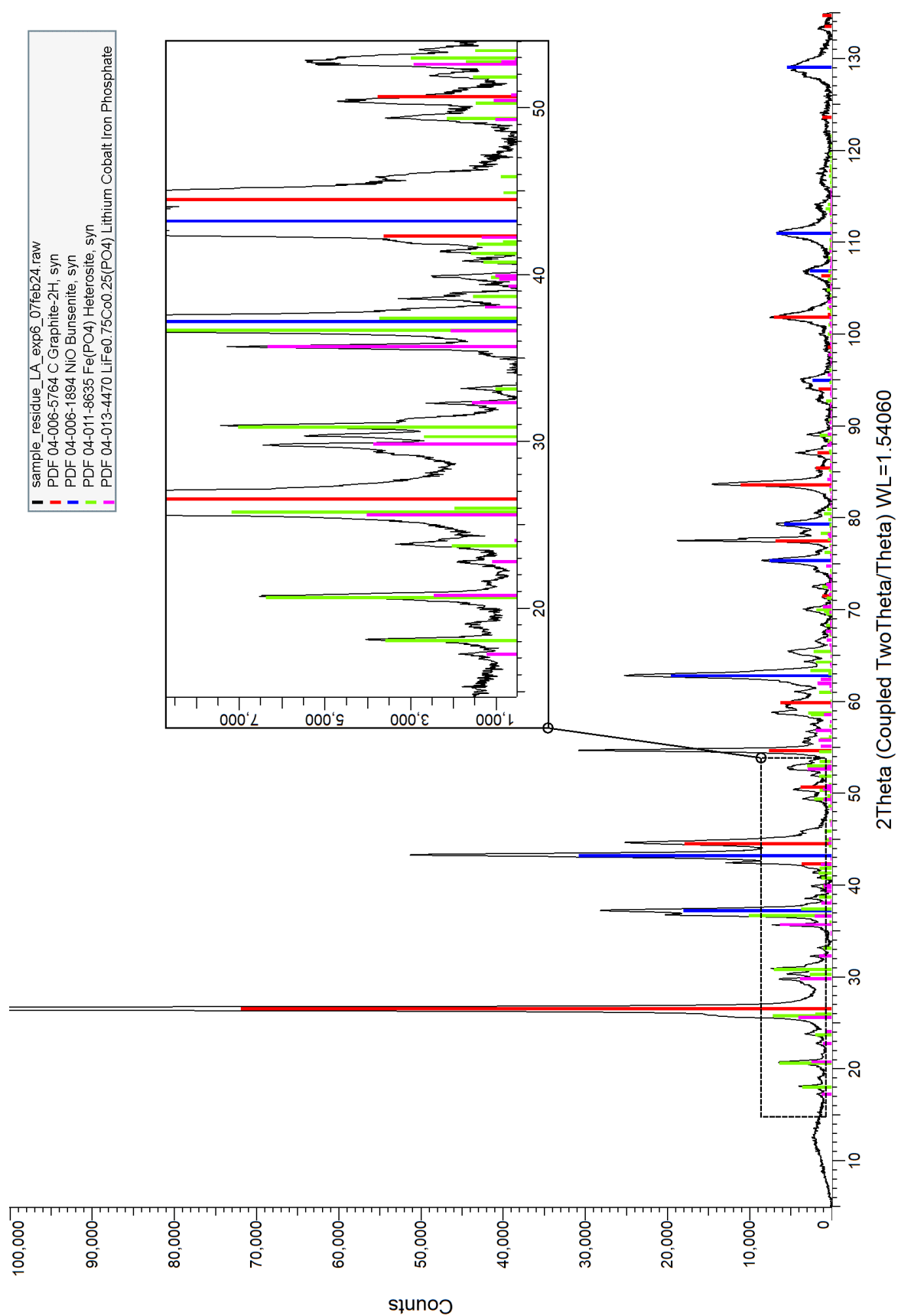


Figure 2 XRD pattern sample "LA exp6 residue"

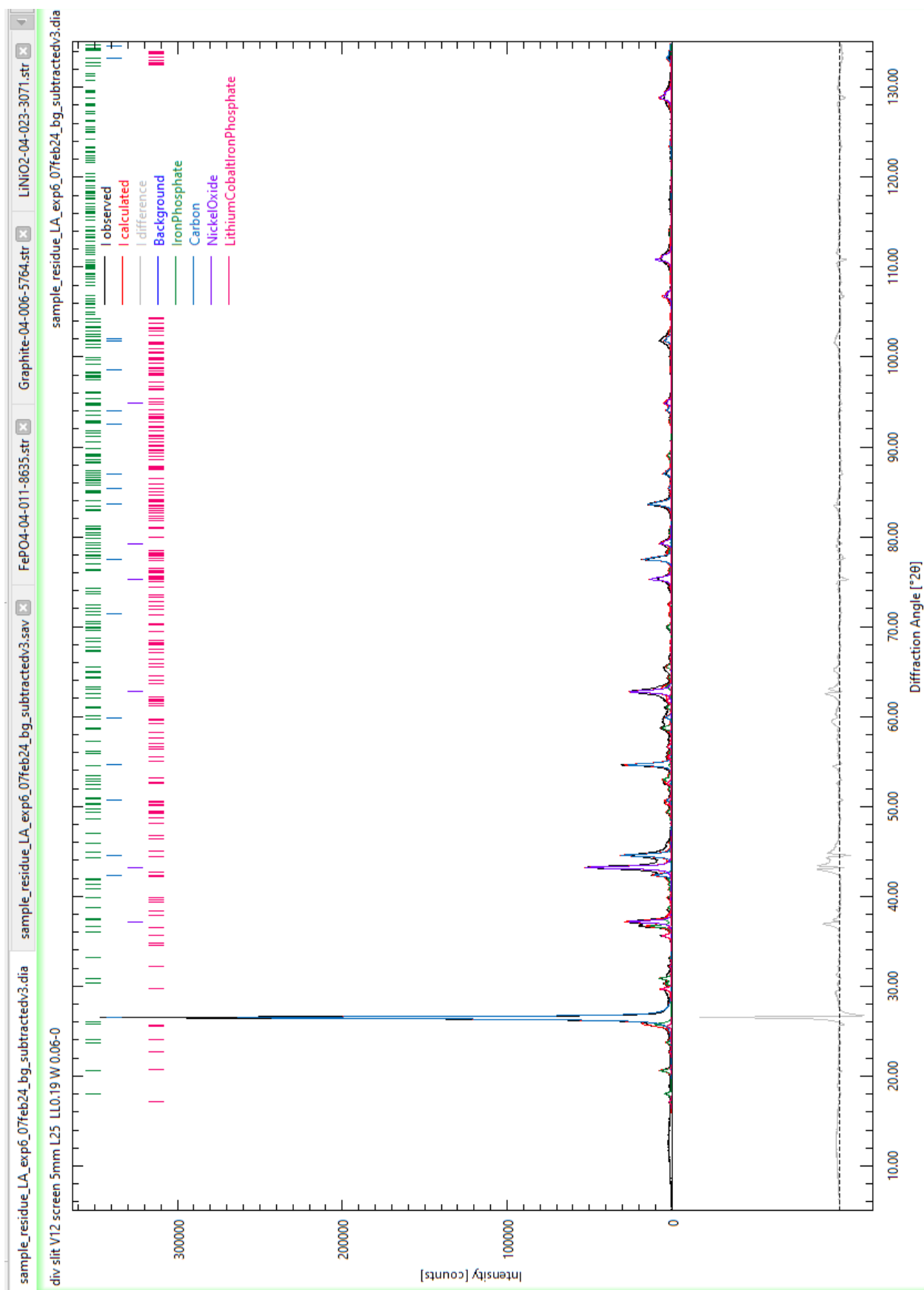


Figure 3 Profex fitsample " LA exp6 residue"