Data Article

Synthesis and structural data of a Fe-base sodium metaphosphate compound, NaFe(PO₃)₃

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This data article contains the synthesis and structure information of a new Fe-base sodium metaphosphate compound, which is related to the research article entitled ‘Synthesis, structural, magnetic and sodium deinsertion/insertion properties of a sodium ferrous metaphosphate, NaFe(PO₃)₃′ by Lin et al. [1]. The research article has reported a new Fe-base metaphosphate compound NaFe(PO₃)₃, which is discovered during the exploration of the new potential electrode materials for sodium-ion batteries. In this data article, the synthesized process of this metaphosphate compound and the morphology of the obtained sample will be provided. The high-power XRD Rietveld refinement is applied to determine the crystal structure of this metaphosphate compound and the refinement result including the main refinement parameters, atomic coordinate and some important lattice parameters are stored in the cif file. Also, the refined structure has been evaluated by checkcif report and the result is also provided as the supplementary materials.

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### Specifications Table

| Subject area          | Chemistry                              |
|-----------------------|----------------------------------------|
| More specific subject area | Crystal chemistry                      |
| Type of data          | Image, figure                          |
| How data was acquired | SEM (FE-SEM, Navo NanoSEM430), polycrystal-powder XRD (X'Pert PRO, PANalytical, Netherlands), GSAS program. |
| Data format           | Raw data                                |
| Experimental factors  | Determination of a new Fe-base sodium metaphosphate compound |
| Experimental features | Sample was obtained by solid-state method by heating at 600 °C. The structure is determined by Polycrystal-powder XRD Rietveld refinement. |
| Data source location  | South China University of Technology, Guangzhou |
| Data accessibility    | The data are supplied with this article |

### Value of the data

- The data provided the base information of the metaphosphate compound NaFe(PO₃)₃.
- The data provided the experimental and calculated XRD patterns of NaFe(PO₃)₃ compound.
- The data provided the structure refinement result of NaFe(PO₃)₃ compound.
- The data also provide the check if report of the refined structure.

### 1. Experimental design, materials and methods

#### 1.1. Sample preparation

NaFe(PO₃)₃ compound was synthesized by conventional two-step solid-state method and Na₂CO₃ (Aladdin, ≥ 99.8%), FeC₂O₄·2H₂O (Aladdin, ≥ 99.9%) and NH₄H₂PO₄ (Codow, ≥ 99.9%) powder reagents were used as the raw materials. The stoichiometric proportions of these raw materials with the molar rate of Na:Fe:P = 1:1:3 was carefully grand homogeneous in the agate mortar, put into the platinum crucible, and then preheated at 573 K for 10 h to expelling NH₃, H₂O and CO₂. After cooling down to room temperature, the samples were reground again in the agate mortar for 30 min, and then sintered at 873 K for 15 h in Pt crucible. After cooling down to room temperature naturally, the NaFe(PO₃)₃ compound was obtained. In order to prevent forming of the trivalent iron, all this sintering process was carried out under the reducing atmosphere (5%H₂ + 95%Ar atmosphere). Fig. 1 show photograph of the NaFe(PO₃)₃ sample synthesized by solid-state method and as can be observed, the white powder can be obtained.

#### 1.2. Morphology of NaFe(PO₃)₃

NaFe(PO₃)₃ powders were dissolved in ethyl alcohol and ultrasound for 30 min to disperse homogeneous. The mixture solutions were then adsorbed in the surface of the mica-sheet and dry in air directly. The typical SEM image of NaFeP₃O₉ powder, as shown in Fig. 2, was obtained by the field emission scanning electron microscopy (FE-SEM, Navo NanoSEM430).

#### 1.3. Polycrystal-powder XRD characterization

There is no special treatment for the sample before the polycrystal-powder XDR characterization. The sample was only slightly grand in the agate mortar after the sintering process and then the white
powder was obtained. The powder diffraction intensity data for the sample were collected using X’Pert PRO (PANalytical, Netherlands) with Cu \( K\alpha \) radiation (\( \lambda = 1.5418 \) Å) and a graphite monochromator was used for diffracted beams. Data were collected by a step scan mode with a scanning step of 0.02° at a sampling time of 3 s. This measurement was carried out at the room temperature.

Fig. 1. The macroscopic feature of NaFe(PO₃)₃ sample obtained by solid state method.

Fig. 2. The typical microstructure of NaFe(PO₃)₃ sample obtained by solid state method.
1.4. Structure refinement and determination

The structure of NaFe(PO\textsubscript{3})\textsubscript{3} compound is determined by the single-phase mode Rietveld refinement using the GSAS program\textsuperscript{[2]} via the EXPGUI interface\textsuperscript{[3]} and a space group of P2\textsubscript{1}2\textsubscript{1}2\textsubscript{1} (No. 19) was selected as the refined model. The starting positional parameters of all atoms were taken by analogy with those of the corresponding atoms in its isomorphic structures\textsuperscript{[4,5]}. The calculated XRD pattern produced by GSAS program was compared with that obtained by experimental method, as shown in Fig. 3. The refinement result including the main refinement parameters, atomic coordinate and some important lattice parameters are stored in cif file (provided in Supplementary materials).
which is produced by GSAS program. The crystal structure of NaFe(PO$_3$)$_3$ compound determined by the obtained cif file is shown in Figs. 4 and 5. Also, the obtained cif file is evaluated on the website of http://checkcif.iucr.org/ and its checkcif report is provided as the Supplementary materials.

Acknowledgments

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Appendix A. Supplementary materials

Supplementary data associated with this article can be found in the online version at http://dx.doi.org/10.1016/j.dib.2015.05.022.

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