Full-Profile Phase Search and Quantitative Phase analysis by the Rietveld Method of Makeup Powder from Ancient Egypt using FullProfAPP

In this exercise we will show how you can use FullProfAPP to discern the constituent phases from a complex archeological sample. The Egyptian makeup pigments that were presented in the original work dated from between 2000 and 1200 BC. and where studied using Synchrotron X-ray powder diffraction using the Rietveld Method.

The experimental data **(EgyptMakeup.xys)** was obtained at the DW22 beam line of the LURE laboratory (λ = 0.9627 Å). An instrumental resolution function file **(LURE.irf)** is also provided in the example.

1. Open FullProfAPP and select the working directory in 'Examples/EgyptMakeup'.

2. Use the 'Import Pattern' option to load the experimental data file into the app.

3. Extract the background using the options provided in the app.

4. The five major phases that were identified in the sample are: Galena (PbS), Cerussite ($PbCO_3$), Gypsum ($CaSO_4{\cdot}2H_2O$), Laurionite (PbOHCl), and Phosgenite ($Pb_2Cl_2CO_3$). Download CIF files from the Crystallographic Open Database using the app's client. For the sake of the exercise, and to show the capabilities of the app, we will perform a broader query and download more phases than needed. Make the following queries:

    a. Phases contain **Pb**, and may contain **C, O, S, and Cl**.

    b. Phases contain **Ca**, and may contain **C, O, S, and Cl**.

    c. Phases contain **Ca, S, O, and H**

    d. Phases contain **Pb, O, Cl, and H**

5. Perform a **Full-Profile Phase Search**. Take care with downloaded CIFs that have a very low symmetry, bad entries (<NA> symmetry number), and empty cells.

What phases did you find? Do the phases correspond with the originally identified ones? You can find the phases that were originally identified in the 'Examples/EgyptMakeup/ICSD' directory. Try to repeat the steps above, but now include the CIF files in the 'Examples/EgyptMakeup/ICSD' directory along with the rest of the downloaded phases.

Is there any change in the detected phases with respect to the previous run? Once you know which are the phases present in the sample, use the **Automatic Refinement Protocol** window to improve upon the refinement provided by the Full-Profile Phase Search. Try out different refinement strategies to see which one fits better. Check the validity of the refinement parameters. Correct bad parameters (negative temperature factors, negative broadening parameters, …) using the **Manual Refinement** window.