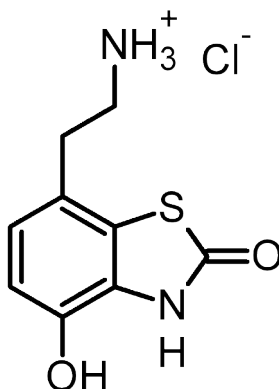# TUTORIAL EXPO: RIETVELD REFINEMENT

INSTRUCTIONS FOR EXERCISE 1

In this exercise, you will perform the crystal structure refinement by Rietveld method of 2-(4-Hydroxy-2-oxo-2,3-dihydro-1,3-benzothiazol-7-yl)ethylammonium chloride ($C_9H_{11}N_2O_2S \cdot Cl$).

Go to directory **Rietveld refinement/ammonium.** You will see the following files:

- **ammonium.xy** this is a file containing the experimental powder diffraction data.

- **ammonium_dm.cif** this is a file containing the crystal structure of the compound obtained from structure solution by direct methods.

- **ammonium_pub.cif** this is a CIF file containing the crystal structure from CSD database (CCDC 247129 DOI:10.5517/cc894xl).

- **ammonium.exp** this is an EXPO2014 input file for Rietveld method

Open and read the file ammonium.exp, it consists of the following lines:

```
%Structure ammonium
%Job ethylammonium chloride (C9 H11 N2 O2 S Cl)
%Data
        Pattern     ammonium.xy
        Wavelength 1.54056
%crystal
        file ammonium_dm.cif
%rietveld
```

The command %crystal is used to load the crystal phase contained in the CIF file ammonium_dm.cif. %rietveld is the command to enable the Rietveld refinement procedure.

To run EXPO2014 on ammonium:

- Open Expo2014 and click on **Load & Go** button in the **File** menu**.**

- Navigate to directory `ammonium` and load the input file `ammonium.exp`.

- Now click **GO**.

- It will open a the **Rietveld refinement window**.

- As you can see, your model does not have hydrogen atoms. For the moment close the Rietveld Refinement window by clicking **Quit**.

- Add hydrogen atoms by using a tool of JAV molecular viewer: **Tools→Add Hydrogens→ Automatic**. Based on the structure formula, hydrogen H2 is not expected and a hydrogen atom should be attached to the O1 atom. To show the name of the atoms click **View→ Label Type→Atom name** or click the toolbar button

- On the vertical toolbar click the selection button

- Delete the H2 atom: select the H2 atom, mouse right click, click **Delete Selected atoms or bonds**.

- Add H atom to O1 atom: select O1 atom, **Tools→Add Hydrogens→Hydroxyl O-H**.

- In the main window click **Refine→Rietveld**.

- Select **Automatic refinement of profile** and **Automatic refinement of structure** in the Rietveld Refinement window. Select **CIF** to export the result of refinement in a file CIF called `riet.cif` that you will find in the working directory `ammonium`.

- Click **Refine**.

- At the end of the automatic refinement process click on **Quit** to leave the Rietveld Refinement window.