# Phase Identification using Powder X-ray Diffraction

## Phase Identification

When samples are chemically identical, but possess different crystalline phases (or polymorphs), powder X-ray diffraction (PXRD) is a key analytical tool as it can rapidly distinguish between different phases of the same chemical composition. This is especially important within pharmaceuticals as PXRD can determine the difference between an active and a non-active polymorph in minutes. In addition, PXRD, can be used in the identification of minerals (such as quartz and cristobalite – both $SiO_2$), unreacted species, corrosion products amongst many other applications, where a solid crystalline form required identification.

Within this case study, we will look at the phase identification within a research sample formed from a high temperature reaction of apatite-wollastonite glass mixture, provided by a user at Newcastle University.

## Powder X-ray diffraction methodology and results

PXRD data were collected on a Panalytical X'pert diffractometer using Cu $K\alpha_{1/2}$ radiation. The peaks were picked using the standard algorithm within the PANalytical software HighScore Plus.[1] The peak positions were then compared to known faces using the Crystallography Open Database (COD)[2] using the Search/Match function within Highscore plus. Selecting the matched phases to those with the highest score reveals the sample contains the minerals Wollastonite, Cristobalite and Whitlockite (Figure 1). The relative intensities of the database phases and the matched peaks within the phase mixture further exemplifies the correct assignment of the identification.

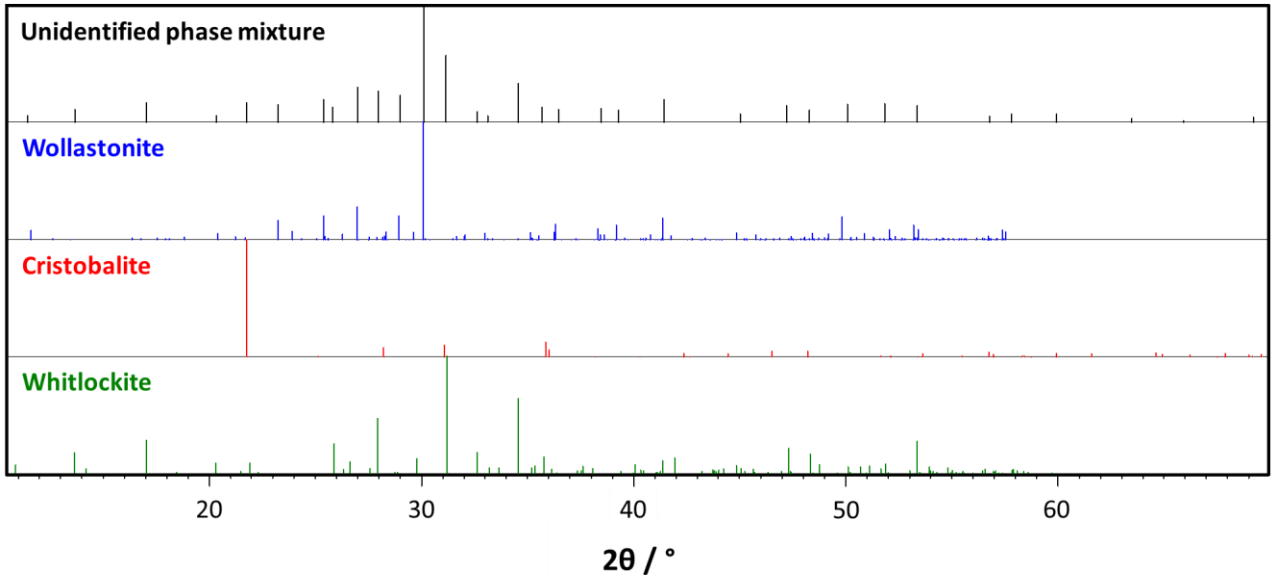


*Figure 1: Phase Identification of an unknown mixture. Analysis was completed using the search/match feature within HighScore Plus, comparing the patterns to the COD database.*

## Conclusions

Through picking the peaks within the powder X-ray diffraction pattern and comparing to known phases within the COD database permitted the assignment of the unidentified phase mixture as a combination of Wollastonite, Cristobalite and Whitlockite. If a whole pattern fit was performed and calibration samples were provided, then the relative phase distribution of the samples could also be determined.

## References


1. The HighScore suite, T. Degen, M. Sadki, E. Bron, U. König, G. Nénert, *Powder Diffr.*, (2014), **29**, S13

2. "Crystallography Open Database – an open-access collection of crystal structures"S. Grazulis, D. Chateigner, R. T. Downs, A. T. Yokochi, M. Quiros, L. Lutterotti, E. Manakova, J. Butkus, P. Moeck, & A. Le Bail, *J. Appl. Cryst.* (2009). **42**, 726.