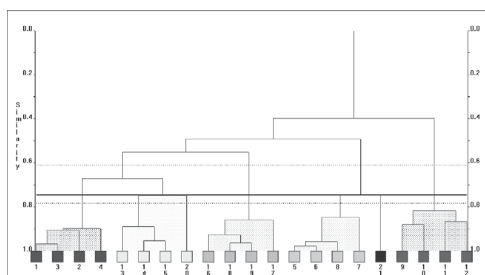


s8.m27.p10 **High Throughput Crystallography - What to do with 1000 Powder Patterns.** Christopher Gilmore, Gordon Barr and Wei Dong, *Department of Chemistry, University of Glasgow, Glasgow G12 8QQ, Scotland, UK. E-mail: chris@chem.gla.ac.uk*

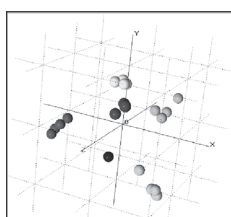
Keywords: Powder diffraction; Multivariate analysis; Classification

With modern robotic systems and data collection methods, it is quite possible to measure 1000 powder diffraction patterns in a few days, and this is becoming commonplace in some pharmaceutical laboratories where the search for polymorphs and salts is of great importance. The problem arises as to what to do with such data in particular how can it be grouped into classes when there is no database of pure phases, and there may well be mixtures present? We have developed two computer programs to address these issues [1,2] that use techniques of multivariate analysis, and classification to solve these problems. The formalism works as follows:

1. Data are suitably pre-processed with background removal, smoothing *via* wavelets, and peak searching.
2. Each of the n patterns is correlated with every other pattern using the Pearson and Spearman coefficients to generate an $(n \times n)$ correlation matrix.
3. This is used to generate a distance matrix which acts as a source of classification to generate dendrograms, multidimensional metric scaling, silhouettes, fuzzy clusters and minimum spanning trees; these are tools that can partition the data into clusters of related patterns. A typical dendrogram is show below:



Here we have partitioned 21 patterns into 6 clusters containing between 1 and 4 patterns. You can also represent the data in three dimensions using multidimensional metric scaling. Every sphere represents a powder pattern:



- [1] Gilmore, C.J., Barr, G. & Paisley, J. (2004). *J. Appl. Cryst.*, in press.
- [2] Barr, G. Dong, W. & Gilmore, C.J., (2004). *J. Appl. Cryst.*, in press.

s8.m27.p11 **Selective and direct localization of cations in zeolite by using anomalous powder diffraction.** H. Palancher^{a, b}, J.L. Hodeau^a, J.F. Berar^a, Ch. Pichon^b, B. Rebours^b, J. Lynch^b, J. Rodriguez-Carvajal^c; ^a *Lab. de cristallographie, CNRS BP166X 38042 Grenoble, France,* ^b *Institut Français du Pétrole, 1 & 4 av. Bois Préau, 92852 Reuil-Malmaison, France,* ^c *Lab. Léon Brillouin, CEA/Saclay, 91191 Gif/Yvette, France.* E-mail: hodeau@grenoble.cnrs.fr

Keywords: Contrast method; Anomalous scattering; Powder diffraction

The variation of the resonant scattering contribution near the edge of a given atom induces a variation of the contribution of this specific atom to diffracted intensities [1]. We present herein an application on powders to extract the contribution of a single element in a given crystallographic site.

This technique is still not largely used in powder diffraction probably due to its complexity and the relatively low contrast improvement. Bragg intensities in powder diffraction data are much weaker than in single crystal ones and the relative weakness of the anomalous f' and f'' contribution (-8 to -5 e.u. for transition metals) *impose* us to collect patterns with very good statistics and without any systematic errors. Furthermore due to the superposition of $(h\ k\ l)$ and $(-h\ -k\ -l)$ reflections in powder diffraction diagrams, the SAD method (based on f'' variation) can not be used for data refinement. In addition the powder diagram refinement are based on the one dimensional profile fit of all overlapping reflection versus Bragg angle and for complex samples (multiphase ones or with large cell parameters) structural information are less accurate and a "acceptable" fit can correspond to a false minimum with an inaccurate localisation or quantification of some atoms compensated by scattering attributed to other elements in the unit cell. The use of resonant scattering for such atoms, by collecting two or more X-ray patterns at different photon energies (or wavelength) corresponding to different resonant (or anomalous) contribution provide us a specific information on their localisation or quantification.

Here the method is validated by analysis of an industrially interesting sample, the hydrated SrX zeolite and application to cation distribution determination in a highly dehydrated SrRbX powder will be demonstrated. It must be stressed that the study of this last sample where both cations have the same number of electrons (35 e.u.), is a particularly difficult case for X-ray diffraction.

To validate our methodology three diffraction patterns with a q range up to at least 0.8\AA^{-1} were collected, in Debye-Scherrer geometry, on a hydrated SrX sample at respectively 900eV, 65eV and 10eV below the strontium K absorption edge ($E_{K-Sr} = 16.106\text{keV}$). The high quality of the diffraction patterns enables intensity variations with energy to be observed which exceed background noise even at q values corresponding to strong reflections. The anomalous effect is clearly measured and larger than the difference between observed and calculated patterns and, thus can be taken into account in the pattern refinement. A clear selective distribution of Sr, Rb atoms and water molecules is obtained.

We show that accurate localization of different atoms in powders is possible, the gain in the multi-wavelength powder data refinement allows a direct visualization of these atoms.

- [1] J.L. Hodeau *et al.*, *Chem. Rev.* **101**, 1843 (2001).