been confirmed with a full Rietveld refinement, and the code VNI
ble connectivity types, Suggestions for further developments to
sumptions of certain structural properties is also limited to materi­
overcome this problem are outlined,
the consequences of relaxing the structural assumptions were in­
connected topologies, The capabilities of
gies
metal phosphonate interactions led to unusual linear
erally, the structures of these compounds are layered, where the
layers which are separated by the organic groups on either side. In
reflections by a combination of direct
3-dimensional4-con­
ners will be discussed using some representative examples.
thermogravimetry, electron diffraction, etc. to
important to use the results from other methods like spectroscopy,
30-70
low angle(CuKα+)
and each trial structure is accepted or rejected
structural model. This technique differs considerably
high brightness, excellent collimation and wavelength
their high brightness, excellent collimation and wavelength
tunability provide optimum conditions for extracting individual
structures.
union of chemical and
important crystalline solids cannot be prepared in the
Many important crystalline solids cannot be prepared in the
powder diffraction data. In this method, a series of structural mod­
intensity information is not directly extracted
from powder diffraction data, in that intensity information is not directly extracted
from the diffraction pattern, and hence the problems of assigning
intensities in the powder pattern. In most cases we have been able
to obtain the best possible diffraction data. Synchrotron sources, with
This talk focuses mainly on the structure determination of
metals. These compounds are difficult
to obtain in single crystalline form and in most cases even
powder samples are poorly crystalline. The compounds yield
strong diffraction peaks whose intensities fall off very rapidly
at higher scattering angles. Despite these difficulties, we have been able
to solve the structures of a large number of compounds. Gen­
eral, the structures of these compounds are layered, where the
metal-phosphate inorganic framework forms a two-dimensional
layers which are separated by the organic groups on either side. In
some cases metal phosphate interactions led to unusual linear
porous structures. General methods used in solving the struc­
tures will be discussed using some representative examples.
In many cases the structures were solved by the use of 30-70
low angle(CuKα: 2θ=60°) reflections by a combination of direct
methods and heavy atom methods. The success comes from expe­
ence both in the structural aspects of these materials as well as
the systematic application of efficient methods. It is equally im­
portant to use the results from other methods like spectroscopy,
thermogravimetry, electron diffraction, etc. to arrive at the solu­
tion when only a limited number of powder diffraction data are available.

\[ \text{Ab initio} \] crystal structure determination from X­
ray powder diffraction data has been demonstrated by its applica­
tion to the solution of several crystal structures including
\[ \text{p-BrC}_6\text{H}_4\text{CH}_2\text{CO}_2\text{H} \]
[K. D. M. Harris, M. Tremayne, P. Lightfoot,
P. O. Bruce, J. Am. Chem. Soc. (1994), 116, 3543]
and the \(2\)-phase
\[ \text{3-CIC}_6\text{H}_4\text{CHCHCO}_2\text{H} \]
[B. M. Karuki, D. S. Zin, M.
Tremayne and K. D. M. Harris, Chem. Mat. (1996), in press]. In
the work presented here, we have extended the Monte Carlo method
for structure solution by a) simultaneous translation and rotation of
a rigid structural fragment within the unit cell - illustrated by the
structure determination of 1-methylfluorene (1) and b) simulta­
neous translation and rotation with additional rotation about in­
tramolecular bonds - illustrated by the structure determination of
7-hydroxyflavone (2).

\[ \text{CH}_3 \]
\[ \text{HO} \]
\[ \text{O} \]
\[ (1) \]
\[ (2) \]

**MS20.05.06 CRYSTAL CHEMISTRY FROM POWDER DATA.** Ian E. Grey CSIRO Division of Minerals, PO Box 124, Port Melbourne, Australia

A strength of structure analyses using powder data is the rel­
atively short time required to obtain high quality data sets. This
makes powder diffraction analysis particularly suitable for sys­
tematic structural studies on series of related compounds such as
solid solutions in minerals and their synthetic equivalents. The
high precision obtained when collecting and processing powder
data sets under identical conditions allows the detailed study of
subtle crystal chemistry correlations, for example variations in
cation site occupancies, anion vacancies and bonding interactions.

The use of variable counting time (VCT) data collections
(Madsen and Hill, 1994) enhances the capability to analyse subtle
structure variations using X-ray data collected using a conven­tional
laboratory configuration. In particular it yields more con­
istent thermal parameters, more accurate site occupancies and
more stable refinement of light atoms.
Examples will be presented of the application of Rietveld analysis of VCT powder X-ray data to investigate chemical and structure variations in synthetic layeringite. (Ca, Ti3+, Ti4+, Mn)2O3 prepared under reducing conditions, studies on anion vacancy and Fe3+/Fe2+ variations in the solid solution of hexagonal BaTiO3 with Ba2Fe2O5, and crystal chemistry systematics of sulphate and oxysulphate phases formed during sulphuric acid digestion of ilmenite.


MS02.05.07 INFORMATION ON SYMMETRY IN POWDER DIFFRACTION DATA. M. Ohsuma1, K. Ohsumi2 and H. Torny3. 1Department of Life Science, Himeji Institute of Technology, Japan; 2Photon Factory, National Laboratory for High Energy Physics, Japan; 3Ceramics Research Laboratory, Nagoya Institute of Technology, Japan.

Since each crystal family except triclinic, monoclinic and orthorhombic ones doesn’t correspond to a Laue class uniquely: (holohedral and hemihedral Laue classes) and no method to identify Laue classes of those families had been presented for powder specimens, their space groups had been assigned only with crystal family and reflection conditions. Because summed intensities are measured by powder diffraction methods and they are not separable to individual intensities. Recently Ohsuma & Ohsumi (1995) indicated that weighted reciprocal lattices named composite reciprocal lattices are constructed from powder diffraction data in such a way that summed intensities are distributed to reciprocal lattice points to hold holoheclral symmetry and indicated that information on Laue classes can be obtained from concentrations of interatomic vectors in Patterson functions evaluated with intensities at the composite reciprocal lattice points (composite Patterson functions). For hemihedral Laue classes, a composite reciprocal lattice is regarded as a superposed record of weighted reciprocal-lattice points of a single crystal. In this case the superposition yields new symmetry generators (extra generators) which are not intrinsic to the Laue class of the single crystal. The same generators as the extra ones are included in holoheclral symmetry and the distribution of the points and their relative weights in the composite Patterson function coincide with those in the Patterson function of the real structure. The same generators as the extra ones are not included in the symmetry generators of the real structure with hemihedral Laue symmetry but the apparent symmetry of the composite Patterson functions is enhanced to holoheclral one by the extra generators. However the distribution of the peaks and their relative weights in the composite Patterson functions are not perturbed by the extra generators and the feature of the concentration of the peaks related to interatomic vectors in the real structure is retained. Consequently, distinction of space groups should in principle be possible by interpretation of composite Patterson functions. Composite Patterson functions of some materials will be indicated as examples.


MS02.05.08 THE TANGENT FORMULA DERIVED FROM PATTERSON FUNCTION ARGUMENTS: A USEFUL TOOL FOR SOLVING ZEOLITE STRUCTURES FROM X-RAY POWDER DATA. J. Rius & C. Miravitles, Institut de Ciencia de Materials de Barcelona (CSIC), Campus de la UAB, 08193-Cerdanyola, Catalunya (Spain).

The viability of solving the structure type of zeolitic and layered materials applying multisolution direct methods to low resolution (a2.2 Angstroms) powder diffraction data is shown. The phases are refined with the tangent formula derived from Patterson function arguments [Rius (1993). Acta Cryst. A49, 406-409] implemented in the KLENS program and the correct phase sets are discriminated with the conventional figures of merit.

The two test examples presented are (a) the already known tetragonal zeolite ZSM-11 (space group I-4m2) at 2.3 Angstroms resolution and (b) the hitherto unknown layer silicate RUB-15 (Ibam) at 2.2 Angstroms resolution. In both cases, the tetrahedral Si units appear as resolved peaks in the Fourier maps computed with the phases of the highest-ranked direct methods solutions.

MS02.05.09 DIRECT PHASING FROM POWDER DATA: THE EXTRA OPTIMIZED PROCEDURE C. Giacovazzo, A. Altomare(+), G. Cascarano(+), A. Guagliardi(+), A.G.G. Moliterni(+), M.C. Burla(*) & G. Polidori(*) (*) Istituto di Ricerca per lo Sviluppo di Metodologie Cristallografiche - CNR c/o Dipartimento Geomineralogico - Campus Universitario Via E. Orabona, 4 - 70125 Bari - Italy, (+) Dipartimento di Scienze della Terra, Universita, 06100 Perugia, Italy.

Present difficulties in solving crystal structures from powder data are not to be found in the lack of efficiency of direct methods, but in the inaccuracy of the process which extracts structure factor amplitudes from a powder pattern. EXTRA [A. Altomare, M. C. Burla, G. Cascarano, C. Giacovazzo, A. Guagliardi, A. G. G. Moliterni & G. Polidori (1995), J. Appl. Cryst. 28,842-846] has been an efficient answer to this problem: friendly to use, the program is able to automatically decompose, via the Le Bail algorithm, quite complicated experimental patterns. EXTRA is now incorporating new important features. It is able to take advantage from: a) the Patterson map; b) the pseudotranslational symmetry; c) the preferred orientation; d) the statistical estimates of the diffraction amplitudes via direct methods; e) a molecular fragment.

All the above sources of information are able to dramatically improve the accuracy of the pattern decomposition process so enlarging the size of crystal structures solvable by powder data. We are merging EXTRA with SIRPOW.92 [A. Altomare, G. Cascarano, C. Giacovazzo, A. Guagliardi, M.C. Burla, G. Polidori, & M. Camalli, (1994), J. Appl. Cryst. 27, 435-436], our direct methods program for powder data, to provide a unique program, transparent to the user, which should be able to solve a structure directly the diffraction pattern.