exactly with heterogeneous structures, and also with neutron powder diffraction data.

The geometric treatment and the analytical results derived in this work [Bricogne (1991), *loc. cit.*] can also be applied to the harmonic overlap problem which affects single-crystal data collected by the Laue method

MS–02.03.02 DETERMINATION OF MOLECULAR CRYSTAL STRUCTURES ON A LABORATORY POWDER DIFFRACTOMETER. P. Lightfoot*, M. Tremayne, K. D. M. Harris and P. G. Bruce, Department of Chemistry, University of St Andrews, St Andrews, Fife, Scotland.

Many problems in materials chemistry require detailed structural information about the materials under study. Traditionally such information has been obtained by single crystal X-ray diffraction, however, many materials cannot be obtained in single crystal form. In recent years it has been shown that simple crystal structures can be determined *ab initio* by powder diffraction techniques, often at a synchrotron source (A. K. Cheetham and A. P. Wilkinson, *J. Phys. Chem. Solids*, 1991, 52, 1199). However, in order for the technique to become viable as a more routine tool for day-to-day use, a strategy for structure determination on laboratory-based powder diffractometers is required. We are currently testing existing methods for structure determination by X-ray powder diffraction on a state-of-the-art laboratory-based diffractometer (P. Lightfoot et al., *J. Mater. Chem.*, 1992, 2, 361; P. Lightfoot et al., *J. Chem. Soc. Chem. Commun.*, 1992, 1012; M. Tremayne et al., *J. Solid State Chem.*, 1992, 100, 191; M. Tremayne et al., *J. Mater. Chem.*, 1992, 2, 1301). We shall present examples covering a range of moderately complex structures, including both organics and organometallics, such as $C_2H_4N_2O_2$, $[(C_5H_5)Fe(C_5H_4)CH_2NMe_3]^+I^-$ and $PhSO_2NH_2$, chosen to illustrate the strategies of structure solution and refinement involved. Our experience suggests that laboratory-based powder diffractometer data will be invaluable in obtaining useful structural information for materials where sufficiently large single crystals cannot be grown.

MS–02.03.03 TEXTURE CORRECTION OF POWDER DATA FOR AB INITIO STRUCTURE DETERMINATION. By R. Peschar* and H. Schenk. Laboratory for Crystallography. University of Amsterdam. The Netherlands and P. Capkova. Faculty of Mathematics and Physics. Charles University. Prague, Czechoslovakia.

The crystal structure determination from powder data by means of Direct Methods relies on the crystallite distribution being random. Deviations caused by the non-uniformity of the distribution can obstruct the solution of the structure by ab initio methods. In general texture correction requires the knowledge of the complete structure and the orientation distribution function, for example via pole-figure measurements. Therefore, an alternative texture correction procedure will be presented, based on a statistical analysis of the deconvoluted intensities, which needs neither a structural model nor pole-figure measurements. The texture correction procedure is valid for disc- or rod-shaped crystallites and requires the use of a specimen spinner to ensure axial symmetry. The only information being necessary, besides the indexed powder pattern, is the contents of the unit cell. Under the above assumptions the texture function P_H can be modelled as a simple exponential form involving a single adjustable parameter G,

$$P_H(\varphi_h) = \exp[-G\sin^2(\varphi_h)]$$

in which the texture direction $\underline{H} = (HKL)$ makes an angle φ_h with a reflection $\underline{h}$ (hkl). Both the texture direction H and the parameter G can be established by means of a least-squares analysis of the integrated intensities. The influence of symmetry-equivalent reflections will be discussed and test results will be presented which demonstrate the potential use of the new method for ab initio structure determination from powder data.

MS–02.03.04 SIRPOW.92: A USEFUL TOOL FOR SOLVING CRYSTAL STRUCTURES FROM POWDER DATA. By A. Altomare, G. Cascarano, C. Giacovazzo*, A. Guagliardi, Istituto di Ricerca per lo Sviluppo di Metodologie Cristallografiche, CNR, c/o Dipartimento Geomineralogico, Universita' di Bari, 70124 Bari, Italy.

A Direct Methods package optimized for powder data is described. It is able in most cases to overcome the difficulties generated by reflections overlapping and, in some degree, by preferred orientation. Among the main characteristics: a) both neutron and X-ray data can be treated; b) care is taken over reflection overlapping, and strategies are designed in order to reduce its effect on the phasing process; c) weak reflections are suitably exploited; d) a procedure for automatic partial refinement of the structure is designed; e) care is taken over the preferred orientation. Several experimental tests have been performed which prove the usefulness of SIRPOW.92.

MS–02.03.05 STRUCTURE SOLUTION FROM POWDER DIFFRACTION DATA - INCORPORATING PRIOR CHEMICAL KNOWLEDGE By W I F David and M A Estermann, ISIS Science Division, Rutherford Appleton Laboratory, Chilton, Didcot, OXON., OX11 0QX, U.K.

Prior chemical knowledge can play a crucial role in structure determination from powder diffraction data. Structure factor data can rarely be extracted from even a high resolution powder diffraction pattern without severe overlap, particularly in the important d-spacing range of 1.0Å to 1.5Å. This usually leads to the failure of conventional crystallographic techniques since atomic resolution has not been obtained. In contrast most powder diffraction patterns are still relatively free of overlap at d-spacings above 2.5Å. This paper focusses on exploiting the combined use of these longer d-spacing reflections with available prior chemical knowledge. A number of algorithms for structure solution are presented that are based upon locating larger structural elements. Examples will be principally drawn from the analysis of silicate framework structures and small organic molecules.