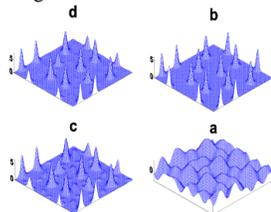


MAXIMUM ENTROPY FOURIER MAPS, A STANDARD FOR CRYSTAL IMAGING

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This talk reviews advances made in Magnetization Density Imaging since 1990. Initially introduced to improve 2D-projections by reducing truncation errors and by taking experimental error bars into account, Maximum Entropy [ME] Fourier Spin density maps are now routinely used to reconstruct full 3D field-induced magnetization densities in crystals. All available data can and should be used, even for 2D projections. The next major improvement was the tackling of acentric structures with a gradual sophistication: as a linear approximation [1994], a mathematical iterative nonlinear process [1997], leading eventually to a modified experimental procedure emphasizing the physical role of the Nuclear-Magnetic interference term over that of the more traditional flipping ratio [2000]. The latest aspect is the crucial part played by adverse non-uniform prior [model] densities towards the retrieval of truly reliable weak magnetic features: the latter should survive the bias towards a plausible but unfavorable model. All those points are embodied in the example shown in the figure below pertaining to the induced magnetization in crystallographically acentric cubic Nd_{3-x}S₄, which may harbor two inequivalent magnetic Nd sites. In a) a generalized conventional Fourier projection along 001 proves to be useless. b) displays our prior density: only Nd sites are magnetic, and they are equivalent c) A ME reconstruction with a uniform prior seems to indicate two magnetically distinct Nd sites and also some [spurious?] extraneous density. d) When the plausible non-uniform prior b) is used: 1) the extraneous density disappears and 2) the magnetic inequivalence of the two Nd sites is confirmed. Finally, some hints about how to compute non-uniform priors will be given.



Keywords: MAGNETIZATION DENSITY MAXIMUM ENTROPY

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SUBTLETIES IN CRYSTAL STRUCTURE SOLUTION FROM POWDER DATA: RANITIDINE HCL

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The paradigm of using known chemical information in support of *ab initio* structure solution from powder data is growing in popularity and productivity. While there have been numerous successes, the overall trustworthiness of solutions from powder data is regarded by many as an open question. One specific issue raised is how closely a result depends on the information built into the search: if one makes an incorrect initial hypothesis, is the error built into the solution, or will an inconsistency come to light? We sought to address that issue with a solution of the structure of the well-known drug, Ranitidine HCl, form II. It is already known from the single crystal structure that the molecule has a degree of conformational disorder. Using our simulated annealing code, PSSP, we were able to get a solution under the assumption of a single conformer. A Rietveld refinement (Monoclinic lattice, $P2_1/n$, 18.808 Å x 12.981 Å x 7.211 Å, $\beta = 95.047^\circ$) had $R_{wp} = 11.12\%$ and $\chi^2 = 10.56$, with some absurd thermal parameters. This would arouse the suspicion of most crystallographers. When we refined from the known (disordered) single crystal structure, we obtained $R_{wp} = 7.41\%$ and $\chi^2 = 4.51$.

Keywords: POWDER SYNCHROTRON PHARMACEUTICAL

X-RAY-NEUTRON EXPERIMENTAL COMPLEMENTARITY FOR MAGNETIC STUDIES

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Modern magnetism is no longer simple; for most of the real cases, we should treat the materials of complicated structure as well as showing the complex phases. Then we challenge the mechanism of the interplay with other freedoms, such as charge and lattice, even super-conductivity. Very precise and detailed experiments with larger energy range as well as larger momentum space are required. This challenge is only achieved by use of both intense neutron source as well as synchrotron X-ray radiations. I emphasize this complementarity by showing our recent experiments on the physics of CMR materials.

Keywords: CHARGE, SPIN AND MOMENTUM DENSITY
MAGNETISATION AND SPIN DENSITIES

USEFULNESS OF X-RAY POWDER DIFFRACTION IN THE PHARMACEUTICAL INDUSTRY

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Most drugs are administered in solid dosage forms, among them a large part are crystalline compounds. In this class of molecules, usually large and complex, frequently flexible, containing both aromatic moieties and hydrogen bonding groups, polymorphism (including solvates and hydrates) is a very common phenomenon. It not uncommon to isolate 10 or more different crystalline phases of such a compound during routine laboratory research activity. Although polymorphs, hydrates and solvates share the molecular structure of the underlying compound, because of their distinct crystalline structures they may display different physical, chemical, physiological properties. The investigation of polymorphism in pharmaceutical compounds is of utmost importance for both regulatory and marketing considerations. When and where available, powder x-ray diffraction (pxrd) is the preferred technique for identification of crystal forms. In many instances, however, pxrd is not feasible, and other techniques, more common in the industrial laboratory, such as ir or dsc, may or must be used. The purpose of this presentation is to show the power of the x-ray powder diffraction technique for analysis of drugs (bulk active ingredients as well as active ingredients within formulations) vs. Spectroscopic techniques, and its usefulness in phase identification, phase quantitation, and detection of phase traces. Examples from day-to-day research tests will be provided.

Keywords: POLYMORPHISM PHARMACEUTICALS
FORMULATIONS X- RAY POWDER DIFFRACTION