structures of square-planar transition-metal complexes. The data in the crystal structures from CSD show mutual slipped-parallel (*offset face-to-face*) orientation between chelate and C_6-aryl rings similar to the slipped-parallel orientation of two benzene rings.

Based on the fact that chelate rings with delocalized π-bonds can form stacking with C_6-aryl rings, one can anticipate that two chelate rings could form mutual stacking interaction. The evidence of chelate-chelate stacking interactions was obtained by analyzing crystal structures of square-planar transition-metal complexes from CSD [3]. The analysis showed that chelate-chelate stacking interactions occur in a large number of the crystal structures of neutral square-planar complexes.

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MS.94.5

Formation and structure of fullerene and cubane based cocrystals

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Fullerenes form a large variety of high symmetry molecular crystals with cubane molecules [1]. The basic material, $C_{60}C_8H_8$ has a face centered cubic crystal structure at ambient condition, with significantly expanded lattice, related to the parent fullerene structure. This cocrystal consists of separated sublattices of rotating and static components. The stabilizing factor is the strong fullerene-cubane attraction, which we explain as a molecular recognition between the convex C_{60} and concave cubane surfaces. Although this strong shape and size recognition of the constituents allows the rotation of fullerenes, it keeps cubane in the equilibrium position and orientation. This unusual structure gives rise to a complex dynamics, called rotor-stator feature, that is different from both the orientationally ordered and the plastic crystals. As a result of this effect, the orientational ordering phase transition of fullerene-cubane takes place at the lowest temperature in all fullerene-based materials [2].

We prepared several member of this new family of heteromolecular crystals: $C_{70}, C_{76}, C_{78}, C_{84}$ [3] and some endohedral fullerenes also form rotor-stator phases with cubane. C_{60} also forms cocrystals with 1,4-disubstituted cubanes. The ball-shaped fullerenes has an fcc structure with cubane, the elongated molecules compose less symmetrical structures. The different size and symmetry of the components slightly modified the rotor-stator properties. The lattice parameter depends on the sizes of fullerenes and cubane and can be predicted accurately.

The materials have rich phase diagrams and unconventional topochemistry. At elevated temperature the unimolecular isomerization of cubane results in a single phase topochemical reaction with the surrounding fullerenes. The product is a random copolymer that is stable up to high temperature.

Here we present the formation and crystal structure of the recently synthethized members of the fullerene-cubane cocrystals and discuss the effect of the molecular geometry on the crystal structure and the rotor-stator dynamics.

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MS.95.1

Automating mail-in data collection at 11-BM at the APS

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The 11-BM powder diffractometer [1] at the Argonne Advanced Photon Source is a high-resolution instrument ($\Delta Q/Q \approx 2\times10^{-4}$) with 12 independent analyzers [2] where a typical dataset is collected in an hour. The beamline is equipped with a robot and cryostream device, allowing completely automated data collection with temperatures of ±200C. The highest priority for the instrument deployment was to offer user access where samples are mailed to the beamline. This was selected, as this service was not widely available and offered the greatest availability to the potential user community.

To allow the high-throughput of the instrument to be accessed by a large number of users with mail-in access, it was necessary to automate not only the operation of the instrument, but all other aspects of the workflow, including: requests for sample mounting kits; filing of safety review forms; programming of the data collection parameters; acknowledging sample receipt; post-collection data reduction; sample storage; dissemination of data to users; sample disposal/return; and query of users for publication information [3]. This is accomplished primarily via web interfaces that offer users additional features, such as tables summarizing samples and datasets. Care is taken to provide privacy for experimental information without demanding user login with password protection. A crucial component of mail-in operation has been adoption of sample bases that regularize sample mounting and where each base has a unique barcode that can be read by the robot.

The sample management system has been sufficiently popular that when on-site data collection software was later deployed, it was requested that on-site users have the option to include their work in this database.

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MS.95.2

Automation and efficiency in the powder structure solution by EXPO package

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The EXPO package [1] is a computing program able to successfully provide the structure solution by starting from minimal information: the experimental powder diffraction pattern and the chemical formula of the compound to investigate. EXPO can be used for solving organic, inorganic, metalorganic structures for a large variety of applications. Automation and efficiency are suitably combined in EXPO for performing all the steps of the solution process: indexation, space group determination, intensity extraction, structure solution, model optimization, Rietveld refinement.

Automation. Default strategies, identified as the most effective, are selected for carrying out the solution process automatically and quickly. They may fail when the experimental data resolution is bad and/or the structure complexity is remarkable. In these cases, EXPO can promptly switch to appropriate strategies by profiting by a user friendly graphical interface.

Efficiency. Innovative theories and computing procedures aiming at making straightforward all the steps of the solution process are integrated in EXPO and widely tested. EXPO is continuously updated and optimized in terms of both computing efficiency and graphical performances.

In EXPO the two stages based *ab initio* approach (in the first stage the intensities are extracted; in the second, the reflections are phased by Direct Methods) [2] is the automatic choice. The model provided by Direct Methods is usually partial and approximate because of well known unavoidable problems in powder diffraction (overlapping, background, preferred orientation). It can be optimized by default [3], [4] and/or non-default strategies [5], [6] which are able to reduce the errors depending on the limited experimental resolution.

A very recent study has regarded a new and more effective figure of merit [7], alternative to the classical one. It is able to pick up the phasing trial corresponding to the best solution among several feasible ones.

Non ab initio method, requesting the knowledge of the expected molecular geometry and based on Simulated Annealing technique [8, 9], can optionally be attempted particularly in case of solution of organic compounds.

Examples of successful automatic runs by EXPO will be discussed in addition to available special strategies.

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MS.95.3

Pushing the limits of powder charge flipping

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In 2004 Oszlányi and Sütö introduced a new way of determining crystal structures from single-crystal diffraction data that they termed charge flipping [1]. The algorithm is an elegant one, based on a very simple perturbation (charge flipping) of electron-density maps that are generated during the structure solution process. Initially, this charge-flipping algorithm was viewed as a curiosity, but in a surprisingly short period of time the approach found application in areas where traditional methods flounder. One of these was X-ray powder diffraction (XPD).

The original algorithm was adapted to accommodate powder diffraction data by two different groups in 2006 [2] and 2007 [3]. The latter includes a second perturbation of the electron-density map based on histogram matching [4] prior to the repartitioning of the intensities of overlapping reflections, and has been implemented in the program *Superflip* [5]. Although diverse structures have been solved automatically with this program, its limits have not yet been reached. By including information from electron microscopy (EM) experiments in different ways, three particularly complex zeolite structures [6,7,8] could be solved. At least part of the algorithm's strength lies in the fact that it operates in both real and reciprocal space, so manipulation in both is possible.

In the first of the zeolite structures, starting phases for the charge-flipping algorithm were generated using an approximate (but incorrect) model derived from several high-resolution transmission electron microscopy (HRTEM) images. In the second case, a structure envelope [9] was generated from a single lower resolution HRTEM image and imposed in real space to enforce the channel system. In the third case, reflection intensities from selected area electron diffraction patterns were used to pre-partition the intensities of reflections overlapping in the powder diffraction pattern.

It has also been shown that precession electron diffraction (PED) data can be used to improve the quality of the intensities extracted from a powder diffraction pattern (by identifying and eliminating weaker reflections) and to generate some better starting phases (by applying charge flipping to 2-dimensional PED data) [10]. In light of the improvements arising from the use of 2-dimensional EM data to supplement the powder diffraction data, an alternative approach using 2-dimensional subsets of the XPD data has recently been devised [11].

Further automation incorporating some of these developments into the software can be easily envisioned.

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MS.95.4

Algorithm and performance of a new powder indexing software *Conograph*

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