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Robotic Adventures in Crystallisation Space
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An automated parallel crystallisation screen has been applied to the systematic recrystallisation of the thiazide diuretic hydrochlorothiazide. The work is carried out as part of the collaborative project "Control and Prediction of the Organic Solid-State" funded under the auspices of the UK Research Councils Basic Technology Programme. The screening approach provides accurate control of key crystallisation parameters, namely: solvent identity; temperature; agitation and rate of evaporation. Polycrystalline samples are characterised using a multi-sample X-ray powder diffractometer equipped with foil transmission geometry, CuKα1 radiation and a linear PSD [1]. The instrument is highly effective where there is a requirement to analyse 20 – 30 recrystallised samples per day, with an emphasis on obtaining the high-quality data that are important in pattern recognition (using e.g. PolySNAP [2]) and imperative in indexing. The results of multivariate analysis of the solvent properties and physical forms produced in the screen is presented along with the crystal structures of the novel forms with a view to identifying the factors underlying the formation of polymorphs and solvates of hydrochlorothiazide.

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Keywords: parallel crystallisation, polymorph screening, powder X-ray diffraction

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Crystal Structure of a Polymorph of Carnidazole from Synchrotron X-ray Powder Diffraction
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The crystal structure of a polymorphic form of carnidazole, a nitroheterocyclic compound active against both anaerobic protozoa and bacteria, has been determined using synchrotron powder diffraction. The simulated annealing approach, as implemented in the program FOX [1], was used to obtain the initial model. The model was refined with the Rietveld refinement program Fullprof [2]. This monoclinic polymorph crystallizes in $P2_1/n$ space group with a = 13.907(3), b = 8.091(2), c = 10.643(2), β = 110.831(5), Z = 4. The imidazole ring is planar. The molecules are held in the crystal forming two infinite zig-zag chains along [010] *via* hydrogen bonds of the type N-H…N. A structural comparison with the previously reported polymorph and the monohydrate forms of this drug is presented.

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Keywords: carnidazole, powder diffraction, polymorphism

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High-throughput Polymorph Screen of Cimetidine and Clarification of its Nomenclature
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Polymorphism evaluation as a part of preformulation studies of a candidate drug is a critical step in the drug development process. The progress of high-throughput experimentation in recent years has led to a significant reduction of the amount of time and the quantity of the active pharmaceutical ingredient needed to perform these studies. In order to investigate the effectiveness and efficiency of a rational high-throughput polymorph screen (HTPS) the compound cimetidine was used as a case study for the formation of its polymorphic forms under different crystallization conditions.

The characterization of the various forms that occurred after crystallization was realized by means of X-ray powder diffraction experiments, differential scanning calorimetry and thermogravimetric analysis. As it turned out the HTPS was an effective means for obtaining all of the commonly reported solid-state forms of cimetidine (A, B, C, D, M1) and led to the formation of a new solid form (F). An analysis of the results will be given and the influence of the various crystallization conditions on the formation of the various solid forms will be discussed.

In order to assess the obtained results a literature investigation was carried out revealing a large confusion with respect to the nomenclature of the various cimetidine forms. A general and unifying nomenclature is proposed and compared with the older literature.
Keywords: high-throughput polymorph screen, rational design, cimetidine

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***In situ* X-ray Diffraction, DSC and Raman Spectroscopy Thermal Investigation of Chlorpropamide**
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Thermal analysis techniques, especially DSC, provide very useful information to identify and characterize APIs. What is more, DSC is widely accepted as a thermo-analytical tool for the study of phase transitions in different type of compounds. Unfortunately, this method does not reveal the identity of the transforming phases and their identification is often difficult without the aid of techniques that give information regarding structures and reactions. In this respect, X-ray diffraction and Raman spectroscopy are reliable techniques for phase identification. Thus, these methods are considered complementary to each other and the development of the instruments combining some of these techniques is a very powerful methodology with can have many applications for the characterization of pharmaceutical solids. For all these reasons, the aim of the present work is to apply Simultaneous WAXD-DSC, using Synchrotron Radiation, and temperature dependent Raman spectroscopy measurements to the study of the model drug chlorpropamide, which presents several polymorphs and phase transitions.
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Keywords: active pharmaceutical ingredients, *ab-inito* calculations, polymorphism

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Quantification of (Pseudo)Polymorphic Mixtures Using Full Pattern Analysis of X-ray Powder Diffraction Data
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The solid-state structure of drug substances influences their bioavailability, chemical stability, formulation properties etc. For that reason, analytical methods for identification and quantification of polymorphs, solvates, and amorphous forms are required. The analysis of X-ray powder diffraction data using a full-pattern analysis is a quantitative method that is both robust and precise. The ratio of