distribution of conformational states represented in the PDB. A systematic review of multiple deposits shows that a single protein is rarely represented by a single structural conformer. This result sheds light on the first link and demands the reformulation of the protein-folding problem. A vast majority of proteins shows significant number of distinct conformational states with, sometimes large, structural divergence (up to ~24Å). The results suggest that every single protein evolved according to its own optimization principles combining different proportions of rigid (solid-like) and mobile (liquid-like) structural elements. The results suggest further that the optimization process that produced the particular combination of those elements is intricately connected with the function of individual proteins. Therefore, the structural description of the protein, besides the folding class (the architecture represented by the SCOP database), should include the natural structural divergence (width of the distribution) as two main attributes. Additionally, our analysis suggested the principles of functional evolution by use of the Dual Personality sequences (sequences with incomplete representation in the atom records that have distinctive sequence features from regularly folded and intrinsically disordered fragments).

Keywords: redundant structure database, distribution of conformational states, protein folding

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Analysis of the organic X-ray powder diffraction database and its use with pharmaceutical substances

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The crystals of small molecule pharmaceutical substances are usually of low symmetry, often have hydrogen-bonding-induced polymorphs [1], and frequently exhibit anisotropic crystal habits leading to preferred orientation in X-ray powder diffraction (XRPD) experiments. Statistical studies and cluster analyses have been used to show the prevalence of polymorphs and low symmetry space groups for these materials. Such statistical analyses can be performed using permutations of 40 different property and data searches with the PDF-4/Organics database (PDF-4). The XRPD patterns for these materials can exhibit overlapping peaks over small ranges of two theta angles, peak asymmetry at low angles, and preferred orientation. However, the proper choice of diffractometer and specimen configuration can minimize the two latter effects. Examples of XRPD data from experiments with thermodynamically stable polymorphs [2] will be given to illustrate this optimization. Modern crystallographic software can index XRPD patterns, determine unit cell dimensions and assign space group symmetry. Using this information and the PDF-4 searches lead to the model selection from the Cambridge Structural Database (CSD), followed by Rietveld refinement to verify both crystallographic parameters and indexing assignments of the experimental XRPD pattern. The experimental XRPD pattern then becomes a powerful reference for quality control measurements, quantitative analysis and polymorph identification. Systematic analysis examples of data from XRPD experiments for active pharmaceutical ingredients will be given.
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Keywords: X-ray powder diffraction, polymorphs, preferred orientation

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Studying conformational preferences for mechanistic purposes: Using database mining and computation

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It has long been known that phosphine and phosphite ligands can adopt various conformations depending on coordination environment. Containing over 400,000 structures more than half of which are metal complexes, the Cambridge Structural database (CSD)[1] has an extensive amount of information regarding ligands, the attached metal centre and the coordination environment of the complex. By combining the information from database mining with computational (DFT) studies it is possible to explore the response of ligands to different coordination environments. This enables us to have a better understanding of conformational behaviour when exploring mechanistic pathways, which often involve coordination changes. The chosen systems, tribenzylphosphine $P(CH_2Ph)_3$ and triphenylphosphite $P(OPh)_3$, are part of a synthetically relevant series of phosphorus ligands for catalytic studies. Although DFT is known to give reasonable agreement with molecular structure,[2] these systems are far too large to be studied by DFT conformational searching. The combination of DFT studies with database mining results has accurately predicted the conformational preference and lowest energy profile for these ligands across a series of coordination environments, and has been utilised in mechanistic studies undertaken in our group. Further analysis of the data retrieved from the CSD has highlighted conformational interconversion pathways. These have been explored and structures along the pathways have been used to predict transition states between conformers. This furthers our understanding of ligand conformation during reactions.
1. F. H. Allen, Acta Cryst., B58, 380-388, 2002
2. A. Muller, J. Organometallic Chem., 691, 5782, 2006

Keywords: conformational analysis, DFT, Phosphorus ligand chemistry

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Ligand substructure validation in macromolecular crystallography using the CSD

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Structural studies of protein-ligand complexes have become essential to modern drug discovery processes. The quality of results delivered by X-ray crystallography has a direct effect on downstream computational chemistry studies, therefore thorough validation - especially that of small molecule substructures - should be considered as a key step in structure determination. However, the final geometry of a ligand is influenced primarily by the restraints applied to it during refinement, as a consequence of the typical resolution range of macromolecular crystal structures. Therefore critical assessment of the initial geometrical parameters should carry equal weight, particularly in a high throughput environment. Validation of chemical structures in the context of macromolecular crystallography

can take several directions including comparison of the actual geometry to either theoretical models or experimental structures from the PDB or small molecular databases. In this contribution we will present integration of search and validation tools from the Cambridge Crystallographic Data Centre, in particular Mogul, into an automated structure solution and refinement workflow. Apart from a post-refinement assessment of structure quality, we explore the use of Mogul to compare programs applied to generate ligand parameter dictionaries for crystallographic refinement. Finally, trends in the quality of ligand geometry as function of structure determination parameters will be discussed based on kinase structures from the PDB as well as those solved in-house. Systematic errors in ligand structures will be highlighted along with potential pitfalls of this validation approach.

Keywords: structure validation, statistical analysis CSD PDB, kinases

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Nucleation of heat storage materials - search algorithms for similarities of crystal surfaces

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The demand for e・ciant use of energy leads to an increasing interest in energy storage. For the storage of thermal energy latent heat storage materials are most convenient. These materials use a phase transition solid - liquid in order to store heat. Because of large melting enthalpies and low melting points salt hydrates are of particular relevance for the storage of heat at moderate temperatures. Latent heat storage materials based on salt hydrates show a strong undercooling of the melt. This inhibits the technical development of latent heat storage as an economic useful product. The controled release of the stored energy is possible by triggering the crystallisation using nucleators. Heterogenous nucleators are found empirically or by comparing geometric crystal properties like lattice parameters. The disadvantage of these methods is, that the nucleating agents known for di・erant materials so far are unreliable. Heterogeneous nucleation is expected if the melt gets in contact with epitactic and che-mical similar surfaces. Search algorithms for similar crystal surfaces were developed in order to improve the developement of nucleators for latent heat storage materials and to gain a de-eper insight to the mechanism of nucleation. This approach provides an extension of existing search routines in crystal structure databases and enables more specific search results. The program ATBEL performs a morphology prediction based on the BFDH-method. For all predicted faces ATBEL creates entries in two di・erant databases. These database are used by the program EPITAX for the search of lattice match and chemical similarity of crystal surfaces. New algorithms for automatic handling of crystal structure data are introduced.

Keywords: nucleation, heat storage, morphology

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Hydration of activated belite cements studied by synchrotron X-ray powder diffraction

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In spite of its universal use in contemporary construction, ordinary Portland cement is one of the most environmentally contentious materials. On average, for every tone of cement produced, 0.97 tons of CO_2 are released into the atmosphere. So, cement industry contributes around 6% of all CO_2 anthropogenic emissions. Hence, cement chemists are making great efforts to find ways to reduce the environmental impact of the cement-production process. Belite cements may reduce 10% de CO_2 emissions but belite reactivity with water is slow and thus these cements develop low mechanical strengths at early stages. The reactivity of these materials may be increased by two complementary ways: i) stabilize high temperature belite polymorphs and ii) produce calcium sulfoaluminate (CSA) belite cements. CSA clinkers contain $Ca_4Al_6O_{12}SO_4$, which reacts rapidly with water forming ettringite, AFt or $Ca_6Al_2(SO_4)_3(OH)_{12.26}H_2O$, and enhancing development of early age mechanical strengths. CSA clinker manufacture may reduce CO_2 emissions up to 35%. Here, we report an in-situ synchrotron powder diffraction hydration study of these cements. Both alkaline oxides activated belite and CSA cements have been analysed. The patterns were collected in transmission in BM08 beamline of ESRF using the translating image-plate detector. This methodology minimise powder averaging errors which are critical for obtaining accurate analyses. All patterns have been treated by the Rietveld method in order to extract the quantitative phase contents. The water/cement weight ratio was kept fixed to 0.5 and the gypsum role has been investigated by adding different amounts. The starting crystalline phase assemblage and the evolution of the hydrate phases will be reported and related to the calorimetric studies.

Keywords: cement hydration, synchrotron powder diffraction, quantitative Rietveld cement phase analysis

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Bath quantitative XRD control at Russia aluminum smelters

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The electrolysis cell used in the production of aluminum is a dynamic system. The concentration of main components in the electrolyte must be rigidly controlled in order to maintain optimum conditions during the production process. Key parameters for control include cryolite ratio (CR) or bath ratio (NaF/AlF_3) and additions of calcium, magnesium or lithium fluorides The bath analysis must be done during about 2 to 3 minutes because there are often several hundred or even thousands of cells to measure. The XRD quantitative phase