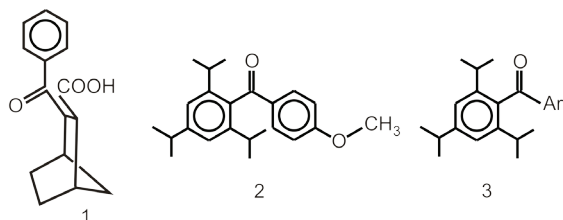


In this poster the results of monitoring photo-induced structural changes in crystals of three compounds will be presented:



Crystals of compounds **1** and **2** are photochemically inert at ambient temperature, however undergo a photo-induced physical process. This process is partially reversible with time for **1**, however the reverse path is different (Fig. 1).

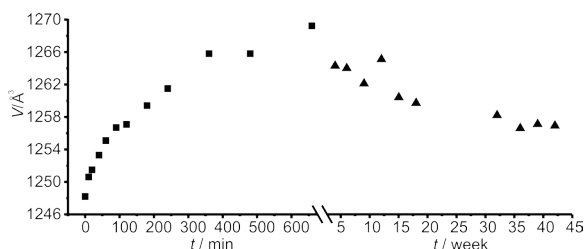


Fig. 1. Variation in the cell volume with time of crystal irradiation and time for the reverse process.

Compound **3** is photosensitive, however contrary to compounds **1** and **2**, it undergoes the Yang photocyclization. There are statistically significant differences between crystal structures determined for different stages of the photoreaction, *i.e.* characterized by different amounts of reactant (and product) molecules. The variations in the cell constants, geometry of the reaction centre and orientation of molecules during the studied reaction were observed and analyzed.

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**Keywords:** structural change; photochemistry; physics of solids

#### FA4-MS05-O4

**Combining X-ray Diffraction and Spectroscopy to Study (Photo)dynamic Processes.** Panče Naumov<sup>a,b</sup>.  
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Techniques which have been traditionally employed to study photoinduced, thermally induced or other dynamic processes are electronic (absorption and emission UV-vis) spectroscopy, vibrational (IR and Raman) and NMR spectroscopies, which are now readily available with commercial experimental setups in steady-state or time-resolved mode. While the spectroscopic techniques are an excellent approach to deduce details on the energy profiles, they provide only limited and, occasionally, ambiguous information on the spatial atomic arrangement and the geometrical changes related to dynamic processes. On the other hand, the X-ray diffraction provides precise information on molecular geometry, but it is not informative about the potential energy profile and the reaction kinetics. In practice, because of the different time-scales between the spectroscopic and X-ray diffraction methods, the information obtained by spectroscopy can be very useful to support diffraction data, for at least the following reasons:

- Spectroscopic data can be correlated to structural data to establish spectra-structure correlations;
- Spectroscopic data can be used to supplement X-ray results in clearing out details within the "grey zones" of the X-ray crystallography (e.g. hydrogen positions, difficult cases of disorder, crystalline-to-amorphous phase transitions, etc.);
- Spectroscopic data can be employed to check or even to correct inaccurate X-ray diffraction data.

In this presentation, the importance of using spectroscopic data for structure elucidation will be illustrated with several examples from our laboratory and from other authors. The results are of broad significance for the fields of structural chemistry and materials science because they demonstrate that the combination of spectroscopic and diffraction methods can provide a more complete and accurate chemical information than either of these methods alone. The development of single crystal and powder X-ray photodiffraction (photocrystallography), as a complementary technique to the spectroscopy and a very convenient analytical tool for direct study of dynamic solid-state phenomena, will be also elaborated.

**Keywords:** reactive intermediates; solid-state photochemistry; spectroscopy and molecular structure

#### FA4-MS05-O5

**Structural Similarity and Reoccurring Hydrogen Bonding Patterns in Acylanilides.** Susanne Huth<sup>a</sup>, Michael Hursthouse<sup>a</sup>, Terry Threlfall<sup>a</sup>.  
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Small molecule X-ray crystallography has become an important tool for the detailed investigation of the solid state with the ultimate aim to understand the (supra)molecular assemblies in crystal structures. The systematic study of crystal packing patterns together with the application of

theoretical calculations can provide an improved insight into the solid state assembly, providing feedback for design and prediction procedures. The identification of common features is key to this process and should not only concentrate on assemblies attributable to strong intermolecular interactions but include patterns caused by medium to weak interactions. It is the latter category that lacks thorough understanding and it is here where 'conventional' graphical programs are of limited use. Libraries of closely related compounds comprise suitable systems for such an analysis since the core structure and its interactions remain the same so that variations in the crystal packing can be directly associated with the effects of the substituents. In recent years, our group has produced several comparative studies of large sets of closely related compounds [1-4] demonstrating the feasibility of this 'structural systematics' method. In continuation of this theme, the project presented here is concerned with mono-substituted acylanilides and their crystallisation and packing preferences. Harvesting the CSD yielded a fragmentary compound library of variable data quality hence we set out to synthesise an expansive array of closely related molecules: To date over 400 acylanilides have been prepared yielding approximately 300 crystalline samples for single crystal XRD, and more than 200 data sets have been collected, corresponding to ca. 160 actual structure determinations. Our initial focus lies on para-substituted acetanilides. These compounds comprise a pharmaceutically important group of molecules; in particular p-hydroxy-acetanilide (aka paracetamol) has attracted exceptional interest. A thorough understanding of substituent effects on crystal packing is hence highly desirable with respect to the utilisability of these compounds. The program XPac [5] is an excellent tool for the search of common structural patterns in solid state assemblies and was thus used for the systematic cross-comparison of the crystal structures of some subsets of mono-substituted acylanilides. Supplementing this information, a variety of calculations has been performed (a) to establish lattice energies using semi-classical density sum (OPiX) methods and (b) to probe the potential energy surface for preferred conformations in correlation with experimental observations. The results of this systematic study will be presented and discussed.

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**Keywords: high throughput crystallography; structural similarity; theoretical calculations**