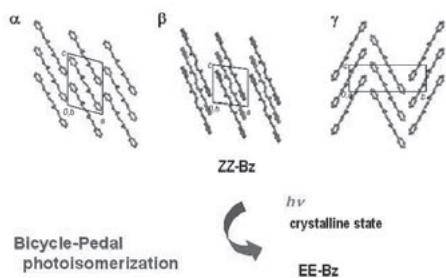


Organic reactions performed in the solid state have many intrinsic features for synthetic and materials chemistry because of the extremely high selectivity of the reactions. A limited number of the *E-Z* isomerizations of olefins in the crystalline state have been reported because of the difficulty in the inevitable change in the size and shape of the space occupied by the substituents of a double bond. Presently, we have revealed a change in the molecule structure of muconic esters, by the direct observation of single crystal structures during photoisomerization in the solid state. Benzyl (*Z,Z*)-muconate (*ZZ*-Bz) afford three polymorphs. During the solid-state photoisomerization of these polymorphs to the corresponding (*E,E*)-muconate (*EE*-Bz), it was revealed that the isomerization occurs via a topochemical reaction process according to a bicycle-pedal model and is finally accompanied by a phase transition to a stable crystal structure. After photoirradiation of powdered *ZZ*-Bz, the conversion to *EE*-Bz depended on the structure of the polymorphs. A difference in the reactivity is discussed on the basis of the molecular structures and the void space in polymorphic crystals.



Keywords: cis-trans isomerization, polymorphs, single crystals

### P08.09.75

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#### Defects in single crystalline Ge-doped silicon revealed by annealing under high hydrostatic pressure

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Germanium - doped Czochralski grown silicon (Cz-Si:Ge) with cGe exceeding 1 at.% has been rediscovered because Ge introduces controlled strain into layered structures, with carrier mobility competitive to the case of III-V semiconductors. Annealing of Cz-Si:Ge with low Ge content under enhanced hydrostatic pressure (HP) at high temperature (HT) results in specific structural transformations, of interest for integrated circuits technology. Defects in Cz-Si:Ge, with cGe = 1.4x10<sup>-3</sup> and 1.4-7.6 at.%, containing also oxygen in a concentration up to 1x10<sup>18</sup> cm<sup>-3</sup>, and processed at up to 1400 K under HP up to 1.2 GPa were investigated by synchrotron topography, high resolution X-ray diffractometry, Infrared Spectroscopy, photoluminescence (PL) and related methods. Topography of Cz-Si:Ge reveals uniform structure in the case of cGe = 1.4x10<sup>-3</sup> at.% while Ge segregation is detected for Cz-Si:Ge with cGe exceeding 1.4 at.%. The presence of dislocations at 10(3) cm<sup>-2</sup> density has been stated after processing. X-Ray diffuse scattering is most pronounced for the samples with cGe = 2.6 at.%; its intensity decreases with HT (HP). Annealing at 1270 K for 5 h resulted in the increased lattice parameter (a), e.g. for Cz-Si:Ge with cGe =1.4

at.%, a=0.5440574 nm, 0.5441908 nm if annealed under 10(5) Pa but 0.5441113 nm after processing under 1.1 GPa. The PL intensity at 1.07 eV, related to the presence of electron-hole droplets, decreased after processing, evidencing out-annealing of point-like defects under HP. The effect of HT - HP on Cz-Si:Ge is related, among others factors, to stress-induced activation of Ge clusters promoting precipitation of interstitial oxygen. On the other hand, the treatment under HP results in the improved overall homogeneity of Cz-Si:Ge.

Keywords: microstructure, Si-Ge, annealing

### P08.09.76

*Acta Cryst.* (2008). A64, C441

#### Structure of synthesized nano-sized perovskite oxide La<sub>1-x</sub>Ce<sub>x</sub>CoO<sub>3</sub>

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Nanosized La<sub>1-x</sub>Ce<sub>x</sub>CoO<sub>3</sub> (x<0.1) cobaltide oxide powder with perovskite structure was synthesized at different calcination temperatures using the amorphous heteronuclear complex La<sub>1-x</sub>Ce<sub>x</sub>Co(DTPA)6H<sub>2</sub>O (x=0.1) as precursor. The effects of the calcination temperature was examined by XRD, TEM and BET methods. The crystal structure of perovskite type La<sub>1-x</sub>Ce<sub>x</sub>CoO<sub>3</sub> (x<0.1) was refined by X-ray Rietveld analysis for three different calcination temperatures, and its correlation with catalytic activities is discussed. The results indicated that the La<sub>1-x</sub>Ce<sub>x</sub>CoO<sub>3</sub> (x<0.1) cobaltide oxide powder is not of pure perovskite type and with increasing calcination temperature from T = 600 °C to T = 900 °C the grain size of La<sub>1-x</sub>Ce<sub>x</sub>CoO<sub>3</sub> (x<0.1) increases. The specific surface area varies from 4.347 to 9.016 m<sup>2</sup>/g. The results reveal that the calcination temperature plays a key role in controlling the morphology, crystallization and phase compositions of La<sub>1-x</sub>Ce<sub>x</sub>CoO<sub>3</sub> (x<0.1) perovskite type oxide. After being calcined at T = 800 °C for 3 h, the La<sub>1-x</sub>Ce<sub>x</sub>CoO<sub>3</sub> (x<0.1) catalyst exhibits the highest catalytic activity for carbon monoxide oxidation.

Keywords: perovskite oxide, X-ray powder diffraction, catalytic combustion

### P08.09.77

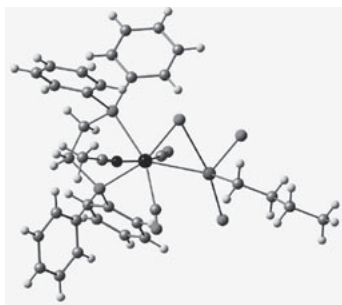
*Acta Cryst.* (2008). A64, C441-442

#### New insights into the Mo-Sn bond in binuclear complexes

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[Mo(CO)<sub>3</sub>(NCMe)<sub>2</sub>(SnBuCl<sub>2</sub>)Cl], **1**, reacted with phosphanes (Ph<sub>2</sub>PCH<sub>2</sub>CH<sub>2</sub>PPh<sub>2</sub>, dppe; Ph<sub>2</sub>PCH<sub>2</sub>PPh<sub>2</sub>, dppm; Ph<sub>2</sub>PNHPPPh<sub>2</sub>, dppa) to afford complexes [Mo(CO)<sub>2</sub>(NCMe)(P-P)(SnBuCl<sub>2</sub>)Cl] (L=dppe **2**, dppm **3**, dppa **4**). When these complexes reacted with [NH<sub>4</sub>][S<sub>2</sub>P(OEt)<sub>2</sub>], the nitrile and the chloride ligands were replaced by the S<sub>2</sub>P(OEt)<sub>2</sub> anionic ligand, yielding [Mo(CO)<sub>2</sub>(S<sub>2</sub>P(OEt)<sub>2</sub>)(P-P)(SnBuCl<sub>2</sub>)] (L=dppe **5**, dppm **6**, dppa **7**). Complexes **5**, **6**, and **7** could also be obtained from a 'one pot' reaction starting from Mo(CO)<sub>6</sub>, SnBuCl<sub>3</sub>, [NH<sub>4</sub>][S<sub>2</sub>P(OEt)<sub>2</sub>] and the appropriate phosphane. Similar reactions took place when SnPhCl<sub>2</sub> was used instead of SnBuCl<sub>2</sub> in the starting material, leading to the three complexes [Mo(CO)<sub>2</sub>(S<sub>2</sub>P(OEt)<sub>2</sub>)(P-P)(SnPhCl<sub>2</sub>)] (L=dppe **8**, dppm **9**, dppa **10**). The structure of complexes **2**, **3**, **5**, **6**, **8**, **9**, **10** were determined using single crystal X-ray diffraction. In these complexes, Mo(II) is heptacoordinate, with a Mo-Sn bond. The tin atom has a tetrahedral environment in all the complexes, except in [Mo(CO)<sub>2</sub>(NCMe)(dppe)(SnBuCl<sub>2</sub>)Cl] (**2**, figure), where the Cl ligand bridges Mo and Sn. DFT (Density Functional Theory) calculations were performed in order to discuss the nature of the Mo-Sn bond.



Keywords: Mo(II) complexes, Sn derivatives, crystal structures

## P08.12.78

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### Guest-dependent high-pressure behavior in a nanoporous metal-organic framework material

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The structural and chemical versatility of metal-organic framework (MOF) materials underlie important technological, industrial, and environmental applications, including in strategic gas storage and separation. The extensive structural complexities now well-documented for these systems are likely to be associated with unprecedented pressure-induced behavior compared with the traditional solid state materials more commonly explored under high pressure conditions. Furthermore, the typically open (low density, often porous) nature of MOF materials is likely to induce such phenomena at more moderated pressures, such as may be routinely encountered in practical applications. For example, densification of powdered nanoporous MOF materials to optimize volumetric gas storage capacity through sample compression (up to several GPa) may distort the framework and pore structure, and accordingly may significantly alter the guest sorption properties (e.g., selectivity). Accordingly, understanding the impact of pressure on MOF systems is not only of considerable fundamental interest, but is relevant to the applications for which MOF materials are being considered. Here we report the structural investigation of a nanoporous MOF material at high pressures within a diamond anvil cell using synchrotron-based X-ray powder diffraction.

Keywords: high-pressure X-ray diffraction, microporous materials, coordination polymers

## P08.12.79

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### Femtosecond to hours structural dynamics of self-assembling systems

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In the following we will present time-resolved small angle x-ray scattering studies on photoactive, self-organising systems like photo-sensitized liquid crystal systems and other reference systems of biological relevance. In the first system, we have studied the kinetics and dynamics of the photo-induced phase transition from a liquid crystal to a microemulsion system and vice versa. In the second system, we mainly concentrate on the structural response function upon photo-irradiation. In the millisecond time domain, the structural dynamics studies have been performed at home-based time-resolved photo small angle X-ray diffraction apparatus [1, 2], the nanosecond and picosecond time scale have been explored employing plasma based home laboratory sources and synchrotron based x-ray scattering sources (ID09B beamline / ESRF, France) [3] and the femtosecond time domain has been investigated employing the FLASH free electron laser facility at DESY, Germany. We will give an overview over the varying time-scales underlying the photo-activated structural transformation processes. We will also discuss the various x-ray sources and compare the different experimental approaches.

[1] W. Quevedo, M. Petri, S. Techert, Z. Krist. (Time-Resolved Photocrystallography) 223 (04-05), 322-328 (2008).

[2] W. Quevedo, M. Petri, G. Busse, S. Techert, J. Chem. Phys., in press (2008).

[3] submitted.

Keywords: time-resolved x-ray scattering, ultrafast processes, structural dynamics

## P08.13.80

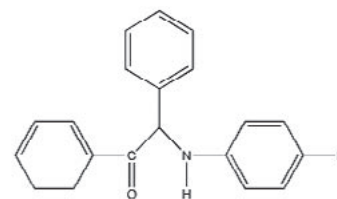
*Acta Cryst.* (2008). A64, C442-443

### Dimers or not dimers in 1-arylanilinoethanones

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Few 1-arylaminoethanone derivatives have been structurally characterised up to date. However, these derivatives are important in synthesis and some of them possess interesting charge-transfer properties while also displaying rather unusual supramolecular arrangements. In the present study we present results from our structural characterisation of four derivatives, with substitutions (Cl-, MeO-, H-, Me-) in the 4-anilino position of the 1-arylaminoethanones. The Cl- and Me- derivatives form distant dimers while MeO- and H- derivatives are found as monomers. The formation of distant dimers has been found to be related to the planarity of the N atom in these structures, in agreement with the formation of similar dimers



R = Cl, MeO, H, Me