

MS14-P7 High pressure X-ray single-crystal and powder diffraction of SF₆ up to 14 GPa. Nadine Rademacher, Lkhamsuren Bayarjargal, Wolfgang Morgenroth, Alexandra Friedrich, Björn Winkler, *Institute of Geosciences, Goethe University, Frankfurt am Main, Germany*
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It is well known that at low temperatures the molecular crystal SF₆ exists in an orientationally disordered body-centered cubic phase (from 90 – 230 K) and an ordered monoclinic phase (below 90 K).^[1] The high-pressure behaviour has so far been only investigated by Raman spectroscopy with pressures up to 10 GPa.^[2] Sasaki *et al.* propose a phase transition at 0.25 GPa from liquid SF₆ to the so-called solid I phase and a second phase transition at 1.8 GPa to the solid II phase. Moreover the assumption is made that the solid I phase crystallizes in the bcc and the solid II phase in the monoclinic structure.

This study presents an in-situ crystal-structure determination of SF₆ in diamond anvil cells (DAC) up to 14 GPa. In order to ensure quasi-hydrostatic conditions, a mixture of 20 vol% SF₆ in helium was loaded into the DAC. During compression of the gas mixture, SF₆ separated from the He at around 0.5 GPa. Crystal growth was observed at 2 GPa and after a pressure increase to 4 GPa SF₆ single crystals were grown using an external heating set-up.^[3] In-situ Raman measurements show the typical SF₆ modes and indicate a phase transition between 1.6 and 2.2 GPa consistent with earlier results.^[2] X-ray diffraction experiments have been performed at the Extreme Conditions Beamline P02.2 at PETRA III.^[4] Single-crystal and powder data were measured using 43 keV radiation at four different pressure points: 1.5 GPa, 1.9 GPa, 4 GPa and 14 GPa. At 1.5 and 1.9 GPa the structure was determined to be the proposed orientationally disordered body-centered cubic and at 4 GPa SF₆ crystallizes in the monoclinic structure. After further compression to 14 GPa only powder rings were observed and the monoclinic phase is still stable.

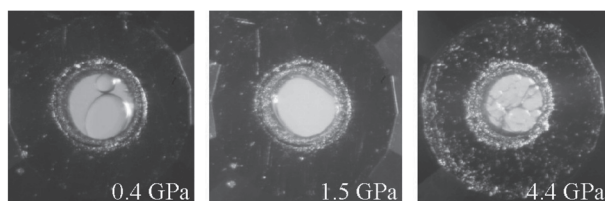


Fig. 1: Left: Phase separation of SF₆ and He in the DAC at 0.4 GPa. Middle: Solid SF₆ at 1.5 GPa. Right: SF₆ single crystals at 4.4 GPa.

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MS14-P8 High resolution electron microscopy study of the early stages of calcium sulfate formation. A.E.S. Van Driessche,^a L.G. Benning,^b J.D. Rodriguez-Blanco,^b M. Ossorio,^a P. Bots,^b J.M. García-Ruiz^a ^a*Laboratorio de Estudios Cristalográficos, IACT, CSIC-U. Granada, Spain,* ^b*School of Earth and Environment, University of Leeds, UK.*
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We used time resolved sample quenching, high-resolution electron microscopy and selective area diffraction to study the precipitation of calcium sulfates from solution. These materials, such as gypsum, play important roles in natural [1] and industrial processes [2] but their precipitation mechanisms remain largely unexplored. Based on our experimental results, we demonstrate that gypsum forms at room temperature via a three stage process: (a) homogeneous precipitation of nanocrystalline hemihydrated calcium sulfate, i.e. bassanite, below its predicted solubility (b) self-assembly of bassanite into elongated c-axis co-oriented aggregates and (c) transformation into dihydrate calcium sulfate, i.e. gypsum [3]. These findings highlight that a stable precursor phase can be formed below its bulk solubility and that in the CaSO₄ system the self-assembly of nanoparticles plays a crucial role. Understanding why bassanite forms prior to gypsum can lead to more efficient anti-scaling strategies for water desalination and help explain the persistence of CaSO₄ phases at low water activities on Mars [4]. Additionally, a different reaction pathway could be responsible for the formation of the massive gypsum deposits in terrestrial evaporitic environments [1] or giant gypsum crystals formed in caves [5,6].

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