ESRF	**Experiment title:** Role of high-pressure clathrates in chemical evolution of icy satellites oceans	**Experiment number**: ES-1218
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Report:

The goal of this proposal was to investigate clathrate hydrates of biologically relevant small organic molecules at high-pressure-low-temperature conditions, in order to illuminate their role in chemical evolution of icy satellites subsurface oceans.

For this purpose, two solutions have been prepared: dimethylamine (DMA)-H_2O (40wt.%, **Experiment 1**) and methylamine (EA)-H_2O (40 wt.%, **Experiment 2**). Membrane-driven Le Toullec-type diamond anvil cells with culet diameter of 500-700 μm were used for pressure generation. The sample chambers with the approximate diameter of 300-400 μm were obtained by drilling stainless steel gaskets preindented to 80 μm. The solutions were loaded in the sample chambers along with rubies for pressure estimation and then pressurised to 0.3-0.4 GPa before placing into cryostat. The solutions were investigated in the range 0-1.7 GPa and 200-300 K. Powder and single-crystal X-ray diffraction has been performed on the solid phases wherether formed.

In **Experiment 1**, in the DMA-H_2O System, two solid phases have been observed over the studied range 0-4.3 GPa and 240-290 K (Figure 1). Over the all P-T range, the low-pressure sI clathrate hydrate is present. At 250 K, formation of a new orthorhombic phase was observed along with the initial sI clathrate with the unit cell parameters *a* = 12.153(15) Å, *b* = 20.516(3) Å , *c* = 21.498(3) Å. This orthorhombic phase has disappered upon further heating.

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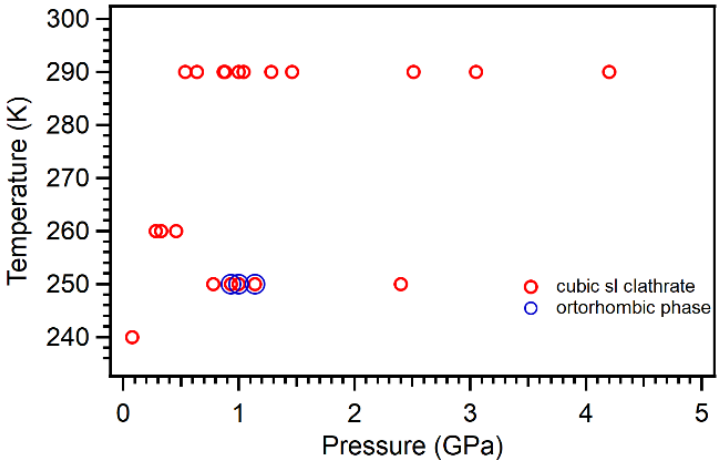

Figure 1. Pressure–temperature phase diagram indicating the conditions at which diffraction data for DMA clathrates were collected in the present study.

EA-H_2O system, studied in the **Experiment 2**, has demonstrated different behaviour (Figure 2). The low-pressure phase is also cubic, sI clathrate hydrate. However, it transforms to another phase at ~0.7 GPa. Above this pressure, a hexagonal phase is present with the unit cell parameters *a* = 16.9008(15) Å, *c* = 20.2337(15) Å. Above 1.5 GPa it undergoes transformation to an orthorhombic phase *a* = 11.376(2) Å, *b* = 23.046(6) Å, *c*= 16.6902(16) Å. This phase was obsered up to ~2 GPa. Above this pressure, a new tetragonal phase *a* = 19.6454(17) and *c* = 11.6835(9) Å was discovered.

Structural refinement of the observed phases is ongoing as well as calculation of the equation of state for the DMA sI clathrate hydrate.

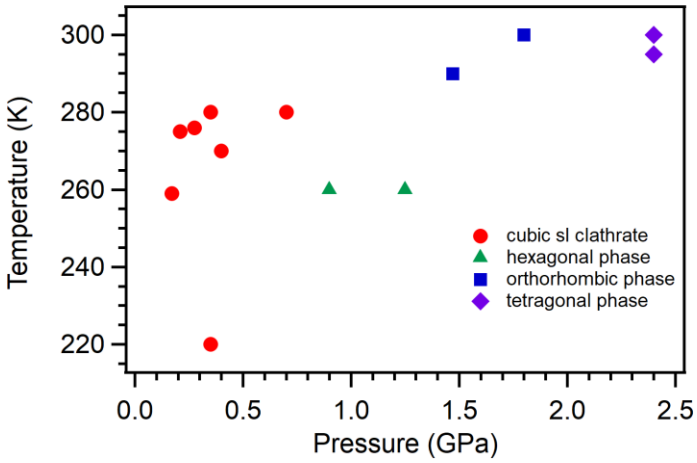

Figure 2. Pressure–temperature phase diagram indicating the conditions at which diffraction data for EA clathrates were collected in the present study.