The crystal structures of the vanadium(V) and the molybdenum(VI) oxides have been investigated by single crystal X-ray diffraction techniques at pressures from 0 to < 55 kbar. The studies have been performed using MoKα radiation from a rotating anode equipment and a conventional diamond anvil cell (Diacell Products). Pressures were estimated with the ruby fluorescence method. Single crystal diffraction data have been collected at several different pressures. Preliminary refinements (giving Rvalues well below 0.10) indicate only smooth structural changes in the selected pressure range. Due to the layered structural packings, the compressibilities are anisotropic, which leads to a pressure-sensitive short V–O contact (< 2.8 Å) in V₃O₅. The experimental studies are supplemented by band orbital calculations with the extended Hückel method.

The high pressure form of Ca(OH)₂, isomorphous with monoclinic ZrO₂, is known from high pressure in situ powder X-ray diffraction at 8 GPa (Kunz et al., High Pressures, High Temperatures, in press). This phase is unquenchable at room temperature, reverting to the Cdl₂-type polymorph at about 1 GPa during decompression.

In the present study, a 170 milligram sample of the high pressure form of Ca(OH)₂, synthesized at 10 GPa and 400°C, was recovered to ambient pressure for powder neutron diffraction. The back-transformation to the Cdl₂ structure was suppressed by chilling the sample with liquid nitrogen during decompression. The sample was transferred to a cryostat and a neutron powder diffraction pattern taken at 11 K. Platinum foil which was originally used to wrap the sample was left on as a dispensing standard.

Rietveld refinement indicates the structure P2₁/c, a = 5.409 Å, b = 6.104 Å, c = 5.965 Å, β = 103.7°. Calcium is in 7-coordination with oxygen, with Ca-O distances ranging from 2.39 Å to 2.89 Å. There are, two O-D bond distances, both close to 1.00 Å. One of the D atoms is hydrogen bonded to oxygen, with an associated O-O distance of 2.81 Å; the other appears to be nonbonded.