performed in the pressure range between 0.2 and 7.9 GPa. For this purpose a diamond-anvil cell with methanol-ethanol mixture (in ratio 4:1) as pressure medium and tungsten gaskets were used [2].

In this pressure range we were able to solve the structures of three different high-pressure phases. The structures of these phases show a stepwise increase of MoO_n-coordination from MoO_4-tetrahedra via MoO_5-pyramids to MoO_6-octahedra with increasing pressure.

At approximately 1.3(2) GPa the first high-pressure phase $K_4Co(MoO_4)_3$–II appeared with additional reflections in the a*-c*-plane of the initial unit cell, corresponding to a doubling of cell volume. Equivalent positions in the α-phase split into inequivalent sites, but only minor changes in the structure are accompanied with this phase transition. At about 2.2(1) GPa the second high-pressure phase $K_4CoMo_3O_{12}$-III was found, showing a change in the coordination of some molybdenum ions: Two neighbouring $[MoO_4]$-tetrahedra transform to a pair of edge-sharing $[MoO_5]$-pyramids. At about 5.97(5) GPa the third high-pressure phase $K_4CoMo_3O_{12}$-IV appeared. More $[MoO_4]$-tetrahedra have transformed into edge-sharing pairs of $[MoO_5]$-pyramids and the $[MoO_5]$-pyramids in phase –III have been transformed further into $[MoO_6]$-octahedra. The $[CoO_6]$-octahedra remain in all phases and are only slightly affected by the application of hydrostatic pressure.

This work is part of a systematic study of complex network structures in quarternary phases A-T-Mo-O with A an alkaline metal and T a 3d-transitional metal.

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MS22 P04

Structures of high-pressure polymorphs: what can different techniques contribute to our knowledge about them? (Crystals of selected amino acids as examples).

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Keywords: polymorphism, high pressure, amino acids

Effect of pressure on the crystalline amino-acids has recently become a hot topic: i) monitoring pressure-induced structural distortions / phase transitions gives a better insight into the intermolecular interactions / intramolecular dynamics in these systems, which can mimic the conformational changes of biopolymers, ii) the control of the polymorphism is important for many applications, for example, for the pharmaceutical industry. Typically, Raman spectroscopy, X-ray, or, more rarely, neutron powder diffraction are used, to characterize the

structures of the new high-pressure forms. In some lucky cases, it is possible to get a single crystal intact through the phase transition, and then a single-crystal diffraction technique can be used. Each of the techniques has its advantages and limitations.

The aim of the present contribution is to compare the results obtained during a few recent years for the same systems studied independently by several research groups in Russia, Germany, UK, India using different experimental techniques. In particular, we compare the complementary knowledge on the high-pressure polymorphs of glycine gained by X-ray powder diffraction, X-ray single-crystal diffraction, Raman spectroscopy and inelastic neutron scattering, as well as the results of the studies of L- and DL-serine by X-ray powder diffraction, neutron powder diffraction, X-ray single crystal diffraction, Raman spectroscopy and inelastic neutron scattering.

MS22 P05

Pressure-induced structural phase transition and negative thermal expansion in ReO_3 Tapan Chatterji[a], G.J. McIntyre,[a] H.E. Fischer[a], M. Brunelli[b], [a]*Institut Laue-Langevin, BP 156, 38042 Grenoble Cedex, France,* [b]*European Synchrotron Radiation Facility, BP 222, 38043 Grenoble Cedex, France.* E-mail: chatterji@ill.fr

Keywords: High pressure, negative thermal expansion, structural phase transition.

We have investigated the pressure-induced structural phase transition in ReO_3 by neutron diffraction on a single crystal. We collected neutron diffraction intensities from the ambient and high pressure phases at T = 7 kbar and refined the crystal structures. We have determined the stability of the high pressure phase as a function of temperature down to T = 2 K and have constructed the (P-T) phase diagram. The critical pressure is P_c = 5.2 kbar at T = 300 K and decreases almost linearly with decreasing temperature to become P_c = 2.5 kbar at T = 50 K. The phase transition is driven by the softening of the M_3 phonon mode. The high pressure phase is formed by the rigid rotation of almost undistorted ReO_6 octahedra and the Re-O-Re bond angle deviates from 180 degree. We do not see any evidence for the existence of the tetragonal (P4/mbm) intermediate phase reported earlier. In order to

check whether Re-O-Re bond angle deviate from 180 degree in the local structure we have determined neutron diffraction intensities up to a very high Q as a function of pressure from a polycrystalline sample of ReO_3 on a hot neutron diffractometer. The preliminary PDF analysis indicates that the Re-O-Re bond angle deviate from 180 degree already at ambient pressure. We have also determined the thermal expansion of ReO_3 on a high resolution X-ray powder diffractometer at the synchrotron source in the temperature range 4-500 K and discovered negative thermal expansion in the low temperature range 4-120 K. The negative thermal expansion in ReO_3 is attributed to the rigid unit M_3 phonon mode.