

**ENHANCING ELECTRON DIFFRACTION STRUCTURES USING MAXIMUM ENTROPY**

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Investigating the structure of a membrane protein using electron diffraction can be very difficult, particularly because of the potential lack of accuracy and the limited amount of data collected because of limitations due to tilting. When these issues become a problem a method which can still be used for solving or enhancing the potential density map is the technique of maximum entropy, an implementation of which is included in the program MICE [1]. The advantage of this method over other similar ones is its ability to incorporate prior information, such as reflection phases from electron microscopy, the molecular envelope, any known partial structure and to employ entropy optimization techniques.

Incorporation of a molecular envelope into the maximum entropy calculation can be vital to impose values on the unit cell where it is most likely to find new places of density. The envelope can easily be produced using the CCP4 program suite [2], however only if a small fragment of the structure is known. To give a starting point for maximum entropy a basis set of known reflection phases is required. These are normally gained through the permutation of the observed reflections, but here they can be found either directly from electron microscopy or the most accurate phases from the Fourier Transform of the partial structure can be used. Using this information maximum entropy can be shown to be a very powerful tool even at relatively low resolutions, enhancing partially known structures and even calculating unmeasured reflections from the missing cone.

## References

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**Keywords: ELECTRON DIFFRACTION MAXIMUM ENTROPY MOLECULAR ENVELOPES****RUBY LUMINESCENCE PRESSURE SCALE REVISED!**

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The shift of the Ruby R1 luminescence line is widely used for pressure measurements in Diamond Anvil Cells (DAC). Even when the most recent version of this Ruby scale[1] is used in static X-ray diffraction experiments with DACs and Ruby, the repeatedly observed discrepancies in these static Equation of State (EOS) data with respect to ultrasonic or theoretical studies are not removed. However, a detailed analysis of the recently published single crystal X-ray diffraction data on diamond up to 140 GPa in a DAC, using He as "nearly" hydrostatic pressure transmitting medium and Ruby as pressure sensor[2], and a comparison of these results with ultrasonic, theoretical, and shock wave data for the EOS of diamond allow together to propose a revision of the Ruby luminescence pressure scale, which is compatible with the previous "quasihydrostatic" calibrations[3] and with the recent "absolute" calibration based on X-ray diffraction and Brillouin scattering of MgO in a DAC with Ruby. The present revision of the Ruby scale resolves to a large extent the previously observed discrepancies in the EOS data for hard materials.

## References:

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**Keywords: HIGH PRESSURE MEASUREMENTS RUBY SCALE EOS****HIGH-PRESSURE POLYMORPHISM IN DEUTERATED UREA**H.P. Weber<sup>1</sup> W.G. Marshall<sup>2</sup> V. Dmitriev<sup>3</sup><sup>1</sup>Universite De Lausanne Institute De Cristallographie Bsp LAUSANNE CH-1015 SWITZERLAND <sup>2</sup>ISIS Neutron Facility, RAL, Chilton, OX11 0QX, UK <sup>3</sup>Swiss-Norwegian Beamlines, ESRF, BP 220, F-38043 Grenoble

The high-pressure properties of simple molecular systems are of great current interest in that they provide important insights into the density dependence of interatomic potentials. Recent examples of systems studied include the classic ices, binary ice compounds and simple organic compounds. A very recent addition to this rapidly growing list is urea OC(NH<sub>2</sub>)<sub>2</sub>. Under ambient conditions, urea crystallizes as phase I (*P*-42<sub>1</sub>m; *Z* = 2) in a simple, comparatively open, strongly hydrogen-bonded structure consisting of ribbons of planar molecules linked head-to-tail along the *c*-axis. Each ribbon is surrounded by four identical orthogonally oriented ribbons pointed in the opposite sense along the *c*-axis. The structure is noteworthy in that it is the only example of a compound in which a carbonyl group accepts four N-H...O hydrogen bonds, each of similar strength, as reflected in their closely similar lengths (2.08 Å average). We have recently determined (POLARIS diffractometer; Paris-Edinburgh pressure cell) the full crystal structure of three additional phases: A. Phase III (*P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>; *Z* = 4) 0.5 GPa < *P* < 2.8 GPa, B. Phase IV (*P*2<sub>1</sub>2<sub>1</sub>2; *Z* = 2) 2.8 GPa < *P* < 7.2 GPa, C. Phase V (*P*mcn; *Z* = 4) 7.2 GPa < *P* < 9.0+ GPa. With increasing pressure, each new phase is a progressively more distorted version of phase I. The more efficient molecular packing in phase III is obtained at the expense of the hydrogen-bonding network. Each carbonyl oxygen atom now accepts only three hydrogen bonds, with N-H...O contact distances ranging from 1.84 Å to 2.30 Å. Intriguingly, phases IV and V both revert back to the four-fold hydrogen-bonding motif.

**Keywords: POLYMORPHISM, PRESSURE, ORGANICS****STRUCTURAL STUDY OF PRESSURE-INDUCED MAGNETIC PHASE TRANSITIONS IN MANGANITES**D.P. Kozlenko<sup>1</sup> V.P. Glazkov<sup>2</sup> R.A. Sadykov<sup>3</sup> B.N. Savenko<sup>1</sup> V.I. Voronin<sup>4</sup><sup>1</sup>Joint Institute For Nuclear Research Frank Laboratory of Neutron Physics Joliot Curie St., 6 DUBNA MOSCOW REG 141980 RUSSIA <sup>2</sup>Russian Research Center "Kurchatov Institute", 123182 Moscow, Russia <sup>3</sup>Institute for High Pressure Physics, RAS, 142190, Troitsk Moscow Reg., Russia <sup>4</sup>Institute for Metal Physics, Ural Branch of RAS, 620219, Ekaterinburg, Russia

Manganites of perovskite type R<sub>1-x</sub>B<sub>x</sub>MnO<sub>3</sub> (R - rare earth, B - alkaline-earth elements) exhibit rich magnetic and electronic phase diagrams depending on the B-cation type element and its content [1]. Their structures and properties were studied in the wide range of temperatures and applied magnetic fields in relation with the recently discovered colossal magnetoresistance (CMR) effect. However, an effect of high pressure on the crystal and magnetic structure of manganites has not been much studied yet.

Two systems with different magnetic properties were chosen for experiments - La<sub>0.67</sub>Ca<sub>0.33</sub>MnO<sub>3</sub> and Pr<sub>0.7</sub>Ca<sub>0.3</sub>MnO<sub>3</sub>. At ambient pressure they have an orthorhombic structure (space group *Pnma*). At *T* < *T<sub>c</sub>* = 250 K La<sub>0.67</sub>Ca<sub>0.33</sub>MnO<sub>3</sub> becomes a ferromagnet. Pr<sub>0.7</sub>Ca<sub>0.3</sub>MnO<sub>3</sub> at *T* < *T<sub>N</sub>* = 140 K transforms to antiferromagnetic phase with two propagation vectors *k*<sub>1</sub> = (0 0 1/2) and *k*<sub>2</sub> = (1/2 0 1/2) and at *T* < 100 K a mixture of this phase with additional ferromagnetic phase appears.

Neutron diffraction experiments were performed with the DN-12 spectrometer (IBR-2 reactor, Dubna) at pressures up to 4 GPa in the temperature range 15-300 K using sapphire anvil high pressure cells. Surprisingly, in both La<sub>0.67</sub>Ca<sub>0.33</sub>MnO<sub>3</sub> at *P* = 3.8 GPa and Pr<sub>0.7</sub>Ca<sub>0.3</sub>MnO<sub>3</sub> at *P* = 2.2 GPa a phase transition to a new antiferromagnetic phase with propagation vector *k* = (0 1 0) was observed at *T* = 15 K. This transition results in shortening of *b*-axis by 3 % and increasing of *c*-axis by 2 % while *a*-axis remains nearly the same. Structural parameters of the new magnetic phase were determined.

## References

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**Keywords: HIGH PRESSURE, NEUTRON DIFFRACTION, MANGANITES**