（ ※本報告書は英語で記述してください。ただし、産業利用課題として採択されている方は日本語で記述していただいても結構です。 ）

CROSS TOKAI **Experimental Report** J-PARC	承認日 Date of Approval 承認者 Approver 提出日 Date of Report
課題番号 Project No. 2016B0190 実験課題名 Title of experiment D/H isotope effect on hydrogen bond geometry of guyanite at high-pressure 実験責任者名 Name of principal investigator Asami Sano 所属 Affiliation Japan Atomic Energy Agency	装置責任者 Name of Instrument scientist Takanori Hattori 装置名 Name of Instrument/(BL No.) BL11 実施日 Date of Experiment 2017. Feb. 11 – 2017. Feb. 14

試料、実験方法、利用の結果得られた主なデータ、考察、結論等を、記述して下さい。(適宜、図表添付のこと)
Please report your samples, experimental method and results, discussion and conclusions. Please add figures and tables for better explanation.

1. 試料 Name of sample(s) and chemical formula, or compositions including physical form.

Sample: Deuterated guyanite (CrOOD, powder), Vanadium (V, solid)
Gasket: TiZr encapsulating gasket
Pressure medium: deuterated methanol-ethanol

2. 実験方法及び結果 （実験がうまくいかなかった場合、その理由を記述してください。）
Experimental method and results. If you failed to conduct experiment as planned, please describe reasons.

EXPERIMENTAL: A high-pressure neutron diffraction experiment at ambient temperature was conducted at PLANET using a Paris-Edinburgh press with tungsten carbide single toroid anvils. The sample of CrOOH and CrOOD was synthesized at 3 GPa and 270 °C from a mixture of CrO_2 and D_2O using a Teflon lined autoclave prior to the neutron diffraction experiments. Synthesized sample was loaded into TiZr null-alloy encapsulating gaskets with deuterated methanol-ethanol mixture to keep hydrostatic condition. Only the experiment on CrOOD was conducted in this term because the signal from the sample was weak and the exposure time was longer than expected probably due to the low crystallinity.

RESULTS: Fig.1 shows the observed pattern obtained at 2.7 GPa and Rietveld fitting. The observed pattern can be explained by β-CrOOD in addition to small amount of CrO_2 that was contaminated during the synthesis. Difference Fourier map (Fig. 2) shows that nuclear density map consists of asymmetric two peaks suggesting hydrogen is partially disordered at this pressure. The diffraction patterns were corrected at ambient pressure, 0.8, 1.6, 2.7, 4.3, 5.5 and 6.5 GPa on the compression. The data of vanadium and empty were also obtained to normalize the intensity.

2. 実験方法及び結果（つづき） Experimental method and results (continued)

Experiments at higher pressure above 7 GPa on CrOOD and also CrOOH will be conducted in future to discuss the evolution of hydrogen bond geometry and its isotope effect.

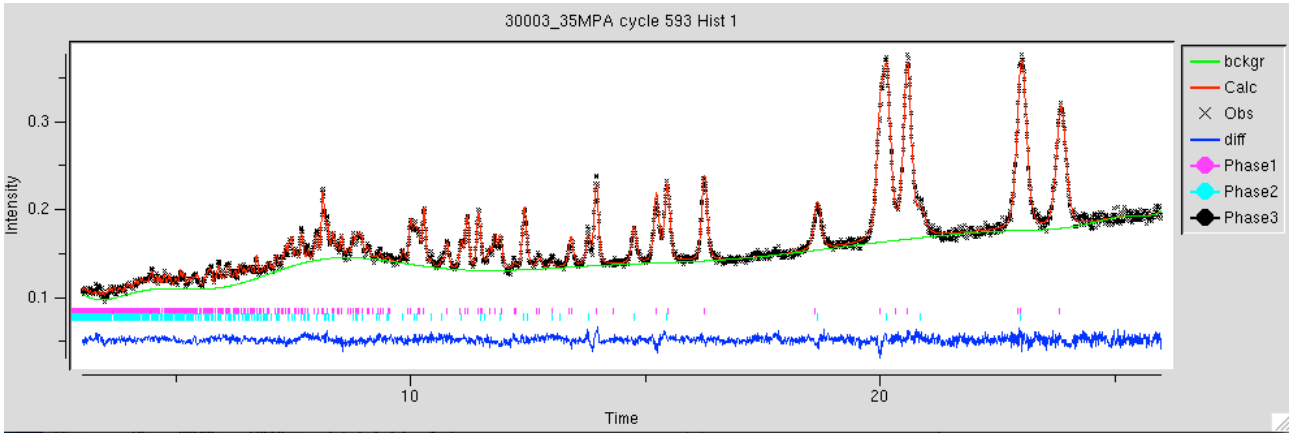

Fig. 1 Observed (crosses) calculated (red line) and residual patterns (blue line) obtained at 2.7 GPa. Tick marks are calculated peak positions of β-CrOOD (top) and CrO_2 (bottom)

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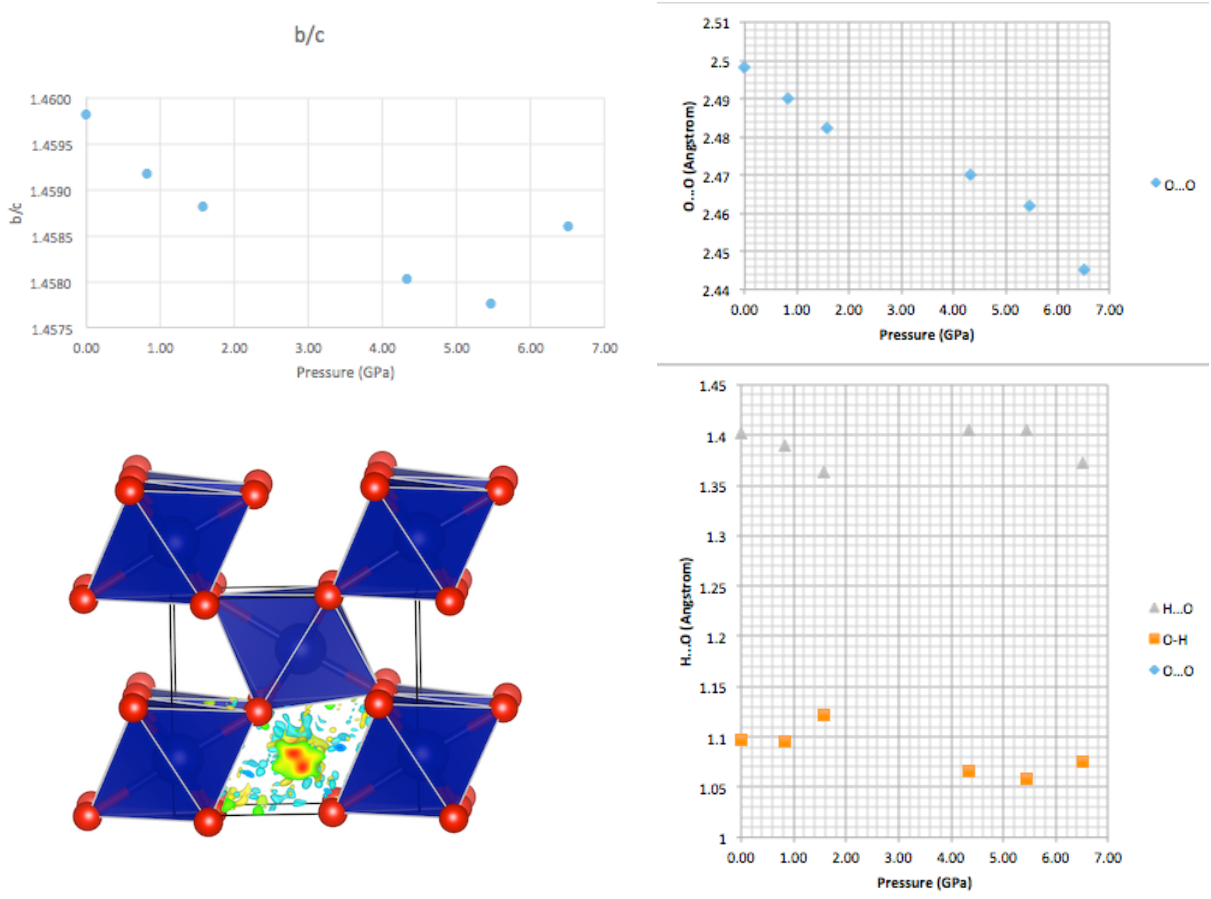

Fig. 2 (top left) Axial compressibility showing anomaly around 5.5 GPa, (bottom left) Difference Fourier map around deutrium at 2.7 GPa, (right) Pressure evolution of O-D...O hydrogen bond geometry.