ESRF	**Experiment title: Structure determination of (n-But4)2[Ru(NO2)4(NO)(OH)]H2O: a new Ru compound with metastable excited states**	**Experiment number:** CH-590
Beamline: ID11	**Date of experiment:** From: 31-3-99 to: 5-4-99	**Date of report:** 31-8-99
Shifts: 18	**Local contact(s):** Gavin Vaughan	*Received at ESRF:* 02 SEP. 1999

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Report:

The experiment consisted of X-ray diffraction data collections on $(n\text{-}But_4)_2[Ru(NO_2)_4NOOH]$ single crystals at 42KeV in order to determine the crystal structure. A previous X-ray diffraction experiment on the $(n\text{-}But_4)_2[Ru(NO_2)_4NOOH]$ compound had shown serious radiation damage preventing the crystal structure determination.
The study of the phase transition of $Na_2[Ru(NO_2)_4NOOH]H_2O$ by powder diffraction was also performed.
The crystals of $(n\text{-}But_4)_2[Ru(NO_2)_4NOOH]$ showed high mosaicity and diffracted upto a maximum resolution of 1Å. Data was collected on the two best crystals using the Brucker CCD-smart system. Despite the bad diffracting power of the samples, 10307 unique reflections could be measured on the second crystal covering 65% of the reciprocal space. The space group turned out to be C2. The structure was solved using the SHELXS-86 and refined with the SHELXL-93 programs, respectively. The lack of high order data and the presence of considerable disorder in the tetrabutilammonium groups precluded an anisotropic refinement. An agreement factor of 13% was obtained for 3928 unique reflections with $I>2\sigma(I)$ and 360 refined parameters. A **bc** projection of the crystal structure is shown in Fig.1.
The chemical analysis of the sample indicated the presence of Na in the compound and suggested the chemical formula $[(C_4H_3)_4N]_3Na[Ru(NO_2)_4NOOH]_2$. The crystal structure confirmed the chemical hypotesis.

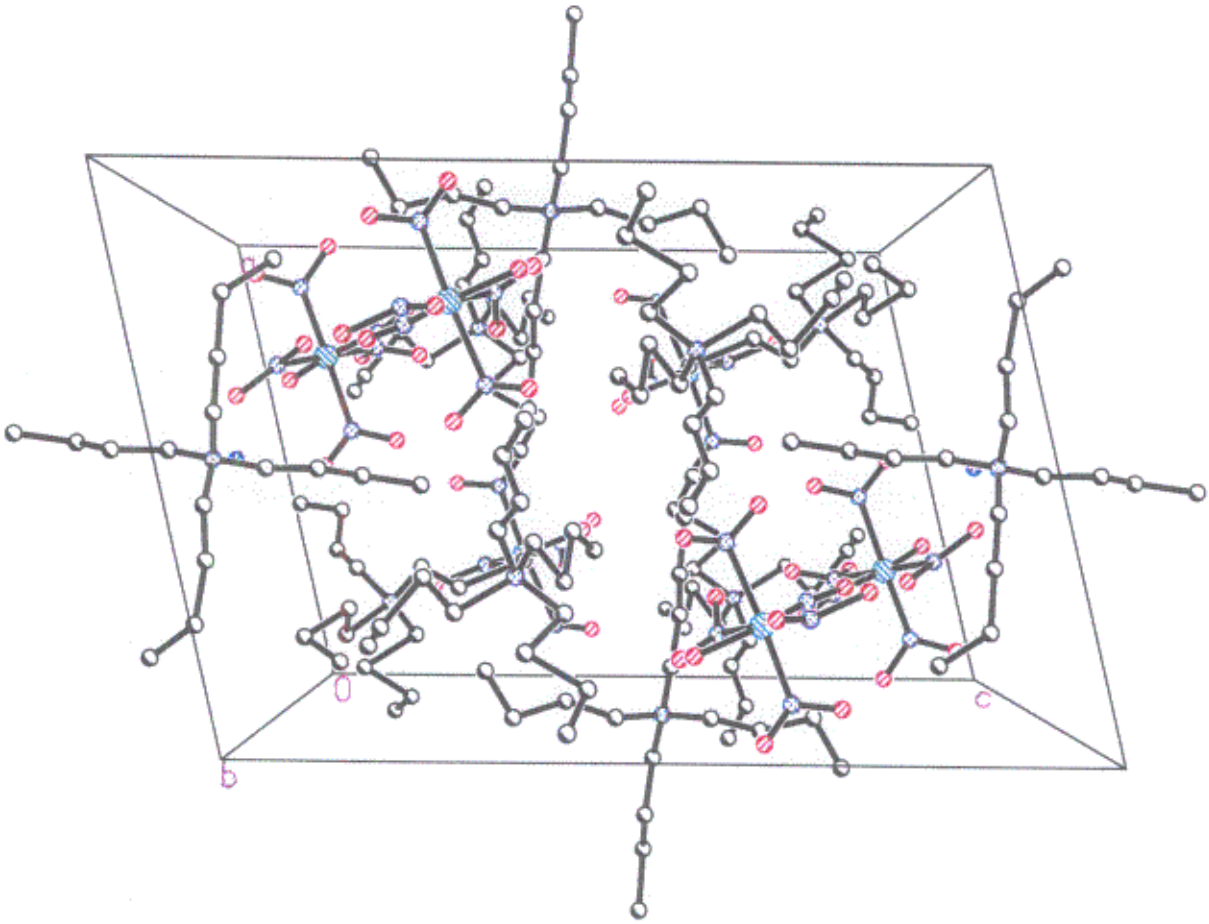

Fig. 1. Crystal structure of $[(C_4H_3)_4N]_3Na\ [Ru(NO_2)_4NOOH]$.

Previous structural studies on $Na_2[Ru(NO_2)_4NOOH]2H_2O$ single-crystals had indicated a phase transition from C2/m to $P2_1/n$ involving a minor reorientation of the $[Ru(NO_2)_4NOOH]^{2-}$ groups. Scanning calorimetry pointed out a phase transition at 242K. In order to further study the phase transition synchrotron X-ray diffraction data were collected on $Na_2[Ru(NO_2)_4NOOH]2H_2O$ powder at 72 different temperatures between 293K and 110K at 42KeV with the Brucker-Smart CCD.

A second order phase transition was observed at 242K characterized by the change in the slope of the **a** and **c** crystallographic axis and the unit cell volume. Fig. 2 shows the evolution of the cell parameters with temperature.

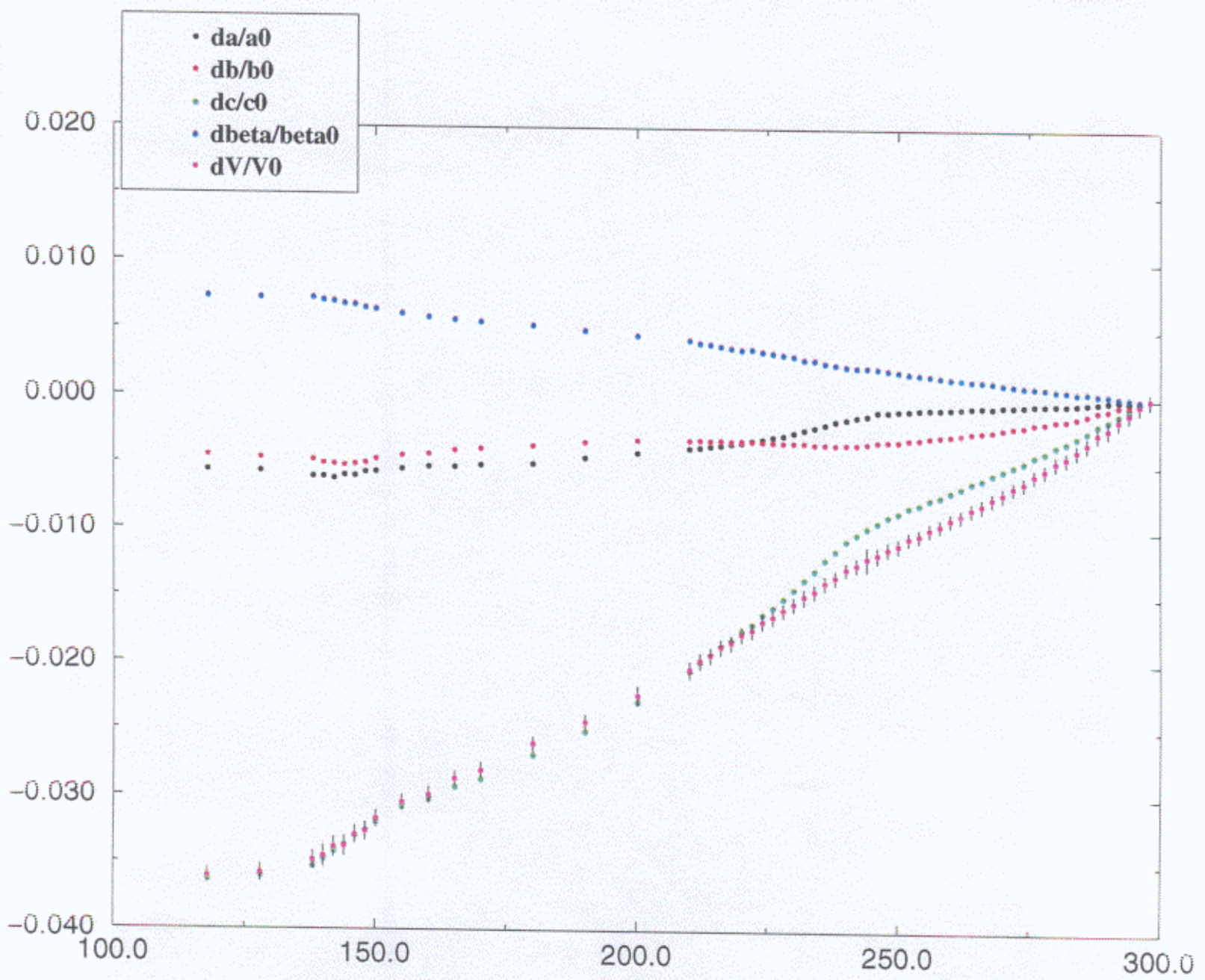

Fig. 2. Changes of the $Na_2[Ru(NO_2)_4NOOH]2H_2O$ unit cell parameters with temperature.