



ESRF

Experiment title: Charge Density Study of
 $k_2[Ru(NO_2)_4(OH)(NO)]$ metastable excited states

**Experiment
number:**

CH186

Beamline:

ID11

Date of Experiment:

from: 4-12-96

to: 9-12-96

Date of Report:

Shifts:

15

Local contact(s):

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Received at ESRF:

1 AOUT 1997

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Report:

The experiment consisted of performing X-ray diffraction of a $K_2[Ru(NO_2)_4(NO)(OH)]$ crystal before and after exciting it with a laser beam in order to determine structural changes between the ground state and the main metastable excited state. The experiments were carried out at ID11, at 50.2keV, in hybrid2 mode, 160mA of current, 20.2mm wiggler-Gap with a beam size of $0.3 \times 0.3 \text{ mm}^2$. The sample had prismatic habit with its maximum length not exceeding 100 microns. Four data sets were collected using the Siemens CCD camera. The sample-camera distance was 6.05 cm. A first data set, consisting of 1000 frames of 0.1 degrees in omega and an exposure time of 10 seconds, was measured at room temperature in order to determine the cell parameters and make sure the sample was of good quality. A second data set, consisting of 2800 frames of 0.1 degrees in omega with an exposure time of 10 seconds, was taken at 150K with a redundancy of 2.5 to determine the ground state structure. The data analysis of this data set gave an $R_{int}=4\%$ and the structure was successfully solved obtaining an agreement factor $R=4.9\%$. A third data set, consisting of 3500 frames of 0.1 degrees in omega and an exposure time of 10 seconds and 4000 frames with an exposure time of 3 seconds, was taken at 150K after having excited the sample during 8 hours with the 457.9nm line of an argon laser with a power density of 50 mW/cm^2 . To avoid any de-excitation induced by the X-rays beam, the laser was kept irradiating the sample during the data collection. The processing of the third data set showed good R_{int} values (5-6%) but the structural study points to an overheating of the sample during the laser excitation and therefore an inefficient excitement of the sample. In that sense we used the K-atoms thermal agitation factors to deduce the sample temperature since these atoms are not involved in the excitation. The sample temperature turned out to be 190-200K. The spectroscopy studies from Woike et al. shows that the main metastable excited state de-excites completely above 200K.

Visible spectroscopy experiments are nowadays being carried out in collaboration with Dr. Dominique Block from the Laboratoire de Spectroscopie Physique at the Université Joseph Fourier of Grenoble to determine the best way to excite the samples. A fourth and last data set consisting of 700 frames of 0.1 degrees in omega and 10 seconds of exposure time was measured at room temperature before taking the sample away. The analysis of this data demonstrates the sample did not suffer any damage during the experiment since we could reproduce the results from the first data set.