| ESRF | Experiment title: Precise determination of the inter-atomic potential in simple molecules by XAS | Experiment number: CH657 |
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| **Beamline:** BM29 | **Date of Experiment:** from: 23-Jan-1999 to: 30-Jan-1999 | **Date of Report:** 25-Aug-1999 |
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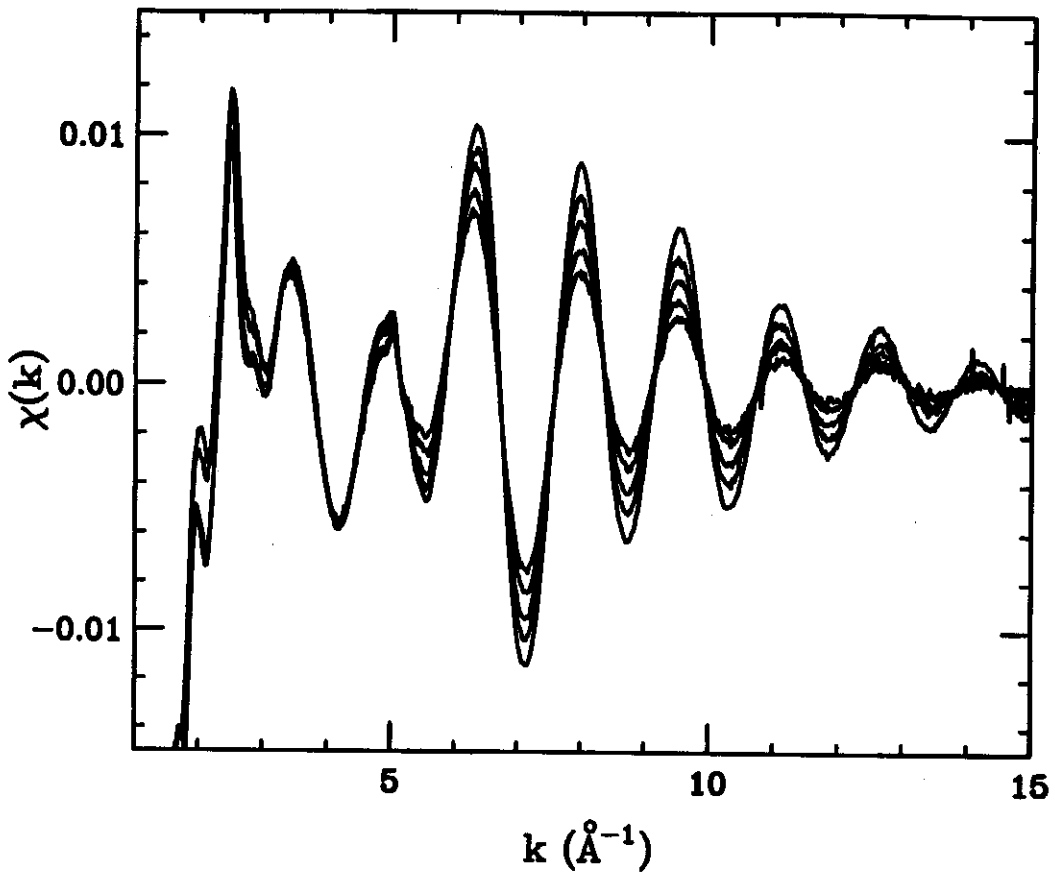
**Report:**

We have, for the first time, installed and commissioned a new BM29 sample-environment device suitable for measurements of gas phase low-density samples in the 300 to 1300 K temperature range. This device is a 80 *cm* three-region tubular oven which guarantees a stable and uniform temperature ±2 K in a 30 *cm* central region. The oven is remotely piloted through a RS232 interface for changing the set-point and for temperature readout, and therefore can be used in automatic temperature runs.

Samples are made using sealed Pyrex or quartz cylindrical cells. Several cells with flat windows in the 0.5-1.5 *mm* thickness range for experiments at E> 20 keV or with blown windows of thickness around 0.1-0.3 *mm* suitable for E> 10 keV were tested. The cells were evacuated and filled, through a capillary, with a suitable amount of sample (as gas, liquid, or solid) and then sealed with a flame. The sample cell is inserted in the middle of the tubular oven and aligned to the beam. Typical beams of 10×0.2 *mm* (h×v) were used which guarantee both an excellent energy resolution and high flux. Pyrex cell operation is limited to about 800 K whereas quartz can be used up to the maximum operation temperature. The temperature range accessible to the measurements is that going from RT (or the minimum temperature required to vaporize the whole sample) up to the molecular dissociation limit. For polyatomic molecules usually the dissociation is not reversible and the sample purity is lost.




Several samples were measured during this beamtime as indicated in the original proposal, including, diatomic molecules like $Br_2$, triatomic linear molecules ($HgI_2$), planar triangular molecules ($BBr_3$), and tetrahedral molecules ($GeBr_4$). In all cases high quality spectra were collected up to $k \approx 20$ Å$^{-1}$ with a fine sampling of $\Delta k = 0.05$ Å$^{-1}$ and at 100 K temperature steps. Due to the absence of irreversible decomposition the spectra of $Br_2$ were measured, for the first time, up to 1200 K. The above figure reports the $\chi(k)$ oscillations of the spectra collected at the temperatures of 120 °C, 320 °C, 520 °C, 720 °C, and 920 °C extracted using a default polynomial spline. These spectra have a much better quality, $k$ extension, and maximum T than those previously reported [1]. The effect of increasing thermal vibrations on the $\chi(k)$ is quite evident.

The analysis of these and other sets of data will be performed following the procedures described in our previous publication [2]. In the present case, due to the high maximum temperature, small deviations from the Gaussian approximation for the bond length distribution are detectable. The precise knowledge of this distribution can provide a valuable information on the shape of the molecular potential around the minimum in diatomic and polyatomic molecules.

[1] T. Yokoyama, K. Kobayashi, T. Ohta, A. Ugawa, Phys. Rev. B, **53**, 6111 (1996).

[2] A. Filipponi and P. D' Angelo, *"Accurate determination of molecular structures by x-ray absorption spectroscopy."*, J. Chem. Phys. **109**, 5356 (1998).