ESRF	**Experiment title:** High pressure x-ray diffraction studies of the actinide metals Am and Cm	**Experiment number:** HS-504
Beamline: ID30	**Date of experiment:** from: 14 June 1998 to: 19 June 1998	**Date of report:** 1 September 1998
Shifts: 11	**Local contact(s):** T. Le Bihan	*Received at ESRF :*

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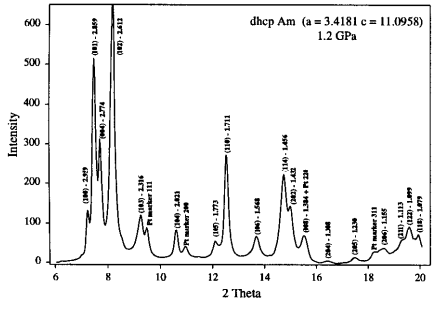
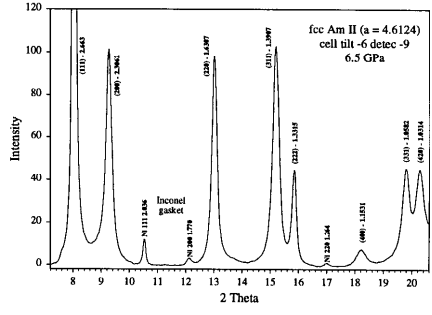
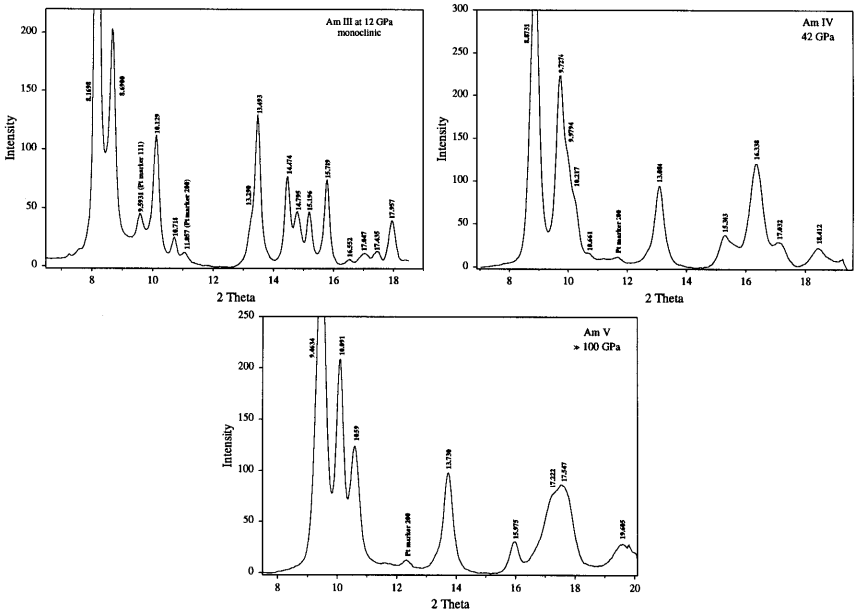
Report:

Samples of ^{243}Am and ^{248}Cm were loaded into three types of high pressure cell at the Oak Ridge National Laboratory in the USA and shipped to the ESRF where they were mounted in specially made containers for experiment HS-504 at ID30.
Extra precautions were taken to ensure that these samples remained confined during the experiments by the use of double or triple sealing of the cells with Mylar, Kapton or Be windows.
The cells used were of the Holzapfel, Mao-Bell and Cornell types to allow as much flexibility as possible with accurate determination of low and high pressure sample phases as well as a certain flexibility to choose optimal wavelengths and diffraction angles for the detector system.
We used the ID30 high pressure beam line in angle dispersive mode and collected data containing the full diffraction cones on the Fast Scan Image Plate Detector. A micro-focused beam of 25x25 μm^2 obtained with two bent mirrors and passed through a pinhole was used for the Cornell cells.
^{243}Am was loaded into Holzapfel and Cornell cells whilst the ^{248}Cm was loaded into the Mao-Bell and Cornell cells allowing a certain redundancy in case of problems with one of the cells.
400 pm diameter beveled diamonds with 200 μm hole size Inconel gaskets were used in the Holzapfel and Mao-Bell cells whereas 95 pm diamonds with 50 pm hole size steel T301 gaskets were used in the Cornell cells.
Exposure times ranged from only 15 seconds for the larger samples in the low pressure range to 60 seconds for the highest pressures.
The aims of the experiment were to probe the fundamental chemistry and physics of these elements by decreasing interatomic distances with applied pressure and in particular to solve the structures of Am III and Am IV as well as hopefully finding a new fourth phase of Cm above 60 GPa.
The selection of Cm and Am for investigation of high-pressure behavior of transplutonium elements was especially attractive as ^{248}Cm and ^{243}Am isotopes, which have a relatively low specific-activity, were available for the study.
We achieved most of our goals except that of reaching the Cm IV phase which was compensated for by the unexpected appearance of a fifth phase of Am. Structure refinement of the Am III and IV phases is in progress.

Results for ^{243}Am:
Both pressure cells behaved correctly and we were able to observe a total of live high pressure phases with a far higher resolution that any previous work.
The initial dhcp structure transformed to fcc at 6 GPa, to a monoclinic structure at 11 GPa, to a possible orthorhombic structure above 18 GPa and then to an as yet unidentified fifth phase around 100 GPa.
The sequence of structural changes is shown below:





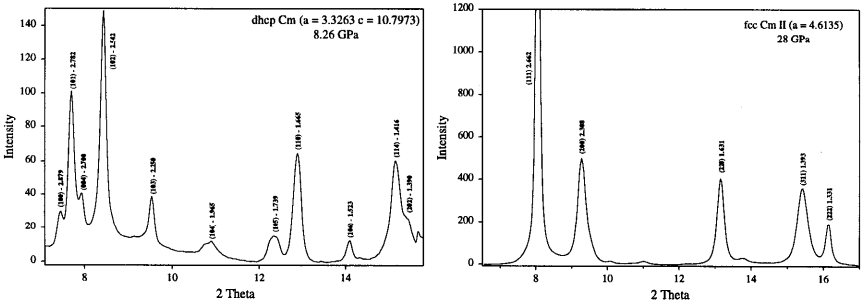


Results for ^{248}Cm:

Cm (element 96) is one of the most interesting transplutonium elements as it has a stabilizing ground state electronic configuration of : [Rn core] $5f^7$ 6d $7s^2$. This half-filled **5f-shell** greatly influences the pressure behavior of Cm; which displays higher structural transition pressures and resists f-electron delocalisation. The low pressure region was studied using the Mao-Bell pressure cell up to 20 GPa.

We were not able to further increase the pressure with this series because of deformation of the gasket hole which could have eventually led to diamond breakage.

The ^{248}Cm sample studied remained in the dhcp phase shown below throughout the measurement.



The high pressure range of ^{248}Cm was studied using a second Cornell cell where we observed a phase transition to an fcc structure shown up at around 26 GPa. This phase occurs at a considerably higher pressure than for Am. A third phase was also observed starting at around 45 GPa which may be the same as that found for Am III but which has yet to be analysed.

Our goal of reaching the predicted fourth phase of Cm was not realised in this measurement due to distortion of the gasket hole above 50 GPa.

Further experiments on Cm as well as alloys of Am/Cm are required and will be the subject of a further proposal for beam time at ID30.