ESRF	**Experiment title: The application of x-ray microscopy to determine the spatial distribution of Cr(VI) across soil grains in remediated and unremediated soils.**	**Experiment number**: CH-546
Beamline: ID21	**Date of experiment**: from: 23 Sep 98 to: 26 Sep 98	**Date of report**: 25 Aug 98
Shifts: 9	**Local contact(s)**: Jean Susini	*Received at ESRF:*

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Report:

This experiment was the first imaging to be performed on ID21. The theory was straightforward, that the height of the pre-edge peak is proportional to the ratio Cr(VI)/total Cr present in the sample (Fig. 1). Calibration of the pre-peak intensity was successfully performed using XANES on known bulk mixtures of Cr(VI)/Cr(III) at constant total Cr concentration in a matrix of $LiCO_3$ (a light element compound causing minimum fluorescence interference). We then transferred to micro-XANES (pixel size 1.0 μm) and scanned across grains which had been characterised previously by SEM/EDX mapping. Scans were performed for the pre-edge peak (6,023 eV) and the adsorption edge (6,037 eV) and then ratioed to give qualitative maps of Cr(VI) distribution (Fig. 2). Because the instrumentation was new, the acquisition times were slow (requiring overnight acquisition times for each energy) so very few grains were analysed. We did not find any definitive evidence of Cr(VI) in the samples although there is reported to be approximately 30% of Cr as Cr(VI). One possibility was that photo-reduction of Cr(VI) had taken place in the intense

micro-beam. However, subsequent experiments by Jean Susini indicate that this should not be a problem (especially if we were to work at LN_2 temperatures). In summary, this pilot project has demonstrated that the technique has enormous potential for mapping Cr(VI) and Cr(III) in contaminated soils. The perfection of this technique is of extreme importance in the remediation of Cr contaminated soils (normally achieved by reduction of Cr(VI) to Cr(III)) as there are very few methods to distinguish between these two oxidation states.