

Chemical characterization of ultra trace metals in small liquid drop

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Recent advances in biomedical sciences have demonstrated that many significant phenomena are influenced by trace metals in the system [1], but when the analyte is limited to a small liquid drop, the characterization becomes extremely difficult. X-ray fluorescence with synchrotron radiation is suitable for determination and also chemical state analysis [2] of such small amount of sample. One promising way for a liquid drop is the use of a micro beam and the other is the use of total reflection sample support with normal synchrotron beam [3]. In the present study, the latter procedure is employed.

The grazing incidence X-ray spectro-reflectometer [4] (Fig.1) has very recently been installed at the beamline. The beam time for the present research program has been spent for the commissioning of the equipment, and some preliminary tests were performed. The measurement was done using 13 keV monochromatic X-rays (ID Gap 17.88 mm) with the size of 0.03 x 2 mm². Higher order harmonics were rejected by

means of the beamline mirror (5 mrad). A Si(Li) detector is placed at 345 mm distance from the sample with an inclination angle of 10 deg to the surface. Figure 2 shows the total reflection X-ray fluorescence spectrum of donor horse serum (ICN Biomedicals, Inc, USA) centrifuged (10,000 G, 30 min at 4 °C) dropped onto a mirror-polished silicon wafer. The amount is 3 µl for both protein-bound and protein-free components. In the future experiments, chemical state analysis will be carried out for some trace metals observed in the spectrum. The authors are grateful to all members of the SPRING-8 spectro-chemical collaboration group for their contribution to the commissioning of the beamline and the present spectro-reflectometer.

References

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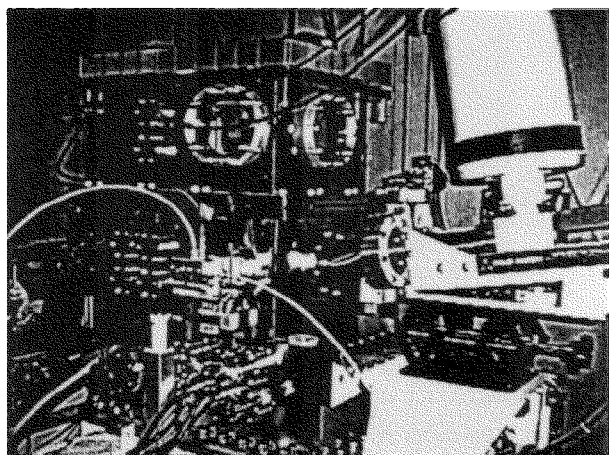


Figure 1 Grazing incidence X-ray spectro-reflectometer installed at the BL-39XU.

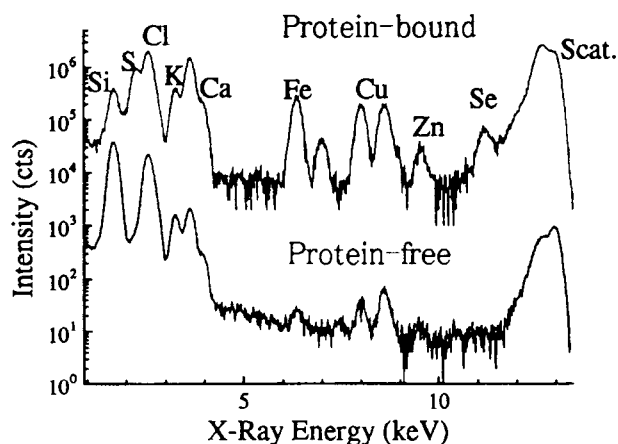


Figure 2 X-ray fluorescence spectrum of centrifuged serum.