

## 2 - 40 Determination of Iodine in Solution Using Energy Dispersive X-Ray Fluorescence Spectrometer

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$^{99}\text{Tc}$ ,  $^{127}\text{I}$ ,  $^{237}\text{Np}$  and  $^{241}\text{Am}$  are high-toxic and long lived-radionuclides in nuclear fuel waste. If these nuclides are transformed into short-lived or stable nuclides in ADS transmutation process, the waste has lower radiotoxicity. The rapid analysis of these nuclides before and after transmutation becomes very important. XRF is a very simple analytical technique; X-rays excite atoms in a sample, which emit X-rays at energies characteristic of each element. A detector measures the energies and intensities of the emitted X-rays. From this, one deduces which elements are present and their concentrations. In this work, we use stable iodine replace radio iodine to establish the rapid analysis method of iodine by XRF in solution<sup>[1-3]</sup>.

The  $K$  characteristic rays of iodine are  $K_{\alpha}$  (28.61 keV) and  $K_{\beta}$  (32.29 keV) (Fig. 1). The intensity of  $K_{\alpha}$  was selected to make the working curve (Fig. 2). The correlation coefficient ( $R^2$ ) was 0.996. The range of determination is 1~14 g/L.

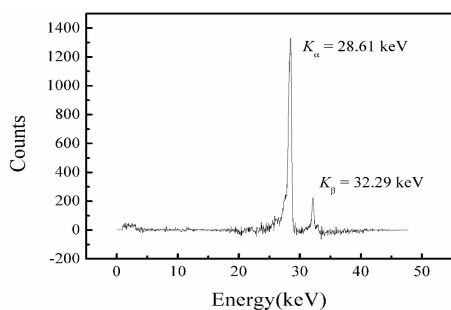


Fig. 1 The  $K$  characteristic rays of iodine.

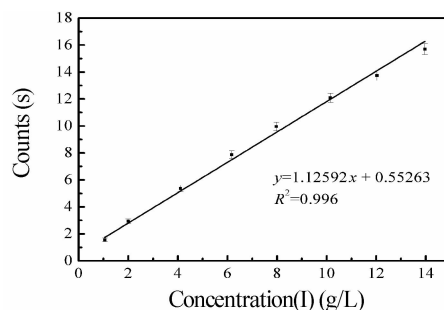


Fig. 2 The working curve.

A certain concentration sample was contained by 10 polyethylene centrifuge tubes (10 mL) and each of these tubes was detected for 100 s. The values were: 6.63, 6.69, 6.55, 6.86, 6.46, 6.59, 6.88, 6.95, 6.63 and 6.66 g/L. The average value was 6.69 g/L and the standard deviation was 0.16 g/L. The relative standard deviation was 2.35% ( $\bar{x} \pm s = (6.69 \pm 0.16)$  g/L). The precision of the analysis was good.

It indicated that EDXRF could be a potential simple method for analyzing iodine in solution.

### References

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- [2] Bai Erjun, Journal of Instrumental Analysis, 17, 1(1998).
- [3] Lu Weiwei, Song Fuxiang, Journal of Isotopes, 25, 4(2012).

## 2 - 41 Gas Phase Chemistry of Short-lived W and Os Carbonyls

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Formation of carbonyls in hot atom reactions has recently been pioneered as a powerful tool for future study of some transactinide elements<sup>[1]</sup>. It was postulated that synthesis of carbonyls at heavy ion accelerators is only possible behind a preseparator. In this study we have investigated carbonyl formation at a

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heavy ion accelerator without any preseparator, hence, by adding carbon monoxide directly to a carrier gas in which the products were stopped (gas-jet technique).

Fig. 1 depicts the set-up used at the IMP sector field cyclotron for our study. Tb or Ho targets of about  $0.7 \text{ mg/cm}^2$  thickness on a 19 m Be foil were bombarded by a cot 101 MeV  $^{19}\text{F}^{7+}$  beam with about 450 nA (corresponding to 64 pA) intensity. At the exit of the chamber the volatile products were transported with the carrier gas through a 5 m long Teflon capillary to an isothermal chromatography device. In this device 2 m long and 2 mm dia. Teflon columns could be kept at temperatures between 0 and  $-80 \text{ }^\circ\text{C}$ . Behind the column the products were transported along a 1 m long Teflon capillary to an activated charcoal trap positioned in front of an HPGe detector to assay  $\beta$ -decaying nuclides.

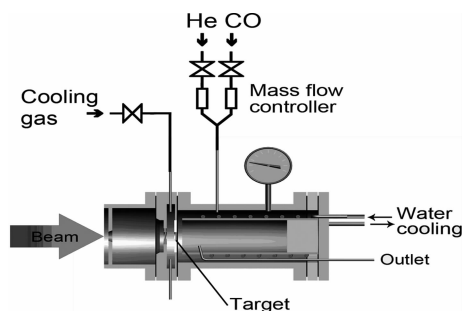


Fig. 1 Set-up used for the synthesis and transport of carbonyls at the IMP cyclotron.

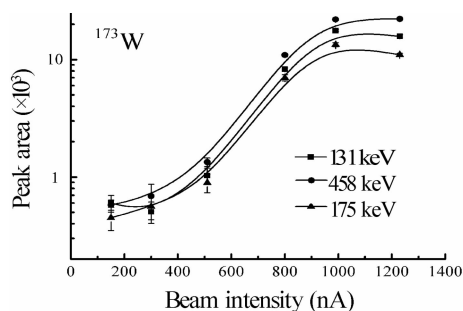


Fig. 2 Yield of  $^{173}\text{W}$ , formed in  $^{19}\text{F} + ^{\text{nat}}\text{Tb}$  reaction as a function of beam intensity.

Fig. 2 shows the yield of  $^{173}\text{W}$  as a function of beam intensity. Clearly, a saturation is observed at an intensity of about  $1 \mu\text{A}$  (equivalent to 143 pA). It was then decided to run the experiments with about 450 nA.

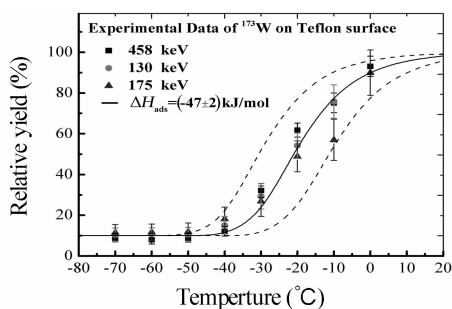


Fig. 3 Yield curve for  $^{173}\text{W}$  in an isothermal Teflon column.

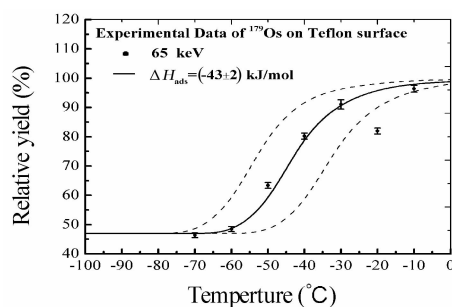


Fig. 4 Yield curve for  $^{179}\text{Os}$  in an isothermal Teflon column.

Fig. 3 shows the yield curve for  $^{173}\text{W}$  as a function of temperature along the Teflon chromatography column. Fig. 4 shows the similar chromatogram for Os using a  $^{\text{nat}}\text{Ho}$  target.

The resulting adsorption enthalpies deduced from the measured break-through curves resulted in  $(-47 \pm 2)$  and  $(-43 \pm 2)$  kJ/mol, for W and Os, respectively. It is important to note, that the yield curves do not drop to 0 % at low temperatures but to about 10 % and 50 %, respectively. We interpret this observation by aerosol transport from carbon clusters formed via interaction of the beam with CO in the collecting chamber. The higher value in case of the Os curve can then be interpreted by an increased contamination of the recoil chamber in the later experiment with Os.

To conclude, though model studies with carbonyls seem possible with the gas jet technique, the clear drawback is the aerosol formation and beam limitation that prevents application of this approach for study of transactinide carbonyls.

## Reference

- [1] J. Even, et al., Inorg. Chem., 51, 12(2012)6431.