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Kβ intensity ratios for Gd and Tb oxides and fluorides

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Introduction

It has become important to develop methods for observing the chemical environments of gadolinium and terbium in light of recent strong demand related to nanotechnology applications. In this report, the use of Kβ X-ray fluorescence spectra has been examined.

Experimental

The samples measured were 10 mm diameter x 5 mm thick pellets of Gd and Tb compounds (metal, oxide, and fluoride). They were mounted in the vacuum chamber that is usually used for Compton scattering measurements at the beamline. Energy-dispersive X-ray fluorescence (XRF) spectra were collected from 90 deg direction by a Ge detector (Canberra) and a digital signal processor (ORTEC DSPEC Jr). The energy of the incident X-rays was set at 69 keV.

Results and Discussion

Figure 1 shows X-ray fluorescence spectra of terbium oxide (Tb_4O_7). The accumulation time (real time) was 7200 sec. and the counting rate was 1.3 k counts/sec. One can see characteristic Tb K-lines as well as their escape and sum peaks. The Kβ peaks measured were higher than those of Kα, because an attenuator (copper plate, 1.5mm thick) was placed in front of the detector. The energy resolution was ca 400 eV at around Tb $K\beta_1$.

The intensity ratio between the $K\beta_1$ and $K\beta_2$ lines (see the inset of Fig.1) is basically constant, and is around 0.75, as long as the sample is thick enough, though the mass absorption coefficient differs for these two energies.

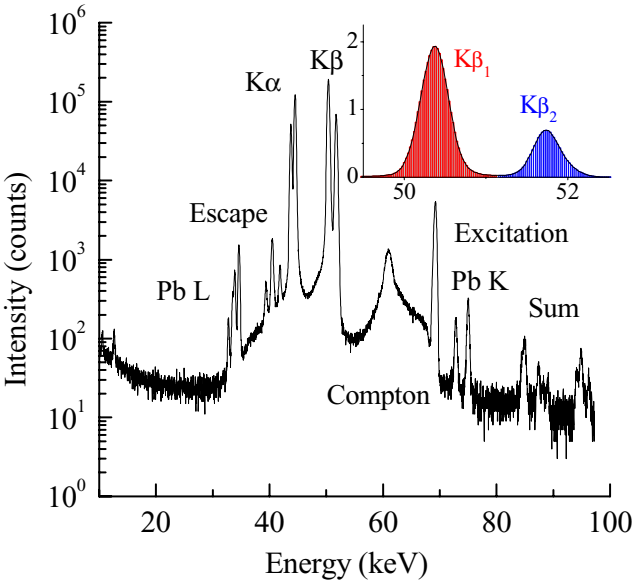

Figure 1 The XRF spectra of Tb_4O_7 pellet.

However, the intensity ratio can change slightly depending on the chemical environment [1]. Some results for gadolinium and terbium are listed in Table 1. The intensity ratio $K\beta_2/K\beta_1$ grows larger for metals rather than oxides and fluorides in the case of both gadolinium and terbium. Figure 2 shows the differences among those samples more clearly. Here, the net intensity ratios of $K\beta_1$ to ($K\beta_1$ + $K\beta_2$) for fluorides and oxides were plotted as a percentage to those of metals. It has been found that there is only small difference between the fluoride and oxide for gadolinium, but some changes are observed for terbium. It is interesting to note when considering the present results that they correspond to the order of the formal oxidation number, i.e., 0, +3, and +3.5 for terbium metal, fluoride, and oxide, respectively. Although the research is in the preliminary stage, further experiments to collect data for many different compounds are in progress. Some theoretical discussion is also important.

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References

[1] M. Harada and K. Sakurai, *Photon Factory Activity Report* **#20**, 286 (2002); **#21**, 268 (2003).

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Table 1 Summary of the intensity ratio $K\beta_2/K\beta_1$ for Gd and Tb samples.

	Gd	Tb
Metal	0.75715±0.00031	0.74719±0.00028
Fluoride	0.75609±0.00027	0.74605±0.00031
Oxide	0.75620±0.00037	0.74406±0.00050

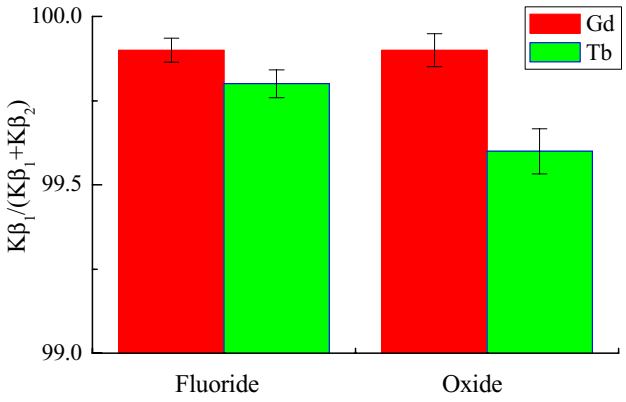

Figure 2 The relative changes of Kβ intensity ratios for fluorides and oxides in comparison to the metals. The left (red) and right (green) bars indicate gadolinium and terbium, respectively.