Tests of internal-conversion theory with precise γ- and x-ray spectroscopy:
The case of [103m]Rh studied via [103]Ru β- decay

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Our program to test internal-conversion theory through precise measurements of K-shell conversion coefficients, α_K, [1] was recently reviewed in Ref. [2]. Eight previously studied transitions, two of $E3$ and six of $M4$ character, showed good agreement with the theory that included the atomic vacancy in the "frozen orbital" approach, while disagreeing strongly with the theory that ignored the vacancy. This conclusion led to the adoption of the "frozen orbital" calculations by the US and international nuclear data networks in the Evaluated Nuclear Structure Data File (ENSDF). The ninth case, described in Ref [2] as still being in progress, is the 39.7-keV, $E3$ transition in [103m]Rh. Its current status is described in this progress report.

The 56-min 39.7-keV isomer in [103m]Rh can be populated either by the 17.0-day [103]Pd electron-capture decay, or by the 39.2-day [103]Ru β- decay. Both parents, [103]Pd and [103]Ru, can be obtained conveniently by thermal neutron activation of stable targets; and by measuring both decay channels we gain access to both α_K and α_T for the transition of interest: [103]Pd decay yields the ratio $\alpha_K/(1+ \alpha_T)$ while [103]Ru decay yields α_K. We reported the former in last year's Progress in Research [3]. In this report we describe our progress on the [103]Ru study, which was started one year later.

Two separate runs were done based on different sources. First, we electroplated [nat]RuO$_2$ on thin 25-μm aluminum backing (99.99% pure natural Al, from Goodfellow, USA). We began by dissolving 4.5 mg of RuCl$_3 \cdot x$H$_2$O powder (99.98% pure, from Sigma Aldrich, USA) in 185 μL of 0.1 M HNO$_3$ and evaporating the solution to dryness under Ar gas. This step converted the ruthenium chloride into ruthenium nitrate. Each sample was then reconstituted with 5 μL of 0.1 M HNO$_3$ and 12 mL of anhydrous isopropanol. This solution was then transferred to an electrodeposition cell [4], and the ruthenium nitrate was electrochemically deposited using the molecular plating technique [5, 6]. The deposition voltage ranged from 150-500 V and the current density was kept between 2 and 7 mA/cm^2. Deposition times ranged from 4 to 5 hr. After deposition, the targets were baked in atmosphere at 200 °C for 30 min to convert the ruthenium nitrate to ruthenium oxide. The resulting targets had thicknesses between 465 and 545 μg/cm^2 as measured by mass. The plating efficiencies were between 40 and 55%. The [nat]RuO$_2$ targets were characterized using scanning electron microscopy (SEM) to ensure uniformity. An energy-dispersive X-ray spectrometry (EDS) analysis was performed to verify the elemental composition, and the EDS spectra showed that Ru and O were indeed the two main components of the target layer. Although we were unable to verify directly the 1:2 Ru:O ratio, this is the most commonly formed oxide of ruthenium. The targets were black in color, as expected of the RuO$_2$ compound.

One target was activated at the Nuclear Science Center of Texas A&M University at a thermal neutron flux of 7.5×10^{12} n/(cm^{2}s) for 20 h in early May 2017. Preliminary spectra were recorded during May, followed by three different main runs in June, after the shorter lived Ru isotopes had decayed out:

the longest lived was ^{97}Ru with $T_{1/2}$ = 2.9 days. Although we used very pure materials and a very thorough procedure for source preparation, with extreme care taken to prevent any external contamination, we found an unexpectedly prominent impurity, ^{153}Gd ($T_{1/2}$ = 240 day), whose $K_{\alpha1}$ and $K_{\alpha2}$ x-ray peaks at 40.9 and 41.5 keV, respectively, were partially superimposed on the relatively small 39.7-keV γ-ray peak of interest. As a result, the analysis of the 39.7-keV γ ray was compromised.

Consequently, we prepared another target using a different material: a pure metal foil of 0.9-μm ^{nat}Ru on 1.5-μm ^{nat}Cu. The sample was activated for 32 hours under identical conditions to the previous target in September 2017, and was then measured, naturally with the Ru facing the HPGe detector. In this case, an impurity analysis of the γ-ray spectrum revealed a number of heavy metal elements (^{182}Ta, ^{185}Os, ^{191}Os, and ^{192}Ir) but fortunately these did not interfere with the 39.7-keV γ ray or with the Rh K x rays of interest.

Although now there were no interfering impurities, the ^{103}Ru β$^-$ decay populates other transitions in ^{103}Rh, which include an internal-conversion component and thus produce rhodium K x rays. We carefully determined the strength of this contribution to be 11.3(2)% compared to that of the rhodium K x rays produced by the internal conversion of the 39.7-keV transition γ ray. In addition, some of the γ rays from these competing transitions, especially the strong one at 497.1 keV, were able to excite the x-ray florescence of the ruthenium target material. In the HPGe spectrum, the Rh and Ru K x rays were unresolved so, in order to determine the contribution from these florescence x-rays, we also collected spectra with a small Si(Li) detector, whose energy resolution was sufficient to enable us to separate the two. As a result we concluded that the contribution of the fluorescence x rays was 3.10(5)% [7].

Analysis is not yet complete.

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