

^{93m}Nb target characterization for K-shell internal conversion coefficient measurements

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Reliable nuclear decay schemes are important in fundamental physics, which also include information on internal conversion coefficients (ICC). This work is aimed at a target characterization that was specifically prepared for ICC measurements of ^{93m}Nb. This element represents the lowest atomic number and transition energy among those studied for K-shell ICC evaluation.

The molecular electroplating technique was successfully utilized in a series of target preparations for the ICC measurements performed in our group [1]. The same approach was used for the Nb target fabrication. To develop the Nb electrodeposition procedure, a non-radioactive inductively coupled plasma mass spectrometry (ICP-MS) standard niobium solution (1 mg/mL in nitric acid) was used. Isopropanol was chosen as the organic solvent for the target preparation. A 170 μ L aliquot of the Nb standard solution was evaporated down to \sim 50 μ L, diluted in 10 mL of isopropanol and transferred to the target cell. The Nb electrodeposition on an aluminum disk (2.275 cm², 10 μ m thickness) was performed for 40 min with a stepwise increasing bias in the range from 500 V to 700 V. The prepared source (\sim 50 μ g air dried, shown in Fig. 1) was characterized at the Texas A&M University Materials Characterization Facility by means



Fig. 1. Picture of Al-disk after stable Nb electrodeposition.

of scanning electron microscope (SEM), energy dispersive spectroscopy (EDS), and x-ray photoelectron spectroscopy (XPS), as well as by visual inspection of the color of deposited layer. Results of these analyses are shown in Figs. 2–4 and Tables I–II. Despite the presence of nitric acid, the EDS and XPS analyses showed that Nb was not electrodeposited as a corresponding nitrate salt. This conclusion is in agreement with literature data on electrodeposition of the metal [2-4]. However, trace amounts of HF in the ICP-MS standard lead to formation of a very stable oxyfluoro complex, NbOF_3 [5], which was most likely deposited along with niobium pentoxide, Nb_2O_5 . Increasing the Nb content (1.3 mL aliquot of the Nb standard solution) and electrodeposition at an average current of 9.5 mA for the first 4 h followed by 3 h more at constant 800 V resulted in an overall electroplating yield of ~50 % [6]. The Nb concentration in the isopropanol solution was determined by neutron activation analysis.

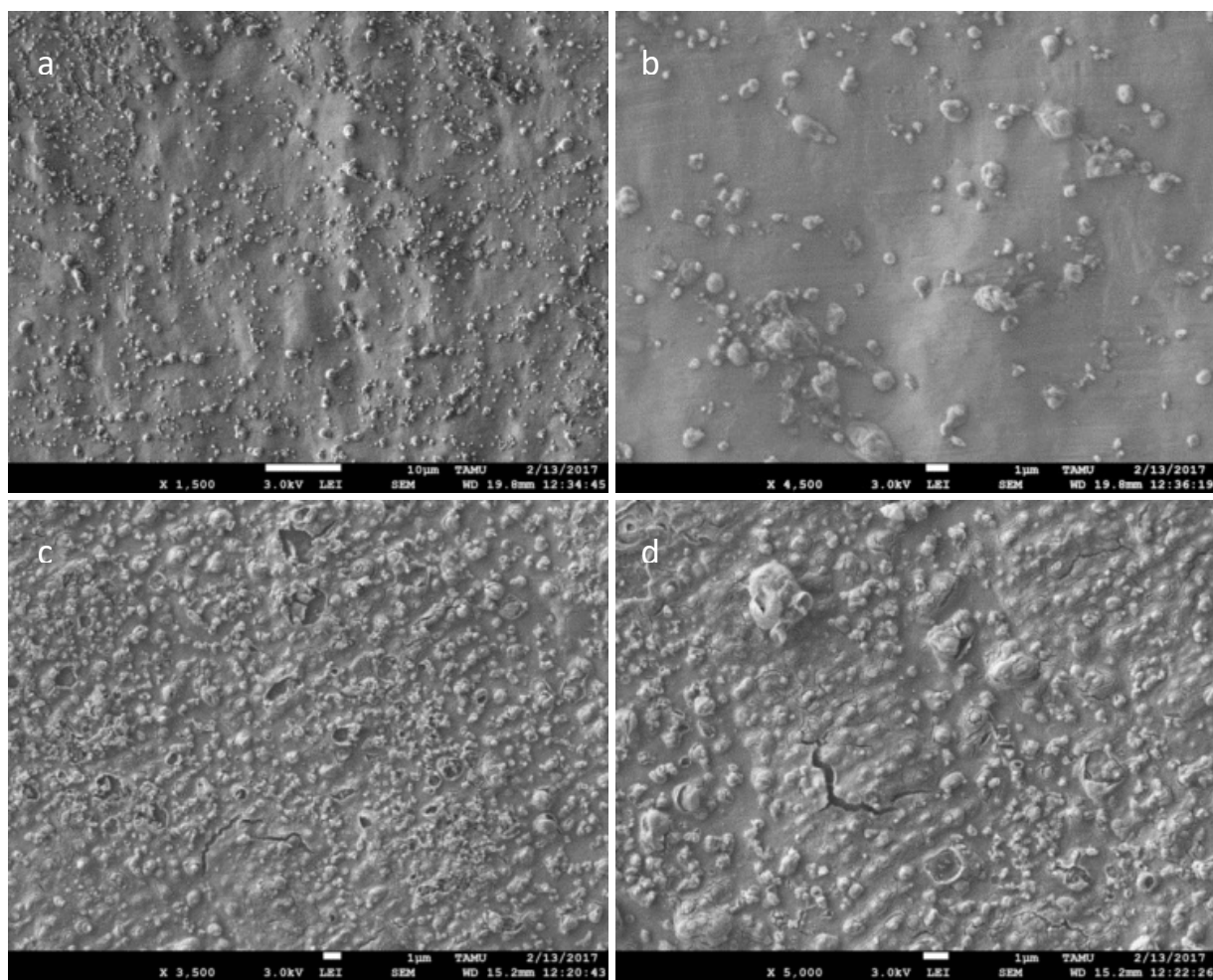


Fig. 2. SEM images of the Al disk at a) x1500 and b) x4500 magnification after bias was applied when pure isopropanol (no stable Nb) was in the target cell; SEM images of Al-disk at c) x3500 and b) x5000 magnification after bias was applied when isopropanol and Nb ICP-MS standard were in the target cell.

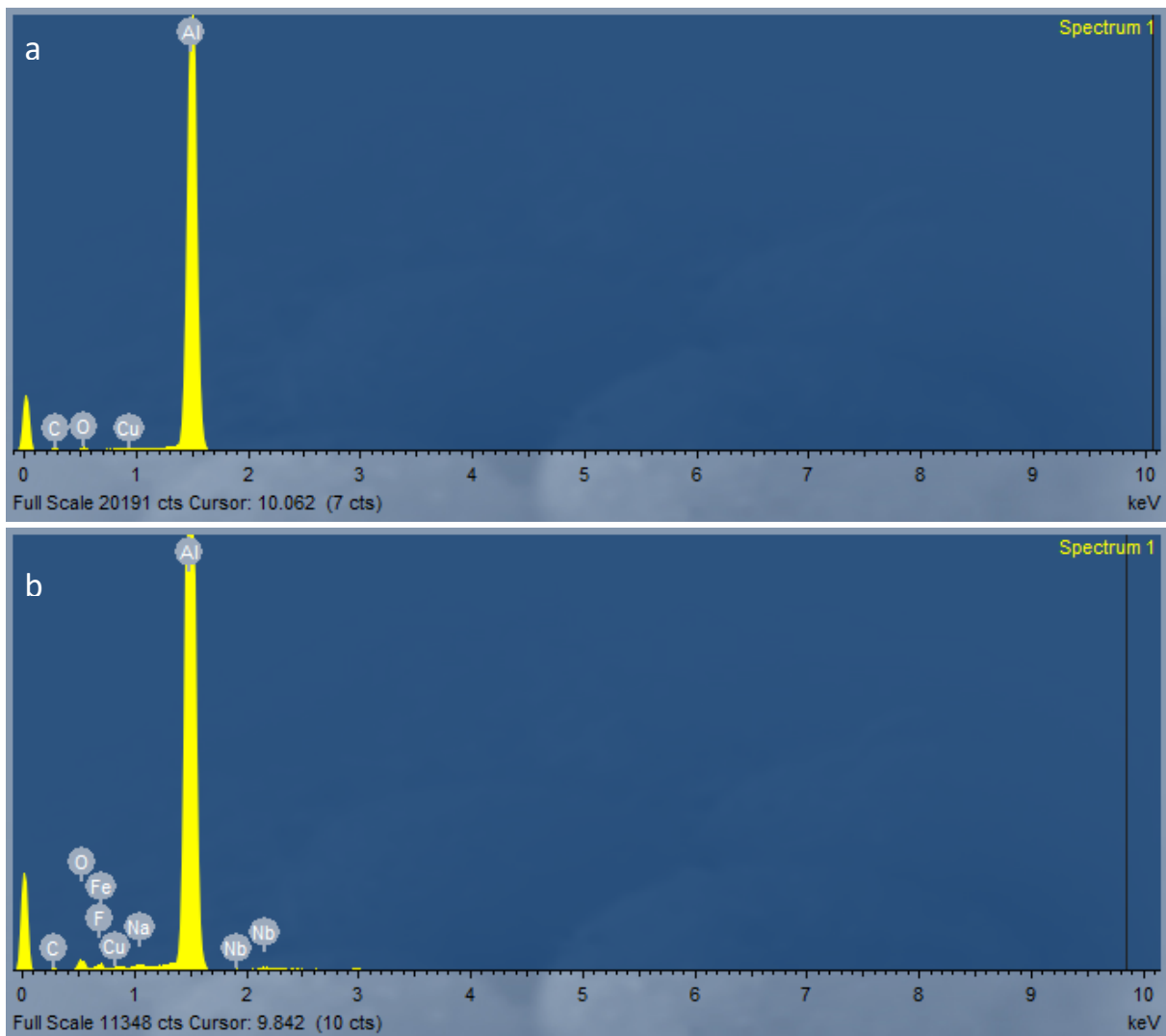


Fig. 3. EDS spectra of the Al disk corresponding to: a) SEM Fig. 2a [isopropanol (no stable Nb) was in the target cell] and b) SEM Fig. 2c (isopropanol and Nb ICP-MS standard were in the target cell).

Table I. EDS report attributed to the spectrum shown in Fig. 3a.

Element	Approximate Concentration	Intensity Correction	Weight%	Weight% Sigma	Atomic%
C K	10.54	0.1659	13.62	0.80	25.52
O K	10.84	0.5457	4.26	0.27	5.99
Al K	478.98	1.2506	82.12	0.79	68.49
Totals			100.00		

Table II. EDS report attributed to the spectrum shown in Fig. 3b.

Element	Approximate Concentration	Intensity Correction	Weight%	Weight% Sigma	Atomic%
C K	4.58	0.1640	6.17	0.75	12.21
O K	22.07	0.6155	7.92	0.30	11.77
F K	5.89	0.3998	3.26	0.21	4.07
Na K	3.12	1.4120	0.49	0.05	0.50
Al K	433.10	1.1863	80.67	0.73	71.06
Nb L	3.47	0.5135	1.49	0.19	0.38
Totals			100.00		

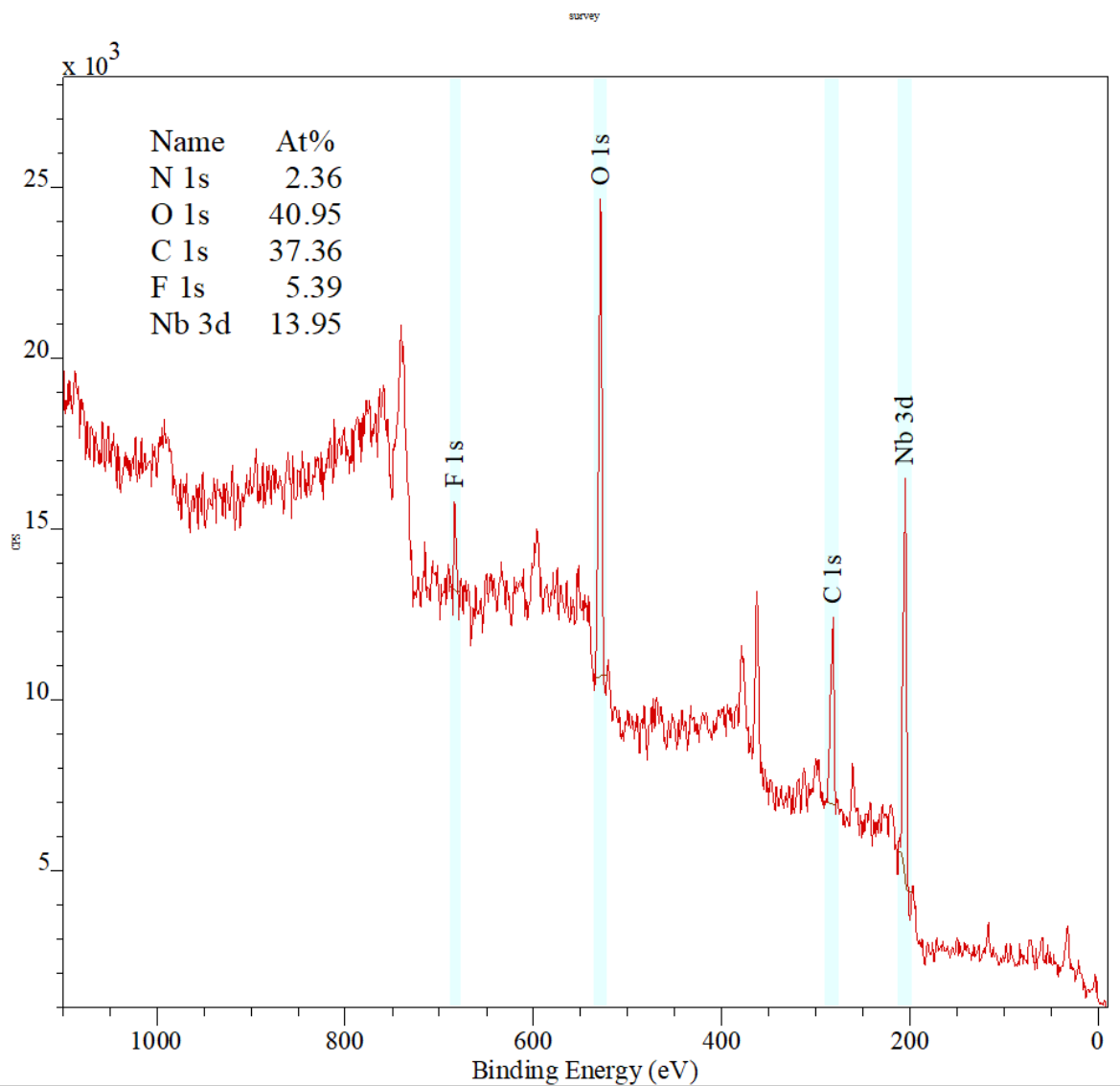


Fig. 4. XPS spectrum of Al-disk after stable Nb was electrodeposited from isopropanol.

After the preparatory step was finished, we purchased 0.85 mL of ^{93m}Nb (40 μCi) dissolved in 1 M HNO_3 + 0.3 M HF solution from Eckert and Ziegler Products, Atlanta, Georgia, USA. A small aliquot (10 μL) of the initial ^{93m}Nb solution was used to spike the solution in the target cell. During the electrodeposition, isopropanol aliquots were taken from the target cell and measured by an NaI γ -counter to simplify the control of electroplating. The efficiency of the Nb electrodeposition was optimized at a current density of 3-5 mA/cm^2 , reaching $\sim 95\%$ for 2 h in the case of relatively low carrier concentration.

The next step was to check the content of stable materials in the purchased ^{93m}Nb sample. For this purpose, an ICP-MS measurement with the radioactive sample was performed at Texas A&M University. The results (Fig. 5) show that the purchased ^{93m}Nb source had 4.1 ± 0.2 mg/mL of stable Nb carrier along with trace amounts of Sb and Ta that usually accompany stable Nb and most likely were added to the solution with the former. Also, 0.43 ± 0.04 $\mu\text{g}/\text{mL}$ ^{238}U were found in the sample.

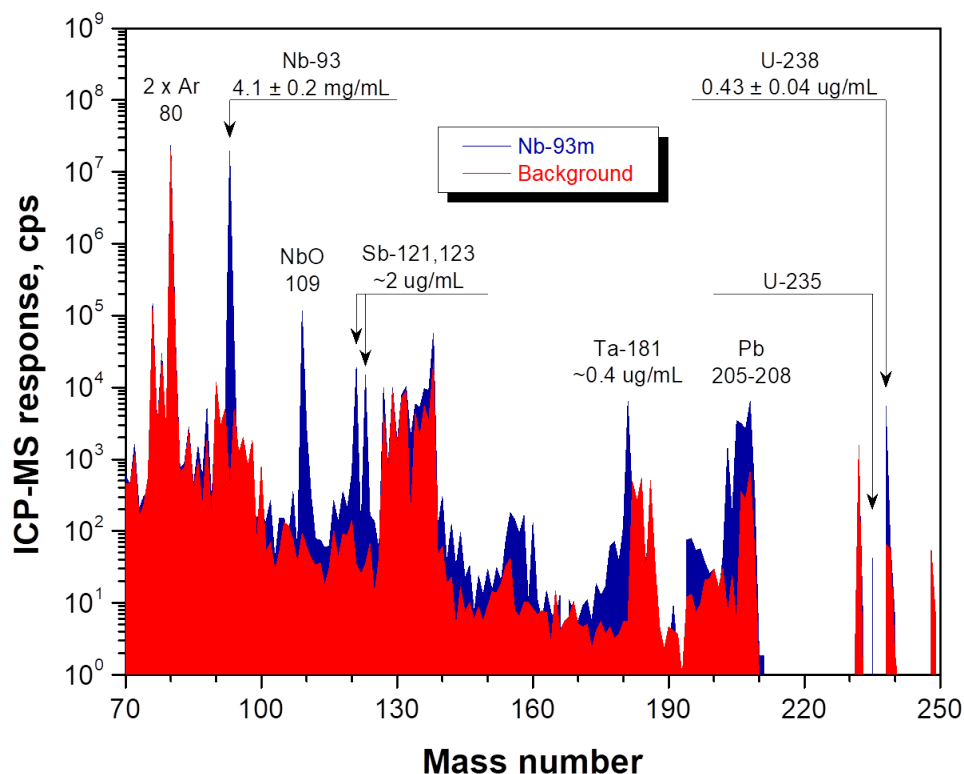


Fig. 5. ICP-MS spectrum of the ^{93m}Nb aliquot.

Finally, we prepared a reliable and convenient ^{93m}Nb source for the ICC measurement. The ICP-MS standard was not used in these experiments. An aliquot of radioactive solution was dried down to a small drop in a Teflon beaker using a heating plate and then transferred to the target cell. The electrodeposition method we employed is limited to 1-2 mg/cm^2 of material deposited. It was decided not

to exceed this layer thickness in order to avoid flaking, cracking, and/or peeling of the film. The procedure to achieve the most active source took 15 h at ~10 mA resulting in a deposition efficiency of ~50 % [7].

The mass of the deposited layer (3.0 mg) was determined by weighing of the backing foil before and after deposition. The use of this foil for ICC measurements is described in another paper submitted to Phys. Rev. C (2020).

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