Accurate Mössbauer line-shape measurements including interference in ^{182}W, ^{183}W, ^{191}Ir, and ^{159}Tb

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We have made precise final state interference measurements using very intense Mössbauer sources and an exact line-shape analysis. Two transitions in tungsten metal, as well as one in iridium metal and one in terbium oxide, have been studied. The interference parameters 100β found are $-1.3(3)$ for the 99.1-keV transition in ^{183}W, $-1.2(1)$ for the 100.1-keV transition in ^{182}W, $-0.71(8)$ for the 129.4-keV transition in ^{191}Ir, and $-0.48(5)$ for the 58.0-keV transition in ^{159}Tb. All values agreed with theory to within error. Our measurement of 1.16(2) cm/s for the ^{191}Ir linewidth agrees with other Mössbauer measurements and shows no indication of hyperfine broadening, but disagrees with some non-Mössbauer measurements reported in the literature. The tungsten and terbium transitions also show no signs of broadening or splitting.

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I. INTRODUCTION

We measured the fundamental line-shape parameters using the Mössbauer effect for the 99.1-keV transition in ^{183}W, the 100.1-keV in ^{182}W, the 129.4-keV in ^{191}Ir, and the 58.0-keV transition in ^{159}Tb. We were particularly interested in the Mössbauer interference parameter for transmission experiments, β, which appears in the resonance absorption cross section and introduces an asymmetry to the otherwise symmetric Mössbauer line shape [Eq. (1)]. The interference parameter arises from final state interference between nuclear and inner electron shell interactions [1–3]. Few existing interference measurements are accurate, due primarily to the use of Lorentzian line-shape approximations which fail to correctly account for finite absorber thickness and hence lead to incorrect values of β [2]. There also is a shortage of experimental confirmation of measured β values, due perhaps to the difficulty of measuring the asymmetric component of the Mössbauer line shape which is usually only a few percent of the total line-shape peak height. Also, where more than one β measurement exists, there is frequently disagreement between the measured values.

Failure to properly take the asymmetry produced by β into account in Mössbauer experiments can result in incorrect line position values (isomer and second-order Doppler shifts) being returned from least-squares fitting routines. This is due to the least-squares routines' "attempt" to compensate for the asymmetry by moving the center position.

The value of β is also of importance to time-reversal invariance (TI) violation experiments of electromagnetic radiation arising from nuclear decay. These TI experiments attempt to measure the multipole mixing phase η, the value of which determines if TI has been broken [4, 5]. What is actually measured, however, is $\eta + \beta_T$, where β_T is the TI interference parameter, and no means have yet been found to measure either η or β_T independently. The practice has been to use theoretical values of β_T [1, 2], which come from the same theories from which the Mössbauer transmission interference parameter β is derived. Accordingly, measurements of β allow a means of testing the theories used to calculate β_T, allowing one means of assessing the degree of confidence which should be placed on the β_T values and hence on the TI experiments of which they are an integral part.

The β value of the 129.4-keV transition in ^{191}Ir is of particular importance since this isotope has been used in TI experiments in the past [6]. This interference value was found earlier by Bullard, Mullen, and Schupp [7]. Because of the importance of this measurement to time-reversal invariance (TI) experiments [6] and the general lack of experimental confirmation of interference values, we chose to redo this measurement and attempt to reduce the error of $\beta = -0.0077(10)$ found in the experiment of Bullard, Mullen, and Schupp.

II. EXPERIMENTAL PROCEDURE

A schematic of the experimental setup [8] used in the tungsten and iridium experiments is shown in Fig. 1. The (200) reflection of LiF was used as a monochromator, removing unwanted photons and hence background from the beam incident on the absorbers. In the terbium experiments the source was Doppler shifted rather than the resonance absorber. The low source intensity in the terbium experiment required dispensing with the LiF crystal and using a direct beam geometry, in which source, absorber, and detector all lay on the same line. All ra-

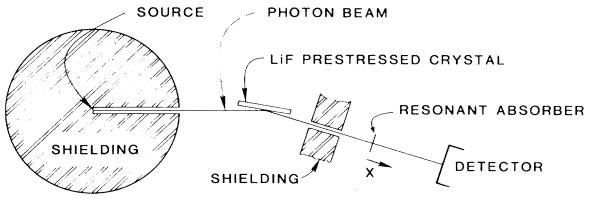

FIG. 1. Schematic drawing of experimental setup.

dioactive sources used were prepared at the University of Missouri Research Reactor facility, at a nominal flux of 3×10^{14} neutrons/(cm^2 s). All the sources used were kept at $\approx$77 K throughout the duration of the experiments. All transitions considered here were found to be single line source and absorber systems, with no evidence of split or broadened lines.

We used ORETC thin Ge detectors, with an energy resolution in the neighborhood of 300 eV for the experiments described in this paper. The count rates varied for different sources, but were usually kept below 20 000 counts/s to minimize dead time corrections. Dead time corrections were applied to the ^{191}Ir data, but they did not alter the results to within experimental error.

The procedures for curve fitting follow our earlier work [7,9], and at the end of the first of these papers the reader will find a summary of our definitions and notation.

The Mössbauer data was fit to the true line-shape expression which required no correction for finite absorber thickness, given by [7, 9]

$$C(x) = C_0\left\{1 - f_s + \frac{2f_s}{\pi} \times \int dx' \frac{\exp[-t(1-4\beta x)/(1+4x'^2)]}{1+4(x'-x)^2}\right\}, \quad (1)$$

with $x = E_0(v - v_0)/\Gamma c$ and $x' = (E - E_0)/\Gamma$, where E is the γ-ray energy which we integrate over, Γ is the Heisenberg level width given by $\Gamma\tau = \hbar$, and τ is the mean life of the excited state. The level width is often given in velocity units given by

$$\Gamma_v = \frac{\Gamma c}{E_0},$$

where c is the speed of light and E_0 is the transition energy of the source nuclei. The remaining parameters are v_0, which is the peak position; C_0, which gives the number of off-resonant counts/channel; f_s, the resonant γ-ray fraction of the source beam incident on the absorber; and the Mössbauer thickness number $t = f_a\sigma_0 n$, where f_a is the absorber recoilless fraction, σ_0 is the maximum resonant absorption cross section, and n is the number of Mössbauer isotopes per area in the absorber. The transmission interference parameter β is the value of primary concern to us and can be written as [1, 3]

$$\beta = \beta_C + \beta_R,$$

where β_C arises from interference between resonant γ-ray absorption followed by internal conversion and inner-shell photoelectric absorption, and β_R arises from interference between nuclear resonant scattering and elastic Rayleigh scattering off the inner electrons.

In the tungsten and iridium experiments we collected two Mössbauer spectra using absorbers with a known 1:2 ratio in their thickness number. In the terbium case, the same physical thickness absorber was used, but one run was performed at room temperature and the other at around 85 K. The two spectra were fit simultaneously with known constraints (the 1:2 thickness constraint in the W and Ir cases, constant Γ and β) in order to reduce errors which result from the high correlation between parameters. In each case the residual plot of the data points minus the best fit values found from simultaneous fitting was a random distribution of points about zero, indicative of a good fit.

We should also note nonlinear least-squares fits were made in which the Mössbauer spectrum was assumed symmetric, that is, where $\beta = 0$. These fits returned reduced χ^2 values greater than 2 for both the individual data files and the simultaneous fit to the data files. This clearly indicates asymmetry in the data. That this asymmetry is due to interference is supported by the fact that even with different drive motors, and frequently reworked reciprocation devices and a variety of electronic data collection devices, no system asymmetry was found. In fact, a variety of 46.5-keV tungsten experiments done over a period of several years [7,10] have consistantly returned the same value for β. When only the symmetric part of the line shape is subtracted from the data, the resulting asymmetric line is as expected and is similar to cases we have shown in earlier papers [11].

A. Tungsten

The sources used for the 99.1-keV and 100.1-keV tungsten lines were made from strips of 0.25 in. $\times$1.25 in. natural tantalum foil 0.003 in. thick. The 99.1-keV and 100.1-keV lines were too close in energy to be completely removed by Bragg scattering from LiF, and so to minimize overlap the SCA (single channel analyzer) window was skewed toward the higher-energy side in the 100.1-keV case and toward the low-energy side in the 99.1-keV case.

In the 100.1-keV case the tantalum foil was left to stand for approximately 4 weeks after irradiated to allow the initially more intense 5.1-day half-life ^{183}Ta 99.1-keV lines to decrease relative to the lines of the 115-day half-life ^{182}Ta isotope of interest. Only one source was used throughout the experiment. As this 99.1-keV "background" diminished, the effective source fraction f_s was found to increase by $\approx$7%, being greatest in the first week and leveling off with time as expected with exponential decay. We were able to divide the 40 days worth of data into periods where f_s could be held constant without distortion of the fit parameters. We note that if f_s is held to be constant throughout the experiment, we get $\Gamma_v = 0.108(4)$ cm/s, $t = 3.7(3)$, and $\beta = -0.013(1)$, values within two standard deviations of the values given in Table I. The agreement in β is particularly reassuring and reflects the weak correlation between interference and the heavily correlated triad of f_s, Γ_v, and t.

TABLE I. Results for 100.1 keV and 99.1 keV transtions in ^{182}W, and ^{183}W, respectively.

Property	100.1 keV	99.1 keV
β	-0.012(1)	-0.013(3)
Γ_v	0.099(5) cm/s	0.190(3) cm/s
v_0 [77(1) K]	-0.001(1) cm/s	-0.004(3) cm/s
f_a [77(1) K]	0.15(2)	0.145(11)
t	4.7(6)	0.82(6)

The 99.1-keV sources had an estimated ^{183}Ta intensity of 50–100 Ci. Newly irradiated sources was used at the beginning of each week to ensure good count rate. A total of five sources was used. The effective source recoilless fraction in the 99.1-keV experiment was found to vary only slightly during the course of the week, and the weekly average was the same for all the sources. This allowed us to restrain the source fraction to be constant throughout the experiment. The origin of this small variation is due to the variation of apparent nonresonance gammas, since the downscatter into the SCA window depended on details of the source age after neutron irradiation, the total irradiation time, and the settings of the SCA window. Also, the source temperature is about 2 K hotter for a fresh source than a 1-week-old source, due to the finite rate of heat transfer for these ultraintense Mössbauer effect (ME) sources. Thus, the effective source fraction is approximately the true recoilless fraction, but we have used the term "effective" to account for these correction terms. Thus, the effective source fraction is the fraction of resonant gamma rays in the beam incident on the Mössbauer absorber, rather than the fraction of transitions in the source that are of the zero phonon type [7].

All tungsten absorbers consisted of natural tungsten foils of 99.95% or better in purity. Tungsten metal is nonmagnetic and body-centered cubic. The foils were all cut from the same larger sheet to ensure uniformity in thickness of 26.7(4) μm. The 1:2 ratio on thickness corresponds to three foils for an absorber thickness of 80(1) μm and to six foils for a 160(2) μm thickness. The tungsten absorbers were placed in a LN_2 Dewar and kept at 77(1) K, and the temperature was monitored throughout the experiment. The data for the 100.1-keV case are shown in Fig. 2, and those for the 99.1-keV case are shown in Fig. 3.

B. Iridium

The sources consisted of 330 mg of natural osmium powder with a ^{190}Os abundance of 26.4%, pressed into a cavity 3.0 mm × 25.0 mm and 0.88 mm deep. The irradiation resulted in an activity of ≈15 Ci for the ^{191}Os. Osmium metal has a hexagonal close-packed structure and so has nonsymmetric sites which might give rise to an electrical quadrupole splitting. Our least-squares-fit results, however, showed no sign of any measurable broadening.

All absorbers were composed of 241.9(3)-μm-thick natural iridium foils 99.95% or greater in purity. Iridium metal is nonmagnetic and face-centered cubic. The two

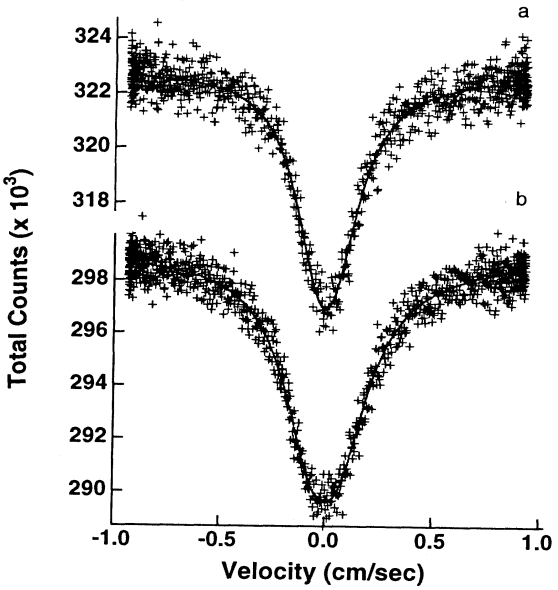

FIG. 2. Mössbauer spectra and best fit for 100.1-keV ^{182}W. Absorbers were at 77 K with thickness (a) 80(1) μm and (b) 160(2) μm.

different absorber thickness used were 241.9(3) μm and an absorber twice this thickness. The absorbers were placed in a continuous-flow helium Dewar and kept at a 4.4(2) K. The data are shown in Fig. 4.

C. Terbium

The source was prepared from 20.8% ^{158}Dy enriched Dy_2O_3 powder. The ^{158}Dy has a very large burnout cross

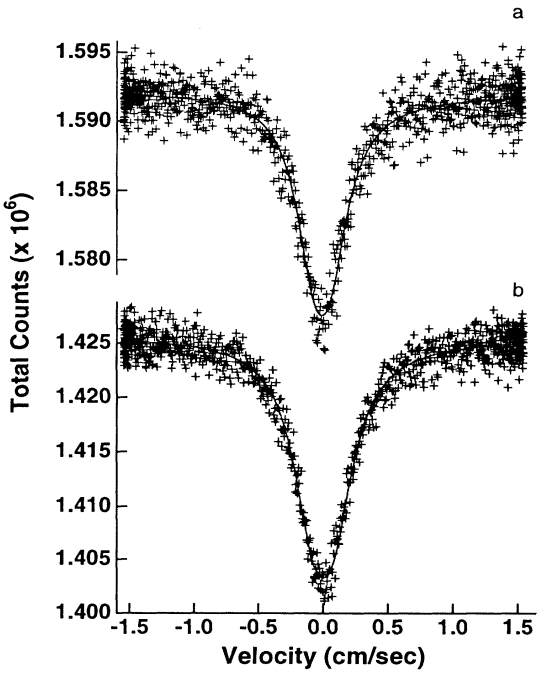

FIG. 3. Mössbauer spectra and best fit for 99.1-keV ^{183}W. Absorbers were at 77 K with thickness of (a) 80(1) μm and (b) 160(2) μm.

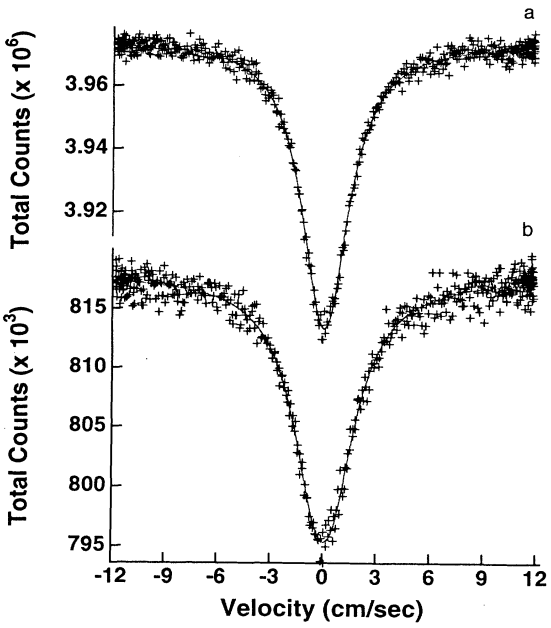

FIG. 4. Mössbauer spectra and best fit for 129.4-keV ^{191}Ir. Absorbers were at 4.4(2) K with thickness of (a) 241.9(3) μm and (b) 483.8(4) μm.

section [12], and so multiple curie sources could not be made. The one used in the present work was estimated to have an intensity of around 55 mCi. The 144.4-day half-life of ^{159}Dy allowed the use of one source throughout the experiment.

The absorber was a fine powder of 99.99% pure Tb_4O_7. The powder was mixed with transoptic powder and then heated under pressure to form a plastic "pill," 1.25 in. in diameter. This absorber pill contained 73.1 mg/cm^2 of Tb_4O_7 which corresponds to 62.2 mg/cm^2 of terbium. Only one absorber was used, and Mössbauer spectra were taken at 297(2) K and at 85(2) K. The data are shown in Fig. 5.

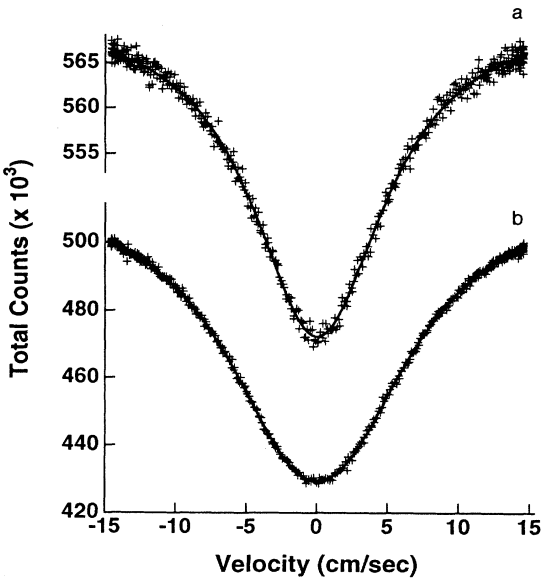

FIG. 5. Mössbauer spectra and best fit for 58.0-keV ^{159}Tb. Absorber was Tb_4O_7 with 62.2 mg/cm^2 of terbium at (a) 297(2) K and (b) 85(2) K.

III. RESULTS AND DISCUSSION

A. Tungsten

The results of our 100.1-keV and 99.1-keV experiments are given in Table I. The reduced χ^2 of the 100.1-keV fit was 0.96, and that for the 99.1-keV was 1.00. The value of t in the table corresponds to the 80(1)-μm tungsten metal absorber at 77(1) K.

Our values for Γ_v are in good agreement with other Mössbauer values such as 0.1045(50) cm/s and 0.200(7) cm/s listed in the Mössbauer Effect Data Index [13] and the values of 0.0999(7) cm/s and 0.193(5) cm/s listed in the Nuclear Data Sheets [14, 15] for the 100.1-keV and 99.1-keV transitions, respectively.

Using the Debye-model expression for recoilless fraction [16, 17] and our measured f_a values, we get a Debye temperature (θ_D) for tungsten metal of 345(18) K using the 100.1-keV value and of 342(10) K using the 99.1-keV value. Both are in good agreement with our earlier reported value of 336.5(7) K found using the 46.5-keV line in ^{183}W [7] and with the value of 344(10) K based on reported absorber recoilless fractions found in a separate 46.5-keV tungsten experiment done earlier [10]. The average f_s was 0.037(1) in the 100.1-keV case and 0.034(2) for the 99.1-keV case, implying a θ_D for tantalum metal of 249(2) K and 242(3) K, respectively. An earlier reported value of f_s for the 46.5-keV tungsten system [10] resulted in a Debye temperature for tantalum metal of 235(2) K. The agreement between measurements here is not as good as that for tungsten metal. This is to be expected since what is measured is the effective source recoilless fraction rather than the true source fraction, which can vary due to different source irradiation times, choice of SCA windows, and small fluctuations in source temperature. When these factors are taken into account the agreement is in fact encouraging and yields an average value of $\theta_D = 242(1)$ K for tungsten impurities in tantalum metal.

The nearness of the 99.1-keV and 100.1-keV lines meant that possible overlap leading to dual resonance absorption may have occurred. Our low χ^2 values of 0.96 and the good agreement between our results and other measurements indicate that any such effect was small at best. Since the production of ^{183}Ta was about 10 times that of ^{182}Ta, the 99.1-keV line was 10 times as great as the 100.1-keV line. When we studied the 100.1-keV transition we used old sources where the contribution of the 99.1 component was small, since the ^{183}Ta half-life is 5 d while that of ^{183}Ta is 115 d. Our low reduced χ^2 value of 0.96 and our agreement with earlier measurements of this width [14] indicates that any errors due to overlap are small.

Our interference values for the 99.1-keV and 100.1-keV transitions agree well with each other, which in turn agree with the theoretical prediction that they are equal. The

two theoretical interference values were −0.0117 [1] and −0.0122 [2], which are in agreement with our measured values to within experimental error.

B. Iridium

Table II gives the best fit parameters for both the previous experiment of Bullard, Mullen, and Schupp [7] and the present experiment. The need to further increase accuracy leads to our simultaneous fitting the earlier data with the current data. The results are given in the last column and are our best current values for the 129.4-keV ^{191}Ir system. The reduced χ^2 for this joint fit was 1.03. The thickness number t in the table corresponds to a 483.4(4)-μm-thick absorber in the 80(1)-K case and to a 241.9(3)-μm-thick absorber in the 4.4(2)-K case.

Our value for Γ_v of 1.16(2) cm/s is in good agreement with other Mössbauer measurements of 1.19(1) cm/s [18] and 1.12(2) cm/s [19], as well as that found in the Mössbauer Effect Data Index [13] of 1.19(3) cm/s. The most recent value published in the Nuclear Data Sheets [20] is 0.86(3) cm/s. This value was a weighted average of various techniques not including Mössbauer measurements. The reason given was that broadening may exist in the Mössbauer data measurements, hence yielding a Γ_v larger than the true value. Our fits assumed no broadening and the results indicated no such effect. If the true linewidth was 0.86(3) cm/s broadened to 1.16(2) cm/s, we would expect that this would have been clearly evident in our reduced χ^2 and associated errors. Also, hyperfine broadening is usually temperature dependent and we might expect different line widths to be measured at 4.4 K and 80 K. But the value for line width using just the 4.4-K data agrees well with that using just the 80-K data. Accordingly, if any broadening effects are present, we believe them to be negligible in magnitude.

The value of $f_s = 0.042(1)$ is 15% larger than that of the the previous 80-K work. This was due to the greater background problem that existed in the previous experiment, done in direct beam where down scatter contributed a larger apparent nonrecoilless fraction to the incident beam. The present value in which a LiF monochromator was employed is therefore closer to the true source recoilless fraction.

The value for the interference parameter of −0.0071(8) is in good agreement with the theoretical values of −0.0071 [1] and −0.0069 [2], but has a large relative error of 11%, making it unsuitable for deciding on questions about the magnitude of interference effects in TI experiments [4–6].

TABLE II. Results for the 129.4-keV transition of ^{191}Ir from previous 80(1)-K [12] and present 4.4(2)-K experiments. Last column is simultaneous fit to both experiments.

	80(1) K	4.4(2) K	Joint fit
β	-0.0077(10)	-0.0060(12)	-0.0071(8)
Γ_v	1.15(4) cm/s	1.16(2) cm/s	1.16(2)cm/s
v_0 (80 K)	-0.009(7) cm/s		-0.013(6)cm/s
v_0 (4.4 K)		0.010(9) cm/s	0.005(8)cm/s
f_a (80 K)	0.036(5)		0.035(4)
f_a (4.4 K)		0.071(8)	0.071(8)
t (80 K)	2.6(4)		2.5(2)
t (4.4 K)		2.5(2)	2.5(2)

TABLE III. Results for 58.0 keV transition in ^{159}Tb.

Property	Measured value
β	-0.0048(5)
Γ_v	4.5(1) cm/s
v_0 [297(2) K]	0.002(13) cm/s
v_0 [77(1) K]	-0.004(13) cm/s
f_a [85(2) K]	0.230(14)
f_a [297(2) K]	0.019(10)
t [85(2) K]	4.9(3)
t [297(2) K]	0.4(2)

C. Terbium

The values returned from our simultaneous fit to the two temperatures are given in Table III, where the reduced χ^2 was 1.04. The t values correspond to the same absorber with 62.2 mg/cm^2 of terbium.

Although Tb_4O_7 is listed as cubic, the actual structure is unknown and the Tb_4O_7 formula is actually a stoichiometric average. Nowik [21] has pointed out that there may be three nonequivalent sites in this oxide, which introduces the possibility of broadening. Our resulting fit parameters, however, indicated only a single unbroadened line of the same magnitude found in earlier work on $TbAl_2$ which is known to be cubic at the Tb site [22].

Values for the level width based on earlier Mössbauer spectroscopy have resulted in values that range from 2.2(4) cm/s [23] to 7.9(9) cm/s [24]. The most recent value (other than those of our group) is 4(1) cm/s [25]. These values suffer primarily from ill-resolved Mössbauer spectra which frequently were collected in very few channels as a means of improving the signal-to-noise ratio. Also in each of these earlier measurements a Lorentzian approximation to the data was used, further leading to distortions. The value based on Coulomb excitation of 4.4(1) cm/s [26] and that found previously by the Purdue group in $TbAl_2$ of 4.40(3) cm/s [22] are in good agreement with our value of 4.5(1) cm/s. The value found in the Nuclear Data Sheets [27] is 4.4(1) cm/s. Even with a large burnout cross section we were able to make sources with significantly greater intensity than past Mössbauer groups, and this coupled with the use of the true lineshape expression allowed measuring the level width to an accuracy comparable to those found using other methods. The average effective source fraction was $f_s = 0.232(4)$. Our value for interference agrees with $\beta = -0.0058(7)$ found previous by our group in $TbAl_2$ [22]. We know of no reported theoretical values for the interference parameter.

IV. CONCLUSION

Our measured values for both iridium and terbium offer the first agreement between two experiments, both

using correct line-shape analysis. In the case of the 129.4-keV line of ^{191}Ir we were able to improve the percent accuracy on β slightly, but the relative error of 11% is still too large for deciding on questions about the magnitude of interference effects in TI experiments [5,6,28]. An earlier measurement of β for terbium was done by our group using $TbAl_2$ as the resonant absorber [22]. Theories of interference predict that the value of β should be highly independent of chemical environment of the isotope. Our current β measurement using Tb_4O_7 does in fact agree with the earlier aluminide value and is the second β measurement of the 58.0-keV terbium transition.

Our reported values for β in 99.1-keV and 100.1-keV tungsten are, to the best of our knowledge, the first reliable transmission interference values for these two transitions. Earlier values by our group [11] were found to suffer from vibration distortion, and even earlier measurements [19] used absorbers with thickness number around 2 but used the Lorentzian line-shape approximation to analyze their results, which as mentioned before is now known to return incorrect β values. Our results confirm, to within error, the theoretical prediction that β is the same for both the 99.1-keV and 100.1-keV transitions, since they have the same Z number, electron wave functions, and nearly identical γ-ray energies.

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