MÖSSBAUER EFFECT STUDY OF THE 105.3 keV TRANSITION IN $^{155}$Gd

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Mössbauer effect studies on the 105.3 keV transition of $^{155}$Gd using the single line source and absorber combination of SmPd$_3$ and GdCO$_2$ have shown that the previously reported narrowing in the linewidth is due to the formation of antipeaks on the spectrum. These antipeaks occur on the 105.3 keV spectrum at positions which correspond to maximum resonant absorption of the 86.5 keV radiation in $^{155}$Gd. They are observed at a very low amplifier output count rate of 6 kHz which corresponds to a multichannel analyzer recording rate of 200 counts per channel per minute for the 105.3 keV spectrum. The antipeaks were not present on the 105.3 keV spectrum after attenuating the 86.5 keV radiation with a thallium acetate filter. The resultant linewidth of the 105.3 keV transition, after correcting for finite source and absorber thickness, was found to be $\Gamma_0 = 1.129$ mms$^{-1}$ in comparison with the theoretical value of 1.112 mms$^{-1}$ calculated from the lifetime of the 105.3 keV energy level.

1. Introduction

Mössbauer effect studies of gadolinium compounds using the $^{155}$Gd isotope have been reported on several occasions over the last twenty years. This isotope has three Mössbauer transitions corresponding to the energies of 60.0 keV, 86.5 keV and 105.3 keV. The 86.5 keV transition is the most suitable since it has a narrow natural Mössbauer linewidth of $2\Gamma_0 = 0.499$ mms$^{-1}$, ($T_{1/2} = 6.33$ ns) and a high intensity. The 60.0 keV transition has a broad linewidth of $2\Gamma_0 = 23.5$ mms$^{-1}$, ($T_{1/2} = 0.194$ ns) and it is, therefore, using this transition, nearly impossible to obtain well resolved spectra for magnetic or quadrupole split samples. The 105.3 keV level has a half life of $T_{1/2} = 1.17$ ns and the natural Mössbauer linewidth is expected to be $2\Gamma_0 = 2.224$ mms$^{-1}$. Using this transition, it is difficult to obtain well resolved spectra because of the small recoilless fraction, even at liquid helium temperatures. The nuclear energy level diagram of $^{155}$Gd is shown in fig. 1. In the source, the short lived isotope of $^{154}$Sm decays to $^{155}$Eu which in turn populates the excited levels of $^{155}$Gd by $\beta^-$ emission. The three Mössbauer transitions are shown as $\gamma_{M1}$, $\gamma_{M2}$ and $\gamma_{M3}$. Table 1 summarizes the gamma transitions in $^{155}$Gd and also the relative intensities and multipolarities of the radiation.

Many of the previous Mössbauer studies using $^{155}$Gd, were involved with the determination of the magnetic and quadrupole moments of the ground and excited states [1–8]. Some of these studies [1–5] were however hampered by the lack of narrow single line sources and in several cases, only sources which were either quadrupole split or had a greatly broadened linewidth were available. As a result, there was a large variation in the reported values of the moments, most of which have been summarized in the earlier paper by Cook and Cashion [8]. Prowse et al. [9] first reported the use of two narrow single lines sources, EuPd and SmPd$_3$ and since that time, more consistent parameters have been published. On several occasions [3,6,8] it was reported that the linewidths obtained from fits to the spectra recorded using the 105.3 keV gamma radiation, were less than the natural Mössbauer linewidth of 2.224 keV.

![Radioactive decay scheme for $^{155}$Gd showing the three Mössbauer transitions $\gamma_{M1}$, $\gamma_{M2}$ and $\gamma_{M3}$. The excited states in the source are populated through the $\beta^-$ decay of $^{155}$Eu.](image_url)

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Table 1
Gamma ray transition energies and their relative intensities in $^{155}$Gd. The three Mössbauer transitions are $\gamma_1$, $\gamma_2$, and $\gamma_3$.

<table>
<thead>
<tr>
<th>Transition</th>
<th>Energy (keV)</th>
<th>Intensity</th>
<th>Multi-polarity</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\gamma_1$</td>
<td>18.8</td>
<td>0.16</td>
<td></td>
</tr>
<tr>
<td>$\gamma_2$</td>
<td>45.3</td>
<td>4.2</td>
<td></td>
</tr>
<tr>
<td>$\gamma_3$</td>
<td>26.5</td>
<td>1.0</td>
<td></td>
</tr>
<tr>
<td>$\gamma_{M1}$</td>
<td>60.0</td>
<td>3.6</td>
<td>M1</td>
</tr>
<tr>
<td>$\gamma_{M2}$</td>
<td>86.5</td>
<td>100.0</td>
<td>E1</td>
</tr>
<tr>
<td>$\gamma_{M3}$</td>
<td>105.3</td>
<td>66.9</td>
<td>E1</td>
</tr>
</tbody>
</table>

Linewidths between 0.64 mms$^{-1}$ and 2.25 mms$^{-1}$ have been reported and in most cases they have been determined from spectral fitting techniques without taking into account the effects of source and absorber thickness. In two previous reports [6,8] specific mention was made of this unusual observation. Armon et al. [6] attributed the narrowing to pile-up effects in the electronics if spectra are recorded using high count rates. This effect caused antipeaks in the 105.3 keV spectrum at velocities corresponding to a resonance in the 86.5 keV spectrum. They suggested that spectra should only be recorded at low count rates. Cook and Cashion [8] reported Mössbauer linewidths between 1.94 mms$^{-1}$ and 2.10 mms$^{-1}$ even when spectra were recorded with a low total count rate of 1600 counts s$^{-1}$ into the multichannel analyzer. They discounted the effects of pile-up, believing that this count rate was sufficiently low to allow the electronics to process the pulses efficiently.

This paper reports on the results of a further investigation into the cause of the line narrowing in the 105.3 keV spectra. It will be shown that the effect is due to the electronics, as suggested by Armon et al. [6], and that the antipeaks are observed on the 105.3 keV spectra even at very low count rates. It is also shown that satisfactory spectra can be recorded with the 105.3 keV radiation if the 86.5 keV radiation is attenuated before reaching the detector. Finally, mention is made of the consequences of subjecting commonly used Mössbauer electronics to periodic fluctuations in count rate, a phenomenon which is inherent in every Mössbauer experiment.

2. Experimental

Mössbauer spectra were recorded using a single line source of $^{154}$SmPd$_3$ and the single line absorber GdCo$_2$. The 51 mCi source was prepared by melting together stoichiometric amounts of $^{154}$Sm metal, (98.69%), and palladium metal, (99.999%), in an argon arc furnace. The alloy was annealed in an evacuated quartz tube for eight days at 900 C. The final mass was 416 mg. The alloy was then powdered for efficient emission of gamma rays. X-ray diffraction analysis confirmed that the structure was cubic and that the alloy was not deficient in samarium or palladium. After being sealed in a high purity cylindrical aluminum container, 0.95 cm in diameter, the material was irradiated for 46 hours in a thermal flux of $3 \times 10^{14}$ neutrons cm$^{-2}$s$^{-1}$. From the mass and density of SmPd$_3$, the actual thickness of the 0.95 cm diameter source, before being powdered, was $d_s = 0.058$ cm.

The GdCo$_2$ absorber was prepared by melting together stoichiometric amounts of gadolinium, (99.99%), and cobalt, (99.997%), metals in an argon arc furnace. The alloy was then annealed in vacuum for eight days at 900 C and X-ray analysis confirmed that no Gd$_2$O$_3$, which can easily form in gadolinium compounds, was present. The absorber was then finely crushed in a porcelain mortar. A total of 400 mg of the GdCo$_2$ was mixed with 100 mg of boron nitride binder and pressed under 10 tons into a cylindrical pellet of cross-sectional area 1 cm$^2$. This mass of GdCo$_2$ was chosen to give an absorber thickness [10] of $1/\mu_\gamma$, for the 105.3 keV spectra. The actual thickness of 400 mg of uncrushed GdCo$_2$, of density $= 8.6$ g cm$^{-3}$ and of area 1 cm$^2$ is $d_a = 0.047$ cm.

The effective absorber thickness, $t_a$, was determined using the standard equation [11]

$$ t_a = f_a n_a a_0 d_a, $$

where $f_a$ is the recoil-free fraction, $n_a$ is the number of element atoms per cubic centimeter, $a_0$ is the isotopic abundance, $a_\gamma$ is the Mössbauer resonance cross-section in cm$^2$, and $d_a$ is the thickness in centimeters. Table 2 summarizes the effective thicknesses of the source and absorber for the 86.5 keV and 105.3 keV radiation. Also included are the linewidths for the source, $\Gamma_s$, and absorber, $\Gamma_a$, calculated, by taking into account the effective thickness, using the standard equation [12]

$$ \Gamma_a = \Gamma_s (1.00 + 0.135 t_a). $$

$\Gamma_a$ is the absorber linewidth for an infinitely thin absorber.

Mössbauer spectra were recorded with the source and absorber inside a liquid helium cryostat. Both were maintained at 5.0 $\pm$ 0.2 K with helium exchange gas. The source, which was connected to the transducer by a drive rod 1 m in length, was driven in constant acceleration mode. The velocity calibration was determined using a $^{57}$CoRh source and either a 6 $\mu$m thick, 95% enriched or a 25 $\mu$m thick, natural iron foil absorber. Isomer shifts quoted in this paper are with respect to the SmPd$_3$ source at 5 K. The gamma rays were detected with a liquid nitrogen cooled p-type high purity germanium detector whose energy resolution for 122 keV $^{57}$Co radiation, of less than 0.5 keV allows the 86.5
Table 2

<table>
<thead>
<tr>
<th>Energy (keV)</th>
<th>Effective thickness</th>
<th>Linewidth (mm s⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Source £ₜₛ</td>
<td>Absorber £ₜₘ</td>
</tr>
<tr>
<td>86.5</td>
<td>0.29</td>
<td>2.36</td>
</tr>
<tr>
<td>105.3</td>
<td>0.18</td>
<td>0.36</td>
</tr>
</tbody>
</table>

keV and 105.3 keV radiation to be easily resolved. The maximum allowable energy rate into the detector, as specified by the manufacturer, was 2500 MeV s⁻¹. The pulses from the detector’s preamplifier were amplified by a high count rate, (up to 150,000 counts s⁻¹), amplifier. The output pulses with 0.5 μs rise time were input to a high stability single channel analyzer capable of resolving pulses in excess of 10⁶ counts s⁻¹. Spectra were recorded over 512 channels of the multichannel analyzer operating in MCS mode. Initially, some of the 86.5 keV and 105.3 keV spectra were recorded simultaneously on two subgroups of the MCA using a multiplexing system. For these experiments the amplified pulses were input to two single channel analyzers, each being set to allow either the 86.5 keV or 105.3 keV pulses to be output to the mixer-router multiplexing system. This arrangement of electronics conforms closely to the configuration used in the previous experiments reported by Cook and Cashion [8]. Finally, several spectra were recorded separately, without the use of the multiplexing system, and with one single channel analyzer which was set to allow either the 86.5 keV or 105.3 keV radiation to be counted by the MCA.

3. Results

Initially, Mössbauer spectra, using the 86.5 keV and 105.3 keV transitions, were recorded simultaneously using the multiplexing system. The results are shown in fig. 2a, b. The counts per channel per minute over the 512 channels of each subgroup of the MCA were 300 and 200 for the 86.5 keV and 105.3 keV spectra, respectively. The 86.5 keV spectrum, fig. 2a, shows a single line approximately 0.9 mm s⁻¹ in width. The 105.3 keV spectrum, fig. 2b, shows the presence of a small bump, at approximately 0.02 mm s⁻¹, on what is expected to be a broad single line. This bump corresponds to the position of maximum absorption of the 86.5 keV radiation. This characteristic was observed on all occasions when the spectrum was re-recorded. It was initially suspected that pile-up effects, as reported by Armon et al. [6], were occurring in the multiplexer and that this led to the observation of an antipeak in the 105.3 keV spectrum. This effect had been observed earlier in our laboratory when the multiplexer was used to simultaneously record two ⁵⁷Fe CEMS Mössbauer spectra from separate Mössbauer systems. As a result the multiplexer was removed and the rest of the spectra were recorded individually, at separate times. When the 86.5 keV and 105.3 keV spectra were recorded separately and at the same count rates as stated above, the same results, as shown in fig. 2a and 2b, were obtained. The 105.3 keV spectrum was recorded several more times as a function of count rate by moving the detector closer to the source. At no stage did the total energy input to the detector exceed the manufacturer’s specification of 2500 MeV s⁻¹. In fact, from studies of the output count rate of the amplifier, of approximately 6 kHz, it was determined that the energy input into the detector was about 600 MeV s⁻¹. It was found that as the count rate increased, so did the amplitudes of the antipeak which at all times corresponded to the position of maximum resonant absorption of the 86.5 keV radiation. Fig. 2c shows the result for the 105.3 keV spectrum recorded with a count rate of 660 counts per channel per minute. In fact a spectrum recorded at 920 counts per channel per minute resulted in an antipeak nearly reaching up to the background level of the spectrum. The counting statistics of fig. 2c are poor because the spectrum was only recorded for 40 hours. It was not our intention to study the antipeaks in detail, but merely to establish the fact that they were present in the 105.3 keV spectra.

These results, along with the previously mentioned observations of antipeaks formed when two Mössbauer spectra from separate experiments were simultaneously recorded, clearly indicate a possibility of pile-up effects in the electronics. The results show that the antipeaks are observable in the 105.3 keV spectra even when the total count rate into the MCA was as low as 4.3 kHz.

In order to study the effect of the 86.5 keV radiation on the shape of the 105.3 keV spectrum, a thallium acetate, Tl(OOCC₃H₇), filter 0.1 cm thick, was placed over the entrance window of the germanium detector. Thallium has an electronic K absorption edge [13] at 85.64 keV and is therefore suitable for attenuating the 86.5 keV radiation. The effective thickness of the thallium filter was 0.04 cm and this allowed the ratio of 86.5 keV to 105.3 keV gamma rays transmitted to be decreased from 1.47 to 0.29. In doing so however, the intensity of the transmitted 105.3 keV radiation was itself decreased to 10% of its original value. This decrease in count rate was compensated for by moving the detector closer to the source. A final count rate of 220 counts per channel per minute was recorded on the MCA. This was very close to the count rate used to record the spectrum of fig. 2b. The pulse height spectra of radiation emitted from the source are shown in fig. 3a, b. The spectrum of fig. 3a was recorded without the filter and fig. 3b shows the spectrum recorded with the
filter placed over the entrance window of the detector. Both spectra were recorded for 6 minutes. The 105.3 keV Mössbauer spectrum, recorded for 12 days with the filter over the detector, is shown in Fig. 4. No antipeaks were observed, substantiating that the radiation from the 86.5 keV transition, as processed by the electronics, does result in antipeaks on the 105.3 keV spectra. This matter is discussed further in section 4.

The 86.5 keV and final 105.3 keV spectra were fitted with a least-squares analysis incorporating the Gauss–Newton method of numerical integration. The program is the same one used to fit the spectra in the previously reported studies of single crystal and powdered samples of GdVO₄ and GdAlO₃ [14–15]. The program includes a choice of fitting using either the thin absorber approximation, or with the transmission integral methods [16] with the effective absorber thickness $\tau_a$, being an iterative parameter. This latter method also allows separate linewidths for the source, $\Gamma_s$, and absorber, $\Gamma_a$, to be used as input parameters. The transmitted intensity using the transmission integral method is of the standard form [12]

$$I(E) = I_0 \left[ 1 - \int_{-\infty}^{\infty} S(E, E_a) \exp \left[ -\tau_a A(E) \right] dE \right].$$

(1)

The lineshape of the source Lorentzian is given by

$$S(E, E_a) = \frac{2}{\pi \Gamma_s} \frac{\Gamma_s^2}{(E - E_a)^2 + \frac{1}{4} \Gamma_s^2}.$$

The absorption of the radiation is given by

$$A(E) = \frac{\frac{1}{4} \Gamma_a^2}{E^2 + \frac{1}{4} \Gamma_a^2} \left( 1 + \frac{2E}{\frac{1}{4} \Gamma_a} \right).$$
The dispersion term, $\xi$, arises from the interference between nuclear absorption followed by internal conversion and photoelectric absorption [17]. Its maximum amplitude is given by

$$\xi = \left( \frac{a_0 c}{6 \pi \lambda} \right)^{1/2}$$

where $a$ is the internal conversion coefficient, $c_0$ is the partial cross section for E1 photoelectric absorption and $\lambda$ is the wavelength of the incident $\gamma$ rays. For $^{155}$Gd, $c_0 = 57.8b$. For the 86.5 keV radiation, $\lambda = 0.144\AA$ and $a = 0.43$ and for the 105.3 keV radiation, $\lambda = 0.118\AA$ and $a = 0.26$.

The linewidths of the 86.5 keV and 105.3 keV spectra are broadened from their natural widths, $2\Gamma_0$, of $0.499\text{ mms}^{-1}$ and $2.224\text{ mms}^{-1}$, respectively. This broadening is due to the finite thickness of both the source and absorber and these parameters are accounted for in eq. (1). GdCo$_3$ is a collinear ferrimagnet [18] and the effective hyperfine field at the gadolinium site has been reported to be $H = +3.5\ T$ [19,20]. This field will also result in a small unresolved broadening of the spectra. Each of the two spectra were fitted using several different constraints in the program. Different combinations of the fitting parameters $\Gamma_0$, $I_0$ and $H$ were constrained for each fit. When constrained, the first two parameters were fixed at values calculated theoretically as shown in table 2. The value of the hyperfine field was constrained at either zero or 3.5 T as reported by Tomala et al. [20]. For each fit the ratio of the magnetic splitting of the excited and ground states was constrained to give [8]

$$g(86)/g(0) = +1.217\text{ and } g(105)/g(0) = -0.55.$$
Fig. 4. Fitted Mössbauer spectra recorded at 5.0 K with the (a) 86.5 keV and (b) 105.3 keV transitions. The 105.3 keV spectrum was recorded with a count rate per channel of 220 min\(^{-1}\) after attenuating the 86.5 keV radiation with a thallium acetate filter.

<table>
<thead>
<tr>
<th>Energy (keV)</th>
<th>IS (mms(^{-1}))</th>
<th>H (T)</th>
<th>(I'_x) (mms(^{-1}))</th>
<th>(I'_y) (mms(^{-1}))</th>
<th>2(\Gamma) (mms(^{-1}))</th>
<th>(\tau_x)</th>
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<td>86.5</td>
<td>0.024</td>
<td>0</td>
<td>0.892</td>
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<td>2.7</td>
<td>0.276</td>
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<tr>
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<tr>
<td>Theory 105.3</td>
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<td>3.5</td>
<td>1.166</td>
<td>1.129</td>
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<tr>
<td>Theory 105.3</td>
<td>3.5</td>
<td>1.166</td>
<td>1.112</td>
<td>0.36</td>
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</table>
The value of $\Gamma_0$ was not constrained in any fit. The results of some of the fits are given in table 3, with the values underlined showing that the parameter was constrained. From these combinations, the contribution of source and absorber thickness to the width of the Mössbauer lines has been determined and the natural linewidth of the source and absorber obtained. The fitted results for the 86.5 keV and 105.3 keV spectra are shown in fig. 4.

The results of table 3 show that, after taking into account the effects of broadening due to the source and absorber and also the presence of the small hyperfine field in GdCo$_2$, the source and absorber linewidths are very close to those predicted theoretically from the lifetimes of the 86.5 keV and 105.3 keV excited nuclear energy levels. The values of $\Gamma_0$ obtained for the 86.5 keV and 105.3 keV levels are 0.249 mm$^{-1}$ and 1.129 mm$^{-1}$, respectively. The interference parameter determined from the fits was found to be $\xi = 0.029$ for the 86.5 keV transition. The theoretical value was calculated to be 0.0314. However for the 105.3 keV transition, the value of $\xi$ consistently converged to zero for all fits, even though the input value was initially set at 0.03. This transition has been reported to be of multipolarity E1 and the theoretical value is expected to be 0.0299.

4. Discussion

The results of the previous section indicate that the linewidth of the Lorentzian lines in the 105.3 keV spectra are not narrowed. In fact, after taking into account the effects of broadening due to the source and absorber thickness, the linewidth obtained is very close to theoretical value. Even for the spectrum of fig. 2b which contained the antipeak, the linewidth does not appear to be narrowed. However the fact that the antipeak corresponds to the position of maximum resonant absorption of the 86.5 keV radiation, leads to a better understanding of why previously reported linewidths appeared to be narrowed. In comparing the 86.5 keV and 105.3 keV spectra of Blumberg et al. [3], Armon et al. [6] and Cook and Cashion [8], it can be seen that the peaks corresponding to maximum absorption of the 86.5 keV radiation lie close to midway between the broader, unresolved peaks in the 105.3 keV spectra. This occurs in two line quadrupole split spectra, for example, because the quadrupole moment of the 86.5 keV level is very small in comparison to that of the 105.3 keV level. As a result, the 86.5 keV resonant lines are always seen at velocities which are nearly midway between the resonant lines of 105.3 keV spectra. The antipeaks are therefore not clearly observable because they add to the regions of the 105.3 keV spectra which are already closer to the background level. This gives the appearance that the 105.3 keV resonant lines are better resolved and narrower than they really are. If however some of the resonant lines from both transitions were nearly coincident in velocity, the antipeak would be observable as was seen in fig. 2b, c. This can occur for magnetically split gadolinium compounds. Close inspection of the spectra of GdFe$_2$ reported by Armon et al. [6], shows, that for this magnetically split absorber, there is an indication of an antipeak in the 105.3 keV spectrum at zero velocity, corresponding to one of the resonant peaks in the 86.5 keV spectrum.

The cause of the antipeaks has been attributed by Armon et al. [6] to pile-up effects in the electronics. The results of this paper support this theory. Yet the antipeaks are observed at count rates far below those needed for appreciable pulse overlap. For the spectra recorded in this study, the total count rate of the combined 86.5 keV and 105.3 keV pulses at the output of the amplifier was 4.3 kHz. The total output of the amplifier due to all radiation being detected was approximately 6 kHz. These figures are far below the maximum count rate processing ability of each of the electronic modules used and it could therefore be concluded that pulse overlap would be negligible. However the fact that the size of the antipeak increases as the count rate increases and also observation that the antipeak is not present if the thallium filter is used to attenuate the 86.5 keV radiation, supports the argument that either pulse overlap occurs or the electronics cannot respond efficiently to the change in count rate due to the 86.5 keV resonance absorption. Since we have previously observed antipeaks when two Mössbauer spectra were recorded simultaneously from two separate Mössbauer systems, it is likely that the latter argument is true.

We have more recently undertaken a detailed study of this problem by simulating two Mössbauer experiments using frequency modulated signals from two function generators. One of these signals was triggered with the sweep frequency of the MCA. We have found that antipeaks are observed, again at very low count rates, as a result of the periodic change in count rate which is synchronized with the sweep of the MCA. It is suspected at the present time that single channel analyzers cannot respond linearly to the change in count rate and that because the dead time decreases as the count rate decreases, a proportionally higher count rate is observed in the second simulated Mössbauer spectrum. A detailed account of these findings will soon be submitted for publication.

5. Conclusions

Mössbauer studies using the 105.3 keV transition in $^{155}$Gd have shown that the previously reported narrowing in the linewidth occurs as a result of antipeaks formed on the spectrum at positions corresponding to
maximum resonant absorption of the 86.5 keV radiation. The effect can be observed at very low count rates and is attributed to a nonlinear response of the electronics as the count rate changes periodically. The effect was noticeable in the 105.3 keV spectra because the recoilless fraction for this transition is much smaller than that of the 86.5 keV transition. It has been shown that after attenuating the 86.5 keV radiation, no anti-peaks are observed. As a result, analysis of the 105.3 keV spectra has shown that after accounting for the effects of source and absorber thickness, the resultant Mössbauer linewidth is very close to that predicted theoretically from the lifetime of the 105.3 keV energy level.

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