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# Measurement of the half-life of excited nuclear states using liquid scintillation

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#### Abstract

This work presents measurements of the half-life of excited nuclear states of <sup>237</sup>Np and <sup>57</sup>Fe using a liquid scintillation (LS) spectrometer and a gamma detector. A novel approach for the determination of half-lifes is presented which uses only LS data from a detector with two PMTs. The life time of the 1<sup>st</sup> and 2<sup>nd</sup> excited states of <sup>57</sup>Fe were obtained without the use of a gamma detector. The half-life results from this study are consistent with the average value found in the reference decay data tables and have a lower uncertainty.

Keywords: Half-life measurements, LSC, Np-237, Fe-57, Liquid scintillation

#### 1. Introduction

LS counting is a powerful method for measurement of activity of  $\alpha$ ,  $\beta$  and electron capture (EC) emitting radionuclides and is widely used in laboratories around the world. It is particularly suited for the detection of low-energy emitters due to its relatively high detection efficiency and  $4\pi$  geometry. The measurements are usually performed with an aliquot of an aqueous solution, containing the radionuclide, which is dissolved in an organic scintillator and placed in a glass or a plastic vial. The scintillation light is then detected by two or more photomultiplier tubes (PMTs) working in coincidence.

LS counting has some specific features over other detection methods, namely the  $4\pi$  detection geometry and the practically 100% detection efficiency for energies over 40 keV [1]. Due to these factors the method is well suited for measurements of the half-life of radioisotopes. In recent years it was successfully used for the accurate determination of the half-lives of <sup>147</sup>Sm (1.08×10<sup>11</sup> years) [2], <sup>176</sup>Lu (3.8×10<sup>10</sup> y) [3], <sup>10</sup>Be (1.4×10<sup>6</sup> y) [4], <sup>212</sup>Pb (10.64 h) [5], <sup>214</sup>Po (3.7 µs) [6] and others, which range from several microseconds to  $10^{11}$  years.

These properties of LS detectors could be also useful for the study of excited nuclear states, especially in short lived nuclides, where lower detection efficiency would require long measurement times which may not be achievable. Moreover, due to the fast response of organic scintillators (typically a few ns, depending on the scintillator) and PMTs (1.3 ns rise time), the timing of scintillation events could be very precise. Thus, it is a useful method for fast timing measurements in the nanosecond range.

In this work we show that LS could be a useful tool for the study of the half-life of nuclear isomeric states. Two measurement techniques are proposed: one which utilizes a 3-PMT LS counter equipped with additional gamma detector and a CAEN digitizer and another with a 2-PMT LS detector - without the use of a  $\gamma$  channel. We analyze the time interval distributions between the events in the different channels of the system and demonstrate that these could be used for accurate determination of the half-lives of some excited states in certain nuclides.

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#### 2. Materials and methods

Measurement systems. Two experimental systems were used for the purpose of this study. The first experimental system is a TDCR portable device developed at LNE-LNHB for primary measurement of activity of LS-samples. It consists of a three PMT LS detector and a 5 mm x 5 mm x 1 mm Cadmium Telluride (CdTe) X-ray detector. The PMTs in the LS system are square, small form-factor Hamamatsu R7600-200 tubes with 350 ps FWHM transient time spread [7]. The solid state detector is an AmpTek XR-100CdTe with a PX2T power supply and shaping amplifier [8]. The three PMTs are positioned in a 120° geometry around a standard 20 ml liquid scintillation vial. The vial is placed in a 3D printed optical chamber made with PLA plastic. The chamber is covered with reflective polymer foil with 98.5% reflectivity [9]. The CdTe detector is placed as close as possible to the vial, in order to achieve maximum geometrical efficiency and between 2 of the 3 PMTs (as described in Figure 1). The detailed specification and performance of the device will be detailed in a future paper.



Figure 1: Simplified schematic of the 3-PMT LS detector and the CdTe  $\gamma$ -detector.

The three PMT channels and the amplified and shaped output of the CdTe detector are connected to a CAEN DT5751 desktop digitizer [10] with 1 GS/s sampling rate. The signals of the three PMTs and the CdTe detector are digitized and a timestamp of their arrival time as well as the peak area is recorded for each event in a file for later off-line analysis.

The second experimental system is an in-house developed LS detector with two XP2020 PMTs looking from opposite sides at a standard 20 ml LS sample. The vial is placed in a 3D printed optical chamber which is optimized for fast light collection. This chamber was also covered with the same reflective polymer foil. Both PMTs are directly connected to the DT5751 digitizer and the incoming events are recorded in a list mode file. This system was used for the LS-LS coincidences measurements and to obtain the half-lifes of the excited states without the use of the gamma channel.

Data analysis. The off-line data is analysed using an in-house developed computer software that counts the coincidences between the first arriving events in each channel and applies an extending type dead-time (20 µs base duration) after the end of the coincidence resolving time. The extending type dead-time ensures that enough time has passed before each detected signal such that no afterpulses are detected. The code was already tested and used for the study of coincidence counting algorithms [11] and the time response of different LS cocktails [12].

Due to the higher delay of the solid-state detector, the PMT channels in the 3-PMT detector are delayed by 4  $\mu$ s using the software. This value was determined by measuring the <sup>241</sup>Am LS-source and changing the

delay until a maximum coincidence counting rate is reached between the LS and  $\gamma$  channels with a 40 ns coincidence window.

The list-mode data analysis software can output the time interval distributions between the PMT channels as well as between a PMT channel and the CdTe detector. All time interval distributions are corrected for accidental coincidences according to the experimental method described in [13]. The obtained time distributions are fitted with the non-linear least squares Levenberg-Marquardt algorithm. The weight of each point is calculated as the square root of the number of detected events and is taken into account in the fitting procedure.

#### 3. Results

#### 3.1. Half-life measurements of excited states in <sup>57</sup>Fe

 $^{57}$ Co decays via EC to the 136.5 keV excited level of  $^{57}$ Fe with 99.8% probability (shown in Figure 2). The decay is detected in the liquid scintillator by the X-ray and Auger electron emissions. The time for the rearrangement of electrons after the EC is in the order of  $10^{-16}$  s and thus this process is considered to be instant compared to the studied half-lives [?]. The energy released in the scintillator after the EC is the range from 5.4 to 7.1 keV. The detection efficiency for a double coincidence in the LS detector for such energies is around 50%.

The second excited level of <sup>57</sup>Fe transitions to the ground level with 9.2% probability or to the first excited level with 85.5% probability with a half-life of around 8.6 ns (see Figure 2). The 2–0 transition can be detected in the scintillator by the 129.4–136.5 keV conversion electrons, produced with about 4% probability, or in can be detected by the gamma emission due to the transition  $\gamma_{2,0}$ (Fe) = 136.5 keV which directly interacts with the scintillator by Compton scattering or photoelectric effect. Similar to the previous transition, the 2–1 transition can be detected by the 115–122 keV conversion electrons, produced with about 4% probability, or by the gamma emission due to the transition  $\gamma_{2,1}$ (Fe) = 122 keV. Finally, the first excited level has a 98 ns half-life and the 1–0 transition can be detected from the 7.3–14.4 keV conversion electrons or gamma emission due to the transition  $\gamma_{1,0}$ (Fe) = 14.4 keV.

With the analysis of this decay scheme and the properties of this specific LS-gamma detector, there are a few possible start-stop combinations from the available events:

- 1. EC is detected (START) and  $\gamma_{2,0}$ (Fe) is detected (STOP). Such coincidences were used to study the half-life of the 2<sup>nd</sup> excited state.
- 2. EC is detected (START) and  $\gamma_{2,1}$ (Fe) is detected (STOP). In this case  $\gamma_{1,0}$ (Fe) would be within the dead time of the detector. These coincidences were also used to study the half-life of the 2<sup>nd</sup> excited state.
- 3. EC is not detected;  $\gamma_{2,1}(\text{Fe})$  is detected (START) and  $\gamma_{1,0}(\text{Fe})$  is detected (STOP). This was used to study the half-life of the 1<sup>st</sup> excited state.



Figure 2: Simplified decay scheme of  ${}^{57}$ Co. Only transitions with intensities above 0.2% are shown. The START and STOP signals that were used to study the half-lives of the two excited states are also shown. Data and figure taken from [14].



Figure 3: Spectrum of the  ${}^{57}$ Co LS-sample obtained with the 2 PMT system. For the study of the 14.4 keV level the START and STOP signals are taken from the lower energy part of the spectrum. For the study of the 136.5 keV level the low energy events are used as START and the high energy events are used as STOP.

4. EC is detected (START),  $\gamma_{2,1}$ (Fe) is not detected (low probability) and  $\gamma_{1,0}$ (Fe) is detected (STOP). Such coincidences were also used to study the half-life of the 1<sup>st</sup> excited state.

The various start and stop possibilities are shown in Figure 2.

Half-life measurement of the 14.4 keV level of <sup>57</sup>Fe using LS-LS coincidences. A 60 hour long measurement of a 5 kBq <sup>57</sup>Co LS-source was performed using the 3 PMT TDCR- $\gamma$  detection system. This system was preferred over the 2 PMT system in order to directly compare results with LS- $\gamma$  coincidences on the same list-mode data. The obtained list-mode files were analysed using the dedicated software; two of the PMT channels were constrained to include only events with medium energy - i.e. removing single photon events and the high-energy 122-136 keV interactions. Removal of the single photon events is done in order to remove spurious events and delayed fluorescence coming from the cocktail. The energy spectrum and the part of the events which were considered for this study are shown in Figure 3. Even after selecting only a part of the energy spectrum that excludes the high energy events all coming from the 2<sup>nd</sup> excited state of <sup>57</sup>Fe there is a non-zero probability to detect events from this state because of Compton scattered electrons from the  $\gamma_{2,0}(\text{Fe})$  and  $\gamma_{2,1}(\text{Fe})$  transitions. Such events will distort the decay curve of the 1<sup>st</sup> excited state as they can serve as both start and stop signals. However, after ten half-lives of the 8.6 ns level, or about 90 ns, only events from the 14.4 keV level can be detected as a stop signal due to its significantly longer life-time compared to the other transitions or uncorrelated events from ther decays. Radeloff et al. have proposed a bi-exponential fit when there is a large probability for accidental coincidence events to serve as stop signals [?]. This was observed for measurements of half-lives in the order of a second. The time range considered in the current experiment is less than 1500 ns and the counting rate in the detector is less than 5000 events per second. The correction exponential distribution due to accidental coincidences in this case would be in the order of  $10^{-5}$  and thus negligible.

Another effect that has to be considered is that the Compton scattered electrons produced from the  $\gamma_{2,1}$  (Fe) transition interacting with the cocktail can act as a start signal. Due to the 8.6 ns half-life of the 2<sup>nd</sup> excited state they cannot be considered to be instantaneous after the decay. This leads to a sum of two exponential distributions: first a rise time equal to the decay time of the 2<sup>nd</sup> excited state and second an exponential decay with the decay constant of the 1<sup>st</sup> excited state. After a sufficient time the effect of the rise time is negligible and only the decay time of the 2<sup>nd</sup> excited state will be observed.

The time interval distribution between the first events in two PMTs within the selected energy range is shown in Figure 4. The total number of coincidence events in the presented measurement is  $10^8$ . The time distribution was fitted with an exponential and the points considered in the fit are between 300 and 1100 ns. The obtained half-life is 97.93(9) ns, where the uncertainty is the statistical uncertainty reported by the fitting algorithm. The residuals are well grouped within  $\pm 2\sigma$ . Some residuals outside of the fit boundaries are also shown in order to illustrate that the goodness of the fit is preserved also at wider range.



Figure 4: LS-LS coincidences measurements of the 14.4 keV level of  ${}^{57}$ Fe. The start signal is given by a low energy event in one PMT and a low energy event in the other PMT. The time interval distribution is obtained with the TDCR detector.

Measurement of the 14.4 keV level of <sup>57</sup>Fe using LS- $\gamma$  coincidences. The same list-mode <sup>57</sup>Co measurement was used in order to study the LS- $\gamma$  coincidences. In this case the start signal is given by an event in the 136.47 keV peak in the CdTe detector and the stop by a low-energy event in the LS (excluding the single photon peak). The time interval distribution is displayed in Figure 5. The fit is performed between 250 and 450 ns with an exponential function. The obtained half-life is 97.8(28) ns, where the uncertainty is only the statistical uncertainty reported by the fitting algorithm. Due to the low geometrical efficiency of the  $\gamma$ -channel detector, it is difficult to obtain the same precision for the same measurement time as with the LS-LS coincidences method.

Measurement of the 136.5 keV level of  $^{57}$  Fe using LS-LS coincidences. The half-life of the 136.5 keV excited state was determined from LS-LS coincidences using the 2 PMT detector by triggering on medium-energy events in one PMT as a start signal and using the 122-136 keV peaks for a stop signal in another PMT. The spectrum of the events considered for start and stop is shown if Figure 3. The study of the 2<sup>nd</sup> excited state is significantly easier compared to the 1<sup>st</sup> as there is only one start event and the two stop transitions 2–1 and 2–0 can be separated well from the rest. The half-life of this level is too short to be studied with the gamma detector due to its timing resolution.

The observed decay curve in such a configuration is shown in Figure 6. The fast response of the PMTs in LS-LS technique allows us to study 9.7 half-lives of this state. The half-life obtained from an exponential fit is 8.78(2) ns, where the uncertainty is the statistical uncertainty reported by the fitting algorithm.

#### 3.2. Measurement of the half-life of the 59.54 keV level of <sup>237</sup>Np

<sup>241</sup>Am decays with the emission of an  $\alpha$ -particle to the 59.54 keV short lived isomeric state of <sup>237</sup>Np with 84.45% probability (see Figure 7). A 1 kBq source of <sup>241</sup>Am in UltimaGold (UG) liquid scintillator in a standard 20 ml glass vial was prepared for the purpose of this study. The source was measured for 60 hours with the described measurement system and the events from all LS channels and the  $\gamma$  detector were



Figure 5:  ${}^{57}$ Co LS- $\gamma$  coincidences. Start signal is given by the 136 keV peak in the CdTe and the stop is given by a low-energy PMT event.

recorded in list-mode files. The files were analyzed to obtain the time interval distributions for the LS-LS and  $\text{LS-}\gamma$  coincidences.

For the usual applications of the LS detectors the PMTs are working in single photon mode with gain in the order of 10<sup>7</sup>. At such high gain the light from the alpha particles of <sup>241</sup>Am would produce large signals with significant afterpulses spanning several microseconds. Thus, single photon mode would be unsuitable to detect the signal from the 59.54 keV de-excitation as the detectors will be saturated. This saturation can be avoided by reducing the PMT high-voltage, from 2100 V to 1700 V for the used XP2020 PMTs, thereby reducing the gain to approximately 10<sup>5</sup>. This is sufficient to reduce the afterpulses to a negligible level and gives the opportunity to use the <sup>241</sup>Am  $\alpha$ -particle in the LS as a start signal and a 59.54 keV conversion electron or  $\gamma$ , again in the LS, as a stop. The obtained decay curve with such a configuration is shown in Figure 9. A major advantage of the LS-LS coincidences with the 2 PMT detector over using a  $\gamma$  detector is the 100% efficiency for  $\alpha$ -particles and close to 100% efficiency for the 59.54 keV emissions.

The result from the LS-LS coincidences was compared with the half-life obtained with LS- $\gamma$  coincidences using the 3 PMT TDCR- $\gamma$  detector. In this case the start signal is given by an  $\alpha$  event in the LS and the stop event is given by an event in the 59.54 keV peak in the CdTe detector.

The alpha particle coming from the decay <sup>241</sup>Am is detected in the scintillator with a 100% efficiency. The 59.54 keV gamma can then be detected in the CdTe detector with a geometrical efficiency of about 0.3%. The performed measurement is 136 h long and the total number of recorded coincidences is  $4.10^5$ . The distribution of the time interval between a PMT event and the CdTe detector event in the 59.54 keV peak is displayed in Figure 10. The number of events per bin are normalized by the total number of detected events. The bottom graph shows the residuals of the fit within the fitting range of 175 ns to 700 ns in red and outside the fitting range in green. The points before 175 ns are excluded in order to remove effects of the gaussian timing response of the CdTe and events after 700 ns are excluded due to too low statistics. The reduced  $\chi^2$  statistic of the fit is 1.037. The observed half-life is 67.48(22) ns, where the uncertainty is the statistical uncertainty reported by the fitting algorithm.



Figure 6: LS-LS coincidences measurements of the 136.5 keV level of  ${}^{57}$ Fe. The start signal is given by a low energy event in one PMT and a 122-136 keV event in the other PMT. The time interval distribution is obtained with the 2 PMT system.

#### 3.3. Analysis of the results with corresponding uncertainty evaluation

An overview of the uncertainty budgets is presented in Table 1. The factors that could influence the half-life assessment considered in this work are: the uncertainty associated with the fitting procedure, the corrections for accidental coincidences and the choice of boundaries for the fitting. The fits of the decay curves were performed with non-linear least squares Levenberg-Marquardt using gnuplot [?]. The fit uncertainties are taken from the output of the fitting procedure. The uncertainty associated with the correction for accidental coincidences was estimated by a manual change of their amplitude until a visible change in the residuals was observed; similar to the technique described in [?]. The uncertainty coming from the choice of fitting boundaries was evaluated by choosing different low and high limits and studying the effect on the final value for the half-life.

The final values obtained for the half-lives of the two <sup>57</sup>Fe states and the <sup>237</sup>Np state, after taking into account the factors contributing to the uncertainty, are shown in Table 1. Due to the low counting statistics the <sup>237</sup>Np LS-LS and the <sup>57</sup>Fe LS- $\gamma$  measurements were not considered for the final result report.

	136.5 keV $^{57}$ Fe	14.4 keV $^{57}$ Fe	59.5 keV $^{237}$ Np
Uncertainty from fit	0.22%	0.13%	0.33%
Corrections for accidental coincidences	0.25%	0.15%	0.15%
Stability & dead time	0.10%	0.10%	0.10%
Fitting boundaries	0.20%	0.24%	0.03%
Total Final result	0.40% 8 78(4) ns	0.31%	0.37% 67.48(25) ns

Table 1: Measured half-life values and relative standard uncertainty budgets. The uncertainty values are standard deviations at k = 1.



Figure 7: Simplified decay scheme of  $^{241}$ Am. Only transitions with intensities above 3% are shown. Data and figure taken from [14].

Comparison with existing decay data. The Decay Data Evaluation Project (DDEP) data on  ${}^{57}$ Co provides 98.0(4) ns for the half-life of the 14.4 keV level of  ${}^{57}$ Fe. It is taken as the weighted average of 7 measurements:

- 1. In [15] Alkilov et al. report 97.8(14).
- 2. In [16] Ahmad et al. report 99.2(4) ns via two HPGe detectors.
- 3. In [17] Hohenemser et al. report 99.3(5) ns via Time-differential perturbed angular correlation.
- 4. In [18] Morozov et al. report 94(4) via autocorrelation single-crystal scintillation time spectrometer with plastic scintillators and a single XP2020 PMT.
- 5. In [19] Eckhause et al. report 97.7(2) ns via two NaI detectors.
- 6. In [20] Kistner and Sunyar report 98(1) ns.
- 7. In [21] Clark reports 97.9(2) ns via two NaI detectors.

It should be noted that there is a significant discrepancy between the different measurements. The obtained half-life value in this work is 97.9(3) ns which is in agreement with reports 1, 5 and 7 within the estimated uncertainties, but not with reports 2 and 3; reports 4 and 6 have large uncertainties, and thus are in agreement.

The DDEP data on  ${}^{57}$ Co states 8.6(4) ns for the half-life of the 136.5 keV level of  ${}^{57}$ Fe. It is taken as the weighted average of 5 measurement, which are consistent within their stated uncertainties. The result from this study, 8.78(4) ns, has the lowest uncertainty compared to previous measurements and is consistent with the already provided average value.



Figure 8: Spectrum of the  $^{241}\mathrm{Am}$  LS-sample obtained with the 2 PMT system. The  $^{241}\mathrm{Am}$   $\alpha$  peak is shown in green and the events from the 59.5 keV transition of  $^{237}\mathrm{Np}$  in red.



Figure 9: LS-LS coincidences measurement of the 59.5 keV level of  $^{237}$ Np. The start signal is given by events in the alpha peak of the LS spectrum and the stop signal is given by a low energy event from the 59.5 keV transition. The time interval distribution is obtained with the 2 PMT system.

The Nuclear Decay Sheets (NDS) state 68.1(2) ns for the half-life of the 59.54 keV level of <sup>237</sup>Np [22]. The value is the weighted average of 5 measurements, the most recent of which is from 1972. The value obtained in this study does not agree with the stated average value which could be caused by the very low uncertainty of the 1972 measurement 68.3(2) ns [23]. This low uncertainty is increased to 0.7 ns (1%) by the evaluator in the ENSDF dataset and the obtained weighted average value is 67.2(7) ns, which is consistent with the result obtained in this work.

#### 4. Conclusions

The half-lives of two excited nuclear states in  ${}^{57}$ Fe and one in  ${}^{237}$ Np were measured in the presented study by means of LS-LS and LS- $\gamma$  coincidences. The half-life of the 59.54 keV level of  ${}^{237}$ Np was measured by LS- $\gamma$  coincidences and the result is 67.48(25) ns, which is in good agreement with already published decay data. The half-life of the 14.4 keV level of  ${}^{57}$ Fe was measured by LS-LS coincidences and the result is 97.9(3) ns which is consistent with some of the published results, however a significant discrepancy exist between the values found in the literature. The half-life of the 136.47 keV level of  ${}^{57}$ Fe was measured also by LS-LS coincidences and the result is 8.78(4) ns, which is consistent with the published data and comes with a significant improvement in the uncertainty.

Liquid scintillation fast timing measurements can be a useful to refine some already known decay times. Another advantage over more-commonly used methods is the relatively short measurement time that is needed, due to the  $4\pi$  geometrical efficiency. This could allow more precise studies of half-lives of excited states of short lived nuclides where long measurements are difficult to perform. The method employing LS-LS coincidences is shown be useful for precise measurements of half-lives down to 8 ns.



Figure 10: LS- $\gamma$  coincidences measurement of the 59.5 keV level of <sup>237</sup>Np. The start signal is given by the alpha particle in the LS and the stop is given by an event in the 59.5 keV peak in the CdTe.

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