MAPP Lab Work - Tübingen

Mößbauer Experiment

by Marc Breisch 30.07.2021

Literature for preparation

IMPORTANT: Remember to bring a USB drive on the day of the experiment.

Relevant keywords for the experiment

This chapter contains a list of keywords that will be relevant for the experiment. On the day of the experiment these keywords will be quizzed by the person in charge.

- Radioactive decays
- Radiation types and their detection
- Decay scheme of ⁵⁷Co
- Multipole radiation
- γ-matter interactions (photo effect, Compton effect, pair production)
- Debye-Waller factor
- Fine structure and hyperfine structure
- Isomer shift
- Quadrupole splitting
- Doppler effect
- Isotop, Isomer, Isobar

Literature

This instruction is merely an overview. To get a full grasp on the underlying mechanisms more preparation is necessary and can be found in the following literature:

- G. Schatz/A. Weidinger "'Nukleare Festkörperphysik"', Teubner (sehr zu empfehlen)
- H .Wegener "' Der Mößbauer-Effekt und seine Anwendungen in Physik und Chemie"', Hochschultaschenbücherverlag
- K. Betghe/G. Walter/B. Wiedemann "'Kernphysik Eine Einführung"', Springer
- W. Demtröder "'Experimentalphysik III + IV"', Springer

Contents

1	Introduction	1			
2	An introduction to the theory	2			
	2.1 Suppression of the recoil energy	2			
	2.2 Suppression of the Doppler broadening	2			
	2.3 Messprinzip	3			
3	The experimental setup	4			
	3.1 The basic setup	4			
	3.2 The source: 57 Co	5			
	3.2.1 The moving source	5			
4	Running the experiment	6			
	4.1 The source spectrum	6			
	4.2 The sample spectra	7			
5	Protocol and analysis	9			
	5.1 Analysis of the source spectrum	9			
	5.2 Analysis of soft iron	9			
	5.3 Analysis of steel	9			
	5.4 Analysis of iron sulphate	10			
A	Hyperfine structure of soft iron	11			
B	Quadrupole splitting of iron sulphate	12			
С	2 Detector efficiency				
D	D Relative absorber thickness				
E	E Software: Settings				

1 Introduction

The measuring principle of the Mößbauer experiment is to bring the process of nuclear emission and absorption into resonance to gain information about for example the splitting or shifting of nuclear energy levels. Taking advantage of the Doppler effect, the energy of the emission line can be varied in a small interval around its original energy value by moving the source with a variable velocity. Having decided for a γ -line, the measurement only works within a very small energy range around this line, but achieves an extremely high energy resolution. Two problems that occur and have to be solved are the energy shift due to momentum transfer on both the emitting and the absorbing nucleus and the Doppler shift due to the thermal motion of the atoms. Either of these effects would change the line energy on a much higher order compared to the measuring energy range and the nuclear energy structures and have to be suppressed. The discovery of the Mößbauer effect by Rudolf Ludwig Mößbauer in 1958 enabled these measurements and was honoured only three years later with the Nobel Prize of Physics. The Mößbauer effect provided the possibility to resolve nuclear spectra down to the quantum mechanical limit and is still a powerful tool today especially for material analysis.

For example the two Mars rovers Spirit and Opportunity were equipped with Mößbauer spectrometers. These spectrometers lead to the discovery of jarosit on the surface of the Mars. Jarosit is an iron oxide, that only forms in a moist environment. This discovery is evidence for an earlier existence of water on Mars' surface. Also worth mentioning is the first experimental proof of a general relativity prediction with the Mößbauer effect only one year after its discovery. Robert Pound and Glen Rebka arranged a source and an absorber vertically 22.5 m apart. Depending on where in the gravitational field of the earth a γ -quantum is emitted, its energy is slightly red shifted. The comparison of the velocities needed to compensate for this effect, with first, the source above the absorber and afterwards with interchanged positions, lead to the experimental confirmation of the general relativity.

The aim of this experiment is to examine different influences on the nuclear spectra of ⁵⁷Fe like the hyperfine structure, the isomer shift and the quadrupole splitting, to measure the magnetic field at the nucleus and to determine the lifetime of the Mößbauer niveau with the natural line width of the Mößbauer line.

2 An introduction to the theory

A γ -quantum emitted by an atomic nucleus can only be absorbed by another nucleus, if the energy of the particle coincides with the excitation energy of the absorbing nucleus. On first sight, resonant emission and absorption should be possible as long as the nuclei and the nuclear transitions match. But a closer look shows the emitting nucleus is being recoiled due to the conservation of momentum and the photon loses some energy. Also at the process of absorption, momentum is being transferred. For ⁵⁷Fe atoms this recoil energy is of the order of $2 \cdot 10^{-3}$ eV. Furthermore, the line energy will undergo a Doppler broadening caused by thermal motion. For a gas at room temperature, Doppler broadening affects the line energy by about 10^{-2} eV. So for the very unlikely case of resonant emission and absorption, a photon shifted by these two effects would have to hit an absorber nucleus, which by chance has to have the exact right velocity to compensate for the energy shifts. The difference in energy of a nucleus before and after the emission is given by:

$$E_i - E_f = \hbar \omega_0 + \underbrace{\hbar (\vec{k} \cdot \vec{v})}_{\text{Doppler effect}} - \underbrace{\frac{\hbar^2 \vec{k}^2}{2M}}_{\text{recoil energy}}$$

with

$$E_i = E_a + rac{ec{p}^2}{2M}$$
 und $E_f = E_g + rac{(ec{p} - \hbar ec{k})^2}{2M}$

where $\hbar \vec{k}$ is the momentum of the quantum, \vec{p} is the momentum of the atom, ω_0 the unshifted energy of the transition and *M* the mass.

2.1 Suppression of the recoil energy

In order to suppress the recoil energy transfer, Mößbauers approach was to use crystals. Because of the conservation of momentum, a γ -emitter in the crystal lattice will either generate phonons or the momentum will be transferred to the whole lattice. The latter will cause the transferred energy $\frac{\hbar^2 \vec{k}^2}{2M}$ to be negligible due to the high mass of the crystal of about 10^{20} nuclear masses, which is the denominator of the energy term. However, this is only the case for a fraction of the emission processes, which is described by the Debye-Waller factor. The Debye-Waller factor is highest for T = 0, but never becomes f = 1 due to the zero point energy. ⁵⁷Fe at room temperature has a Debye-Waller factor of f = 0.8. Since the absorption is just a reversed emission, the Debye-Waller factor for absorption is the same. The γ -emission with suppressed transfer of recoil energy is known as the Mößbauer effect.

For a classic description of the Debye-Waller factor see [1]. The full quantum-mechanical description can be found in [2].

2.2 Suppression of the Doppler broadening

For a gas at room temperature, the Doppler effect typically causes a line broadening of the order of 10^{-2} eV. The thermal motion of atoms and molecules follows the Maxwell-Boltzmann distribution, so the mean velocity goes with $\frac{1}{\sqrt{M}}$, where *M* is the mass of the atoms or molecules. But in the case of an emitter in a crystal lattice, *M* is given by the mass of the whole crystal and the Doppler broadening affects the line energy by only about 10^{-12} eV.

2.3 Messprinzip

The suppression of the recoil energy and the thermal Doppler shift enables resonant emission and absorption between nuclei of the same kind. Small changes in energy or the splitting of energy levels caused by e.g. hyperfine structure, isomer shift, or non-spherical deformations of the nuclei can be examined by taking advantage of the Doppler effect. The source is put into an oscillating drive system and moved forward and backward relative to the absorber, with velocities of a few millimetres per second. The emitted electromagnetic waves are slightly blue or red shifted, depending on the velocity and its direction. Resonant absorption occurs exactly if the Doppler shift of the source matches the energy shift of the absorbers transition line. For the examination of the absorber spectra we arrange the source, the absorber and the detector in a line, so that the photons have to pass through the absorber on their way to the detector. We trigger on the Mößbauer line energy and with every trigger the velocity of the source is being digitalized and the result put into a velocity histogram. If the Doppler shifted source energy matches a transition line in the absorber, we expect an absorption line in the velocity histogram. The Doppler effect allows to resolve lines down to the natural line width.

The natural line width is caused by the Heisenberg uncertainty principle and can easily be calculated with the lifetime of the excited state of ⁵⁷Fe 14.4 keV above the ground state.

$$\Gamma = \frac{\hbar}{\tau_N} = 4, 7 \cdot 10^{-9} \text{eV}$$

with Γ as the natural line width, τ_N the lifetime of the excited state, $\hbar = \frac{h}{2\pi}$ and *h* as the Planck constant. The measuring principle described above allows a relative energy resolution of 10^{-13} !

3 The experimental setup

3.1 The basic setup

The setup of this experiment is quite simple. It consists of a source in a driving system, an absorber and a detector plus read out electronics (see figure 1 and 2). We use an Amptek CdTe semi conductor detector, because of its very high quantum efficiency in the relevant energy range. For further information see their website [3]. The Mößbauer experiment can be set up in two different configurations, as a scattering spectrometer and as a transmission spectrometer. We use it as the latter. The X-rays are passing through the absorber and we get a decrease in rate at the Mößbauer resonance. Therefore, we expect absorption peaks in the spectrum.



Figure 1: Experimental setup: On the left side the detector and on the right side the source. The different absorbers are put in between detector and source.

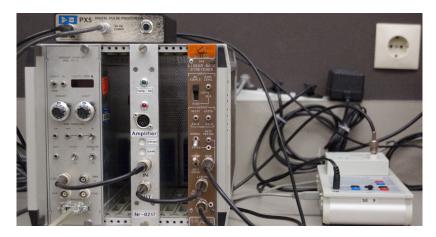


Figure 2: Readout electronics: From left to right the readout electronics consist of a transducer to move the source, an amplifier with a temperature control unit, a linear gate stretcher and a CASSY module. On top is a PX5 module to supply voltage and take data.

3.2 The source: ⁵⁷Co

The used source is a ⁵⁷Co source. In figure 3 the decay scheme is showing the decay from cobalt to iron as well as different de-excitations of an exited iron state $I = \frac{5}{2}$.

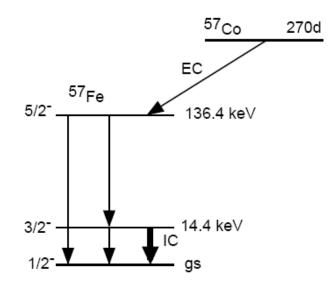


Figure 3: Decay scheme ⁵⁷Co [4].

For the preparation and quiz the following questions should be answered: What are the probabilities for the different decays and how do these values come about? Why do these values differ?

3.2.1 The moving source

As earlier mentioned, to scan an energy range around the line energy, the source is being moved forward and backward relative to the absorber with velocities of a few $\frac{mm}{s}$ (it would also work to move the absorber). The source is being driven by a transducer unit. The velocity range can be adjusted at the 'VELOCITY' knob. The Transducer should show a velocity of -5 to $5 \frac{mm}{s}$.

Important for the Protocol: The exact velocity calibration for all Mößbauer spectra should be done by using the literature value of $v = 10.65 \frac{\text{mm}}{\text{s}}$ for the distance between the two outer most lines of the hyperfine structure.

4 Running the experiment

The experiment itself is split into two parts. The first part covers the recording of a source spectrum to deepen the understanding of the used source and the second part is the measurement of the spectra for our three samples: soft iron, steel and iron sulphate.

CAUTION!

Before the detector is switched on via the PX5 module, the crate for the temperature control has to be turned on to ensure that the detector is cooled. As soon as the green LED turns on the detector can be turned on as well.

4.1 The source spectrum

To record the source spectrum, no absorber is put between detector and source and the detector is moved as close to the source as possible. Additionally the transducer should be turned off to measure an unshifted source spectrum. With this the hardware setup is done and now the software has to be set up.

To do this the PX5 software (see figure 4) is necessary and has to be opened. If the PX5 module is already turned on a window should open to connect the PX5 module to the software (cmp. appendix E figure 11). Next several pre-settings have to be made by opening the settings window "Acquisition Setup" with



Figure 4: PX5 Software.

F9. Under "General" most of the pre-settings can be loaded using "Open Configuration File". The now open explorer window should have a folder called "Setup_PX5" pinned on the left side and a file called "CdTe_Co57_MOS" should be inside. A double click will open and load the settings.

On the left side of the screen are several sensor data updates. Among them are the supply voltage of around 500 V and the temperature which should always be around 240 K. Occasionally they have to be updated using F4.

Next the settings for the input channel have to be selected. Therefore switch to the tab "Misc" and choose a dropdown menu called "AUX1". For this part it should be set to "DETRES" (cmp. appendix E figure 12) which allows us to take the entire width of our source spectrum.

Once everything is set confirm with "OK" and start the measurement with **F3**. The spectrum should now start growing on screen, eventually the limit for the y-axis has to be re-adjusted.

The spectrum can be saved with "File \rightarrow Copy Spectrum Data", which copies the data like "Crtl+C". This can then be pasted into a new text file.

4.2 The sample spectra

To measure the sample spectra, first the software has to be adjusted again. The first and most important setting is switching on the software side single-channel-analyzer (SCA). A SCA gives us the possibility to change the "window" of the recorded spectrums width. In our case we want to limit our measurement to the Mößbauer line at 14.4 keV.

To do this open the "Acquisition Setup \rightarrow Misc" window again and set "AUX1" from "DETRES" to "SCA8" which is the SCA channel we use. To set the limits of this "window" use the lowest menu in the "Misc" tab

Single-Channel Analyzer (SCA)						004.0	
SCA Index	Low Threshold	High Threshold	Output	t Level	Pulse V	Nidth	SCA 8
V SCA8	178	209	\sim	HIGH	\sim	1000	Defaults
Preview SC	A Settings	Enable Single-Channel Analyzer (SCA)					

Figure 5: Single-channel-analyzer settings in the Software.

which can be seen in figure 5. In this menu three settings have to be made. The first is to set the "SCA Index" to "SCA8". The second and third thing to change are the lower and upper thresholds of the "window" around the Mößbauer line.

To determine these, the peaks appearing in the source spectrum have to be understood. Once the 14.4 keV peak is found a small window around it is read from the x-axis and entered into the corresponding window. Once done confirm with "OK" again.



Figure 6: CASSY Mößbauer software.

The difference to the measurement before is that the data is now taken by using CASSY. On the desktop a short-cut (see figure 6) opens an almost completely set up CASSY window (cmp. appendix E figure 13). The only setting which has to be changed is the measurement time. This time varies from sample to sample and can be found in table 1.

Table 1: Measurement times for the different samples in minutes.

Sample	Measurement time in min	
Soft iron	30	
Steel	30	
Iron sulphate	60	

An important note for these measurement times is that they also vary heavily with the strength of our source and thus may be adjusted by the supervisor on the day of the experiment.

With this the software is set up and we continue with the hardware setup. First of is placing the sample

between the detector and the source. Therefore the sample is mounted onto the available holder and put onto the rail the detector is on (see figure 1). All three components (detector, sample and source) should be as close as possible to minimise losses.

CAREFUL: We use radioactive sources! Never put any part of your body in front of the opening in the lead!

Lastly switch on the transducer with the corresponding switch to move the source (see 3.2.1). With this the measurement can be started by pressing **F9**. It will stop automatically after the time runs out and the measurement can be saved once as .txt/.csv and once as .cassy.

5 Protocol and analysis

The protocol should contain three parts. First, all the theory which is important for the understanding of the experiment, a description of the setup and the data that were taken, and the actual analysis of the data. For every value the corresponding reproducible error has to be listed.

5.1 Analysis of the source spectrum

The first task for the source spectrum is to calibrate it using the 14.4 keV and 122 keV peaks. The next task is to identify the monitored structures, they should be identified, labelled and explained (Hint: Appendix C, figure 9). The positions of the photo peaks, the Compton edges and the backscattered photo peaks have to be marked in the spectrum.

5.2 Analysis of soft iron

As preparation the question why only six of eight possible transitions are visible should be answered.

First of the velocity scale has to be calibrated by using the literature value (see 3.2.1) and the two outermost absorption lines. Assign the different absorption lines to the nuclear transitions between the different hyperfine structure levels. Calculate the energy splitting ΔE_g of the ground state and ΔE_a of the excited state by using the velocity calibration factor and the Doppler relation.

Using the schematic of the hyperfine splitting of soft iron (see figure 7 in appendix A) it becomes obvious that the velocity difference between the second and fourth and the third and fifth peak correspond to the energy splitting of the ground state, while the difference in velocity between the peak one and two, two and three, four and five, fife and six represent the energy splitting of the excited state.

With ΔE_g and $\mu_g = +(0,0903 \pm 0,0007)\mu_k$ (see [5]), calculate the magnetic field at the nucleus and afterwards using the magnetic field and ΔE_a the magnetic momentum μ_a of the excited state.

5.3 Analysis of steel

Use the FWHM of the 14.4 keV ⁵⁷Fe line to calculate the lifetime of the Mößbauer level. Take into consideration, that the finite line width plays a roll in both the emission and absorption process and include the absorber thickness in the calculation. For more information on the influence of the absorber and the source thickness see [6]. The source can be considered to be thin with an effective thickness of $T_S = 0$. Assuming this the lowest curve in appendix D figure 10 can be used.

The relative broadening $\frac{\Gamma_a}{\Gamma}$ can then be found by calculating T_A , which should then be compared to $\frac{\Gamma_{exp}}{\Gamma}$.

$$T_A = f'_A \cdot n_A a_A \sigma_0 \cdot t_A$$

with f'_A : the absorbers Debye-Waller factor, which is 0.8 for iron at room temperature.

 n_A : the absorber nucleus density $(8, 84 \cdot 10^{22} \frac{\text{nuclei}}{cm^3}$ for iron) a_A : the isotope concentration for ⁵⁷Fe in the absorber (0,022 for our absorber)

$$\sigma_0 = 2\pi \left(\frac{\lambda}{2\pi}\right)^2 \frac{2I^* + 1}{2I + 1} \cdot \frac{1}{1 + \alpha}$$

with

λ = wavelength of the 14.4 keV-γ-line of ⁵⁷Fe I^* = spin of the excited state of ⁵⁷Fe I = spin of the ⁵⁷Fe ground state α = conversion coefficient = 9,7 t_A = absorber thickness: t_A = 2,54 · 10⁻³ cm

5.4 Analysis of iron sulphate

Determine the isomer shift (in eV) relative to the position of the stainless steel line. In addition calculate the quadrupole constant $B = eQV_{zz}$ out of the energy splitting of the FeSO₄ line. Information on the quadrupole splitting can be found in [2] (page 95-100).

A Hyperfine structure of soft iron

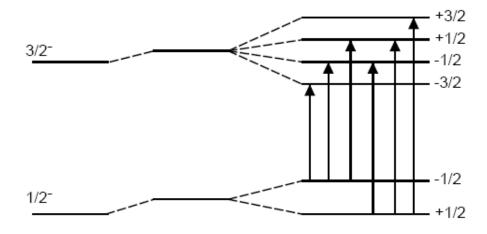
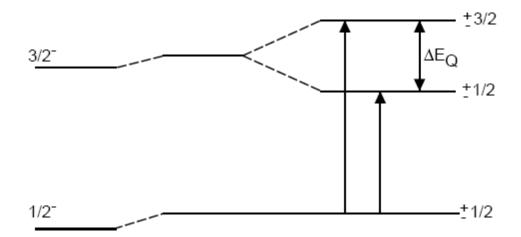


Figure 7: Hyperfine structure of soft iron [4].



B Quadrupole splitting of iron sulphate

Figure 8: Quadrupole splitting of iron sulphate [4].

C Detector efficiency

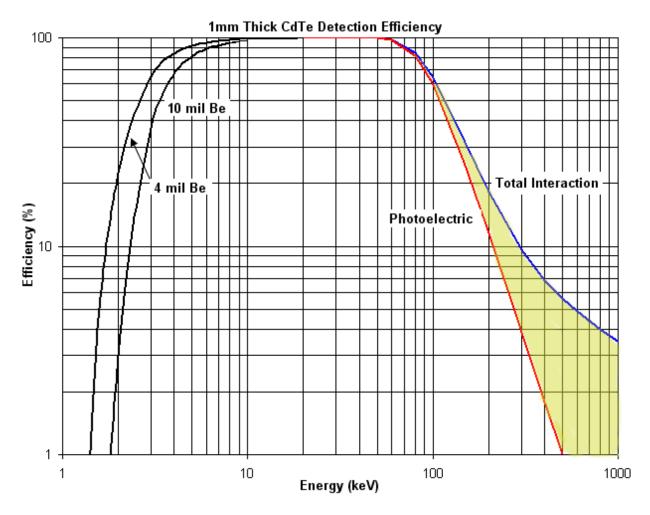
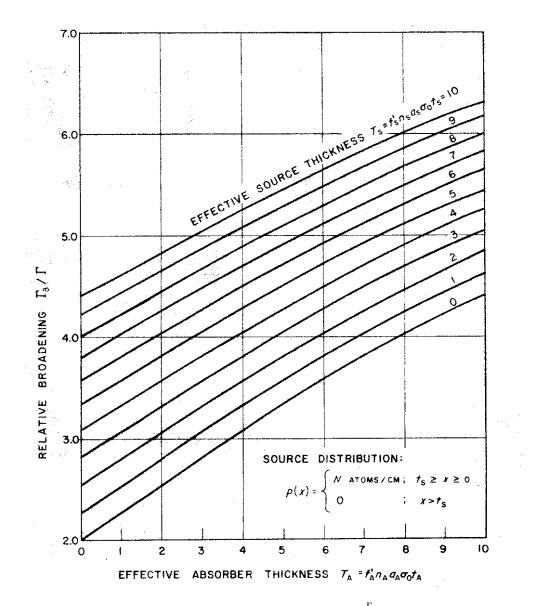


Figure 9: Detector efficiency curve [3].



D Relative absorber thickness

Figure 10: The relation between the relative broadening $\frac{\Gamma_a}{\Gamma}$ and the effective thickness of the source [6].

E Software: Settings

Select DPP Communications Port		?	\times
USB Find Device FOUND Select Device 1 Close Device	Amptek PX5 - S/N 2756 Connection: USB Device 1 of 1 (no description) MAC Address: 00-0b-3c-6b-9d-72 Time Powered: 4 minutes, 6 seconds		
CEthernet Find DP5 IP			
IP Address Port 192 168 0 255 10001 Type In IP Address			
RS232 Serial Find Device			
Close Port			
Open Port			
Spectrum Request Rate Request Spectrum Every 1000 ms	Default Connect	Do Not Conn	ect

Figure 11: Connection window for the detector.

DPP Configuration		×
General MCA Shaping Gain Power Misc		
Quickset Shaped Output	ADC Input Pile-Up	
Advanced		
DAC	General Purpose Counter	
Analog Out V SHAPED	Counter Edge 🗸 RISING	
Output Offset 50 -500 to +499mV	Counter Uses MCA_EN? ~ ON	
Output Offset 50 -500 to 4455mV	Counter Uses GATE? V ON	
AUX1 V DETRES	Clear Counter With MCA Counters? V ON	
Test Pulser V OFF		
AUX2 V ICR	Counter Input V AUX1	
Connector 1 V AUXOUT1		
Connector 2 V AUXOUT2	Scope Trigger Edge V FALLING	
PX5 Speaker	Trigger Position V 12	
Speaker On/Off V OFF	Gain 🗸 1 x	
Single-Channel Analyzer (SCA)	SCA 1	
SCA Index Low Threshold High Threshold Output Leve		
	FF V 1000 Defaults	
Preview SCA Settings Enable Single-Channel An	alyzer (SCA)	Misc
1		
	OK Abbreche	n Übernehmen
Ready		

Figure 12: Misc window for detector settings.

h	Firstellungen	џ	~		
ł	Einstellungen CASSYs	*	^		
-	Sensor-CASSY				
1	Eingang A1 (VKA-Box, 524058)				
1	Ereignisse N _A				
1	Kanal n _A				
	Energie E _A				
1	Spannung U _A				
1	Rate R _A				
1	Totzeit t _A Eingang B1 (ohne Sensorbox)				
	Relais R1				
1	Spannungsquelle S1				
1			~		
-					
1	Ereignisse N _A				
1	Messung: 512 Kanäle 🗸 🗸				
	Impulse / Zeit: 36132960 / 86400,0 s				
1	Verstärkung				
1					

	negative Pulse: 4950 mV Faktor: 1,01				
	Energiekalibrierung				
1	Global für alle Spektren auf diesem Eingang				
1	Kanal: Energie: V				
ł	🗌 Kanal: 🔹 Energie: 🗸 🗸				
1	Tipp: Kanalwerte durch senkrechte Linie im Diagramm, Achsenumschaltung durch Drag&Drop von n oder E.				
1	1147-				
+	Hife				
	Aufnahme: automatisch V 🗌 Neue Messreihe anhängen				
1	Messzeit: 1+0:00 h ~ < > Anzahl: 86401				
1	Intervall: 1 s V V Pretrigger: 0				
	Trigger:				
1	Messbedingung: 1				
1	Stoppbedingung: 0				
1	□ Wiederholende Messung □ Akustisches Signal				
1	Hilfe				
j					
1					

Figure 13: Settings for CASSY.

References

- G. Schatz and A. Weidinger: Nukleare Festkörperphysik: Kernphysikalische Meßmethoden und ihre Anwendungen (Vieweg+Teubner Verlag), 2013, URL https://books.google.de/books?id= ni-cBgAAQBAJ.
- [2] H. Wegener: Der Moessbauer-Effekt und seine Anwendungen in Physik und Chemie, 1996.
- [3] Datasheet for xr100cdt Detektor, 09.2017, https://www.amptek.com/-/media/ametekamptek/ documents/products/user-manuals/xr100-cdte-user-manual-b2.pdf.
- [4] J. Loebell: Anleitung Moessbauer Effekt, 2014, previous lab work manual.
- [5] G. W. Ludwig and H. H. Woodbury: Magnetic Moment of Fe⁵⁷, Phys. Rev., volume 117, Mar 1960, 1286, URL https://link.aps.org/doi/10.1103/PhysRev.117.1286.
- [6] S. Margulies and J. Ehrman: Transmission and line broadening of resonance radiation incident on a resonance absorber, *Nuclear Instruments and Methods*, volume 12, 1961, 131, URL https://www. sciencedirect.com/science/article/pii/0029554X61901227.