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PAC SPECTROMETER FOR CONDENSED
MATTER INVESTIGATION

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Спектрометр ВУК для исследования конденсированных сред

Для исследования сверхтонких взаимодействий (СТВ) в конденсированных средах создан 4-детекторный спектрометр возмущенных угловых $\gamma\gamma$ -корреляций (ВУК), позволяющий проводить измерения практически с любыми типами детекторов. Оригинальное схемотехническое решение с использованием специально разработанного блока «Master PAC» в сочетании с ЭВМ по сравнению с традиционными спектрометрами ВУК позволило существенно повысить эффективность, сократить время настройки и упростить эксплуатацию спектрометра. Криостат и высокотемпературная печь позволяют проводить измерения в диапазоне температур от 120 до 1300 К. Электромагнит броневое типа дает возможность создавать на образце магнитное поле до 2 Тл. В измерительный комплекс входит пресс со специально разработанной камерой высокого давления, дающей возможность проводить измерения ВУК в образцах при давлении до 60 ГПа в режиме «on-line».

Работа выполнена в Лаборатории ядерных проблем им. В. П. Дзелепова ОИЯИ.

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PAC Spectrometer for Condensed Matter Investigation

A four-detector spectrometer of perturbed angular $\gamma\gamma$ correlations is developed for investigation of hyperfine interactions in condensed matter. It allows measurements with practically any types of detectors. A unique circuit design involving a specially developed Master PAC unit combined with a computer allows a substantially higher efficiency, reduced setup time and simpler operation in comparison with traditional PAC spectrometers. A cryostat and a high-temperature oven allow measurements in the temperature range from 120 to 1300 K. An encased electromagnet makes it possible to generate a magnetic field up to 2 T on a sample. The measurement system includes a press with a specially designed high-pressure chamber allowing on-line PAC measurements in samples under pressure up to 60 GPa.

The investigation has been performed at the Dzelepov Laboratory of Nuclear Problems, JINR.

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INTRODUCTION

Hyperfine interaction (HFI) physics deals with a wide range of phenomena where interaction of atomic nuclei with electric and magnetic field generated by electrons in free atoms and in condensed matter is manifested. This allows the electron structure of solids, chemical compounds, and biological objects to be studied. The HFI parameters are quite sensitive to details of the electron wave functions for atoms and ions, which makes it possible to gain unique information unobtainable by other methods.

The HFI Hamiltonian may be written down as a series

$$H_{\text{cr}} = H(E_0) + H(M_1) + H(E_2) + \dots,$$

where E_0 is the electric monopole interaction, M_1 is the magnetic dipole interaction, E_2 is the electric quadrupole interaction. Each term of the series is a product of one of the nuclear moments by the corresponding component of the extranuclear electromagnetic field. As the interaction energy rapidly decreases with increasing multipolarity, the dipole/quadrupole interactions alone are usually dealt with. Thus, knowing the magnetic dipole moment and the electric quadrupole moment of the probe nucleus, we may determine the electromagnetic fields of the probe nucleus surroundings.

The main methods used to study HFI are the nuclear orientation method, nuclear magnetic resonance method, Mössbauer effect, and method of perturbed angular correlations (PAC) of nuclear radiation cascades.

One of the major advantages of the PAC method is that it allows experiments to be carried out with samples in any aggregate state, in a wide range of pressures, temperatures, and external electromagnetic fields, with extremely small concentrations of probe nuclei (probe radionuclides) down to 10^{-15} M. As a result, it is possible to investigate a wide range of objects from gaseous substances to biological samples measured *in vivo*.

The PAC method requires spectrometers that provide data taking with a good statistical accuracy within reasonable measurement time. The efficiency of PAC spectrometers is mainly increased by increasing the number of detectors to four or six, as a rule.

To decrease the load of the accumulation equipment in the PAC spectrometers, only signals passed through the preliminary energy selection are applied to the time-to-amplitude converter (TAC) [1–3]. Since the energy selection requires time, either fast analogue signals are delayed [2], which may result in worse

time resolution, or only the fast component of scintillation (BaF_2) is used [3], which worsens the energy resolution. Sometimes trade-off decisions are taken, e. g. partial collection of the slow component (BaF_2) is carried out with a small time signal delay [1]. Note that in all above-mentioned spectrometers the energy selection of gamma-quanta is carried out by hardware. In addition, the above detectors are made to work only with a particular type of detectors.

PAC spectrometers were designed and constructed by groups of physics engaged in solving nuclear physics problems. Recently PAC methods have found wide use in other sciences, such as solid state physics, chemistry, medicine, etc. To make PAC widely used, it is necessary to have spectrometers which do not require sophisticated setup procedures and which allow a wide range of radioactive isotopes to be used as probes. Development of electronic and computer circuitry and components make it possible to develop such spectrometers.

We have developed a four-detector spectrometer which can be used with different types of detectors and with all radionuclides suitable for PAC measurements. Preliminary selection for reducing the load of the equipment is done by time, and the energy selection is carried out by using the accumulation program and codes corresponding the total detector charge collection. Special devices are built into the spectrometer to increase the measurement accuracy and to make setup (operation) easier. This spectrometer is used to carry out investigations in solid state physics [4] and physical chemistry [5].

1. BLOCK DIAGRAM AND OPERATION OF THE SPECTROMETER

The block diagram of the PAC spectrometer is displayed in Fig. 1. Detectors of the spectrometer (DET1–DET4) are cylinder-shaped BaF_2 or NaI(Tl) crystals, 40×40 mm in size, optically connected to the XP2020Q photomultiplier.

Time referencing is by the anode signal of the detector, the CANBERRA 2126 constant fraction discriminator (CFD) is used. The dynode output of the photomultiplier is connected to the shaping spectrometric amplifier (Ampl.).

The hardware and software of the spectrometer provide two modes of operation:

- basic mode, in which 12 time spectra and 4 energy spectra are recorded;
- setup mode, in which energy windows may be set up and operation of detector sections may be controlled.

During the experiment (basic mode) the Master PAC unit provides selection of two events coinciding in time for any pair of the detectors. The coincidence time range is set by the switch on the front panel of the unit. In the case of coincidences the Master PAC produces GATE signals turning on the recording instruments while at the START and STOP outputs information pulses are generated. The time interval between them is equal to the interval between signals

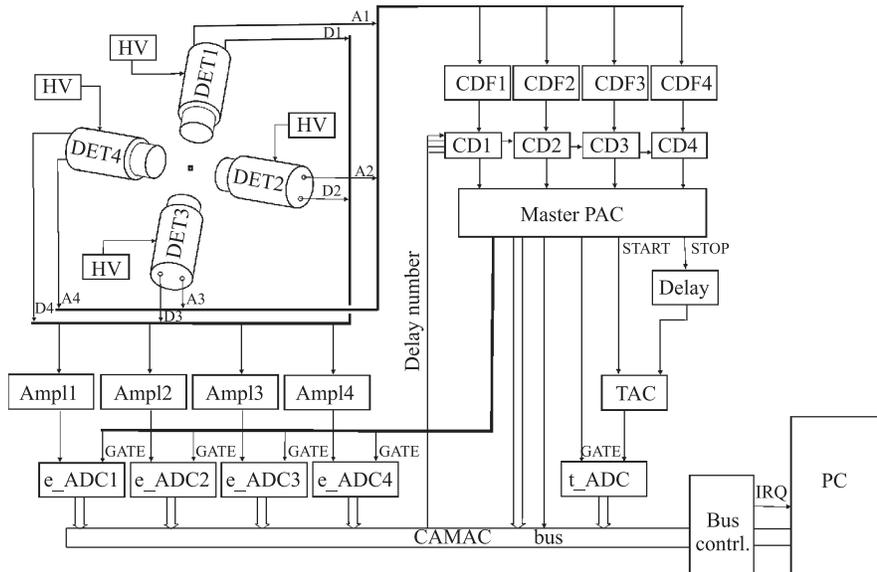


Fig. 1. Block diagram of the four-detector PAC spectrometer

from the detectors. The time selection in the Master PAC reduces the accidental signal load of the recording instruments by two-three orders of magnitude.

The TAC unit (ORTEC 467 time-to-amplitude converter) receives START signals directly from the Master PAC and STOP signals through the cable delay (DELAY) and generates pulses with an amplitude proportional to the START-DELAY/STOP time interval. Positions of switches on the Master PAC and TAC and the DELAY value determine the time interval measurement range. Signals from the TAC and the amplifiers Amp1–Amp4 arrive at the analogue-to-digital converters, time t_ADC and energy e_ADC1 – e_ADC4 ones respectively. Master PAC pulses GATE trigger the ADC and LAM-IRQ signals launch the data recording procedure in the PC.

The data recording procedure involves the «hardware interruption» mechanism. The data reading program itself is a resident, i. e. it is permanently stored in the main memory of the computer. The resident procedure and «hardware interruptions» ensure a high rate of the PC response to the LAM-IRQ signal and comfortable display of accumulated data. After the procedure is started in the PC and conversion is completed in the ADC, the data arrive at the computer through the controller «Bus contrl.». After the data transfer energy selection begins and the measuring instruments are set ready for recording the next events while processing of the last event is still on, that is the instrumentation and the PC operate in parallel in the «pipeline» mode. This organization allows the dead time to be reduced by 20–30%.

In the Master PAC fast time selection is effected at high input loads, in the computer energy selection-sorting is effected by e_ADC codes. Before the experiment two energy windows are set in the program for each detector, ΔE_{γ_1} corresponding to the γ -quantum populating the intermediate level and ΔE_{γ_2} corresponding to the γ -quantum de-exciting it. The code of each e_ADC is compared with the windows ΔE_{γ_1} and ΔE_{γ_2} set for it. If the code of one of the time-coincident e_ADCs falls within the window ΔE_{γ_1} and code of the other e_ADC falls within its preset window ΔE_{γ_2} , the event is considered useful. The operation of a pair of detectors (6 variants for 4 detectors) and the result of the energy selection (2 variants) form 12 time spectra to which the t_ADC code will be added.

The codes from the e_ADC are used not only for selection of t_ADC codes but also for accumulation of four energy spectra, i.e. the code of each triggered e_ADC is recorded in the corresponding energy spectrum. The shape of these spectra is used to check the operation stability of the detectors during the experiment and may later be used to normalize time spectra.

The spectrometer makes it possible to carry out experiments in the multidimensional analysis mode. In this case ADC codes for each event may be stored on an event-by-event basis, i.e. files are formed where each event consisting of the codes of two e_ADC and one t_ADC is successively written. After these files are written, time spectra of interest may be formed by optimizing boundaries of energy windows and other parameters.

In a spectrometer like this the number of detectors can be easily varied. There are no problems with increasing the number of energy windows for any detector and with adding pressure, temperature detector and others.

Checking and setting-up are particularly troublesome procedures in PAC spectrometers. To cope with most problems arising in the set-up mode, additional procedures in the program and a special mode of operation of the Master PAC are provided.

When the programmed set-up/test procedure is started by a command from the computer, the Master PAC is switched to the test mode. In this case e_ADCs are triggered by each detector signal. After conversion the code of an e_ADC is stored in the computer. Energy spectra are constructed for each detector. On the basis of their shape

- operation of the detectors is appraised;
- energy ranges of detector operation are selected by varying the high voltage of the photomultiplier (HV unit) and the amplification factor (Ampl. unit);
- CFD thresholds are set;
- boundaries of the energy windows ΔE_{γ_1} and ΔE_{γ_2} are set.

All settings are made without an oscilloscope and without reconnection of units. The spectrometer can be periodically switched to the test mode during data taking without interrupting measurements.

Energy selection of events in the computer, unlike the traditional one with the use of differential discriminators, allowed the operation of the spectrometer to be much simpler, the data taking control to be improved, the energy selection to be optimized, and the spectrometer set-up time to be reduced.

2. MASTER PAC

The only specialized unit of the spectrometer is the Master PAC. As mentioned in the previous section, the Master PAC may operate in two modes, basic and set-up. The desirable mode is selected by the PC command. Basic mode functions are:

- determination of signals from any two detectors which coincided within the preset time range (for four detectors there are 6 variants);
- generation of the START and STOP pulses with the interval between them equal to the interval between the CFD signals;
- triggering of ADCs and launching the procedure of reading data into the computer;
- transmission of the numbers of the detectors whose signals appear at the START and STOP outputs to the computer.

Depending on the time interval between the signals from the detectors two versions of the basic mode of operation are possible.

The first is when operation of one of the detectors is not followed within the present time interval by operation of the other three detectors for they did not detect radiation, i. e. there are no coincidences. In this case the Master PAC returns to the initial state as fast as possible for receiving next signals. This initialization time is less than 100 ns (i. e. considerably smaller than initialization in TAC). With the event rate 100000 events/s at all inputs the counting errors do not exceed 1%.

The second is when two detectors detected radiation simultaneously or one after the other within the preset time interval, i. e. coincidence is detected. Interaction of spectrometer units in this case is described in the previous section. While conversion of the analogue signals to code and their transfer to the computer take place, the Master PAC inputs are closed. For our spectrometer this time varies from 10 to 20 μ s. After the data transfer is completed, the computer sends the Reset signal. By this signal all spectrometer units are initialized and the Master PAC inputs switch to the open state. The number of coincidences for a four-detector PAC spectrometer do not normally exceed 2000 events/s. At such a load the dead time of the spectrometer is not larger than 4%, and the total dead time, with non-coincidence cases (1%) taken into account, will be less than 5%.

In the set-up mode the Master PAC supplies GATE signals to the e-ADC each time there appear a signal from the CDF (irrespective of coincidences).

Setting-up of the Master PAC itself consists in setting the coincidence time, which is done by the «Time of coincidence» switch on its front panel. There are eight options: 100, 200, 300, 400, 500, 600, 700, and 800 ns.

3. SOFTWARE FOR THE $\gamma\gamma$ PAC SPECTROMETER

The operation of the spectrometer is controlled by the IBM-compatible computer which is a part of the spectrometer. Most of the software is written in PASCAL. Parts of the programs critical for their execution are in ASSEMBLER.

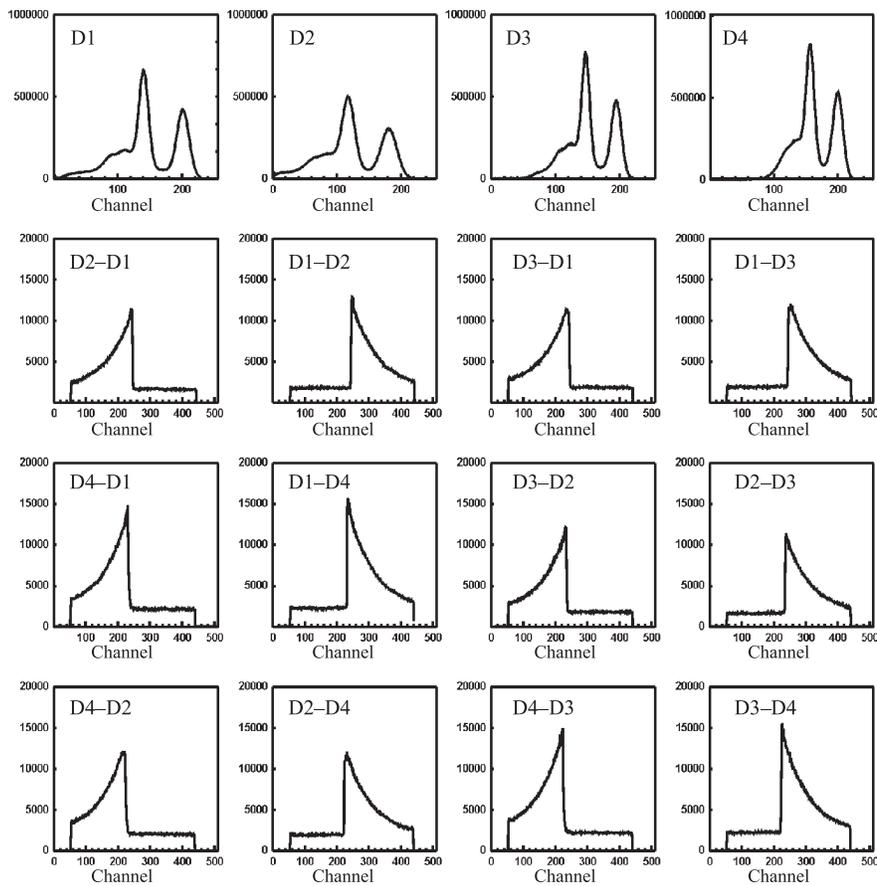


Fig. 2. Four energy spectra and 12 time spectra obtained with ^{111}In

Now a version of the accumulation program with on-line visualization is developed for working under the Linux operating system with using ROOT 3.10 environment.

The program allows accumulation of four energy spectra and 12 time spectra (Fig. 2). To each of 12 time spectra there corresponds its own combination of the triggered detectors and the energy windows $\Delta E\gamma_1$ and $\Delta E\gamma_2$. The program regularly saves the data on a disc without interrupting measurements.

4. SUPPRESSION OF INTERCHANNEL INTERFERENCE

Interchannel interference (pick-up) is a common problem of multichannel spectrometers. In our case this interference manifests itself as wave-like structures in the spectra of delayed coincidences. Figure 3 shows a spectrum of accidental

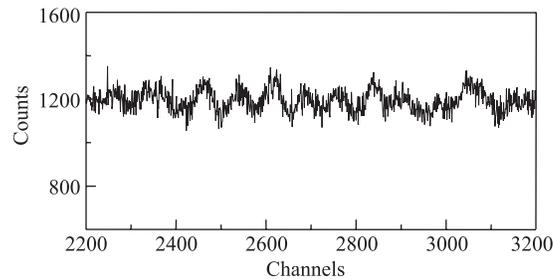


Fig. 3. Spectrum of accidental coincidences measured with a ^{137}Cs source

coincidences from two detectors measured with a ^{137}Cs source. A wave-like structure is seen in the spectrum.

It was decided to solve this problem by using the statistical level-off method («sliding scale» method) developed by Gatti [6]. An additional unit CD (see Fig. 1) with a few cable delays (four in our case) is installed in each of four channels of the spectrometer. The delays vary from 1 to 20 ns, which corresponds to the pick-up «period». The delay duration is changed by the PC command.

During measurements the program periodically (e. g. once a minute) changes the combination of delays in the units. On detection of an event the time ADC code changes by a value corresponding to the given combination of delays. Thus, the spurious effect is «leveled off» while the useful signal remains as it is. Figure 4 shows two time spectra measured with the ($^{137}\text{Cs} + ^{60}\text{Co}$) source without (a) and with (b) the use of the «sliding scale» method. The interference is seen to be appreciably suppressed while the peak of prompt coincidences is not smeared out.

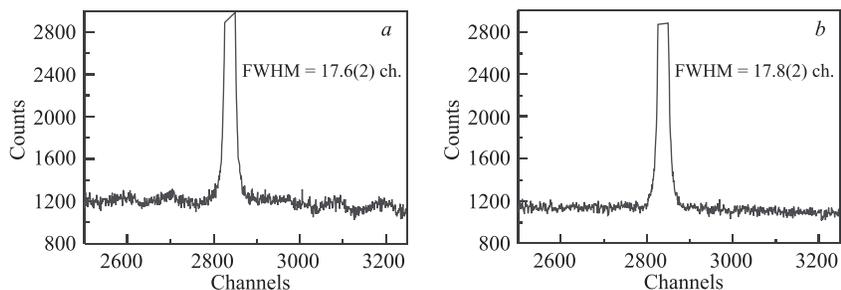


Fig. 4. Time spectra measured with the ($^{137}\text{Cs} + ^{60}\text{Co}$) source: a) without sliding scale; b) with sliding scale

5. PAC MEASUREMENT AT HIGH PRESSURE

The main advantages of the PAC method for studying materials under high pressure are

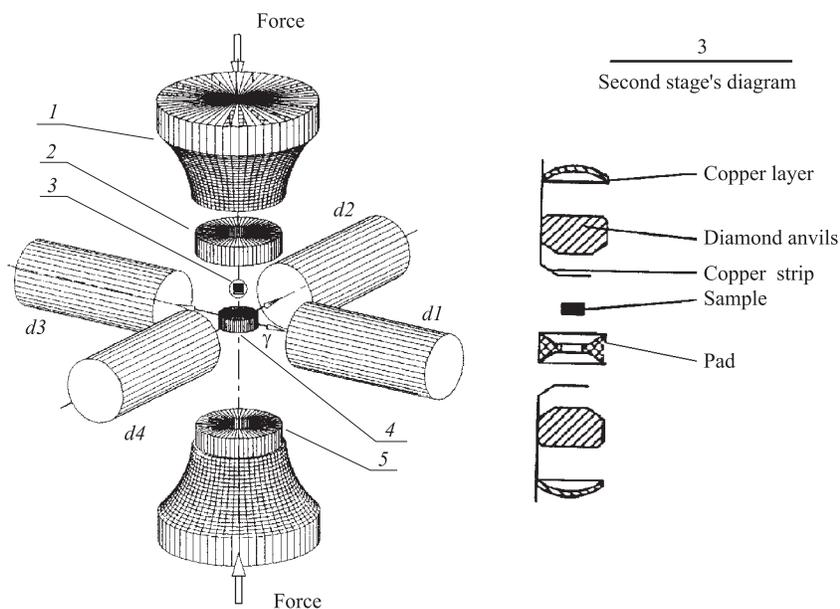


Fig. 5. Schematic view of the apparatus for high-pressure PAC measurements: 1 — support transferring force from the press; 2, 5 — first stage of the high-pressure chamber, upper and lower parts; 3 — second state of the high-pressure chamber and a sample (right: schematic view of the second stage assembly); 4 — catlinite cell transferring and maintaining pressure

- the possibility of using isotopes with a sufficiently high γ -radiation energy, which considerably simplifies technical support of the experiment;
- a small (0.3–0.5 mm³) volume of samples.

A many-stage high-pressure apparatus of the «matryoshka» type [7] allows a pressure up to 60 GPa to be generated in a volume sufficient for PAC measurements in a sample under pressure at the temperature of liquid nitrogen and higher.

To carry out under-pressure PAC measurements, the four-detector spectrometer is equipped with a small-size hydraulic four-arm press of capacity up to 300 t, which makes it possible to use the many-stage high-pressure apparatus (Fig. 5). A «toroid»-type chamber [8] is used as the first stage. Its holes 10 and 15 mm in diameter allow generation of pressure up to 10 and 8 GPa, respectively. Bridgman anvils are used as the second stage. They are made either of carbonado (sintered artificial diamonds) or of sintered natural diamonds, which allows pressures about 60 GPa to be attained.

Resistance transitions of reference substances or residual quartz density-against-pressure curves were used to calibrate the apparatus [7].

Quartz and catlinite (Ca and Al oxide) or pyrophyllite were used as a medium transferring and maintaining pressure.

6. THERMOSTATING SYSTEM

The spectrometer is equipped with a thermostat and a high-temperature oven to measure temperature dependence of the HFI parameters.

The thermostat chamber (Fig. 6) is tightly fixed on the upper end of the evacuated siphon with vacuum insulation. The siphon enters the Dewar vessel with liquid nitrogen through the air-tight seal in its neck. The pressure in the Dewar vessel is a few millibars higher than the atmospheric pressure. Cold nitrogen vapour moves over the siphon from the vapor region through the heat exchanger at the bottom of the Dewar vessel to the experimental chamber. The cold nitrogen vapour is heated to the required temperature by the electric heater inside the isothermal unit. A thermocouple and commercial electronic devices intended for controlling industrial processes are used to control and stabilize temperature. Then the heated gas uniformly cools the experimental ampoule on all sides and passes to atmosphere over the spiral groove between the foam plastic plug and the body of the experimental chamber. The thermostat makes it possible to cover the temperature range from 120 to 350 K and to maintain the temperature with an error of 0.5 K.

The main element of the high-temperature oven (Fig. 7) is the refractory block with four cone-shaped windows to let radiation from the sources under study pass to the detectors and a vertical channel to accommodate the heater. The block is

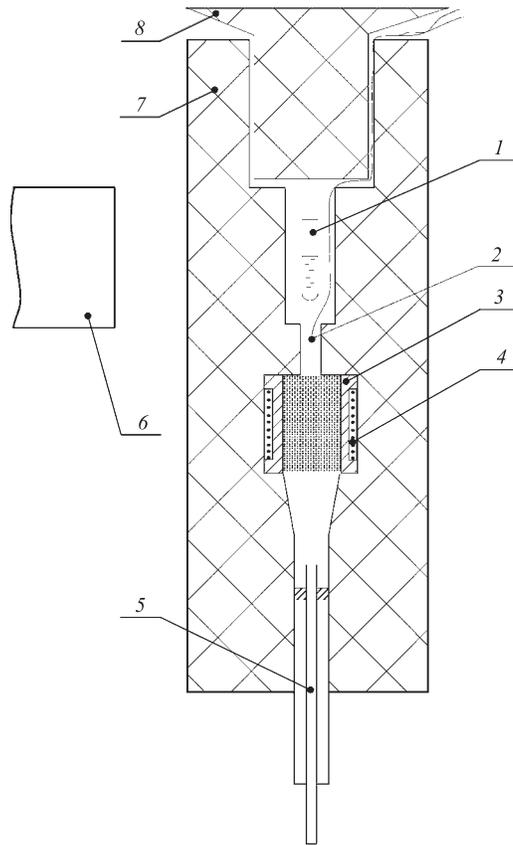


Fig. 6. Head part of the thermostat: 1 — capsule with a sample to be studied; 2 — thermocouple; 3 — isothermic heat exchanger of sintered large-grain copper powder; 4 — heater; 5 — evacuated siphon; 6 — detector; 7 — foam plastic heat insulation; 8 — foam plastic plug

made of foam chamotte brick. The cone-shaped space of the windows is insulated with Goodfellow refractory wool of non-woven aluminium oxide fibres. The wool is kept in the windows by a thin capton film. The electric tantalum-wire heater of the oven is wrapped on an aluminium oxide pipe inserted with its closed end up in the channel of the block. The rest of the oven space inside the steel body is filled with foam chamotte crumb. Temperature is stabilized and controlled in the range up to 1300 K by a computer through the above-mentioned electronic devices.

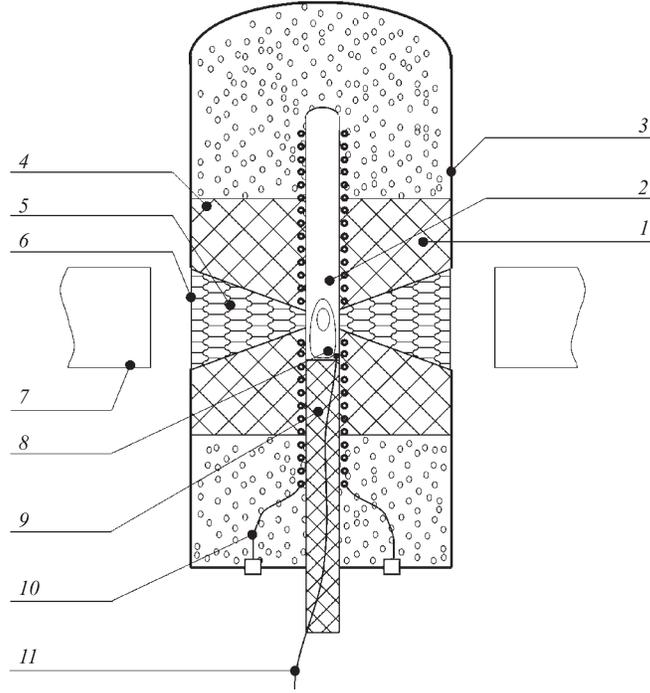


Fig. 7. High-temperature oven: 1 — refractory block; 2 — aluminium oxide pipe; 3 — stainless steel body; 4 — chopped foam firebrick; 5 — refractory wool of thin aluminium oxide fibres; 6 — thin capton foil; 7 — detector; 8 — refractory air-tight capsule with a sample; 9 — heat plug; 10 — heater; 11 — thermocouple

7. CONSTRUCTION OF THE ANISOTROPY FUNCTION

The procedure for construction of the anisotropy function from the time spectra is as follows.

1) For all time spectra «zero» channels are determined from the independent measurement of the spectra of prompt coincidences with ^{60}Co . Six spectra are rotated with respect to their «zero» and then all spectra are adjusted by the «zero» channels.

2) The time spectra (TS) are normalized to the number of counts in the windows $\Delta E\gamma_1$ and $\Delta E\gamma_2$. The normalization coefficient K_i for the i th TS is

$$K_i = \frac{SS_i}{\min \{SS_i\}}, \quad (1)$$

where $SS_i = S_{\text{start}}S_{\text{stop}}$; $S_{\text{start}}, S_{\text{stop}}$ are the numbers of events in the windows

of energy spectra corresponding to the «start» and «stop» γ -quanta for a particular TS. A spectrum is normalized:

$$n_i^\theta(t) = \frac{N_i^\theta(t)}{K_i}, \quad (2)$$

where $N_i^\theta(t)$, $n_i^\theta(t)$ are the numbers of events in the i th TS for the angle θ (90 and 180°) before and after normalization.

3) The background from accidental coincidences is taken into account:

$$C_i^\theta(t) = n_i^\theta(t) - F_i^\theta(t), \quad (3)$$

where $C_i^\theta(t)$ is the number of true coincidences, $F_i^\theta(t)$ is background from accidental coincidences.

4) TS are multiplied channel by channel:

$$X_{90}(t) = \left[\prod_{i=1}^8 C_i^{90}(t) \right]^{1/8}, \quad (4a)$$

$$X_{180}(t) = \left[\prod_{i=1}^4 C_i^{180}(t) \right]^{1/4}. \quad (4b)$$

5) The anisotropy function is calculated

$$R(t) = 2 \cdot \frac{X^{180}(t) - X^{90}(t)}{X^{180}(t) + 2 \cdot X^{90}(t)}. \quad (5)$$

The statistical error $\sigma_{R(t)}$ is calculated:

$$\sigma_{R(t)} = 6 \cdot \frac{X^{180}(t) \cdot X^{90}(t)}{(X^{180}(t) + 2 \cdot X^{90}(t))^2} \cdot \left[\sum_{\theta} \frac{1}{N_{\theta}^2} \cdot \sum_{t=1}^{N_{\theta}} \frac{C_i^{\theta}(t) + 2 \cdot F_i^{\theta}(t)}{C_i^{\theta}(t)^2} \right]^{1/2}. \quad (6)$$

The channel division value in the TS for further calculations is determined from the independent measurement with a calibrated delay introduced before time-to-amplitude conversion.

8. EXPERIMENTAL RESULTS AND CONCLUSION

A four-detector spectrometer of perturbed angular $\gamma\gamma$ correlations is developed with the following characteristics:

- time range 1–800 ns;

- energy resolution (^{137}Cs 662 keV) 10% with BaF_2 , 8% with NaI(Tl) ; (Fig. 8.)
- time resolution (^{60}Co 1173–1332 keV) 200 ps with BaF_2 , 2.0 ns with NaI(Tl) . (Fig. 9.)

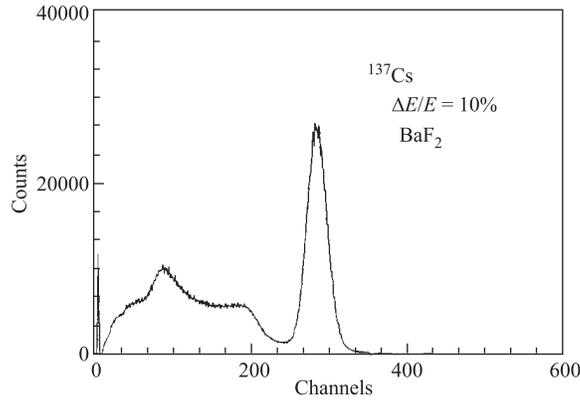


Fig. 8. ^{137}Cs energy spectrum

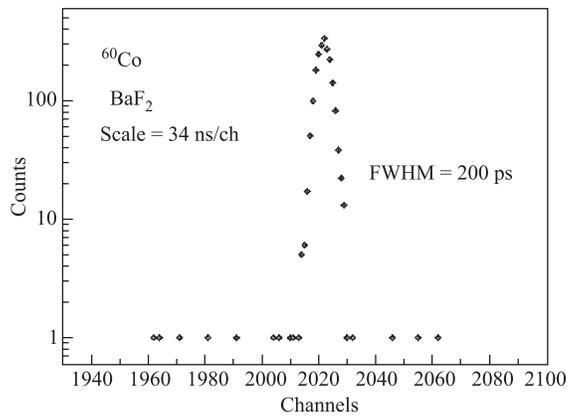


Fig. 9. Spectrum of prompt coincidences with the ^{60}Co source

A wide range of investigations in solid state physics and physical chemistry were carried out with the PAC spectrometer, in particular PAC investigations in frozen aqueous solutions with the use of $^{111}\text{In(EC)}^{111}\text{Cd}$. One of the measured anisotropy spectra is shown in Fig. 10.

These measurements revealed the mechanism for the dynamic character of PAC in ice. Fluctuation of the electric field gradient in this system was attributed to mobility of ion and orientation defects [5].

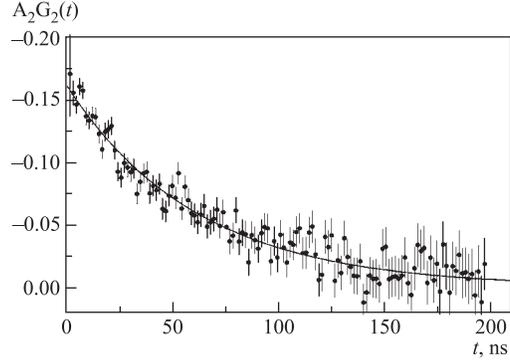


Fig. 10. Dynamic character of PAC ($^{111}\text{In}(\text{EC})^{111}\text{Cd}$) in ice

The angular correlation anisotropy spectrum for ^{111}Cd nuclei in LaRu_2 (Fig. 11) processed by the DEPACK program [9]. The hyperfine interaction parameters ($\nu_Q = 242(3)$ MHz, $V_{zz} = (1.2 \pm 0.2) \cdot 10^{18}$ V/cm 2) derived from this spectrum are in good agreement with the results derived from the spectra measured with a three-detector spectrometer of the Institute of Nuclear Physics, Moscow State University [10].

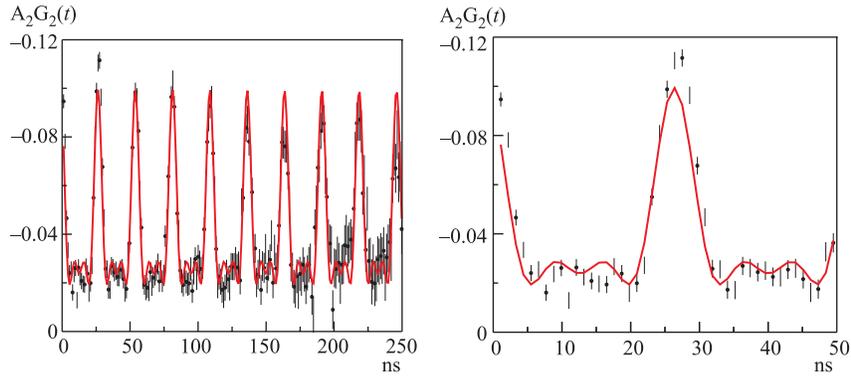


Fig. 11. Angular correlation anisotropy spectrum for ^{111}Cd nuclei in LaRu_2

Temperature dependence of the EFG on ^{111}Cd nuclei at boron sites was measured in the superconductor MgB_2 synthesized at a pressure of 8 GPa. The experimental and calculated EFG values were in good agreement.

The measuring complex described in the paper will allow on-line studies of variation in the electric field gradient on ^{111}Cd impurity nuclei with valence of rare-earth ions in Laves cubic phases.

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