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THE INNOVATIVE DIRECTION OF SCIENTIFIC INSTRUMENTATION — MÖSSBAUER SPECTROSCOPY AS A FACTOR OF IMPROVING THE BRANCHES OF THE RUSSIAN ECONOMY.

PART 1. BREAKTHROUGH SCIENTIFIC RESEARCH IN THE FIELD OF MÖSSBAUER SPECTROSCOPY

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Breakthrough scientific research in the field of Mössbauer spectroscopy is described. The mass-produced IAI RAS Mössbauer spectrometers are presented, which largely determine the development of Russian materials science and are used in the development of new materials. A complex of scientific instruments illustrating the development of one of the most promising areas of scientific instrumentation, that is necessary for studying the magnetic and physico-chemical properties of new materials, investigating the high-temperature superconductivity of compounds, ferroelectrics, and multiferroics is demonstrated.

Keywords: innovations, import substitution, Mössbauer spectrometer, gamma-resonance spectrum, Doppler energy modulation, gamma optical scheme

1. DEFINITION OF A PERSPECTIVE DIRECTION OF DEVELOPMENT OF DOMESTIC SCIENTIFIC INSTRUMENT MAKING

At present, the fundamental science is supported by the state, and its financing is carried out by the Federal Agency of Scientific Organizations of Russia (FANO of Russia). Along with the traditionally developed areas of Russian science: space exploration, medical research, fundamental nuclear and nuclear physics, Russian scientists successfully create innovative technologies in such areas as IT technology, nanotechnology, biotechnology, and the creation of new structural materials [1].

The prerequisites for the successful development of modern domestic instrumentation are the widespread use of IT technologies, as well as the fact that modern scientific instruments make measurements with an error in instruments equal to 10^{-8} cm, comparable to the size of one atom, with speed of light being the basis for accuracy of many parameters.

In retrospect, the development of Russian instrument making is as follows. In the period from 1990 to 2014, Russia was able to compensate for the backlog from world leaders in the field of scientific instrument making, using significant imports of scientific instruments from the EU, US and Japan.

Due to a variety of imported devices from abroad, competition arose in the Russian instrument-making market between domestic instrument making and foreign. Domestic instrument-making firms, through the import of instruments, were able to familiarize themselves with systems and technologies of foreign production. Simultaneously, domestic instrument-making firms continued to produce their own products, carrying out its modernization, retaining their share in the strategically important high-tech sector of the instrument-making market, due to the competitive advantage in the form of lower prices.

Foreign developers of the latest scientific instruments hide the innovative technologies used in them. In Russia, foreign devices are often purchased without knowing the algorithm of its functioning, which is detailed in the device. Such an approach in acquiring foreign devices does not lead to the successful development of innovative technologies in Russia and does not meet the priorities laid down in the national security strategy. The interests of national security in particular imply that it is technologically impossible to depend on the import of foreign equipment and devices. Reflecting the state position on this issue, FAO Russia supports domestic instrument manufacturers [2].

The most obvious national interests in the innovation sphere are illustrated by the Global Positioning System (GPS), which is analogous to the national navigation system GLONASS. China is developing its national navigation system "Beidou", the European Union is forming its national navigation system "Galileo". It should be noted that the cost of each national navigation system is billions of US dollars, while the EU countries and the United States at the same time are "reliable partners".

FANO of Russia together with the Russian Academy of Sciences develops the available innovative potential in scientific instrument making, and chooses breakthrough directions for the further development of instrument making in the priority areas of development of science and technology in the sectors of the Russian economy. Russian scientists are trying to pass several stages of development of the industry, knowing the dead-end directions of development of advanced scientific instrument making in various sectors of the foreign economy.

IAI RAS analyzes the main world trends in the development of scientific instrument making. One of the directions of the development of domestic and world instrument making is the creation of the Mössbauer Spectrometer (hereinafter referred to as the Spectrometer) is a multipurpose instrument for the nuclear gamma-resonance spectra acquisition and data processing.

2. MÖSSBAUER SPECTROMETER

Spectrometer can be used for fundamental scientific research and applied studies in physics, chemistry, geology, biology, medicine, agriculture, industry, and other areas of scientific research, where it is required to investigate for a certain depth the surface of materials that are different in chemical composition [3–6].

IAI RAS has developed and serially produces a set of instruments for multidimensional parametric Mössbauer spectroscopy. Spectrometer assures measurements with resonance transformers: resonance counters, filters, polarizes, locks etc.

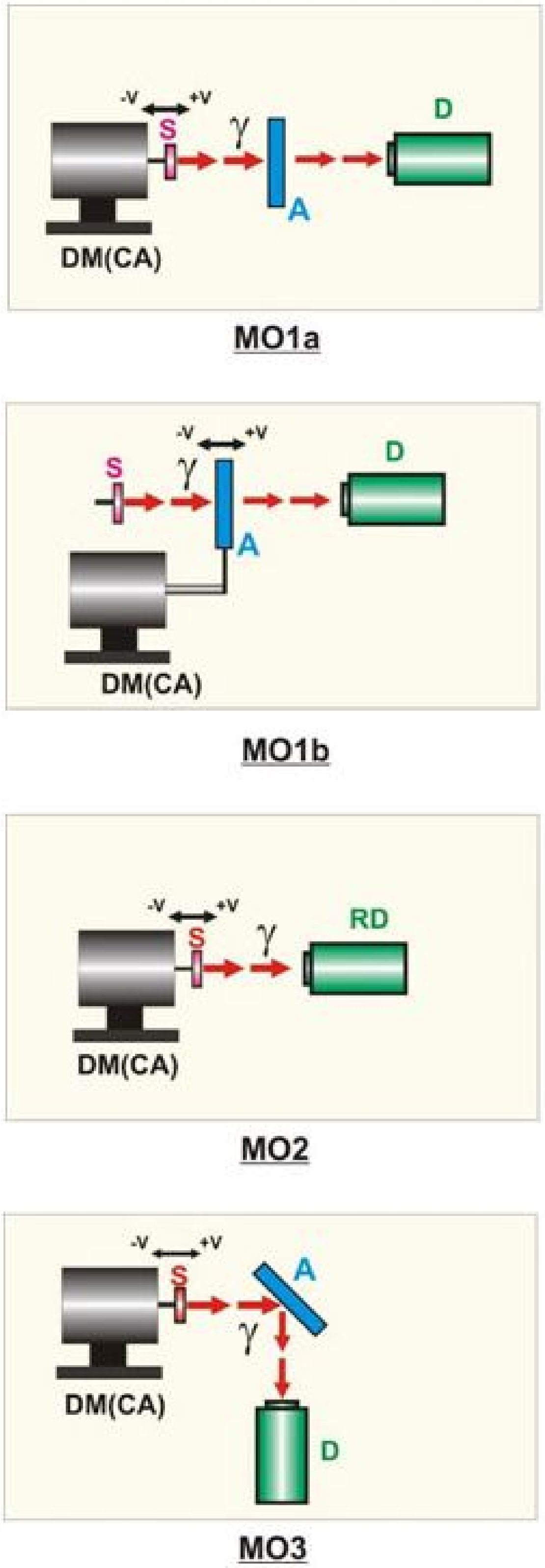
Spectrometer allows simultaneous and independent accumulation of the two pulse height and two Mössbauer spectra, displaying the accumulated spectra on to monitor, normalization and expansion of spectra, data and graphic output to the printer, data saving on disk, data retrieving from disk, mathematical data processing in a friendly dialogue manner, documentation of the results in tabular or graph forms.

Spectrometer provides simultaneous storage, data processing and display of the incoming information.

A spectrum can be accumulated in one of the following modes: preset counts per channel, preset number of cycles, preset time.

Spectrometer is a multifunctional instrument, which makes possible multiple Doppler modulations with automated gamma-resonance spectrum acquisition.

Spectrometer provides automated spectra accumulation in the following Mössbauer Gamma-Optics (MO): transmission; emission; forward-, back- and angular scattering; Rayleigh scattering of Mössbauer



MO1a

MO1b

MO2

MO3

Fig. 1. When operating with a single Doppler modulation system the Spectrometer performs the following measurement functions: (MO1, MO2, MO3)

Radiation (RSMR); Selective Excitation Double Mössbauer Effect (SEDM) and any combinations of the above.

When operating with a single Doppler modulation system the Spectrometer performs the following measurement functions (Fig. 1, Fig. 2):

– Resonance transmission, emission and scattering

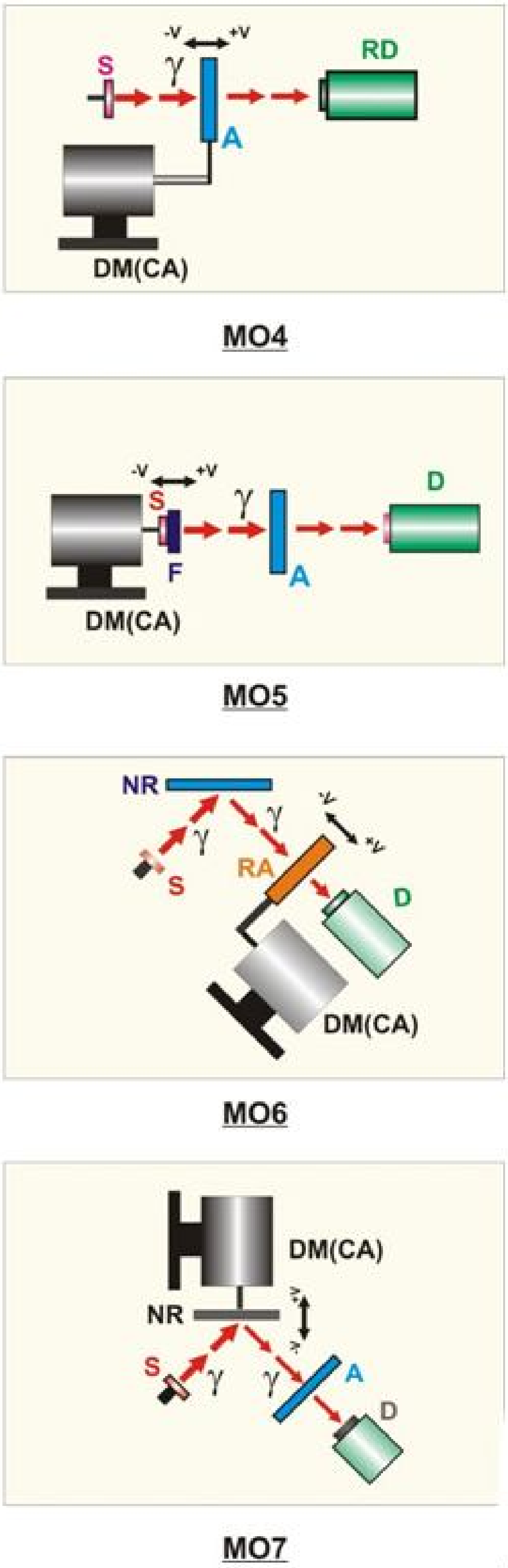


Fig. 2. When operating with a single Doppler modulation system the Spectrometer performs the following measurement functions: (MO4, MO5, MO6, MO7)

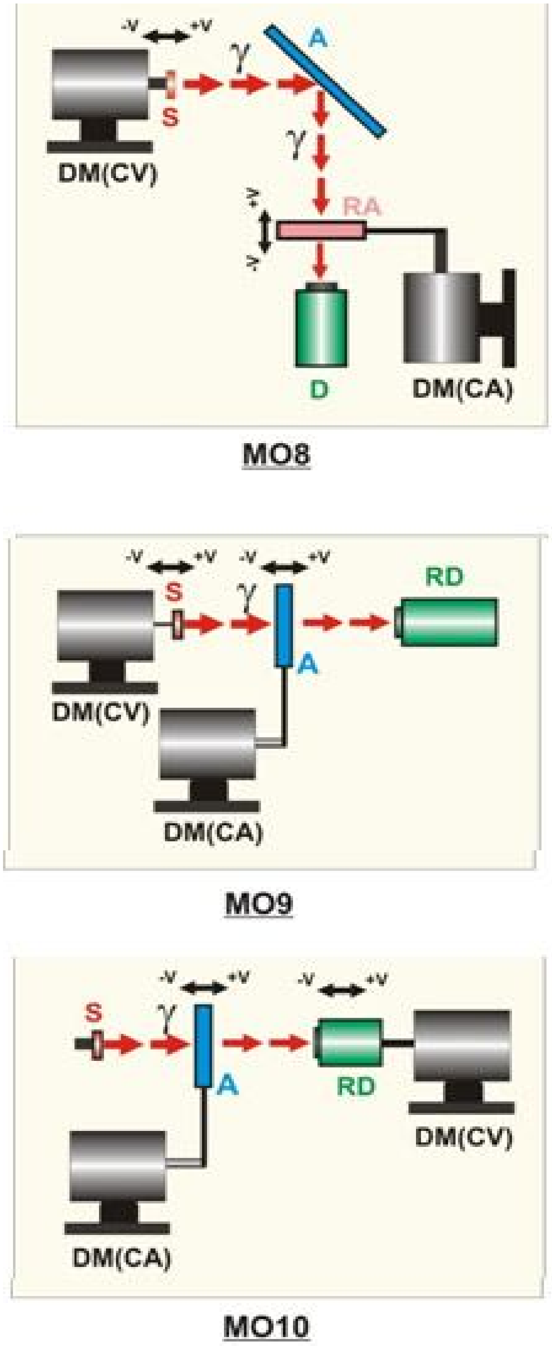


Fig. 3. When operating with the double Doppler modulation system the Spectrometer performs the following additional functions: (MO8, MO9, MO10)

spectra (MO1, MO2, MO3).

– Spectrum with resonance counter, when source and converter lines coincide (MO4).

– Spectrum with selected resonance filters or polarizers (MO5).

– Rayleigh scattering of Mössbauer radiation (MO6).

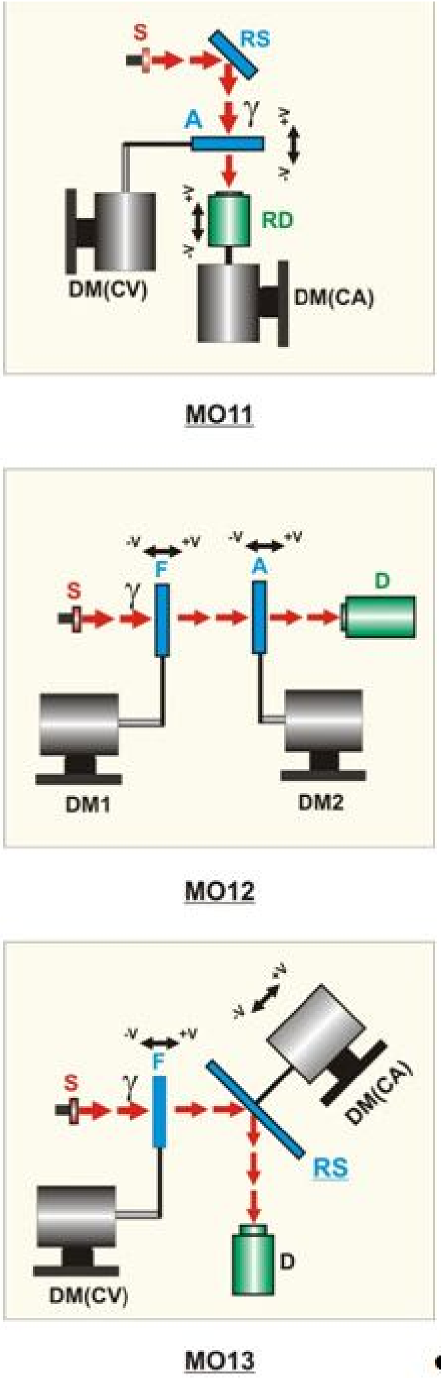
– Transmission or scattering spectrum with intermediate imposed nonresonance scatterer with fixed source and absorber (MO7) [7–10].

When operating with the double Doppler modulation system the Spectrometer performs the following additional functions (Fig. 3–6):

– Selective Excitation Double Mössbauer Effect (MO8).

– Resonance spectrum with resonance counter by compensation technique (MO9).

– Transmission or scattering spectrum with polarized resonance counter (MO10, MO11).

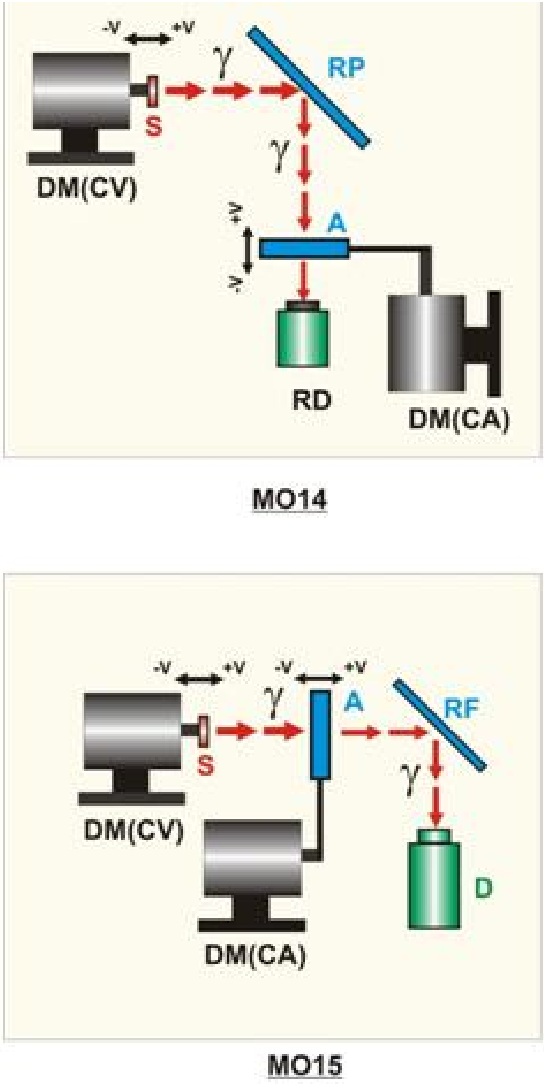


MO11

MO12

MO13

Fig. 4. When operating with the double Doppler modulation system the Spectrometer performs the following additional functions: (MO11, MO12, MO13)



MO14

MO15

Fig. 5. When operating with the double Doppler modulation system the Spectrometer performs the following additional functions: (MO14, MO15)

– Transmission or scattering spectrum with a single or multiline resonance filters by compensation technique (MO12, MO13).

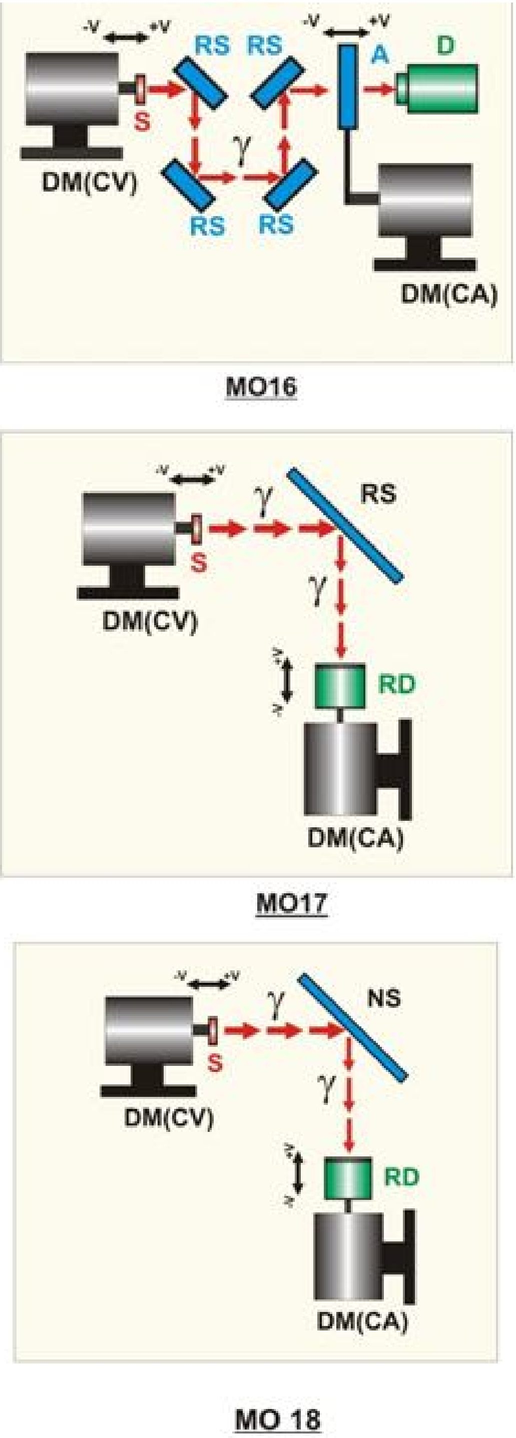


Fig. 6. When operating with the double Doppler modulation system the Spectrometer performs the following additional functions: (MO16, MO17, MO18)

– Transmission spectrum with the use of polarized beams, when polarization is induced by intermediate single crystal polarizer-scatterer (MO14).

– High resolution spectrum, when line narrowing is obtained by tuning to resonance the centers of the source emission and resonance scatterer absorption lines (MO15).

– High resolution spectrum with multiple scattering technique (MO16).

– High resolution, high sensitivity and time saving experiments on Selective Excitation Double Mössbauer Effect with resonance counter (MO17).

– High resolution, high sensitivity and time saving experiments on Rayleigh scattering of Mössbauer radiation in a resonance counter mode (MO18).

– Furthermore, the Spectrometer allows performing investigation of the region of interest part of resonance spectrum (ROI-mode) for any Mössbauer Gamma-Optics.

3. PRINCIPLE OF OPERATION OF A SPECTROMETER USING THE MÖSSBAUER EFFECT

The choice of a particular function is determined by Mössbauer Gamma-Optics, i.e. assembly's arrangement in the analytical cabinet (optical bench) and by the required program loaded into the automated control system. Block-diagram of Spectrometer in MO8 optics is shown in Fig. 7.

Spectrometer consists of an analytical cabinet and control and energy selection systems interfaced to a personal computer.

The analytical cabinet includes vibrodamping and carrier platforms, two Doppler modulators (DM(CV), DM(CA)), gamma-ray detectors with built-in preamplifiers (D1, D2), guard and shielding collimators, various sample and source holders installed on the latter platform.

The control and energy selection systems together with necessary equipment consist of: two Modulator Drivers (MD1, MD2), two Discriminators (Single Channel Analyzers) with built-in amplifiers (SCA), two Low Voltage Power Stabilizer units (LV), two High Voltage Power Supplies (HV), two Specialized Multichannel Accumulator units (SA), and other accessories, i.e. interface card, coaxial and power cables, connectors, etc.

In Mössbauer Gamma-Optics, shown in Fig. 7, the radioactive source S is rigidly attached to the moving part of Doppler modulator DM(CV), while the resonance filter-analyzer RA to the other Doppler modulator DM(CA).

Modulator drivers MD(CV) or MD(CA) provide start and memory (channel) advance pulses for the synchronization of the entire system. These pulses are then fed to the Specialized Accumulator units SA.

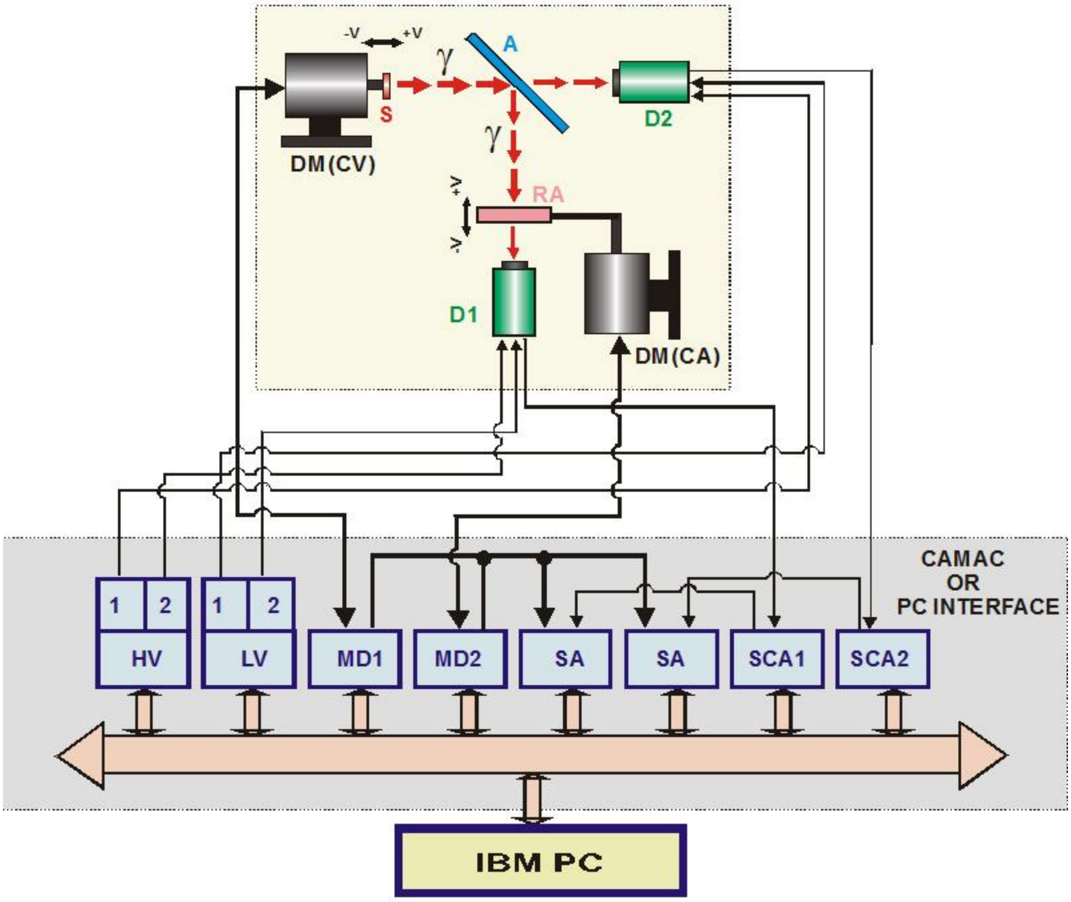


Fig. 7. Block-diagram of Spectrometer in MO8 optics

Modulator Drivers MD(CV) and MD(CA) provide the control voltage signals for the Doppler Modulators DM(CV) and DM(CA), thus making them vibrate according to the preset law of motion.

After the scatter, gamma-quanta are recorded by the detector D1. The electric output signals of the detector are received by the preamplifier.

The preamplifier, which is located in the detector housing, converts a charge pulse of the detector to voltage and provides matching between detector output and SCA1 input. After amplification and shaping of the detector signal, the pulses from the preamplifier are sent to the input of SCA1 with built-in amplifier.

The amplitude selected and normalized pulses from SCA1 output are fed for counting to the information input of Specialized Accumulator unit.

The exact matching of the working part of moving cycles with the channel of SA is provided by start and channel advance pulses from master MD unit.

High voltage for the detector is provided by HV (bias) and the stabilized voltages for preamplifiers are supplied by the Low Voltage Power Stabilizer unit LV.

The counting cycle begins with the arrival of starting pulse and stops after 4096 channel advance pulses have passed.

The path D2→SCA2→SA serves as monitor channel.

During accumulation, the spectrum can be forwarded to the display monitor using the main program.

In the pulse height analysis mode the main program realizes the settings of:

- Levels of the High Voltage Power Supply;
- Single Channel Analyzer parameters: polarity, gains, window;

In the Mössbauer spectrum acquisition mode (multiscalor mode) the main program realizes the settings of:

- Single Channel Analyzer parameters;
- The law and the parameter values for DM;
- Mossbauer Spectrum display;
- Memory cell number and its volume.

In both cases, the software allows to save spectrum and to retrieve it from computer memory, to expand and compress the spectrum pattern, to normalize the spectrum, to output it to the printer in tabular or graphic form.

4. ADVANTAGES OF THE RUSSIAN MÖSSBAUER SPECTROMETER

The advantage of the spectrometer relative to foreign analogs can be illustrated by comparing the capabilities of MO1b and MO9.

Fig. 8 shows Mössbauer spectra of the products from the reaction used to produce SnS compounds measured with the conventional MO1 scheme (left) and with the isomer shift compensation technique MO9 [11–14]. The results in Fig. 8 demonstrate that in the transmission mode Spectrometer makes it possible to enhance the resolution up to 27% and sensitivity up to 10 times.

The most common Mössbauer Spectrometer SM 2201DR. Mössbauer Spectrometer SM 2201DR is an automated problem-oriented spectrometer allowing experiments on the selectively-induced double Mössbauer effect and Rayleigh scattering of resonant radiation. Over some period of time the main techniques for studying dynamic processes of nucleus interaction with the electron shell have been Nuclear Magnetic Resonance (NMR) and Electron Spin Resonance (ESR).

The Mössbauer Spectroscopy technique, owing to its high sensitivity to hyperfine interactions between the nucleus and its environment, has greatly extended the range of substances that can be investigated, and in certain cases has become a unique method for studying these phenomena.

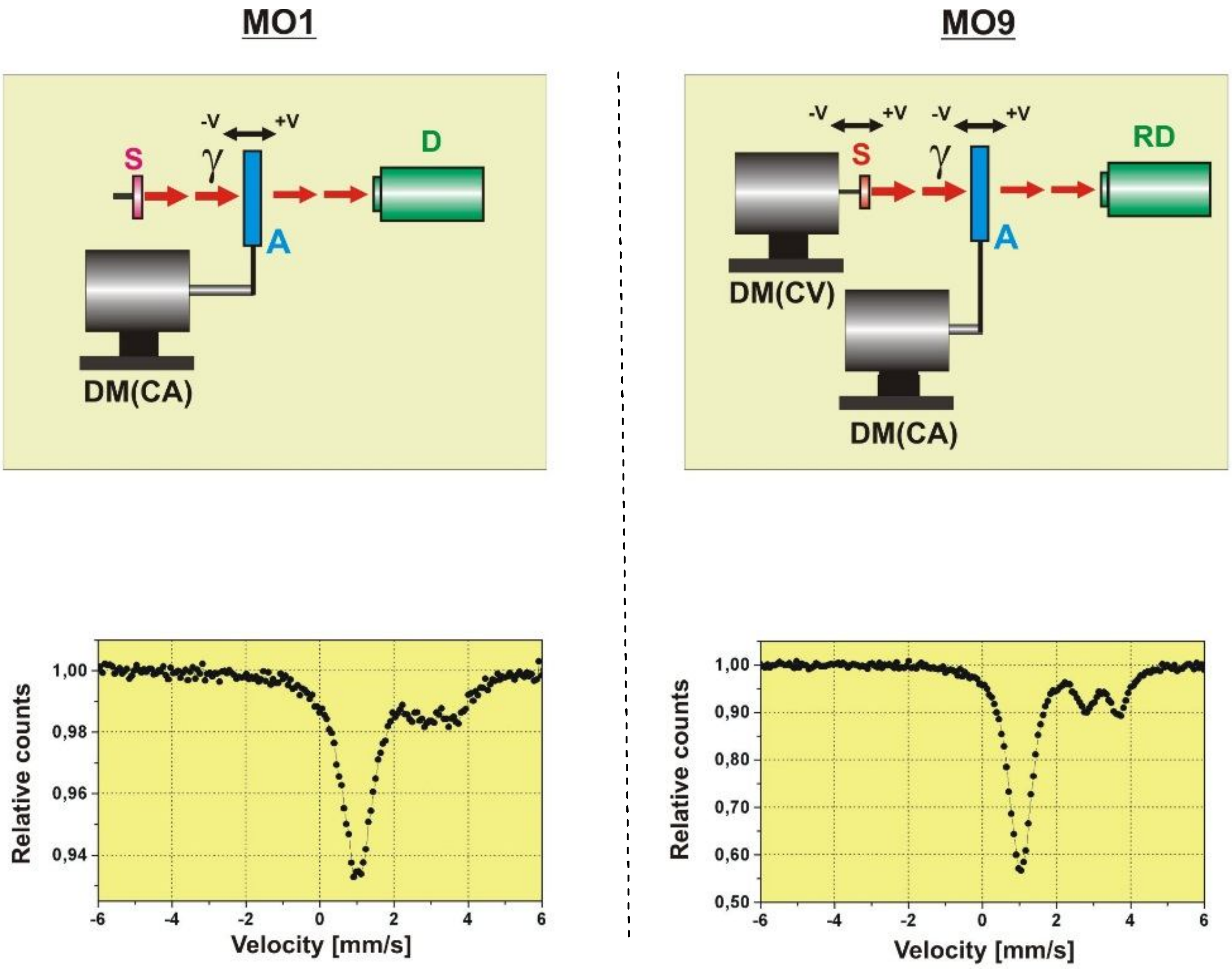


Fig. 8. Mössbauer spectra of the products from the reaction used to produce SnS compounds measured with the conventional MO1 scheme (left) and with the isomer shift compensation technique MO9

Most of experiments [15, 16] that used Mössbauer spectroscopy to study various dynamic processes (e.g., diffusion, paramagnetic, spin-spin, spin-lattice relaxation) were done in the conventional transmission geometry. This technique is simple enough, but in some applications it leads to difficulties in resolving the contribution from dynamic processes to the total spectrum. For example, in the case of slow relaxation the transmission spectrum is characterized by the line broadening, only. However, the broadening may be caused also by instrumental effects, finite thickness of the sample under investigation, multiphase effects, unresolved hyperfine electric and magnetic interactions, that lead to an ambiguity in spectrum interpretation.

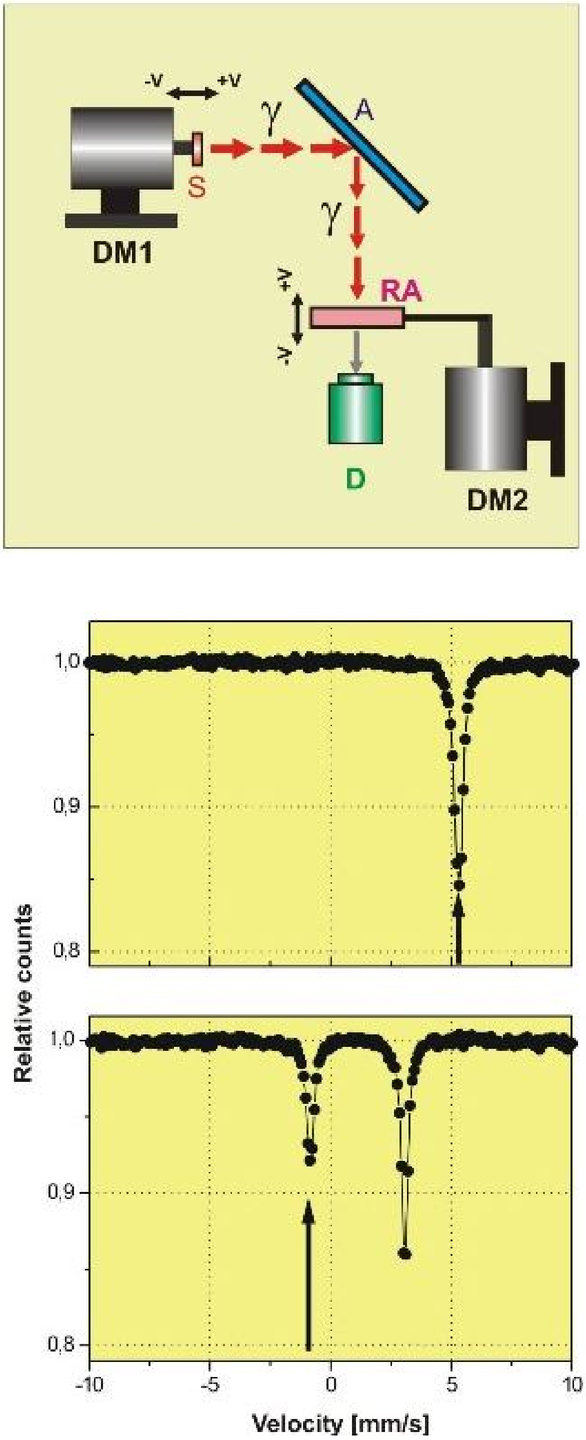


Fig. 9. Conventionally used gamma-optics on SEDM

These problems may be solved through application of Selective Excitation Double Mössbauer effect (SEDM) technique.

The SM 2201DR Mössbauer Spectrometer is intended for investigations of dynamic processes in condensed media containing resonance isotope nuclei (Selective Excitation Double Mössbauer Effect, SEDM) or not (Rayleigh Scattering of Mössbauer Radiation, RSMR). The SM 2201DR Mössbauer Spectrometer can also be used in the studies with conventional Mössbauer Gamma optics schemes: transmission, emission, back and Bragg scattering.

Physically, the selective excitation double Mössbauer effect technique is based on excitation of a certain sublevel of the nucleus hyperfine structure of the sample under investigation and subsequent analysis of the radiation scattered by the specimen by means of a resonance filter-analyzer.

The Mössbauer spectrum measured in this case bears information on solid-state processes that occur during nucleus lifetime: if within this time the nucleus manages to exchange energy with its environment, the energy of the re-emitted gamma-quantum will differ from that of the gamma-quantum exiting the nucleus.

Therefore, the selective excitation spectra can provide direct proof for the existence of dynamic processes and information on their rate and kinetics.

The relaxation time t_R range covered by this method is in the mean lifetime t region of a nucleus in the exited state (e.g., for ^{57}Fe isotope, $t = 10^{-8}$ s). At $t_R << t$ there is no marked redistribution of energy during the nucleus lifetime and addition lines in the scattered radiation spectrum become too weak to be observed.

A conventionally used gamma-optics on SEDM is shown in Fig. 9.

The experiment requires two Doppler modulators. Source S intended for the excitation of a selected sublevel in a specimen A under investigation is rigidly fixed on the first Doppler modulator DM1 moving in a constant velocity mode. Spectral analysis of resonance-scattered radiation is accomplished with the resonance filter-analyzer RA having a single narrow line and driven by the second Doppler modulator DM2 that operates in a constant acceleration mode. A detector D detects the scattered radiation.

Block-diagram of SM 2201DR Mössbauer Spectrometer for this gamma-optics is shown s shown in Fig. 10.

Spectrometer consists of an analytical cabinet and control, energy selection, storage systems interfaced to a personal computer.

The analytical cabinet includes vibrodamping and carrier platforms, two Doppler modulators (DM(CV), DM(CA)), gamma-ray detectors with built-in preamplifiers (D1, D2), guard and shielding collimators, various sample and source holders installed on the latter platform.

The control and energy selection systems together with necessary equipment consist of: two Modulator Drivers (MD1, MD2), two Discriminators (Single Channel Analyzers) with built-in amplifiers (SCA), two Low Voltage Power Stabilizer units (LV), two High Voltage Power Supplies (HV), two Specialized Accumulator units (SA), and other accessories, i.e. interface card, coaxial and power cables, connectors, etc.

In Mössbauer Gamma-Optics, shown in Fig. 10, the radioactive source S is rigidly attached to the moving part of Doppler modulator DM(CV), while the resonance filter-analyzer RA is placed on the other Doppler modulator DM(CA).

Modulator drivers MD(CV) or MD(CA) provide start and memory (channel) advance pulses for the synchronization of the entire system. These pulses are then fed to the Specialized Accumulator units SA.

Modulator Drivers MD(CV) and MD(CA) also provide the control voltage signals for the Doppler Modulators DM(CV) and DM(CA), thus making them vibrate according to the preset law of motion.

In spite of obvious advantages of SEDM experiments over the transmission one it has not so far found wide application. The principal difficulty in this kind of experiments is the low aperture of experiment: the intensity of the gamma-radiation scattered into a given solid angle is very low and becomes even lower because of losses caused by conversion and absorption of radiation and its passing through the resonance filter-analyzer.

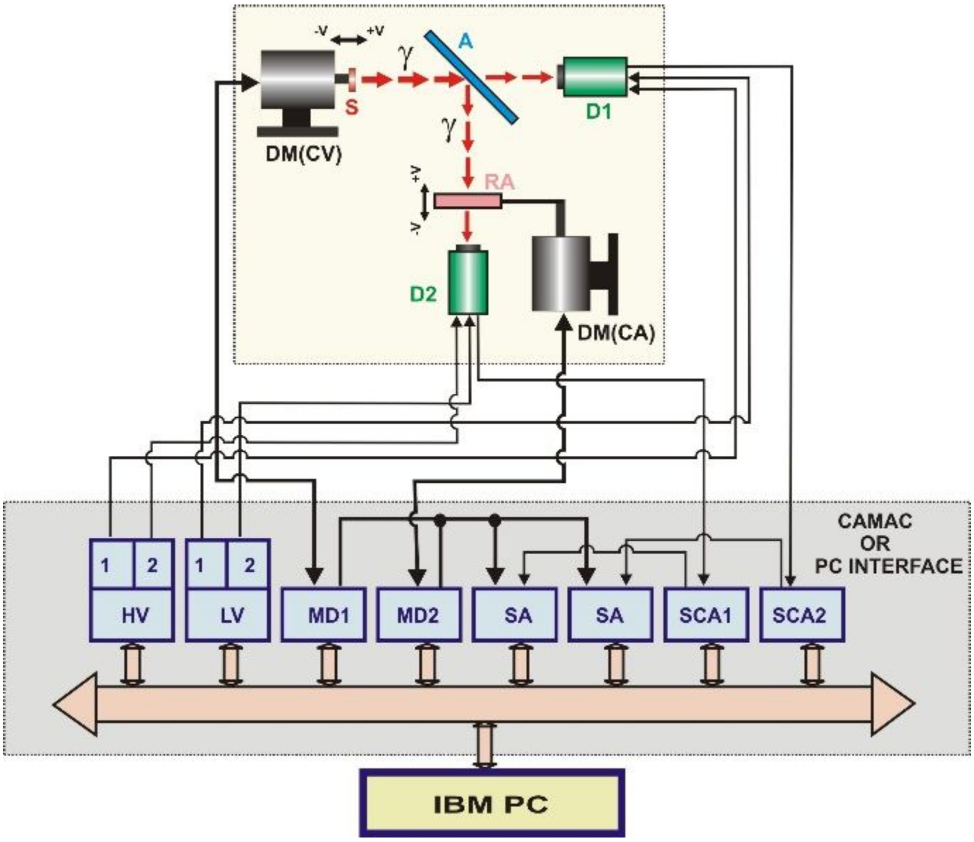


Fig. 10. Block-diagram of SM 2201DR Mössbauer Spectrometer

Furthermore, the signal-to-noise ratio defined by the product of resonance radiation and absorption probabilities by the squared resonance scattering probability, wherein each of the factors is less than unity, is very low and to obtain a high-quality spectrum, extended periods of measurement are required (the average running time should be tens of days). To overcome these difficulties, the following were suggested: using high-intensity sources (with at list 100 mCi activity), maximum enrichment of the sample under investigation and resonance filter-analyzer with a resonance isotope, optimum shielding of the detector from extraneous radiation, minimization of distances between the source, sample and detector, and ensuring long-time stability of the equipment.

The optics of SM 2201DR Mössbauer Spectrometer differs from the existing laboratory setups in that respect that the scattered radiations are detected by resonance detectors RD (Fig. 11), thus eliminating the need of the resonance filter-analyzer in energy analysis and ensuring higher sensitivities and lower running time.

A comparison of SEDM spectra measured using the two gamma-optics scheme (conventional and resonance) shows that the new gamma-optics leads to a 50-fold increase in signal-to-noise ratio and hence dramatically cuts the measurement time (from 28 to 2 hours) necessary to achieve the same statistical accuracy of experiment.

It should be noted that the gamma-optics of SM 2201DR Mössbauer Spectrometer make is also possible to carry out highly-sensitive and rapid investigation of the coherent Rayleigh scattering of Mössbauer radiation (RSMR).

This extends functional capabilities and application fields of the instrument in studying dynamic processes connected with atomic and molecular motion in the 10^7 to 10^9 s^{-1} range, such as phonon spectra, diffusion, low-frequency excitation near phase transition, short range order in liquids, biological objects, amorphous substances, crystals, etc. In this case the sample under investigation may be substances containing resonance isotope nuclei or not.

5. FIELD AND EXAMPLE OF SPECTROMETER APPLICATION

The SM 2201DR Mössbauer Spectrometer will find application in interdisciplinary basic research and in solving applied problems in physics and chemistry of nanotechnology and advanced materials science.

To evaluate the potential of the spectrometer, it was used to find the reasons of spectral line broadening in the $Fe_{(1-x)}Al_xOOH$ aluminosubstituted goethite system of two compositions.

Figs. 12(a) and (b) show Mössbauer spectra for two compositions x = 8 mol% (a) and 2 mol% (b) measured in the transmission geometry (upper spectra) and in geometry of selective excitation of different sublevels of the sample (lower spectra). Arrows indicate excitation energies.

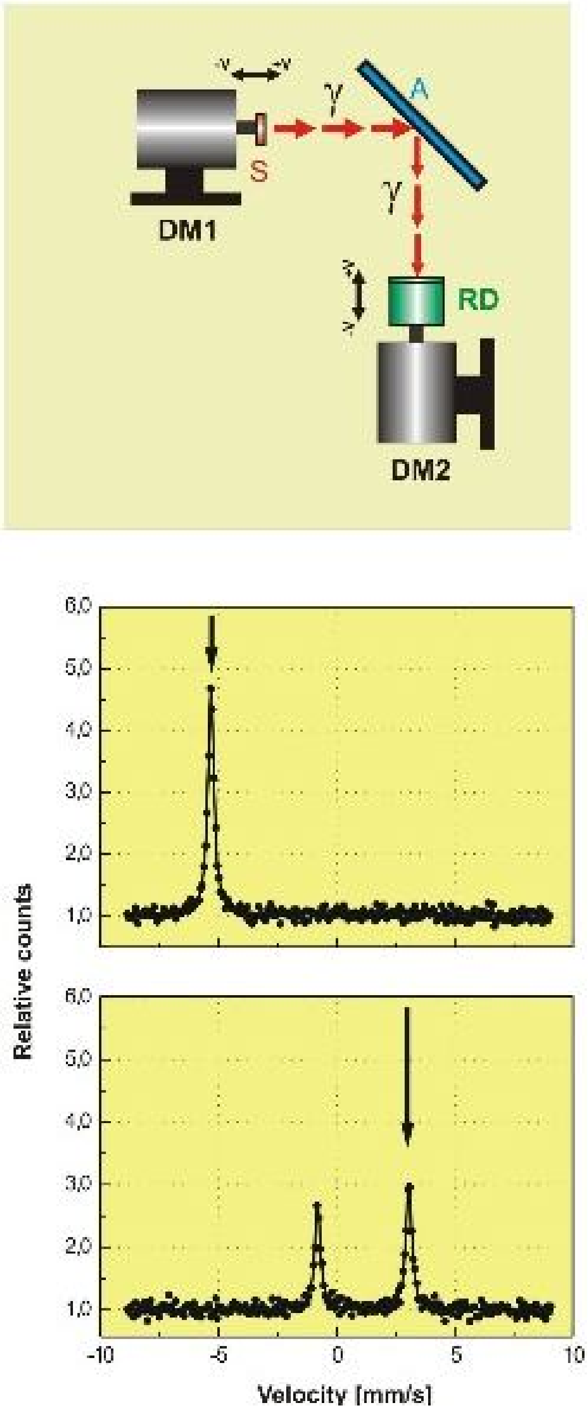


Fig. 11. Scattered radiations detected by resonance detectors RD

The reasons of hyperfine spectral line broadening

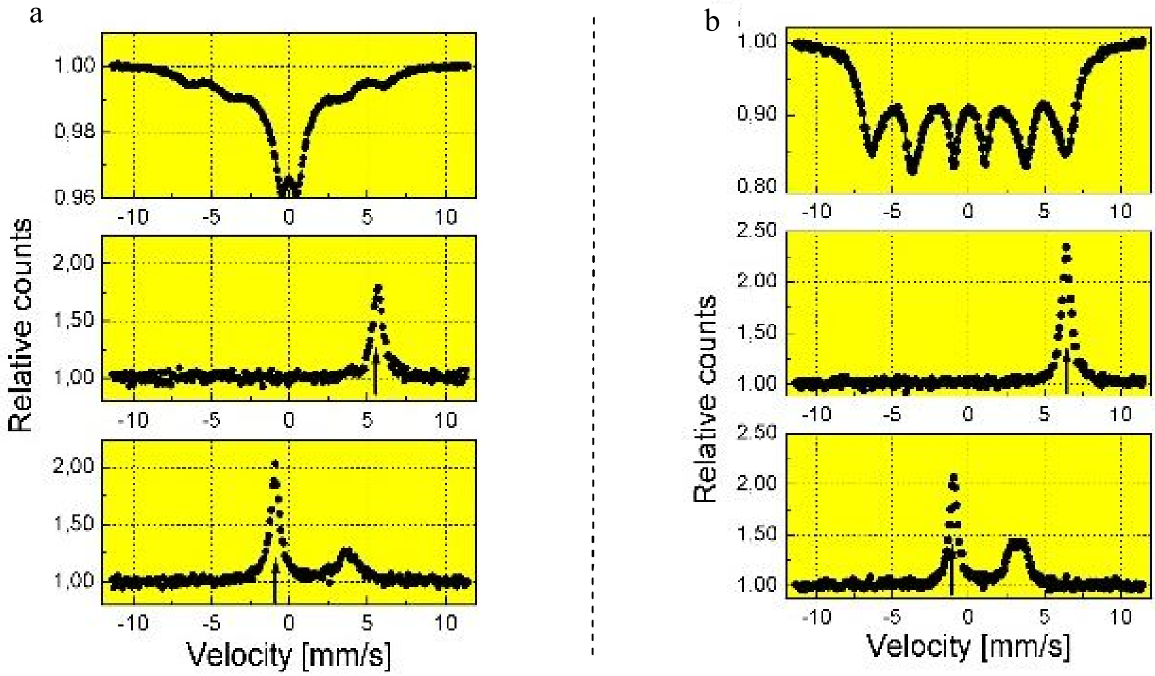


Fig. 12. Mössbauer spectra.
a — x = 8 mol%, b — x = 2 mol%

measured in the transmission mode could be distribution of the hyperfine static interactions caused by difference in the environment of resonance atoms or dynamic processes caused by hyperfine field fluctuation. In some papers, those spectra measured in the transmission geometry were considered as a direct proof of the existence of paramagnetic relaxation in those compounds. However the measurements carried on in the SEDM mode unambiguously revealed the true nature of hyperfine interactions. The lower spectra in Fig. 12, a and b show no satellite line for both samples, which points to the presence of statically set of hyperfine fields in aluminosubstituted goethite independent of time.

6. COMMERCIALIZATION OF THE MASS-PRODUCED IAI RAS OF MÖSSBAUER SPECTROMETERS

Mössbauer spectrometers serially produced by the IAP RAS compete in the domestic and foreign markets for scientific instrumentation with the producers of Mössbauer spectrometers: Wissenschaftliche Elektronik GmbH (WissEl), Starnberg, Germany [17] and Regional Center of Advanced Technologies and Materials, Olomouc, Czech Republic [18].

Russian Mössbauer spectrometers are successfully used in scientific research, academic and educational institutions. Russian Mössbauer spectrometers are used by organizations that work in the field of materials science, developing and researching the properties of new functional and structural materials. These new materials are used in biomedicine, mining and processing industries, in the analysis of mineral raw materials and processed products.

Russian Mössbauer spectrometers are used to study the magnetic and physic-chemical properties of new materials, to study high-temperature superconductivity of materials, ferroelectrics, and multiferroics.

A successful scenario for the formation of Russian scientific instrumentation, in particular, Mössbauer spectrometry instruments, largely depends on the financial support of this direction of scientific instrumentation by the Federal Agency of Scientific Organizations of Russia.

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ИННОВАЦИОННОЕ НАПРАВЛЕНИЕ НАУЧНОГО ПРИБОРОСТРОЕНИЯ — МЁССБАУЭРОВСКАЯ СПЕКТРОСКОПИЯ КАК ФАКТОР СОВЕРШЕНСТВОВАНИЯ ОТРАСЛЕЙ РОССИЙСКОЙ ЭКОНОМИКИ. Ч. 1

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Описаны прорывные научные исследования в области мёссбауэровской спектроскопии. Представлены серийно производимые ИАП РАН мёссбауэровские спектрометры, которые во многом определяют развитие отечественного материаловедения и используются при разработке новых материалов. Показан комплекс научных приборов, иллюстрирующих развитие одного из наиболее перспективных направлений научного приборостроения, необходимый при исследовании магнитных и физико-химических свойств новых материалов, исследовании высокотемпературной сверхпроводимости соединений, сегнетоэлектрики, мультиферроики.

Кл. сл.: инновации, импортозамещение, мёссбауэровский спектрометр, гамма-резонансный спектр, доплеровская модуляция энергии, гамма-оптическая схема

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