

Laboratory work

X-ray emission (nature and experimental spectroscopy)

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Introduction

X-rays are electromagnetic emission generated at the braking of a charged particle in the atoms of a material, or during de-excitation of the atom as a result of the electron transition in the inner shells of the atom. In other words, it is emission that occurs either during the braking of free charged particles (not necessarily electrons) in the material, or during electronic transitions in atoms, and the energy spectra of emission of these processes differ significantly. The first case is characterized by a continuous **bremsstrahlung** spectrum, and the second - by a line spectrum.

(1)

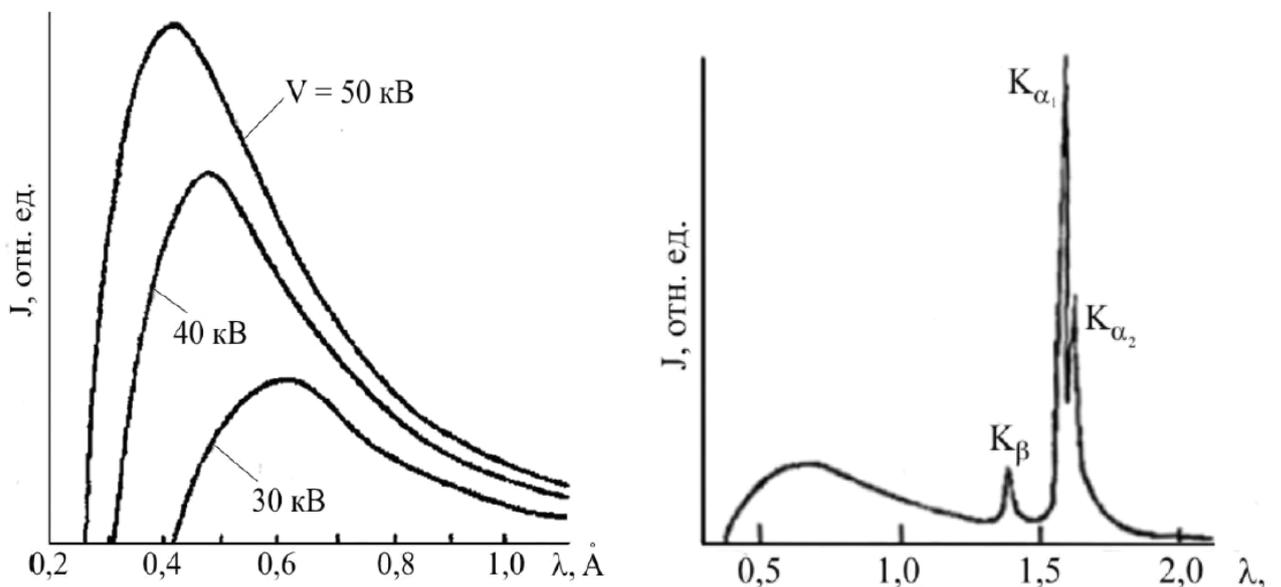


Fig.1. (left) *Bremsstrahlung spectrum in λ -representation*
 (right) *Characteristic X-ray spectrum in λ -representation*

The **bremsstrahlung** spectrum generated during the braking of charged particles is characterized by a continuous distribution by X-ray energy (by wavelength λ see Fig.1; high-energy (short-wave λ) part of the spectrum is cut off abruptly at the energy equal to the maximum kinetic energy of the charged particle. In the case of using an X-ray tube as the radiation source the short-wave limit of the spectrum is inversely proportional to the difference of the tube potential U and is determined by the formula:

$$\lambda_{MIN} = h \cdot c \cdot e^{-1} \cdot U^{-1}$$

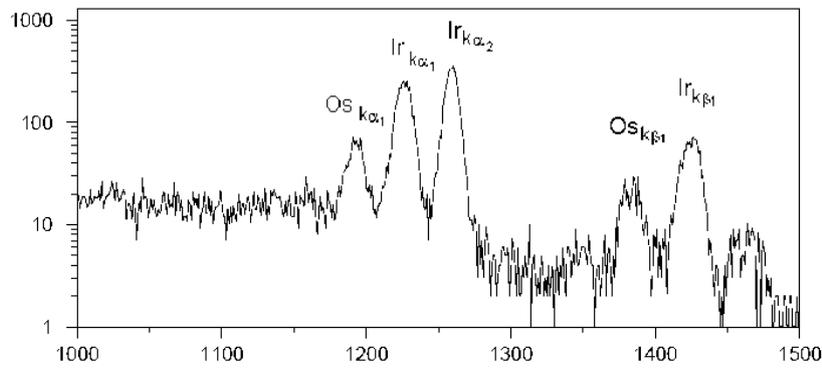


Fig.2. X-ray spectrum of an irradiated sample Os

The shape of the bremsstrahlung spectrum is not dependent on the material in which the charged particle braking occurs. In contrast to braking processes, atom de-excitation processes are accompanied by a line spectrum. Atoms of each chemical element have their own characteristic line spectrum, as the energy of occurring X-rays depends only on the energy of the electron levels of the atom involved in the process. Since they are specific to each element, X-ray line spectra are called **characteristic**, see Fig.1 (right).

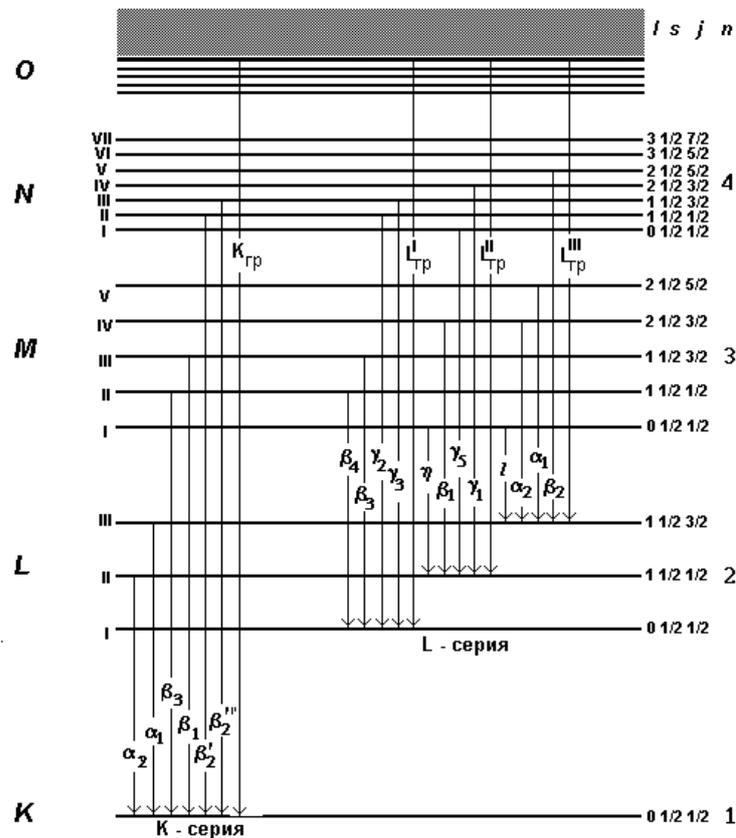


Fig.3 Schematic diagram of the X-ray lines of K and L series

A typical energy spectrum of characteristic X-ray emission detected by a HPGe semiconductor detector is presented in Fig.2.

It should be noted that characteristic X-ray spectra for different atomic elements are qualitatively similar - they are all made of identical groups of spectral lines. This spectrum similarity is due to the fact that the source of characteristic radiation is the processes of

transitions between the inner electron shells of atoms, which are filled identically, in contrast to the outer electron shells.

Groups of spectral lines formed in the transition of electrons from the upper levels to one of the lower levels are identified by the name of series in which this lower level is located. Lines of K-series correspond to transitions to the level with the main quantum number $n = 1$. Lines of K, L-series correspond to transitions to the levels with the main quantum number $n = 1, 2$, respectively. Lines of K-series have the highest energy, see Fig. 3.

X-ray emission corresponds to a range of photon energies E_x 100 eV - 1 MeV.

X-rays have a penetrating ability that depends on the energy E_x , which has resulted in the widespread use of X-rays in various techniques, such as methods of quantitative elemental analysis, flaw detectors, scanners, etc.

1. X-rays

For the first time, X-rays were observed by Roentgen, an achievement that earned him the first Nobel Prize in Physics in 1901. The method was based on the electron bombardment of anode. The electron flux emitted from the hot cathode is accelerated by an electric field in a vacuum and is directed at a target consisting of atoms with a great charge number. The potential difference of acceleration field between the cathode and anode of the tube is several tens of kilovolts. As a result of the bombardment of anode atoms by the electron flux bremsstrahlung and characteristic radiation occur. Provided the condition $E_U \geq E^*$ is fulfilled, where E^* - binding energy of the electron of the corresponding shell, lines of the anode element will be generated.

A necessary condition for the regeneration of the characteristic X-ray emission is the presence of vacancies in the inner electron shells of atoms, therefore the cause of the emission of spectral lines can be not only the electron bombardment of the anode in the X-ray tube, but also other methods of ionization of the inner electron shells of atoms, such as ion excitation, X-ray fluorescence, etc.

Excitation of characteristic X-rays can be carried out using α -particles of a radioactive isotope.

Emission of characteristic X-rays from the decay of radioactive isotopes. The atoms of radioactive isotopes emit X-ray spectra as a result of electron-nuclear interactions, such as:

1. *Internal conversion* - the effect of a direct transfer of energy from the excited nucleus to an atomic electron leading to the ionization of the atom;
2. *Internal photoelectric effect* - when γ -photon emitted from the nucleus knocks the electron out of its atom,
3. *K-capture* - capture of one of the inner electrons of an atom by the nucleus.

The predominant X-ray emission mechanisms are *internal conversion* and *K-capture*.

This paper presents three radioactive sources that emit X-ray spectra of the elements resulting from radioactive transformations. Radioactive isotopes emit the following spectra:

1. Ra^{226} - spectrum of bismuth.
2. Co^{57} - spectrum of iron.
3. Am^{241} - spectrum of neptunium.

2. General properties of semiconductors

Semiconductor germanium- and silicon-based detectors are commonly used in heavy-ion physics for detection of both charged and gamma particles and X-rays.

Such widespread application of semiconductor detectors in modern experimental nuclear physics is primarily due to the possibility to manufacture Ge(Li), Si(Li) detectors with a large-volume sensitive area maintaining high energy resolution parameters. Note that the complexity of manufacturing germanium and silicon detectors causes the limited number of manufacturers.

The most popular manufacturers of germanium and silicon detectors are the firms: Canberra (USA), ORTEC (USA), Baltic Scientific Instruments - BSI (Latvia), and the IPTP (Institute of Physical and Technical Problems, Dubna, Russia). With the development of techniques of manufacturing ultra-pure semiconductor material germanium crystals with a very low concentration of impurities were obtained (less than 10^{10} atom/cm³). These techniques have made it possible not to dope Ge-crystals with compensating Li-impurities and thus, to create HPGe (High Pure Ge) detectors.

These detectors can be stored at room temperature and cooled shortly prior to the measurement performance.

Detectors based on cadmium telluride (CdTe and CdZnTe) are used for gamma- and X-ray spectrometry. Gamma-detectors based on cadmium telluride do not require cooling to cryogenic temperatures when making measurements. X-ray detectors based on cadmium telluride are cooled by small-size thermoelectric coolers based on the Peltier effect in order to reduce their own thermal noise.

Due to a small volume of the sensitive area of CdTe-detectors they are applied for detection of both X-rays and soft γ -rays with energies up to 1 MeV. Energy resolution for line $E_{\gamma} = 122$ keV is $\Delta E(\text{FWHM}) \sim [5-12]$ keV, and for line $E_{\gamma} = 661$ keV $\Delta E(\text{FWHM}) \sim [15-60]$ keV. CdTe-detectors are significantly inferior to germanium detectors both in the efficiency and in the energy resolution.

The main manufacturers of CdTe-detectors are the firms: eV-Products (USA), and BSI.

3. Operating principle of the semiconductor detector

At first sight, the operating principle of the semiconductor detector is the same as that of the ionization chamber: under the influence of ionizing radiation in the material of the semiconductor detector free charge carriers - electrons and holes – are produced. These charges are collected on the detector electrodes by the electric field. Electrical impulse, which is formed at the detector electrodes in the process of charge collection, is converted (amplified and shaped by special electronic devices) into an electronic signal. This impulse carries quantitative information on the number of produced electrons and holes, i.e. information about ionizing radiation. A more detailed description of the detector operation is presented below.

4. Requirements to materials for manufacturing the semiconductor detector

The main materials used for manufacturing various types of semiconductor detectors are germanium and silicon. Table 1 shows the main physical characteristics that are essential in the manufacture of semiconductor detectors. Let us consider some of the characteristics presented in the Table. One of the main requirements for the semiconductors is the minimum value of the average energy ε spent on the formation of an electron-hole pair. It is obvious that the greater the number of charge carriers generated per unit of energy of the detected particle is, the higher the value of the pulse of the desired signal from the detector, with a smaller relative fluctuation (better energy resolution), is obtained.

As can be seen from Table 1, the values of the average energy spent on the formation of electron-hole pairs for silicon and germanium is $\varepsilon = 3.66$ eV and $\varepsilon = 2.96$ eV, respectively. Thus, when detecting an X-ray photon with energy $E_X = 50$ keV in Si and Ge, on average, 13600 and 17000 electron-hole pairs are obtained, respectively; the relative error of energy measurement by the Ge detector, in equal conditions, will be less.

Important parameters of semiconductor material are density ρ , atomic number Z , and μ -mobility of the charge carriers. ρ and Z parameters mostly determine the cross-section values for the interaction of γ -rays with atoms of the detector material, and thus, the detection efficiency.

Mobility of charge carriers is characterized by the completeness of collection of the generated charge carriers on the electrodes of the semiconductor detector.

The average values of the number of electron-hole pairs calculated above represent the ideal case of a complete collection of the charge carriers in the detector material, which necessarily requires a high mobility and long lifetime of the charge carriers. Lifetime is determined by recombination of the charge and depends on the presence of impurity atoms and other charge traps. Charge carrier mobility μ characterizing dependence of drift velocity of the charge carriers v on the electric field intensity E , $v = \mu \cdot E$, largely depends on the temperature of the semiconductor, see Table 1, which defines the requirements to the lifetime of the charge carriers, and thus, to material purity. For example, since the mobility in Si at the boiling point of nitrogen ($T = 77^\circ$ K) is $\mu = 1.8 \times 10^4$ for electrons and 4.2×10^4 [cm²/V·s] for holes, the drift velocities of the charge at an intensity of $E = 100$ [V·cm⁻¹] will be $v = 0.25$ ms and $v = 0.55$ ms for electrons and holes, respectively. Since the charge collection time dT on several-cm-thick detectors is in the order of microseconds, then to ensure the complete charge collection the charge carrier lifetime should be several milliseconds. This is what determines the permissible amount of impurities in the detector material.

Table 1

Physical characteristics	Si	Ge
Atomic number of the element	14	32
Atomic mass	28.6	72.60
The number of atoms per cm ³	4.96×10^{22}	4.41×10^{22}
Density [gr/cm ³] (at $T=300^\circ$ K)	2.33	5.32
Relative dielectric constant ε	12	16
Bandgap E_g [eV] (at $T=300^\circ$ K)	1.115	0.665
Bandgap E_g [eV] (at $T=0^\circ$ K)	1.165	0.746
Average energy of pair production ε [eV]	3.76	2.96
Mobility of electrons at $T=77^\circ$ K [cm ² /V·s]	4×10^4	3.6×10^4
Mobility of electrons at $T=300^\circ$ K [cm ² /V·s]	1350	3900
Mobility of holes at $T=77^\circ$ K [cm ² /V·s]	1.8×10^4	4.2×10^4
Mobility of holes at $T=300^\circ$ K [cm ² /V·s]	1900	480
Intrinsic resistivity ρ [Ohm·cm] ($T=300^\circ$ K)	2.3×10^5	47
Intrinsic resistivity ρ [Ohm·cm] ($T=77^\circ$ K)	∞	∞
Fano factor ($T=77^\circ$ K)	$0.084 \div 0.16$	$0.085 \div 0.143$
Intrinsic carrier concentration $n_i=p_i$ [1/cm ³] ($T=300^\circ$ K)	1.5×10^{10}	2.4×10^{13}
Intrinsic carrier concentration $n_i=p_i$ [1/cm ³] ($T=273^\circ$ K)	1.3×10^9	4.7×10^{12}
Intrinsic carrier concentration $n_i=p_i$ [1/cm ³] ($T=77^\circ$ K)	3.3×10^{-7}	2.4×10^{-20}

5. Moseley's law

Moseley's law is an empirical law that defines a linear relationship between the square root of the emission (energy) frequency of characteristic X-ray spectral lines of the chemical element atom and its atomic number Z in the periodic table. It has been discovered by a British physicist Henry Moseley in 1913. The law states that the square root of the inverse wavelength of the atom emission is linearly related to its atomic number.

$$\sqrt{\nu} = M(Z - a)$$

where M is a constant, a is a screening constant.

Thus, for all series of the X-ray spectrum Moseley's law establishes a linear relationship between the atomic number and nuclear charge. Fig.4 shows a Moseley's diagram for the main lines of K and L series.

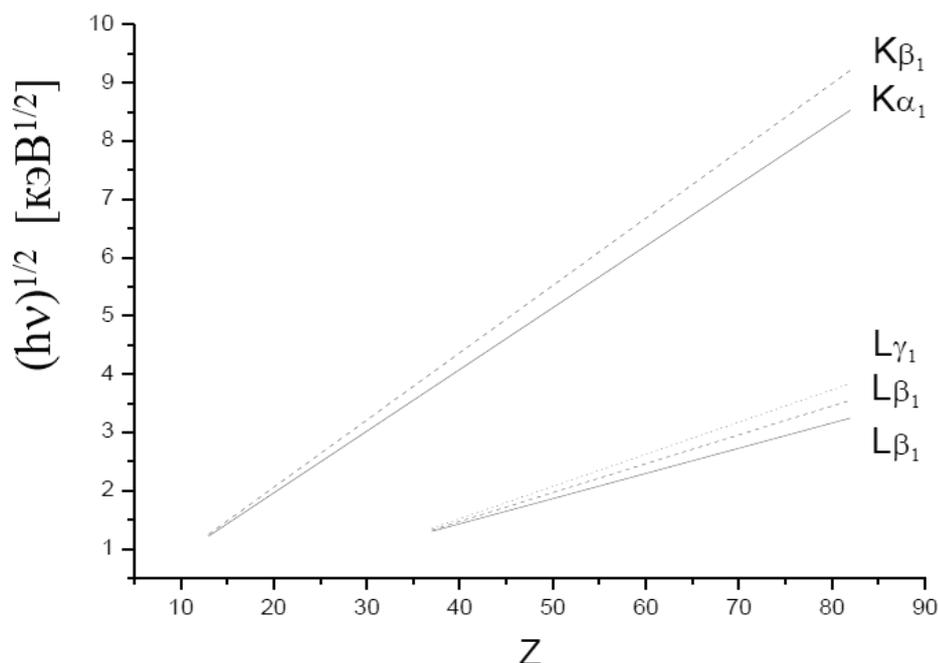


Fig.4 Moseley's diagram for the main lines of K and L series

This law is the first to show that it is not the atomic mass, but the atomic number equal to the nucleus charge, that determines the chemical properties of the atom. Simplicity and monotonous character (absence of periodicity) of X-ray spectra change with increasing atomic numbers confirm the fact that X-ray spectra arise from electronic transitions in the inner shells of the atom.

6. Application of Moseley's law

Measurement of the atomic number of an unknown element.

The measurement of X-ray spectrum of an unknown element and the application of Moseley's law allows one to determine atomic number Z for the unknown element. This method was of particular relevance during the drafting of the periodic table of elements, when the order of the elements was not clear. Before the discovery of Moseley one could only measure the atomic mass. The presence of isotopes led to the fact that the atomic mass of an element with a

large number Z was less. Fractional values of the atomic mass led to discussions about the place of an element in the periodic table.

Moseley's experiments have established, for example, that number Z for cobalt is greater than number Z for iron (26) and is less than number Z for nickel (28). Nevertheless, the atomic mass of cobalt is 58.9, and the atomic mass of nickel is 58.7. Also Moseley's experiments allowed one to identify the gaps in the periodic table - spaces for the items not yet discovered, which could not be achieved by the method of determining the atomic mass. Thus, a vacant space was established, with $Z = 43$, which was later occupied by a newly discovered Technetium.

Diagnostics of alloys and compounds of elements.

In chemical compounds the levels of the outer shells change, therefore it is impossible to tell anything about a complex compound composition by the spectrum in the optical range. However, chemical bonds virtually do not affect the inner shells involved in the production of the characteristic radiation. Therefore, the X-ray spectrum of the complex compound element will be the same as the spectrum of the individual atoms of this element. Thus, the measurement of the characteristic radiation and its comparison with Moseley's law is an important method for the diagnostics of complex compounds.

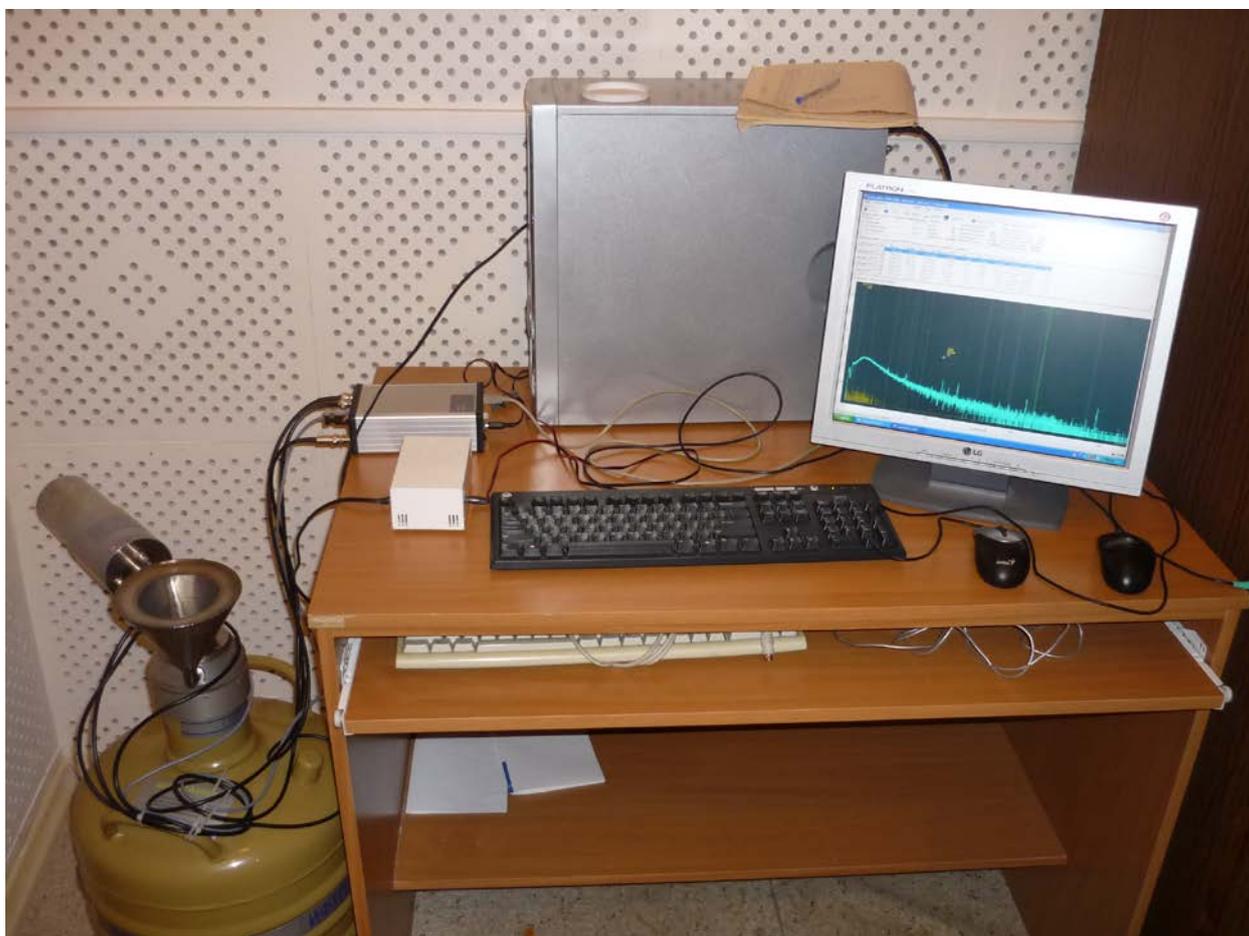


Fig.1. *General view of the semiconductor X-ray spectrometer.*

7. Test questions and tasks

- 1) Write Moseley's law for $K\beta$ -lines ($L\alpha$, $L\beta$).
- 2) What does the screening constant show?
- 3) Explain why the presence of K-series of characteristic X-ray emission is necessarily accompanied by the occurrence of other series (L, M) in the heavy elements.
- 4) Starting from what element can a) L-series, b) M-series appear?
- 5) Does Moseley's law manifest the features associated with the periodicity of properties of the elements?
- 6) Why are the K-lines more intensive than, for example, the M-lines?
- 7) Using Moseley's law, calculate the wavelength and photon energies corresponding to the lines of aluminum and cobalt.
- 8) Perform experimental research of Moseley's law.
 - 8.1. Read the description of the experiment and the theoretical explanation of Moseley's law.
 - 8.2. Read the instruction on operating the X-ray spectrometer.
 - 8.3. Switch on the spectrometer and conduct energy calibrations by the known sources.
 - 8.4. Measure the spectrum of an unknown source and define it using the Table.
 - 8.5. For each series ($K\alpha$, $K\beta$), knowing the energies $h\nu_i$ and Z_i , plot the graph of dependence of the energy square root as a function of the atomic number of the radiation source, and check the linearity of the dependence obtained (i.e. Moseley's law in action).
 - 8.6. Compare the Table and experimental values of the energies of the lines. Calculate the error.