NUCLEAR METHODS IN MATERIAL RESEARCH

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Contents

Summary

In this chapter, some methods of material research are collected in which the analytical information is gathered on the basis of the interaction of particles and the material.

1. Introduction

Nuclear radiation interacts with materials. This is a well known fact since the discovery of nuclear radiation. During the years, it was discovered that such interactions depend not only on the radiation but on the material, as well. As more and more data were collected, it was proved in numerous experiments that these interactions can be used for analytical purposes.

The methods described below are similar in the sense that they all use particles to scan the features of materials. The particles may be charged or neutral; electrons, alpha particles or photons; produced by radioactive sources or accelerators; but they are all particles in the general sense.

As a matter of fact, nuclear radiations and accelerated particles enhanced the chances of analysis, which became no longer limited to chemical methods or light spectroscopies. A great number of new, non-destructive analytical methods were developed based on nuclear radiations. High energy photons, electrons, protons, and other particles can reach deep layers of a material without destructing its structure.

2. Methods Based on the Absorption of Radiation

The absorption of radiation can be used in material research and industry because of its dependence on certain material properties. Since the energy of the radiation used in nuclear methods is usually high, chemical information other than the atomic number or its average cannot be expected. An exceptional case is Mössbauer spectroscopy which is extremely sensitive to the chemical environment and is based on nuclear resonance absorption of gamma rays. The method is discussed in a separate section below.

In most cases, the absorption of radiations is considered electronic because due to the large mass of nuclei, collision with them results in relatively small energy transfer, mainly the direction of the motion of the particle changes and therefore it should rather be considered as scattering.

Alpha particles, which lose energy very fast in interaction with matter, end up as He atoms when the absorption process is accomplished.

Beta minus particles (i.e. electrons) are eventually captured by atoms. Beta plus particles (i.e. positrons) will end up in annihilation radiation when absorbed. This radiation bears very special structural information on the host material. The related positron annihilation spectroscopy is discussed in a separate section.

Gamma radiation, when absorbed, creates electrons and positrons (Compton electrons, photoelectrons, electron-positron pairs) and the process continues the same way as for beta radiations. Finally the gamma energy is dissipated as thermal energy or may be consumed by chemical transformations. As mentioned above, nuclear resonance absorption used in Mössbauer spectroscopy is a special type of gamma–matter interaction.

Although neutron radiation is not observed with radioactive sources (let us neglect now spontaneous fission), nuclear reactors and other neutron sources make possible the application of neutron radiation in material research. Neutrons when absorbed always end up in nuclei, thus the absorption is only nuclear. We do not consider high energy nuclear reaction with neutrons as absorption events [e.g., (n,p), (n,d), (n,α) etc.], because thermalization of neutrons is fast, and the cross section of neutron capture for thermal neutrons is usually so much higher than any other reaction that it dominates the absorption process.

Absorption of radiation triggers secondary radiation in many occasions. Most important examples are bremsstrahlung, Cherenkov radiation, characteristic X-rays, Auger electrons, and gamma radiation of activated nuclei. There are several nuclear methods based on the measurement of these radiations, but we restrict our discussion here to the absorption process and the consequences are not considered.

2.1. Measurement of Absorption

Absorption measurements have a very simple experimental arrangement involving a radiation source, an absorber and a detector (Figure 1).

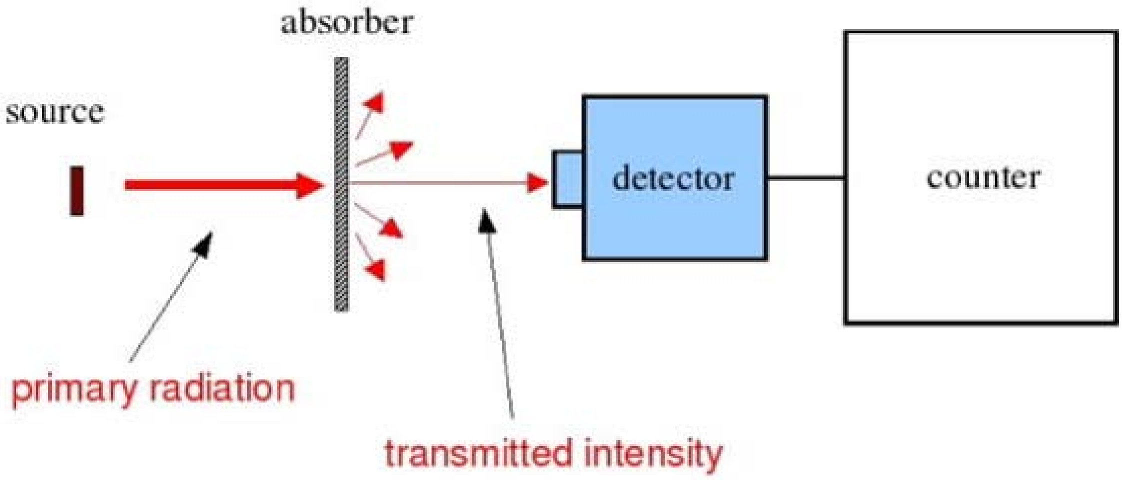

Figure 1: Usual arrangement of an absorption/attenuation measurement.

As visualized in Figure 1, it should not be forgotten that the drop in the observed count rate is rarely due to absorption only, because scattering is also responsible for the observed "effective absorption" called attenuation. With special geometries (like either a high-surface flat source/detector combination or, just the opposite, a well-collimated source/detector assembly), using a thin absorber can reduce the contribution of scattering.

The extent of absorption/attenuation depends on two major parameters for a certain type of radiation and one particular material: sample thickness and energy of the radiation.

Dependence on the sample thickness can be approximated with a Beer–Lambert type of formalism for gamma, beta and neutron radiation.

$$I = I_0\,\mathrm{e}^{-\mu d} \qquad (1)$$

where I is the transmitted intensity, I_0 is the initial intensity, d is the (linear) sample thickness and μ is the (linear) attenuation coefficient. In this formula, μ is not a very good characteristic of the given material (especially if both gaseous and condensed phases are considered), because absorption/attenuation trivially depends on the mass density. Recall that the interaction of radiation with matter is mostly considered as interaction with electrons, and since mass density and electron density are more or less proportional to each other, a new quantity μ/ρ is introduced, where ρ is the mass density. This will be a much better measure of material properties (like molar attenuation coefficients in spectrophotometry). This new quantity is called mass absorption/attenuation coefficient (μ_m) and defined as follows:

$$I = I_0\,\mathrm{e}^{-(\mu/\rho)(d\rho)} = I_0\,\mathrm{e}^{-\mu_\mathrm{m} d_\mathrm{s}}, \qquad (2)$$

where d_s is the surface density (also called mass thickness in the literature) frequently given in g/cm^2 units. Accordingly, the unit of μ_m is cm^2/g,.

This phenomenological treatment is readily applicable to gamma radiation because the penetration depth is high, so close to total absorption is rarely reached. It is also applicable to neutrons for the same reason, however, because the process responsible for neutron absorption is a nuclear reaction, the neutron capture cross section (measured in barns; 10^{-24} cm^2) is used instead of μ_{m}. For a multicomponent material, cross sections for the constituent elements can be summed up. For beta radiation, the exponential dependence is a good approximation for not very high level of absorption, well below the penetration range (typically a few mm in condensed phase). For alpha radiation, absorption is so fast that total absorption is usually reached and penetration depth (typically a few tens of micrometers in condensed phase) is used to characterize absorption.

Dependence on the energy is very different for the different kinds of radiations. However, the general rule is that the lower the energy the faster is the absorption. For alpha and beta rays it can be rationalized by the simplified Bethe-Bloch equation (valid for charged particles of low to intermediate energy passing through an absorber made from a single chemical element):

$$-\frac{\mathrm{d}E}{\mathrm{d}x} = K z^2 \frac{Z}{M} \frac{1}{\beta^2} f\left(\beta^2\right), \qquad (3)$$

where $-\mathrm{d}E/\mathrm{d}x$ is the linear energy transfer (LET), K is a constant, z is the charge number of the particle (2 for alpha and ±1 for beta radiation), Z is the atomic number of the absorber material, M is the mass of the absorber atom, $\beta = v/c$ (where v is the speed of the bombarding particle, c is the speed of light). The function $f\left(\beta^2\right)$ only depends weakly on β^2. The basic message of the formula is that the kinetic-energy related part, β^2 ($E_{\mathrm{kin}} \propto v^2 \propto \beta^2$), is in the denominator of the coefficient before the bracket, thereby explaining the increase of transferred energy (absorption) with decreasing energy of the particle

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For gamma radiation the photoelectric effect will dominate at low energies because the chance for resonance with bound electrons is getting higher. Since both the probability of the interaction and the transferred energy in one event increases, the energy of the radiation gets absorbed over a shorter distance.

For neutrons, the huge increase of the capture cross sections with thermalization explains the increasing absorption at low energy. This is very nuclide-dependent; cross sections for different nuclei can differ by several orders of magnitude due to resonance effects.

2.2. Applications

Since the absorption of various radiations is not very specific to elements, molecular structures, lattice structures, etc. applications are mostly focused on the measurement of macroscopic physical properties (thickness, density) in applied science and industry.

Some special applications are available with neutrons.

2.2.1. Level Monitoring

Especially in industrial and agricultural facilities, where the level of a “fluid” material (e.g., molten metal, concrete mixture, grains) in a huge vessel or container cannot be measured with simple methods, radiation absorption measurements can help. This method has numerous beneficial features:

- discrete levels can be measured or the level can be continuously monitored using the same measurement principle;
- applicable for open and closed systems;
- applicable for solid and liquid systems;
- it can be used for aggressive materials;
- insensitive to changes in temperature, pressure, humidity;
- the detector output signal can be easily used for automatization.

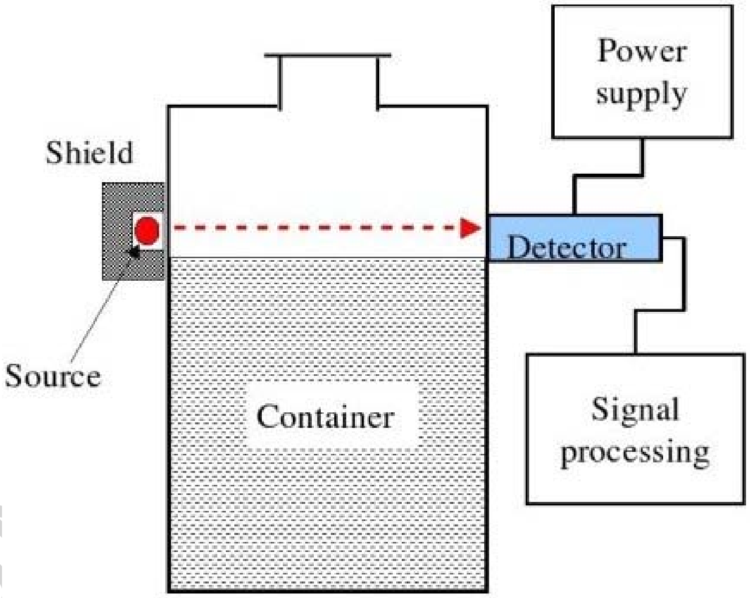

Figure 2: Level monitoring in industrial containers using outer source.

As shown in Figure 2, the source and the detector both can be placed outside the container and when the level of the material reaches the height of the source and detector, an abrupt decrease is expected in the count rate. For higher radiation safety, the source may be placed inside the container if it is possible. The most commonly used detectors are GM tubes.

Since the radiation should pass through the wall of the container, in heavy industry, high energy gamma sources are required. ^{60}Co and ^{137}Cs are commonly used with activities 50-5000 MBq. For light materials and small containers (e.g., tooth paste) beta sources like ^{90}Sr of 100-200 MBq can be applied. A special case is when the vessel is filled with a material of high hydrogen content, for example crude oil and oil derivatives. In this case neutron sources can be advantageously applied. The neutron source can be a mixture of the alpha emitter $^{241}\mathrm{Am}$ and natural Be. The nuclear reaction producing 4 MeV neutrons is $^{9}\mathrm{Be}(\alpha,\mathrm{n})^{12}\mathrm{C}$.

2.2.2. Thickness Measurements

If in a production line the chemical composition and the density of a material are constant, the absorption of radiation will only depend on the thickness of that material, which can be used for thickness measurement. This application is typical in rolling metals, plastic foils and sheets, glass, etc. The measuring arrangement is shown in Figure 3.

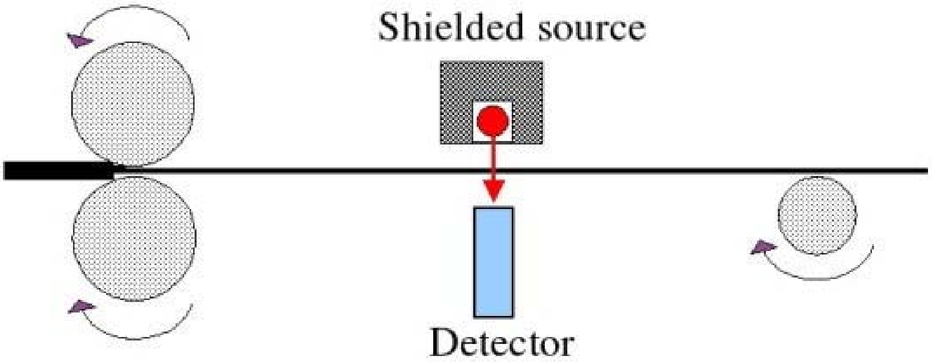

Figure 3: Thickness measurement in a rolling line.

The thickness and the elemental composition determine what kind of a source can be used. For very thin materials (5-50 g/m^2), alpha sources like $^{210}\mathrm{Pb}$ and $^{239}\mathrm{Pu}$ can be applied. Beta emitters can be applied in the surface thickness range 50 to 10 000 g/m^2. The potential radionuclides are $^{85}\mathrm{Kr}$, $^{90}\mathrm{Sr}$, $^{147}\mathrm{Pm}$, and $^{204}\mathrm{Tl}$. The maximum thickness for steel that can be reliably measured with this method is 1.2 mm. With intermediate and high-energy gamma sources ($^{60}\mathrm{Co}$, $^{137}\mathrm{Cs}$, $^{241}\mathrm{Am}$) thicknesses between 10 000 and 100 000 g/m^2 can be measured. $^{241}\mathrm{Am}$ is preferentially used in sheet glass production where the absorption of the beta radiation of $^{90}\mathrm{Sr}$ is too high, and that of the gamma radiation of $^{137}\mathrm{Cs}$ is too low. The half thickness of about 1 cm for the gamma radiation of $^{241}\mathrm{Am}$ in silicon glass proved to be ideal for this purpose.

Thickness measurements are performed sometimes in reflection arrangement, i.e., based on scattering of the radiation.

2.2.3. Measurement of Density

If in an absorption measurement the geometry of the object (i.e., thickness) is constant, the absorption of radiation will be characteristic of the density of the material.

This simple principle can be used to measure the pressure of gases with alpha sources in pressure ranges (usually low pressures) where other methods fail or cannot be applied conveniently.

A typical application of gamma sources (^{60}Co, ^{137}Cs, ^{241}Am) is the monitoring of the flow of crude oil or other oil products in a pipeline. If the source and the detector are set vertically, the possible air bubbles or other voids can be sensitively detected. With horizontally placed source and detector pairs in the lower half of the pipe or in buffer vessels, high-precision density measurements can be carried out using scintillation detectors. Scintillation detectors are more sophisticated and more expensive than GM tubes, but they have much higher detection efficiency for gamma rays, which makes them a better choice for this kind of application. With high-precision density measurement the quality of the oil product can be monitored and one can distinguish between shipments from different oil fields, oil wells or from storage facilities of different suppliers. The difference in absorption is mostly due to the sulfur content, since photoelectric effect occurs with much higher probability on the relatively heavy sulfur atoms than on the lighter constituents of oil (C and H).

Density measurements with beta sources can be applied for light materials with a few centimeters thickness as maximum. Typical application can be found in the tobacco industry where the compactness of tobacco in cigarettes is monitored.

2.2.4. Determination of Chemical Composition

Detailed chemical composition may not be determined by a simple absorption method, however in certain cases, e.g., when the system has only two components (or it is a multicomponent system but only the concentration of one particular component is varied), the chemical concentration may be derived from density. In such cases, under well defined measuring conditions and with proper calibration, the observed count rate can be directly related to the concentration.

Apha radiation can be used for gas analysis. Two- or three-component mixtures can be analyzed. The higher the difference between the atomic masses of the constituents, the more sensitive is the concentration determination. The method is much simpler than using mass spectrometry (MS), the most popular technique for gas analysis today, however, the resolution and sensitivity is nowhere near to that of MS.

A more special application of density measurement with alpha rays is the analysis of gas-solid (aerosol) systems. The most widespread use of it can be found in smoke detection. It is based on the fact that the density of condensed phases is about three orders of magnitude higher than that of gases. Thus even a small volume fraction of aerosol components in air causes an abrupt change in the alpha absorption. The sensitivity of smoke detection can be increased by using pairs of detectors where the reference detector is kept closed (free of smoke). ^{239}Pu sources are commonly used in smoke detectors.

Beta absorption can be used for the determination of hydrogen content. This is based on the fact that hydrogen has about twice as many electrons per mass (or mole) than any other element, because in other elements minimum one neutron is found for each proton in the nucleus, while the nucleus of hydrogen is a single proton. Since beta absorption is due to collisions with atomic electrons, hydrogen has an anomalously high mass attenuation coefficient. This is the basis of the measurement of the H to C ratio in the oil industry using ^{90}Sr sources of 80-120 MBq. The precision of the measurement is about ±0.03% H by mass.

With gamma radiation the sulfur content of oil can be sensitively detected and measured (as already mentioned). The lower the photon energy, the higher is the difference in the mass attenuation coefficient of the radiation for S, C and H. This is the reason why instead of gamma radiation the even softer characteristic X-rays of ^{55}Fe are used. For this 5.9 keV K-radiation, the mass attenuation coefficient for sulfur is 20 times higher than that for carbon, and 450 times higher than that for hydrogen! Soft bremsstrahlung triggered by ^{3}H (i.e., tritium, T) beta particles can also be used. Any other heavy constituents in oil or gasoline can be measured this way (e.g., metal impurities due to wear etc. or the formerly used lead additive). The chlorine content of organic materials has also been measured by soft gamma sources.

Neutron absorption may be used to measure the concentration of an element in a material if that particular element is the only one with anomalously high neutron capture cross section relative to all other constituents. Such elements are B, Cd, and Li. Gd and Sm have also very high neutron capture cross section but their technical importance is little. Ores of these elements can be analyzed without the necessity of any chemical treatment (cross sections can be 1000 and 1 000 000 times higher than for silicon and oxygen, respectively, typical constituents of the host rock).

2.2.5. Radiography

If the absorption of radiation is to be measured over a large area or distance, for example, along a pipeline to find possible defects, it can be done with a mobile source and a mobile detector. Alternatively, keeping the mobile source, the outer surface of the tubes can be covered with films sensitive to the radiation. This method is classified as radiography.

Radiography is frequently used in industry to check the integrity of tube walls at welds, but other imaging tasks can also be solved.

The radiation applied can vary from soft characteristic X-rays to beta to gamma to neutron radiation, depending on the material to be “photographed”. For electromagnetic and beta rays a little bit modified (Photosensitized AgBr based film can be used. For neutrons, the same type of film should be covered with foils containing elements which are easily activated (Ag, Au, Cd, Ga, In, etc.). The activation products of these elements will expose the film that can be developed and the possible defects and the internal structure of the object revealed.

3. Scattering Methods

In this section, we have collected several methods based on scattering of particles. All of them apply the same principle: the scattering of particles by a material is governed by its elemental constitution. Besides the application of nuclear radiation, these methods are nuclear in the sense that – in most cases – the scattering is ruled by the nuclei of the material.

In this chapter, we ignore diffraction methods in which the crystal structure plays the significant role. These methods are discussed elsewhere.

3.1. Beta Backscattering

The phenomenon that materials back-scatter (“reflect”) some of the incident beta particles has been well known for almost a hundred years. E. Rutherford, in his famous article introducing the concept of the nucleus, used not only alpha but beta backscattering data, as well. Also he was the first to state the most surprising fact about the scattering of beta particles, i.e., a large number of them are scattered by the electric field of the nucleus and not by inelastic scattering on other electrons. This is a surprising fact considering that the electrons of a material are ideal targets for beta particles having the same mass. However, inelastic scattering does play a significant role in beta scattering and beta particles usually leave quite complicated tracks in materials (see: *Radiochemistry and Nuclear Chemistry*).

3.1.1. Dependence on Atomic Number

Since backscattering is ruled by the Coulomb force between the nucleus and the beta particle, the atomic number of the target (i.e., the charge number of the nucleus) plays a significant role in backscattering. This atomic number dependence makes beta backscattering an analytic tool. Unfortunately, most beta particles are back-scattered not in a single event but after multiple scattering on several nuclei and electrons. As the mathematical handling of multiple scattering is quite complicated, we still have to use empirical relationships. Moreover, radioactive beta sources produce electrons of a continuous energy distribution limiting the analytical possibilities of beta backscattering.

According to numerous backscattering data, the number of back-scattered particles depends linearly on the logarithm of the atomic number, Z or, rather, on $\log(Z+1)$. This relationship works well for elements and, sometimes, even for compounds and mixtures. For compounds/mixtures, the weighted average of the atomic numbers is used, but the $\log(Z+1)$ relationship gives poor results for hydrogen-containing materials.

3.1.2. Saturation Effect

The probability that a particle becomes scattered increases with the thickness of the target material. This, for thin films, increases the number of back-scattered beta particles with the increase of the thickness. However, the range of beta particles (see: *Radiochemistry and Nuclear Chemistry*) limits the increase and a saturation value is reached when the thickness reaches the half of the beta range. (Actually the saturation thickness is a little shorter than half of the range.) The correlation between thickness and the number of back-scattered particles is so strict that the phenomenon is applied in automated thickness gauges.

3.1.3. Equipment

The equipment for beta backscattering measurements is simple. Besides the sample, it needs only a beta source, a detector and a counter. The source, the detector and the sample holder are arranged in a fixed geometry to gain comparable data. Some types of equipment apply ring sources and the detector is placed into the center of the ring. The source must be a "pure" beta source emitting only beta particles, usually ^{14}C, ^{147}Pm, ^{90}Sr/^{90}Y, or ^{204}Tl. The choice is made on the basis of the maximum beta energy, which determines the penetration depth of beta particles.

3.1.4. Applications

Due to the atomic number dependence, beta backscattering can be used to determine concentrations of certain elements in solvents and mixtures. However, the method requires a precise calibration process to gain useful data. This limits the use of the method to applications where a frequent or continuous concentration checking is needed but the constitution of samples does not vary much. Automated quality control is a good example, although non-nuclear techniques have replaced beta backscattering in most areas.

The main area of application for beta backscattering is thickness control. These applications are based on the principle discussed in Section 3.1.2. Obviously, the beta range and the saturation effect restrict the use of beta backscattering gauges to certain thicknesses. However, the availability of different energy beta sources provides a certain kind of flexibility: for very thin films, ^{14}C might be a good choice, while ^{90}Sr/^{90}Y is better for thicker films. Beta backscattering can even be used to determine the thickness of films on laminated surfaces but, in this case, the atomic numbers of the cover layer and the substrate must differ significantly. Polymeric films (paints) on metal substrates are good examples of the successful use of the method.

3.1.5. New Areas

A revived interest is expressed in the literature for beta backscattering nowadays. However, these new works do not consider beta backscattering as a material research tool but a side effect, wanted or not. Electron backscattering is rather rigorously reconsidered – by Monte-Carlo simulations and measurements – for two reasons. The first is that precise beta measurements should consider the backscattering of electrons from the detector. This is not a real problem in everyday work but, e.g., the study of the beta decay of free neutrons requires much greater precision.

The other area, where a more detailed knowledge is needed on beta backscattering is radiation therapy. Electrons are back-scattered at any boundary where the density is changing abruptly: at interfaces, at tissues and at the surfaces of organs or tumors. Thus, the beta dose absorbed around a point inside the human body can be strikingly different from the value observed for a homogeneous body of similar constitution.

3.2. Rutherford Backscattering

Alpha backscattering (or Rutherford backscattering, RBS) is much better suited for being an analytical method than beta backscattering. It is the elastic scattering of alpha particles on nuclei and the electrons of the material play a much simpler role than in beta scattering. Moreover, unlike beta sources, alpha sources produce monoenergetic particles simplifying the picture even more. It is also a fortunate condition for analytical applications that multiple scattering does not play a significant role in the backscattering of alpha particles.

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Biographical Sketches

Károly Süvegh was born in Oroszlány, Hungary, in 1962. He received his MSc in chemistry from Eötvös Loránd University (ELTE), Budapest, in 1986. He received his CSc (PhD) in nuclear chemistry from the Hungarian Academy of Sciences in 1995.

He has been working for ELTE ever since graduating there. Presently he is Associate Professor in the Laboratory of Nuclear Chemistry, Institute of Chemistry, ELTE. In the meantime he was Visiting Scientist at Lehigh University, Bethlehem, USA (1987). His research field has been chemical applications of positron annihilation spectroscopies. He teaches nuclear chemistry, particle physics and physics lab to chemistry majors. He co-edited with Prof. Vértes and Dr. Nagy a book listed in the Bibliography. He also co-authored several chapters in different books.

Zoltán Homonnay was born in Eger, Hungary, in 1960. He is a professor at Eötvös Loránd University, Budapest, where he teaches nuclear chemistry, radiation safety and nuclear environmental protection. His main research interest is Mössbauer Spectroscopy applied in various fields of chemistry and materials science. Ha has received his first degree (Candidate of Science awarded by the Hungarian Academy of Sciences, an analogue of PhD) in the field of Al-Fe alloys, and then turned toward high-temperature superconductors on which he published his most important papers and received his Doctor of Academy title (DSc). He had substantial contribution to a chapter about Mössbauer Spectroscopy in the Handbook of Nuclear Chemistry [Kluwer Academic Publishers Dordrecht-Boston-London (2003)]. He is heading subdivisions in the Hungarian Chemical Society (Radioanalytical Chemistry) and in the Hungarian Academy of Sciences (Nuclear Methods in Structural Chemistry).