

ANNUAL REPORT 2017



INSTITUTE OF NUCLEAR CHEMISTRY AND TECHNOLOGY

ANNUAL REPORT 2017



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EDITORS

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GENERAL INFORMATION

The Institute of Nuclear Chemistry and Technology (INCT) is Poland's leading research institution in the fields of nuclear and radiation chemistry, and radiobiology. Scientific activities of the INCT are dedicated to a wide range of nuclear topics, involving basic and applied research. Basic research is focused on separation processes of actinides and lanthanides, novel radiopharmaceuticals, radiation effects in molecules of biological importance, cells and polymers. With its six electron accelerators, the Institute is one of the most advanced centres in the field of electron beam applications. The various types of accelerators are used for radiation sterilization of medical devices and transplantation grafts, modification of polymers, removal of SO₂ and NO_x from flue gases, and for food irradiation.

The INCT Scientific Council has the right to confer D.Sc. and Ph.D. degrees in the field of chemistry. In 2017, one D.Sc. and six Ph.D. theses were defended in the INCT.

The Institute's expertise and infrastructure made possible the participation in numerous international and domestic projects. In 2017, the INCT started research activity in four projects of the EU programme Horizon 2020, in which leading European institutes and industrial companies participate:

- ARIES: Accelerator research and innovation for European science and society;
- GENIORS: GEN IV integrated oxide fuels recycling strategies;
- TeamCABLES: European tools and methodologies for an efficient ageing management of nuclear power plant cables;

• CHANCE: Characterization of conditioned nuclear waste for its safe disposal in Europe. Besides, four large projects of the National Centre for Research and Development (NCBR) were carried out, including the specially important for environmental protection project TANGO 2 "Plasma technology for removal nitrogen oxides from flue gases".

There were also 14 projects of the National Science Centre (NCN) led by the INCT researchers, majority of them dedicated to the development of new radiopharmaceuticals for medical diagnosis and therapy.

As the Collaborating Centre in Radiation Technology and Industrial Dosimetry of the International Atomic Energy Agency (IAEA), the Institute coordinated a large-scale intercomparison test on alanine dosimetry measurements for electron beam and gamma irradiations in which 10 European countries participated. Under the framework of the IAEA Coordinated Research Programme, the INCT researchers took part in 10 IAEA projects cooperating with scientists from numerous countries all over the world. Together with the IAEA, in the framework of ARIES project, the INCT organized the conference "Summer School on Advanced Application of Electron Beam Accelerators" in which 40 scientists from 14 countries participated. The other conferences or seminars organized in 2017 concerned developments and applications of nuclear technologies (together with AGH University of Science and Technology, Kraków, Poland) and radiation modifications of polymers.

For the Ministry of Energy, the INCT prepared two reports important for development of Polish nuclear energy programme:

- the analysis of nuclear waste and burnt fuel treatment in Poland,
- the analysis of ionizing radiation applications in Polish industry together with recommendations for Polish enterprises.

In 2017, the INCT scientists published 80 papers, including 60 publications in scientific journals with an impact factor (IF).

MANAGEMENT OF THE INSTITUTE

MANAGING STAFF OF THE INSTITUTE

Director

Prof. Andrzej G. Chmielewski, Ph.D., D.Sc.

Deputy Director for Research and Development Prof. Jacek Michalik, Ph.D., D.Sc.

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- Centre for Radiochemistry and Nuclear Chemistry
 Prof. Grażyna Zakrzewska-Kołtuniewicz, Ph.D., D.Sc.
- Centre for Radiobiology and Biological Dosimetry
 Prof. Marcin Kruszewski, Ph.D., D.Sc.
- Laboratory of Nuclear Control Systems and Methods Jacek Palige, Ph.D.
- Laboratory of Material Research **Wojciech Starosta**, Ph.D.

- Laboratory of Nuclear Analytical Methods Halina Polkowska-Motrenko, Ph.D., D.Sc., professor in INCT
- Stable Isotope Laboratory **Ryszard Wierzchnicki**, Ph.D.
- Pollution Control Technologies Laboratory **Yongxia Sun**, Ph.D., D.Sc., professor in INCT
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 $\label{eq:constraint} \textbf{Anna Korzeniowska-Sobczuk}, M.Sc.$

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- **Bilewicz Aleksander** 1 radiochemistry, inorganic chemistry
- 2. Bobrowski Krzysztof radiation chemistry, photochemistry, biophysics
- 3. Chmielewski Andrzej G. chemical and process engineering, nuclear chemical engineering, isotope chemistry
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- 6. Dybczyński Rajmund analytical chemistry
- 7. Gniazdowska Ewa, professor in INCT chemistry
- +Jamróz Michał, professor in INCT 8. chemistry, physics
- Kruszewski Marcin 9. radiobiology
- 10. Lankoff Anna biology
- 11. Lipkowski Janusz physical chemistry

- radiation chemistry, surface chemistry, radical chemistry
- 13. Migdał Wojciech, professor in INCT chemistry, science of commodies
- 14. Ostyk-Narbutt Jerzy radiochemistry, coordination chemistry
- 15. Pawlukojć Andrzej, professor in INCT chemistry
- 16. Pogocki Dariusz, professor in INCT radiation chemistry, pulse radiolysis
- 17. Polkowska-Motrenko Halina, professor in INCT analytical chemistry
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- 21. Trojanowicz Marek analytical chemistry
- 22. Zakrzewska-Kołtuniewicz Grażyna process and chemical engineering

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- 2. Bartosiewicz Iwona chemistry
- Bojanowska-Czajka Anna 3. chemistry
- 4. Brykała Marcin chemistry
- 5. Brzóska Kamil biochemistry
- 6. **Chajduk Ewelina** chemistry
- Danilczuk Marek (Ph.D., D.Sc.) 7. chemistry

- Dobrowolski Andrzej 8. chemistry
- 9. Dudek Jakub chemistry
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- 11. Głuszewski Wojciech chemistry
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- **16. Kiegiel Katarzyna** chemistry
- **17. Kocia Rafał** chemistry
- **18. Kornacka Ewa** chemistry
- **19. Koźmiński Przemysław** chemistry
- **20. Kunicki-Goldfinger Jerzy** conservator/restorer of art
- **21. Latek Stanisław** nuclear physics
- 22. Lewandowska-Siwkiewicz Hanna chemistry
- **23. Łuczyńska Katarzyna** chemistry
- 24. Łyczko Krzysztof chemistry
- 25. Łyczko Monika chemistry
- 26. Majkowska-Pilip Agnieszka chemistry
- 27. Męczyńska-Wielgosz Sylwia chemistry
- 28. Miśkiewicz Agnieszka chemistry
- **29.** Nowicki Andrzej organic chemistry and technology, high-temperature technology
- **30. Ostrowski Sławomir** chemistry
- **31. Palige Jacek** metallurgy
- **32.** Pawelec Andrzej chemical engineering
- **33. Pruszyński Marek** chemistry
- **34. Ptaszek Sylwia** chemical engineering
- **35. Rafalski Andrzej** radiation chemistry

- **36. Rode Joanna** chemistry
- **37. Roubinek Otton** chemistry
- 38. Sadło Jarosław chemistry
- **39. Samczyński Zbigniew** analytical chemistry
- **40.** Sartowska Bożena material engineering
- **41. Sochanowicz Barbara** biology
- **42. Sommer Sylwester** radiobiology, cytogenetics
- **43. Starosta Wojciech** chemistry
- 44. Sterniczuk Macin chemistry
- 45. Szreder Tomasz chemistry
- **46. Waliś Lech** material science, material engineering
- **47. Walo Marta** chemistry
- **48. Wawszczak Danuta** chemistry
- 49. Wierzchnicki Ryszard chemical engineering
- **50. Wiśniowski Paweł** radiation chemistry, photochemistry, biophysics
- 51. Wojewódzka Maria radiobiology
- **52. Wójciuk Grzegorz** chemistry
- 53. Wójciuk Karolina chemistry
- **54.** Zimek Zbigniew electronics, accelerator techniques, radiation processing
- 55. Zwolińska Ewa chemistry

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CENTRE FOR RADIATION RESEARCH AND TECHNOLOGY

The electron beams (EB) offered by the Centre for Radiation Research and Technology located at the Institute of Nuclear Chemistry and Technology (INCT) are dedicated to basic research, R&D study, and practical applications of radiation technology.

The Centre, in collaboration with the universities from Poland and abroad, applies EB technology to fundamental research on electron beam-induced chemistry and the transformation of materials. Research in the field of radiation chemistry includes studies on the mechanism and kinetics of radiation-induced processes in liquid and solid phases by the pulse radiolysis method. The pulse radiolysis experimental set up allows for direct time-resolved observation of short-lived intermediates (typically within the nanosecond to millisecond time domain), and is complemented by steady-state radiolysis, stopped-flow absorption spectrofluorimetry, and product analysis using chromatographic methods. Studies on radiation-induced intermediates are dedicated to the processes of energy and charge transfer, to radical reactions in model compounds of biologically relevant aromatic thioethers, peptides and proteins, and also to the observation of atoms, clusters, and radicals by electron paramagnetic resonance (EPR) and electron nuclear double resonance (ENDOR). Further, these studies are also focused on research problems in nanophase chemistry and the radiation-induced crosslinking of selected and/or modified polymers and copolymers.

This research has a wide range of potential applications, including creating more environmentally friendly and sustainable packaging, improving product safety, and modifying material properties. Electron accelerators provide streams of electrons to initiate chemical reactions or break down chemical bonds more efficiently than the existing thermal and chemical approaches, thus helping to reduce energy consumption and thereby decrease the cost of the processes. The Centre currently offers five electron accelerators to study the effects of accelerated electrons on a wide range of chemical compounds with a focus on electron beam-induced polymerization, polymer modification, and the controlled degradation of macromolecules. EB technology has great potential to promote innovation, including new ways to save energy and reduce the use of hazardous substances, as well as to enable more eco-friendly manufacturing processes.

Advanced EB technology offered by the Centre provides a unique platform to be used for some of the following applications: sterilization of medical devices, pharmaceutical materials, shelf-life extension food products, advanced polymer materials, air pollution removal technology, and others. EB accelerators replace frequently thermal and chemical processes for cleaner, more efficient, lower cost manufacturing.

The Centre offers EB in the energy range of 0.2 to 10 MeV with an average beam power up to 20 kW and three laboratory-size gamma sources with Co-60. Research activity is supported by unique laboratory equipment such as:

- nanosecond pulse radiolysis and laser photolysis set-ups,
- stopped-flow experimental set-up,
- EPR spectroscopy for solid materials investigation,
- pilot installation for polymer modification,
- experimental stand for removal of pollutants from gas phase,
- polymer characterization laboratory,

• pilot facility for radiation sterilization, polymer modification, and food product processing. The unique technical core makes it possible to organize a wide internal and international cooperation in the field of radiation chemistry and radiation processing including programmes supported by the European Union and the International Atomic Energy Agency (IAEA). It should be noted that currently there are no other suitable European experimental platforms for the study of radiation chemistry, physics and radiation processing in a full range of electron energy and beam power.

Since 2010, at the INCT and based on the Centre for Radiation Research and Technology, an IAEA Collaborating Centre for Radiation Processing and Industrial Dosimetry is functioning. That is the best example of the capability and great potential of concentrated equipment, as well as of the employed methods and staff working towards the application of innovative radiation technology.

PARAMAGNETIC DEFECTS IN GRAPHENE OXIDE IRRADIATED WITH AN ELECTRON BEAM

Grażyna Przybytniak, Jarosław Sadło, Małgorzata Dąbrowska, Bożena Sartowska

Studies on graphene and its derivatives are objects of interest for many researchers representing various disciplines. Radiation chemistry provides the specific tools that are able to enhance our current understanding of the features of these unique materials, answering questions about the nature of the interactions between high energy electrons and the layered structure of graphene.

So far, the issue of whether high energy electrons decrease or increase the order in the structures of graphene (G) or graphene oxide (GO) remains unresolved [1, 2]. Previous studies on



Fig. 1. SEM images of graphene oxide with different magnifications.

carbon nanotubes showed that the effect might be a function of temperature, as in the range of 200-300°C a reduction in the number of the lattice defects was confirmed, whereas at ambient temperature their population was raised significantly [3]. Similar phenomena might be expected in G and GO. These problems are important because irradiation might influence electron transfer and heat conductivity throughout GO lattices, as well as the content of chemical groups.

The general objective of the research was to determine the effects initiated by ionizing radiation in GO with regards to the qualitative and quantitative changes in the population of paramagnetic defects. Graphene oxide was synthesized at the Institute of Electronic Materials Technology (ITME) using a modified Hummers' method [4]. Changes induced by a 10 MeV electron beam generated in an Elektronika accelerator (Institute of Nuclear Chemistry and Technology, INCT) were monitored by electron paramagnetic resonance (EPR) spectroscopy, scanning electron microscopy (SEM) and thermogravimetric analysis (TGA) methods.

The SEM images presented in Fig. 1 reveal the morphology of graphene oxide. The porous structure was constructed from thin GO flakes. The multilayered structures of large, wrinkled surfaces had a thickness which depended on the number of stacked sheets. As seen from the SEM images, the GO flakes showed a thickness of about 20 nm. The wrinkles on the surfaces were probably formed during synthesis of the carbon nanostructures. The images of the material did not change noticeably upon electron beam irradiation with doses up to 1200 kGy, which indicated a negligible influence of electrons on the morphology of GO.

During heating in an air atmosphere, GO decayed showing three characteristic stages (Fig. 2). At temperatures below 150°C, water and weakly bound functional groups were emitted. The second step, around 200°C, was attributed to the release



Fig. 2. TGA and DTG (derivative thermogravimetric analysis) relationships of GO determined in an air flow.



Fig. 3. EPR spectra of graphene oxide measured at two different microwave powers: (A) 1 mW and (B) 100 mW.

of labile oxygen-containing groups, predominantly CO and CO₂. After a mass loss of about 35%, the rest of the functional groups were separated from each other and needed higher temperatures to be removed [5]. They were emitted simultaneously with thermal oxidation of the carbon lattice occurring in the last stage of decomposition. In this case, differences in the thermograms of irradiated and non-irradiated GO were negligible.

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EPR spectroscopy of GO disclosed all the paramagnetic defects in graphene oxide (Fig. 3A). At low microwave power (1 mW), only a narrow singlet was observed with a peak-to-peak linewidth of about 0.22 mT and g = 2.0029. As shown in Fig. 4, the initial intensity of the spectrum increased with the doses used. The spectral parameters remained almost the same, whereas the height of the signals increased significantly. However, for doses over 200 kGy, the population of paramagnetic defects did not change significantly. When the microwave power was increased up to 100 mW, additional low- and high-field wings separated by $\Delta H = 1.28$ mT appeared, while the central singlet became wider and partly saturated (Fig. 3B).



Fig. 4. Relative content of paramagnetic centres in GO irradiated with various doses determined by double integration of the experimental signals.

Based on the analysis of the EPR spectra we concluded that:

• There are at least two types of paramagnetic defects in graphene oxide, situated either at the edges of the GO flakes or on their surfaces.

- The wider signal might be tentatively assigned to defects in which unpaired spin interacts with oxygen atoms. Spectra attributed to such structures are usually broader and insensitive to microwave power saturation.
- Electron beam irradiation initially increases the population of paramagnetic centres. Their nature is similar to the defects existing before exposure to ionizing radiation because although the intensity of the EPR spectra increases, the signal parameters remain unchanged.
- Electrons are presumably trapped by the preexisting non-paramagnetic defects.
- As seen from EPR spectra, upon absorption of a dose greater than 200 kGy the amount of paramagnetic centres reaches a plateau. It seems that with increasing doses the number of suitable defects playing the role of electron traps is reduced, which results in more efficient recombination of electrons due to a cage effect. The process might be enhanced by repulsion from other captured electrons.

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RADIATION MODIFIED ELASTOMERIC COMPOSITES FOR EMC MICROWAVE ABSORBER

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EMC (electromagnetic compatibility) is the branch of electrical engineering concerned with the unintentional generation, propagation and reception of electromagnetic energy, which may cause unwanted effects such as electromagnetic interference (EMI) or even physical damage to operational equipment. The goal of EMC is the correct operation of various types of equipment in a common electromagnetic environment. EMC absorbers tested by the Military University of Technology used elastomer sheets containing magnetic material. They include species targeted to absorb specific frequency bands, as well as components that absorb noise across a wide frequency band.

In some circumstances, there is a need to perform an electromagnetic compatibility investigation not in a specialized anechoic chamber, but in an open space. However, the typical absorbers used in anechoic chambers to absorb the incident radiation and to reduce the reflected rays from walls and floor, such as ferrite tiles and graphite cones, are not suitable for use in open spaces. In this work, the design and investigation of an absorbing material intended to be used to make tents or to produce other light and portable constructions are described. The proposed composite material has good absorbing properties as well as low reflectivity.

Radiation techniques have been used to modify both electronic materials [1] and polymers [2]. In the above example, both processes were combined. The aim of the study was to find new microwave absorbing materials with the following parameters: reduced weight compared to typical ferrites, elasticity, the possibility of being used on the ground outdoors and mechanical resistance to stress and strain, e.g. driving with a motor vehicle. Engage™ polyolefin elastomers (POEs) of the ethylene/octene or ethylene/butene type were selected for testing as composite matrices [3]. It was assumed that suitable composite materials would be based on a new generation of compounds containing ferromagnetic components that exhibit very good damping properties for electromagnetic radiation over a wide frequency range. Various compositions of materials were measured, and the following composition was selected as the final one: metallic glass (70%) with graphite admixture (1%) and Engage 8200 elastomer (29%).

A new composite material, based on a nanocrystalline-amorphous alloy based on iron, was used to synthesize a new absorber. Nanocrystalline alloys are very attractive soft magnetic materials. Metallic glass is used as a precursor to produce nanocrystalline alloys. A nanocrystalline alloy with a composition of Fe_{73.5}Si_{13.5}B₉Nb₃Cu₁ (FINEMET), formed by Yoshizawa *et al.* [4], was the first alloy of this type, and was used as the absorbing material in this work. The quenched, amorphous ribbon was annealed for 1 h at a temperature of 550°C to induce partial crystallization, allowing us to obtain a mean grain size of 10-15 nm. Afterwards, the annealed ribbon was crushed and ball milled to obtain a powder with a particle size of 25-50 μ m (Fig. 1).



Fig. 1. Comparison of (A) the crystalline structure with (B) the amorphous structure of the metallic glass.

To obtain improved electrical properties, the nanocrystalline alloy powder was mixed with graphite. In addition, in order to improve the absorption properties of the metallic glass, it was radiation modified with a dose of 100 kGy.

Previously, the authors investigated the effect of various crosslinking methods (peroxide, ionizing radiation, ionizing radiation/peroxide and peroxide/ionizing radiation) on the properties of the elastomer.

The properties of Engage 8200 are the following: melt index $(190^{\circ}C/2.16 \text{ kg}) - 5.0 \text{ dg/min};$ density $- 0.870 \text{ g/cm}^3$; Mooney viscosity - 8 ML(1 + 4) at $121^{\circ}C$; ultimate tensile strength - 5.7MPa; ultimate tensile elongation - 1100%; 100%Modulus - 2.3 MPa; hardness, Shore A (1 s) - 66, Shore D (1 s) - 17; tear strength type C - 37.1kN/m; Vicat softening point $- 37^{\circ}C$; DSC melting point, $10^{\circ}C/\text{min}$ rate $- 59^{\circ}C$ (Dow method); glass transition temperature $- 53^{\circ}C$ (Dow method); Tc peak $- 4^{\circ}C$ (Dow method).

Initial studies on the effect of radiation dose on the elastomer properties were carried out. Finally, the elastomer was crosslinked with a dose of 50 kGy. The measurements (tensile strength, modulus hardness, Shore A, elongation at break, permanent elongation and compression set) were performed in a certified laboratory at the Institute for Engineering of Polymer Materials and Dyes (PCA accreditation certificate No. AB 147 for the inspection of raw materials, blends and thermoplastic elastomers). The work was performed in accordance with Polish ISO standards. The test results are summarized in Table 1.

Table 1. The impact of radiation dose on the properties of Engage 8200 elastomer.

Managered mean anter		Dose [kGy]						
Measured property	0	50	100	200	300			
Tensile strength [MPa]	6.7	9.7	9.5	8.4	6.2			
Elongation at break [%]	901	869	828	696	509			
Modulus 100% [MPa]	2.3	2.2	2.2	2.2	2.2			
Modulus 200% [MPa]	2.7	2.7	2.7	2.8	2.8			
Modulus 300% [MPa]	3.0	3.0	3.0	3.2	3.4			
Hardness Shore A [°ShA]	71	69	68	67	67			

To analyse the electrical properties of the absorber, to see whether it fulfils the desired electromagnetic requirements, the constitutive material parameters should be determined. The electromagnetic properties of an isotropic material can be described macroscopically by scalar material properties in term of relative complex permittivity (ϵ) and permeability (μ):

$$\varepsilon = \varepsilon' - j\varepsilon''$$

 $\mu = \mu' - j\mu''$

where: ε' – the electric constant, μ' – the magnetic constant, ε'' – the electric loss factor, μ'' – the magnetic loss factor and j – the imaginary number.

The permittivity and permeability broadband measurements of materials are commonly performed using coaxial fixtures. The basic measurements are based on the scattering parameters of the sample. Coaxial line technique allows the constitutive parameters of materials to be measured over a wide frequency band. In a typical configuration of a solid material for measurement of its permittivity and permeability in a coaxial line, the sample completely fills the cross-section of the holder (Fig. 2).



Fig. 2. Schematic configuration of the sample in the co-axial line.

Measurements of electrical and magnetic permeability were made in a coaxial line using a modified measurement method. An electron beam (an Elektronika 10/10 accelerator with a dose rate of 15,000 kGy/h) and gamma radiation (cobalt source GC 5000 with a dose rate of 3.5 kGy/h) were used in the polymer studies. The best absorption properties of the composite were at 1 GHz, where the material did not exhibit electromagnetic field reflections. From the measurements and results obtained, it can be concluded that the composite is a promising absorbing material for solving problems concerning the ability of an electrical or electronic device to work properly in specific electromagnetic environments, such as in antenna systems [5]. Developing an optimal composite requires further research.

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A CONTINUOUS, NON-INTERFERING LINAC ELECTRON BEAM ENERGY MEASUREMENT SYSTEM FOR RADIATION PROCESSING INSTALLATION

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Continuous electron energy measurement is currently obligatory for accelerator installations intended for radiation sterilization. This is one of several values, such as dose rate, beam current, transporter speed and the sweep parameters, which must be recorded in accordance with the conditions listed in the accelerator validation requirements code. While determining the energy of electrons in electrostatic accelerators is relatively simple and leads to the measurement of the accelerating voltage, in the case of RF linear accelerators it cannot be done by means of a simple and direct measurement.

Some accelerator installations have an integrated magnetic electron energy analyser, but

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Fig. 1. Essential components of the measurement system.

such a measurement procedure cannot be carried out during sterilization. Also, the use of an aluminium wedge does not meet the requirements for an on-line measurement.

A typical solution is indirect energy estimation based on the knowledge of certain parameters, such as microwave power level and beam current, which affect the output energy of the electron beam. These parameters can be measured on an ongoing basis, and energy can be calculated using known relationships and calibration curves.

Unfortunately, this method does not give a result with satisfactory accuracy, mainly due to the necessary simplification of the computational model and the inability to measure many other physical parameters that this model would have to take into account.

One of the possible solutions [1] is the use of a sensor located directly under the window of the scanned electron beam in such a way that it does not interfere with the distribution of the dose in the irradiation area and provides data to determine the energy of electrons in the beam and the spectral distribution of this energy.

The measurement method based on the use of a secondary electron-collecting electrode was tested in an electron beam scanned installation, typically used for radiation sterilization. The concept of the measurement is shown in Fig. 1. The collecting electrode can be made of a strip of thin metal (Al) or wire. The high energy electrons passing through the electrode material knock out a number of secondary electrons, some of which are able to leave the electrode. This is recorded as the flow of a small, positive current from the electrode, and it is directly proportional to the intensity of the primary electron stream. For different energies, the magnitude of the deflection will also be different, and therefore the trajectory of electron passage will be different. If there are components with various energies in the beam, then it will be split and stretched over a certain section of Δx . When analysing the intensity



Fig. 2. Waveform oscillogram: A – current in sweeping magnet coil, B – current from the collecting electrode.

of the passing electron stream at subsequent points along Δx , the energy spectrum of the electron beam can be obtained.

The physical movement of the electrode along Δx is unnecessary, because the sweeping current



Fig. 3. Visualization of electron beam energy spectrum.

causes the beam to move linearly along the X-axis, thus all points of the Δx section will move over the electrode at some time. As a result, the current collected by the electrode is in the form of a pulse with an amplitude modulated by the instantaneous intensity of the stream (Fig. 2).

The data delivered *via* the interface is stored in the computer operating memory. After applying the error elimination procedures and averaging, the data are normalized and sent for visualization, as shown in Fig. 3. provide information on the working conditions of various accelerator components, including their alignment and wear level.

The developed and tested system, after calibration using other methods of energy determination, currently works in a continuous mode at the accelerator installation at the Sterilization Station in the Institute of Nuclear Chemistry and Technology (INCT). The current control of the distribution of the electron beam energy spectrum and the automated measurements of other parameters are re-



Fig. 4. An example of electron beam energy recorded over the facility workday.

In addition to the requirement of archivization of electron energy results of measurement during the sterilization process (as shown in Fig. 4), knowledge of the distribution of the electron beam energy spectrum allows the operator to set up the accelerator device in such a way that the machine performs the process at its optimum efficiency. During repair or maintenance works, such measurements are a very important diagnostic tool. They corded in accordance with the applicable requirements and enable multi-parameter optimization of the irradiation process.

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CENTRE FOR RADIOCHIEMIISTRY AND NUCLEAR CHIEMIISTRY

Chemical issues of nuclear power and radiopharmaceutical chemistry are the main research areas of the Centre for Radiochemistry and Nuclear Chemistry. In 2017, the Centre worked on research projects financed in the form of grants from the National Centre for Research and Development (NCBR) and the National Science Centre (NCN), as well as in the form of founding the Institute's statutory research and development and international cooperation from by Ministry of Science and Higher Education.

International collaboration was focused on the development of the European Commission (FP7 Euratom, Fission) and other (IAEA, COST) projects. The individual projects of young scientists funded under the specific subsidy of the Ministry of Science and Higher Education were important elements for the development of the human resources of the Centre.

The teams of three Centre laboratories (Radiochemical Separation Methods, Membrane Processes and Technologies, and Sol-Gel Technology) continued their studies on radioactive waste management, reprocessing of spent nuclear fuel, and on special nuclear materials. In this respect, the Sol-Gel Technology team continued research on the synthesis of potential nuclear fuel for future reactor systems based on uranium oxides and carbides, as well as mixed thorium-uranium dioxide, in the form of microspheres. The sol-gel method was also applied for preparation of phosphate glasses with potential use for radioactive waste stabilization.

The team of Radiochemical Separation Methods Laboratory continued the research on actinide/lanthanide separation by solvent extraction, which was focused on the verification of hypotheses previously set during the European Collaborative Project SACSESS implementation, regarding the formation of heteroleptic complexes of actinides with TODGA and SO_3 -Ph-BTP ligands. The present Euratom project in this field, namely GENIORS (GEN IV integrated oxide fuels recycling strategies, Horizon 2020), focuses on future multiple recycling strategies for spent fuel from the GEN IV reactors. In 2017, the research work of the Centre team concerned the study on new extraction systems for the separation of americium(III) from light lanthanides. Advanced quantum chemical calculations were continued, which allowed explaining the reason of actinide selectivity of some ligands used for solvent extraction separation of actinides from lanthanide fission products. The knowledge based on molecular modelling is crucial for design and synthesis of novel, more selective ligands for An/Ln separations.

Experimental and computational design of chiral molecules for applications in medicine and manufacturing of conductive polymers were performed within the frame of the grant financed by the National Science Centre for research executed in a few Polish universities and research institutes, and supervised by the Spectroscopy and Molecular Modelling group of our Centre.

Recovery of uranium and accompanying metals from various types of industrial wastes like phosphogypsum or waste from flotation of copper ores was studied in the scope of the IAEA CRP. Various aspects related to the management and storage of spent nuclear fuel and radioactive wastes formed in the course of exploitation of nuclear power plants (NPPs), with a special emphasis on the Polish Nuclear Power Programme, were studied.

The team of the Centre participated in the consortium of six Polish institutions working on the development of a safety assessment methodology and indication of the optimal location for shallow storage of low- and intermediate-level radioactive waste. At the request of the Ministry of Energy, the team prepared in collaboration with ZUOP an expert report "Management of radioactive waste and spent nuclear fuel in Poland – current status and prospects". Radioactive waste aspects concern the new Euratom Horizon 2020 CHANCE project (Characterisation of conditioned nuclear waste for its safe disposal in Europe) in which the Centre participates. The project carried out by the international consortium, commenced in 2017, is focused on development of new non-destructive methods of radioactive waste characterization, such as muon tomography, calorimetry and innovative techniques based on laser spectroscopy for gaseous radioactive emissions monitoring. Applications in the field of radioactive waste management are complemented by the studies on new sorbents of radionuclides, among others, novel magnetic cobalt cyanoferrate nanoparticles, sufficiently stable for practical application in the treatment of the radioactive effluents.

Novel methods were examined by the Membrane Processes group, for the treatment of "problematic" nuclear waste, based on integrated processes (membrane filtration combined with sorption, advanced oxidation-membrane process), as the basis for further technological advancement in radioactive waste processing field. The application of membrane systems in nuclear desalination was tested within the frame of another IAEA CRP. The possibility of the application of such methods as reverse osmosis and membrane distillation, for desalination as well as radioactive waste treatment within nuclear power plants, was proved.

Basic research on the phenomena occurring in membrane units was continued in the scope of the NCN research project on the development of sensitive methods for studying concentration polarization and membrane fouling. The combination of radiotracers with optic techniques like SEM (scanning electron microscopy), FTIR/PAS (Fourier-transform infrared/ photoacoustic spectroscopy) has brought data for the future elaboration of the methodology of testing membrane units.

Social and socio-economic effects of implementation of the Polish Nuclear Power Programme with the development of macroeconomical tools for assessment were studied within the frame of the IAEA CRP in cooperation with the governmental institutions.

Research on radiopharmaceutical chemistry (Laboratory of Radiopharmaceuticals Synthesis and Studies) was focused on obtaining and studying novel potential radiopharmaceuticals, both diagnostic and therapeutic. Novel biomolecules, derivatives of tacrine, agonists (*e.g.* substance P) or antagonists (*e.g.* aprepitant, Spantide I (5-11)) of NK-1 receptor, as well as antibiotics used in medical treatment of bacterial infections, were labelled with ^{99m}Tc or ⁶⁸Ga, resulting in potential diagnostic tools for Alzheimer's disease, glioma brain tumours, breast cancer and diabetic foot, respectively. A part of the research was carried out in cooperation with the Department of Pharmaceutical Chemistry and Drug Analyses, Medical University of Łódź and with the Department of Nuclear Medicine, Medical University of Warsaw. New methods for cyclotron productions of diagnostic radionuclides, both SPECT (^{99m}Tc) and PET (⁴³Sc, ⁴⁴Sc, ⁷²As) were developed in cooperation with the Heavy Ion Laboratory of the University of Warsaw, and the National Centre for Nuclear Research – POLATOM, within two projects awarded by the NCBR.

The development of new cyclotron method for ⁴⁷Sc production was continued in the scope of a new IAEA Research Contract. Also potential therapeutic radiopharmaceuticals were obtained and studied. Peptides and proteins were labelled with alpha emitters (²¹¹At, ²²⁵Ac and ²²³Ra) *via* functionalized soft-metal chelates (metal bridge), and by the use of functionalized nanoparticles such as nanozeolites and gold nanoclusters.

Multifunctional nanoparticles for magnetic hyperthermia and indirect radiation therapy were studied in cooperation of the international consortium in the frame of COST project and in the scope of NCN OPUS project.

The teams of the Centre were awarded with the Director's prize for publications presented in 2015-2016 and the Director's award for application achievements in the same period.

The international and national scientific cooperation of the Centre was successfully continued and enhanced making the Centre teams desired partners not only on the national scale, but also over the European research area.

The Centre participated in the organization of several meetings, conferences, seminars and trainings of students.

The scientists of the Centre were involved in the activities of a large number of organizations, societies, and editorial boards of scientific journals in the country and abroad.

⁶⁸Ga- AND ¹⁷⁷Lu-RADIOPHARMACEUTICALS BASED ON NEUROKININ-1 RECEPTOR ANTAGONIST SPANTIDE I (5-11) PEPTIDE FOR GLIOBLASTOMA MULTIFORME TREATMENT

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Gliomas are extremely serious and aggressive brain tumours in adults. For patients with the most malignant glioma - glioblastoma multiforme (GBM, glioblastoma) the prognosis for survival is 9-12 months. All malignant gliomas are primary brain tumours associated with a high morbidity and mortality due to very difficult treatment regimes. It is caused by the fact that tumour cells are relatively resistant to chemo- and radiotherapy, which is highly damaging to healthy brain tissue. Moreover, an accurate surgical treatment is also ineffective and leads to cancer resumption due to the infiltrating and diffusive character of glioblastoma cells spreading beyond the primary tumour area [1]. Therefore, novel therapeutic strategies are needed for the effective management of GBM.

In the past two decades, targeted glioma therapy has become the subject of extensive scientific reflection and study. The development of biomolecular techniques has allowed the recognition of characteristic tumour cell markers, showing potential for treatment. Many published reports have indicated the participation of the Substance P (SP)-Neurokinin 1 receptor (NK-1R) system in the evolution and metastasis of various neoplasm, including malignant gliomas [2-4].

NK-1R is a seven transmembrane helix protein receptor belonging to the family of G-protein coupled receptors (GPCRs). Its endogenous ligands are tachykinins, from which Substance P shows the highest affinity to the receptor. SP is undeca-neuropeptide (sequence Arg-Pro-Lys-Pro--Gln-Gln-Phe-Phe-Gly-Leu-Met-NH₂) distributed mainly in the nervous system, but also in peripheral tissues. Both SP and NK-1R are responsible for cell proliferation, vasodilation, transmission of pain, endocrine secretion, immune system inflammatory response and modulation of neuronal sensory transmission related to stress, emesis and anxiety. SP is also a mitosis and haematopoiesis regulatory agent [5].

In pathological conditions the NK-1 receptor is upregulated. This fact makes SP with its recep-

tor system a potential target for recognition and diagnosis of many diseases related with an excessive NK-1R stimulation. However, to date, clinical studies have confirmed application effectiveness of one NK-1R antagonist only against nausea and vomiting induced by chemotherapy. NK-1R overexpression is widely presented in a variety of tumour cell lines, also in glioma cancer lines [4, 6]. Preclinical trials proved the feasibility of targeted local radiotherapy based on the SP/NK-1R system. For GBM treatment, studies have been performed using a labelled SP derivative (²¹³Bi--DOTA-[Thi⁸,Met(O_2)¹¹]-Substance P) proposed in 2010 by Cordier *et al.* [7]. Such treatments were also applied at the Department of Nuclear Medicine, Central Clinical Hospital, Warszawa, in cooperation with the Institute for Transuranium Elements (JRC-ITU, Karlsruhe), where ²¹³Bi/ ²²⁵Ac-DOTA-[Thi⁸,Met(O₂)¹¹]-Substance P radio-pharmaceuticals were examined in patients diagnosed with recurrent critically located GBM. The results of this experiment showed good toleration of the preparation without acute or serious side effects. Although the disease was stabilized, applied radiobioconjugates showed poor migration into the marginal tumour area and instability in blood presence conditions.

To eliminate these disadvantages, we propose a change of biological vector in applied radiopharmaceuticals to the shorter peptide NK-1R antagonist, Spantide I (5-11), SPE (5-11), (sequence Gln--Gln-D-Trp-Phe-D-Trp-Leu-Leu-NH₂). Spantide I is a SP analogue, (D-Arg¹, D-Trp^{7.9}, Leu¹¹)SP(1-11) with similar affinity to NK-1 receptor [8]. The shorter peptide was chosen according to our experience with fragments of SP [9], where the radiobioconjugate ¹⁷⁷Lu-DOTA-SP(5-11) has showed the highest log P value from analysed compounds. Moreover, Spantide I is more resistant to blood peptidases than SP, because of the replacement of L-amino acids with D-isomers.

In this work we have focused on the labelling of the peptide NK-1R antagonist Spantide I (5-11)



Fig. 1. Structures of the ¹⁷⁷Lu-DOTA-SPE (5-11) and ⁶⁸Ga-DOTA-SPE (5-11) radiobioconjugates.

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'	Table 1. Physicochemical properties of ⁶⁸ Ga-DOTA-SPE (5-11) and ¹⁷⁷ Lu-DOTA-SPE (5-11) radiobioconjugates.							
	Radiobioconjugate	log P	Human serum stability [%] (after certain time)	Radiobioconjugate bound to the biomatr components [%] (after certain time)				
	⁶⁸ Ga-DOTA-SPE (5-11)	-0.56 ± 0.02	> 99 (4 h)	8.7 (1 h)	-	11.9 (4 h)		
	¹⁷⁷ Lu-DOTA-SPE (5-11)	-0.19 ± 0.03	> 99 (7 d)	5.3 (1 h)	8.3 (2 d)	9.1 (7 d)		

with two radioisotopes – ⁶⁸Ga and ¹⁷⁷Lu (Fig. 1), using a macrocyclic chelator DOTA (1,4,7,10-tetraazacyclododecane-1,4,7,10-tetraacetic acid) in the form of active mono-N-hydroxysuccinimide ester (NHS) – DOTA-NHS (1,4,7,10- tetraazacyclododecane-1,4,7,10-tetraacetic acid mono-N-hydroxysuccinimide ester). Our aim was to obtain a novel peptide radiobioconjugate, elaborate on the conditions for effective labelling and examine the stability parameters of the obtained radiobioconjugates.

SPE (5-11) was coupled with DOTA-NHS with addition of Et_3N in DMF at 50°C over night (the molar ratios of reagents were 1 : 1.3 : 4, respectively). Crude DOTA-SPE (5-11) product was purified on a semi-preparative HPLC and lyophilized giving a yield of 60%. MS of DOTA-SPE (5-11): calculated – 1405.63 m/z, found – 1405.73 m/z [M+H⁺]. The labelling reactions of the obtained

Then the stability studies were performed, where the percentage of the unchanged form of each radiobioconjugate in certain time periods was determined. Experiments included analysis of the stability in human serum verified by the HPLC method with radiometric detection.

All examined radiobioconjugate parameters are presented in Table 1. The lipophilicity values of both molecules of interest are slightly negative (-0.56 and -0.19 for ⁶⁸Ga and ¹⁷⁷Lu, respectively). These values, compared to the lipophilicity parameters of radiobioconjugates based on SP and its fragments [9], are significantly higher; this is promising from a pharmacokinetic point of view. Moreover, the presented radiobioconjugates showed total stability in human serum for the entire duration of the experiments (4 h and 7 d for ⁶⁸Ga and ¹⁷⁷Lu, respectively), verified by the radiometric chromatograms (Fig. 2).



Fig. 2. Radiometric chromatograms of liquid phases of ⁶⁸Ga-DOTA-SPE (5-11) and ¹⁷⁷Lu-DOTA-SPE (5-11) radiobioconjugates in human serum solution after 4 h and 7 d, respectively. HPLC conditions: Phenomenex Jupiter Proteo semi-preparative column (4 μ m, 90 Å, 250 mm × 10 mm), γ detection; elution conditions: solvent A – water with 0.1% TFA (v/v); solvent B – acetonitrile with 0.1% TFA (v/v); gradient: 0-20 min 20 to 80% of B, 20-35 min 80% solvent B; 2 ml/min.

bioconjugate were performed according to the following procedures: to the vial containing about 50 µg of lyophilized DOTA-SPE (5-11) in 300 µL of acetate buffer (pH = 5.0), about 100 µL of concentrated ⁶⁸GaCl₃ solution (80-100 MBq) from ⁶⁸Ge/⁶⁸Ga generator or about 20 µL of ¹⁷⁷LuCl₃ solution in 0.04 M HCl (10-15 MBq) were added. In the ⁶⁸Ga labelling reaction mixture pH was strictly set below 4.0. Both reactions were performed at 95°C for 30 min and 60 min, respectively, with progress HPLC verification. The radiochemical yield of the synthesized radiobioconjugates was higher than 97%.

The lipophilicity parameters for both radiobioconjugates were determined as the log P values, which are the logarithms of the partition coefficients of each molecule in a biphasic mixture of n-octanol and phosphate buffered saline (pH 7.40). Considering ligand exchange, reactions with amino acids or other strongly competing natural ligands present in human serum did not take place. In the case of stability studies in human serum, we have measured the radioactivity of precipitated matrix proteins (using ethyl alcohol) and the supernatant fractions. A few percent of ⁶⁸Ga/¹⁷⁷Lu-DOTA-SPE (5-11) molecules were shown to be bound by the biomatrix components, while the rest of the studied radiobioconjugates remained in the liquid phase in an unchanged form.

In conclusion, we have obtained novel ⁶⁸Gaand ¹⁷⁷Lu-radiobioconjugates based on peptide antagonist of NK1 receptor. Both examined molecules showed promising physicochemical properties for further consideration as potential radiopharmaceuticals for glioblastoma multiforme treat-

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ment, competitive to presently applied methods in oncology.

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PREPARATION OF MICROSPHERES OF CARBON BLACK DISPERSION IN URANYL-ASCORBATE GELS AS PRECURSORS OF URANIUM CARBIDE

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A novel technique to fabricate precursors of carbide-ceramic nuclear fuel *via* the synthesis of spherical particles (with diameters below 150 µm) of carbon black dispersed in uranyl-ascorbate gels by a combination of the complex sol-gel process (CSGP) and the double extraction process was elaborated. The application of ascorbic acid as a complexing agent in the complex sol-gel process allows a stable and uniform dispersion of carbon in a uranyl-ascorbate sol to be obtained. In the double extraction process, a dispersion of carbon black in sols is emulsified by drops in the organic phase consisting of 2-ethylhexanol-1 and 1 vol% of Primene JM-T. The microspheres are created by gelation of droplets, through simultaneous extraction of water with 2-ethylhexanol-1 and nitrate ions with Primene JM-T. The elaboration of the optimal gelation conditions requires the consideration of many issues which are described in this paper.

This paper deals with an extension of the previously described complex sol-gel process to the production of microspheres of carbon black dispersion (without dispersing agent) in uranyl-ascorbate gels which are recommended as precursors in the synthesis of a new type of fuel-like uranium carbide or uranium nitride. The obtained dispersion after carbonization to UO2-C can be converted by carbothermic reduction to UC (eventually with UO₂) and additionally by the nitriding process to UN. Medium-sized spherical particles of uranium carbide (with diameters of approximately 100 μ m) can be used in nuclear fuel technology for spherical TRISO fuel [1-4]. A flowchart of the applied combination of CSGP and the double extraction process to synthesize spherical particles of carbon black dispersed in uranyl-ascorbate gel is shown in Fig. 1.

The first step was the preparation of concentrated 1 M uranyl-ascorbate sol solutions with





Fig. 1. Flowchart detailing the preparation of uranium carbide microspheres (diameters below 150 μm) by CSGP with gelation by the double extraction processes.

molar ratio (MR) of ASC/U equal to 1, in which carbon black was dispersed with molar ratios of C/U equal to 1, 2 and 3. Carbon black was dispersed in prepared uranyl-ascorbate sol solutions by mixing for 3 h. The as-obtained dispersions have a high degree of homogeneous distribution of the components. Traditionally, the dispersability of carbon black is improved by using dispersing agents (surfactants) which is needless in the complex sol-gel process. As is shown in Fig. 2 and previous works [5], ascorbate sols perfectly wet the carbon black and the formation of agglomerates were not observed. A complexing agent, such as ascorbic acid, also aids dispersion. No crystallization of any added metallic compounds was observed.

(ADUN) with hexamethylenetetramine (HMTA) and urea [10]. The last step was the filtration and washing of the microspheres. The gelation process was carried out in a glass-enclosed reactor with very rapid stirring (maximum 1500 rpm) and a volume of 1000 mL.

The size distribution and sphericity were sensitive to the mixing speed of the organic phase when all other experimental parameters were held constant. The size distributions and sphericity of microspheres between the minimum and maximum mixing speeds were considerably different. At low mixing speeds (< 600 rpm), populations of microspheres with diameters greater than 100 μ m, but not greater than 200 μ m, were mainly obtained (82%), with sphericity (Eq Ø) equal



Fig. 2. Optical microscope photographs of a carbon dispersion in 1 M uranyl nitrate solution (A) and in 1 M uranyl-ascorbate sol solution (B).

The second step was gelation of the sols, which is crucial for obtaining the desired average size, size distribution and shape of the microspheres. The gelation of spherical particles requires partial hydrolysis by addition of ammonia solution to $pH \sim 4.5$ before precipitation. Without ammonia, spherical particles of gel cannot be obtained. With increasing pH an increase in viscosity is observed which additionally has a positive influence on droplet generation. Viscosity seems to be a crucial parameter in determining the stable emulsion droplets' diameters and size distribution.

Medium-sized spherical particles (diameters below 150 μ m) were obtained by applying the double extraction process [6] which is based on the Oak Ridge National Laboratory (ORNL) method of water extraction from sol drops dispersed in an organic solvent [7-9]. The gelation process consists of three steps. The first step was the formation of a sol emulsion in 2-ethylhexanol-1 containing surfactant SPAN-80 and Primene JM-T. SPAN-80 has a low hydrophilic-lipophilic balance (HLB) which prevents coalescence and sticking of sol droplets. The second step was the gelation of droplets by the simultaneous extractions of water, with 2-ethylhexanol-1, and nitrate ions, with Primene JM-T. The gelation process was carried out at room temperature but heated 2-ethylhexanol-1 can also be applied to the internal gelation of acid deficient uranyl nitrate to 1.092 ± 0.074 . For a given sol, higher stirring speeds (900 rpm) favour the formation of smaller diameter microspheres with good sphericity. If the mixing speed was higher than 1000 rpm, microspheres mainly with diameters below 50 µm were obtained (89%).

Dried gel microsphere dispersions were analysed using scanning electron microscopy (SEM) with a Zeiss DSM 942. A measuring optical microscope (Motic BA300) was utilized to evaluate the morphological features of microspheres such as shape, diameter and dispersity. An optical analysis



Fig. 3. SEM micrograph of spherical particles of a carbon dispersion in a uranyl-ascorbate gel.

MR	Ø _{max} [µm]	Ø _{min} [μm]	Ø _{av} [µm]	Eq Ø			
C/U = 1	143.2	33.8	103.4 ± 29.9	1.007 ± 0.007			
C/U = 2	149.1	46.6	111.2 ± 26.1	1.009 ± 0.011			
C/U = 3	169.1	49.6	119.0 ± 24.5	1.011 ± 0.011			

Table 1. Size and sphericity characterization of dried carbon black microsphere dispersions in uranyl-ascorbate gels as a function of molar ratio of C/U.

showed a spherical particle shape with good surface integrity (Fig. 3).

As shown in Table 1, the size of the microspheres is directly proportional to the molar ratio of C/U. With the increasing molar ratio of C/U, the proportion of spherical particles with a larger diameter is higher.

This is connected to the higher amount of amorphous carbon in the formed droplet dispersions in the organic solvent and with the higher viscosity. Moreover, the increase in microsphere diameter has an influence on the sphericity of the particles, but to a negligible extent.

The energy dispersive spectrometry (EDS) measurements of quality and quantity of elements in the dried gel microspheres were performed with the use of a Bruker AXS Microanalysis GmbH Berlin operated at 15 keV. The EDS analyses (Fig. 4) were performed to confirm the presence of carbon in the microspheres of gels with molar ratio of C/U equal to 1 and 3. Analyses of elements by the EDS were performed on a dried sample. The results of the EDS confirmed the presence of carbon in the uranyl-ascorbate gel and with an increasing amount of carbon, rising intensity of peaks from C is observed. Additionally, the molar ratio of C/O changes with an increase in the molar ratio of C/U in the obtained gel microspheres.

The last step of the process consists of the thermal treatment and requires some special procedures to preserve the spherical shape. In this step, the conditions should be selected to obtain the desired properties of the final product. A slow heating rate $(1-5^{\circ}C/min)$ is required to maintain a non-cracked surface of the particles during the carbonization of organic compounds and elimination of nitrates. The whole step should be carried out in an oxygen-free atmosphere to avoid oxida-



Fig. 4. EDS analysis of carbon black microsphere dispersions in uranyl-ascorbate gels with molar ratio of C/U equal to 1 and 3.

tion of the carbon. The carbonization of gels to a homogeneous mixture of $UO_{2,x}$ -C is carried out in an inert gas atmosphere containing hydrogen (95% N₂ + 5% H₂). The last step is the carbothermic reaction (1600°C) under reduced pressure to uranium carbide.

The complex sol-gel process can be successfully applied to the preparation of homogeneous amorphous carbon dispersions in sols, using complexation with ascorbic acid, of uranyl nitrate. In the CSGP, a dispersing agent is needless. It was observed that, in contrast to water or pure uranyl nitrate solutions, ascorbate sols perfectly wet the carbon black. It is necessary to point out that the preparation of microspheres of uranyl gels from uranyl nitrate by extraction of water and nitrate ions with the addition of ascorbic acid enabled the gels to be formed. The dispersion can also be gelled into medium-sized microspheres (diameter $< 150 \mu m$) by water and nitrate extraction from drops of the dispersion in sols. The size of the microspheres is directly proportional to the molar ratio of C/U. With the increasing molar ratio of C/U, the proportion of spherical particles with a larger diameter is higher. This is connected to the higher amount of amorphous carbon in the formed droplets of dispersion in an organic solvent, and therefore a smaller amount of water and nitrate is extracted during the gelation.

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RECOVERY OF URANIUM AND ACCOMPANYING METALS FROM SECONDARY RAW MATERIALS

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The development of nuclear energy in the world causes an increase in the demand for uranium. Presently, the exploitation of rich uranium ores is profitable when the cost of production of uranium is lower than 130 USD/kg U_3O_8 . According to the OECD-NEA Red Book (2016) [1], such deposits of uranium in the world are estimated at about 5.7 million tonnes of metal. An increase in research on uranium recovery from secondary resources has also been observed. In this investigation, industrial waste and by-products from the copper and phosphorous fertilizer industries, as well as phosphate rocks, were examined as sources of uranium. During the recovery of uranium from secondary sources, which are assumed to be poor quality, the economy of the process can be improved by the simultaneous extraction of accompanying metals such as rare earth elements or other valuable metals present in the processed material.

Before selecting a method of extracting uranium and other selected metals from the available secondary raw material, it is important to adequately characterize this material and determine the type and content of the metallic components of interest. The compositions of the examined materials were determined with the use of ICP-MS (inductively coupled plasma mass spectrometry) analysis. The results are shown in Table 1.



Fig. 1. The leaching efficiencies of metals from copper wastes with various oxidizing agents: CW1 – copper waste 1, CW2 – copper waste 2, CW3 – copper waste 3. Process conditions: 10% H_2SO_4 , 60°C, 2 h.

ing efficiency of 40-67%) were obtained only for copper waste 3.

Phosphogypsum is a very difficult material to leach because of its complex composition. It was found that this material can be leached using a double-stage procedure. In the first stage phosphogypsum was treated with mixture of 30% NaOH and 30% H_2O_2 and heated at 60°C. The solid residue was separated from the post-leaching solution, washed with distilled water and dried at 105°C.

Table 1. The concentration of selected metals in the examined materials: copper flotation tailings, phosphogypsum and phosphates.

Comula	Metals [ppm]								
Sample	U	Th	V	Fe	Со	Ni	Cu	Zn	La
Copper waste 1	15	5	788	nd*	558	330	27 500	1 217	21
Copper waste 2	16	5	833	nd*	589	269	25 000	987	21
Copper waste 3	5	2	55	4 750	79	31	12 225	237	8
Phosphogypsum	2	4	5	2 758	6	14	5	17	40
Syria phosphate	61	1	134	2 911	13	36	13	307	26
Morocco phosphate	117	4	250	3 880	13	69	39	332	90
Tunisia phosphate	32	5	53	3 398	14	20	5	142	64

* nd - not determined.

The efficiencies of uranium leaching from copper flotation tailings with 10% sulphuric acid were not high (Fig. 1). Satisfying results (uranium leachThe obtained solid was leached with 10% HCl in the presence of 30% H₂O₂, as an oxidizing agent, at 60°C. Uranium and rare earth elements were



Fig. 2. The leaching efficiencies of metals from phosphogypsum. Process conditions: step one – 30% NaOH, 30% H_2O_2 , 22-60°C; step 2 – 10% HCl, 30% H_2O_2 , 60°C.

extracted with quite high efficiencies – 61% for uranium and 81% for lanthanum (Fig. 2).

Phosphates were leached using hydrochloric acid, sulphuric acid and alkaline solution. The efficiencies depend strongly on the origin of the phosphate. All three phosphates, from Syria, Morocco and Tunisia, were leached with high efficiencies (40-100%) in acidic conditions; however, only the phosphate from Morocco was leachable in alkaline solution (Fig. 3). It is worth noting that phosphates from Morocco are more easily leached with an alkaline solution than with acid. An interesting



Fig. 3. The leaching efficiencies of uranium from phosphates. Process conditions: 30% H₂O₂, 22° C, 1 day.

result was obtained for the percolation leaching of Tunisia phosphate. A solution of 2.5% Na₂CO₃/ NaHCO₃ with 30% H₂O₂ was used as a lixiviant. The solution was recirculated within the phosphate-filled column. The efficiency of uranium leaching reached 19% after 2 h.

Phosphogypsum and flotation tailings from the copper industry are very difficult materials to leach because of their complexity; however, the obtained results are very promising and these studies will be continued. The uranium leaching efficiency of phosphogypsum reached 60%. Moreover, it should be noted that phosphogypsum could be an interesting and valuable source of lanthanides.

The acidic leaching of phosphates was very efficient. In some cases, the leaching efficiency reached 100%.

The isolation of uranium from post-leaching solutions can be obtained by liquid-liquid extraction [2] or ion exchange chromatography [3].

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NOVEL MAGNETIC COBALT CYANOFERRATE NANOPARTICLES AS A POTENTIAL SORBENT FOR SOLID PHASE EXTRACTION OF RADIONUCLIDES FROM AQUEOUS SAMPLES

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Numerous inorganic sorbents at present find increasing use in applied radiochemistry. The most common use is removing caesium and strontium radionuclides from cooling water in a nuclear reactor, as well as water in the storage pools of spent nuclear fuel. The main advantages of using inorganic sorbents are their chemical strength, thermal stability and radiation-chemical resistance [1, 2].

Insoluble hexacyanoferrates of the transition metals are well-known sorbents and ion exchangers [3-8]. They are hardly soluble in a wide acidity interval (*i.e.* in amounts from 10^{-6} to 10^{-12} g cm⁻³), have high mechanical strength, chemical resistance, small specific gravity, good magnetic properties and are cheap to prepare. Their good sorp-

tion properties (distribution coefficients, K_d , often exceed 10^4 cm³ g⁻¹) depend on their synthesis method as well as on their composition. Inorganic sorbents most frequently occur in the form of fine monocrystals in which the regular arrangement of atoms is cut by a series of channels and free spaces of the size corresponding to those of some ions. Typically, the material is a mixture of hexacyanoferrates containing iron in both the second and third oxidation states [9].

For the prospective use of hexacyanoferrates for the selective removal of *e.g.* Cs-137 from liquid nuclear wastes, a great number of hexacyanoferrates with different transition metals have been studied. An excellent review of the present state of knowledge was recently published by Vincent *et al.* [10].

In the recent past, magnetic nanoparticles have started to be used as sorbents for the removal of trace metals from aqueous solutions [11-15]. The attractiveness of these materials, besides excellent sorption ability and good mechanical properties, is based on their easy separation from the solution using a magnetic field (Scheme 1) [16-17]. process. The results published by Zhao *et al.* [11] for a similar magnetic sorbent, $K_2[CoFe(CN)_6]$ composite containing magnetite, show that sorption of caesium(I) reaches equilibrium within 2 h. The same contacting time for the contaminated water with MHC was applied in the present work. For each factor, magnetic $K_2[CoFe(CN)_6]$ composite with magnetite, studied by Zhao *et al.* [11], was used as the reference material. The aforemen-



Scheme 1. Schematic representation of the usage of a magnetic sorbent.

In this contribution, being a continuation of research published by Zhao *et al.* [11], we report our studies on the possibilities for the application of the cobalt hexacyanoferrate sorbent containing magnetic iron carbonyl (MHC). Iron carbonyl is a highly pure iron, prepared by the chemical decomposition of purified iron pentacarbonyl. Because of its broad applications in electronics and medicine, the material is extremely cheap and, if bought wholesale, the price is significantly lower than 1.0 USD/kg.

In order to synthesize the magnetic composite material, a co-precipitation method was used, as described by Zhao *et al.* [11]. Magnetic particles of the commercially available iron pentacarbonyl (Fe(CO)₅), solid K_4 Fe(CN)₆ and Co(NO₃)₂ were used. All studies of the MHC were performed using unfractionated sorbent.

We investigated sorption of Cs(I), Sr(II), Co(II), Eu(III) and Am(III) ions on the magnetic cobalt cyanoferrate nanoparticles (MHC) in batch experiments by analysing decontamination factors of the contaminated water as a function of the main factors important for designing the technological tioned studies by Zhao *et al.* have been extended by determining the decontamination factors for Sr(II), Co(II), Eu(III) and Am(III) radionuclides.

Figure 1A reveals that the iron carbonyl MHC completely removes caesium(I) from aqueous solution, the adsorption percentage (E_r) being 99.6 ± 0.3%. This observation corresponds well with our referent sorbent containing magnetite: for this material, E_r value is 98.6 ± 1.0%. So, the proposed MHC sorbent resembles that recommended by Zhao *et al.* [11].

The aim of the presented work was, however, checking the possibility of using the novel sorbent for the recovery of metals other than Cs(I). It was also found that the efficiency of removal of Co(II) and Am(III) is high and the respective E_r values are 99.18 \pm 2.13% and 98.4 \pm 5.27%. For the reference composite formed with magnetite, the corresponding values are 92.50 \pm 9.13% and 98.43 \pm 11.42%, respectively. However, the proposed sorbent may be applied poorly for the recovery of the other metals. It was found that europium(III), and especially strontium(II), are sorbed below expectations. While the E_r value for the



Fig. 1. Dependence of the distribution constants of Cs(I), Sr(II), Co(II), Eu(III) and Am(III) by the MHC nanocomposite with iron carbonyl on the reciprocal of their Shannon ionic radii [18] (A) and the analogue sorbent containing magnetite (B). The data are normalized to the respective values for caesium(I). Acidity of the purified solution – pH 3.5.

former metal was determined as $61.02 \pm 1.39\%$, sorption of strontium(II) appeared to be completely insufficient. The E_r value for this metal does not differ from zero by more than 5%. Results obtained for the magnetite sorbent agree within statistical error with the above values.

It is textbook knowledge that the Gibbs free energy (ΔG°) of the interaction between the investigated metal cation and the counter-ions plays an important role in the interpretation of numerous processes in aqueous solutions. Usually, the well-known Born equation may be used to describe the interactions if they are of pure electrostatic origin. When plotting the results recalculated to the distribution constant values as a function of the reciprocal of the Shannon ionic radii no continuous relationship is observed (Fig. 1). This means that even if metal sorption is governed mainly by the electrostatic cation-anion interactions, certain supplementary phenomena should be taken into account.

Furthermore, acidity of the purified contaminated solutions varied in the range from pH 1.5 to 12. Results of the metal sorption by both magnetic $K_2[CoFe(CN)_6]$ materials are presented in Fig. 2.

To check if the sorbents may be used in the purification of real solutions, we decided to compare the aforementioned results obtained for the artificial solutions with the uptake of metals from the solutions of the composition resembling the real radioactive liquid waste. In the available literature, data for this composition are rare and difficult to obtain. To achieve this objective, we have prepared a mixture of Na⁺, K⁺, Mg²⁺ and Ca²⁺ chlorides in concentrations resembling those found in solutions obtained from the National Radioactive Waste Repository, Poland. Results of the comparison are presented in Figs. 3A and 3B. It may be seen that in the case of caesium (to recall, cobalt hexacyanoferrates are dedicated to the selective sorption of this radionuclide), presence of the weighted amounts of salts does not affect sorption in the case of both magnetic components studied. For the other radionuclides, salts present in the solution successfully compete for the sorption sites of the sorbent. So, due to the co-sorption of the salts, one may observe a decrease in the uptake of the radiometals.

Radioactive liquid wastes may also contain certain chelating agents, formerly used for the cleansing of different contaminated items. So, to check



Fig. 2. Effect of pH on adsorption of Cs(I), Sr(II), Co(II), Eu(III) and Am(III) by cobalt cyanoferrate nanocomposites with iron carbonyl (A) and magnetite (B).

It can be seen that Cs(I) may be completely sorbed from both the acidic and basic solutions, meaning that the acidity of the purified solution has no significant influence on the adsorption process of Cs(I). The observation resembles that observed by Zhao *et al.* [11]. On the contrary, sorption of Sr(II) in the whole range of pH is extremely poor, as the efficiency is close to 0%.

Finally, removal of Co(II), Eu(III) and Am(III), metals with smaller radii than the aforementioned cations and with partially filled *d*- and *f*-orbitals, does not exhibit simple dependence on the initial acidity of the solution. This observation may be related to the diverse phenomena appearing for these metals in aqueous solutions. Of primary importance are changes in their speciation. the potential sorbents in processing of the low--level radioactive wastes, we tested them by sorption of the radionuclides from aqueous solutions containing a mixture of popular chelating agents. Composition of the mixture resembled that in CANDEREM (Canadian decontamination and remediation) lotion: about $2 \cdot 10^{-3}$ M of oxalic and citric acids and also Na-EDTA [19]. Then, the obtained results were compared with the data gained previously for the aqueous solution with no chelating agents. As is seen from Figs. 3C and 3D, only for certain radionuclides the E_r values do not differ from each other by more than the experimental errors. So, it may concluded that the cobalt hexacyanoferrate composites may be successfully applied for the recovery of caesium(I)



Fig. 3. Effect of the salting: (A) nanocomposite with the iron carbonyl, (B) nanosorbent containing magnetite. Plot (C) presents the effect of the chelating agents present in the solution for the nanocomposite with the iron carbonyl while (D) shows that for the nanosorbent containing the magnetite.

and americium(III), but in the processing of other radionuclides their value is rather limited.

Future applications of both materials for radioactive liquid waste management requires high radiation stability. So, the composites were treated with a radiation dose of 250 kGy and checked to see if their properties had been changed due to the radiation. In the first step, sorption of the radionuclides was studied. It was found that the E_r values are comparable within the experimental error for both pairs: raw materials and the appropriate irradiated sorbents (Fig. 4). Measurement of the zeta potential (ζ_n), which describes the electrostatic potential near the surface of the particle, was determined for both pairs of sorbents. Relatively small changes of the potential occurring upon irradiation may be interpreted as indicating stability of the materials. The already determined ζ_n potential of the K₂[CoFe(CN)₆] composite containing magnetite fluctuates around -30 mV for solutions between pH 4 and pH 10 [11]. Such a value seems to be high enough to produce a significant potential barrier preventing coagulation of the dispersed



Fig. 4. Stability of sorbent adsorbed with metals: (A) raw MHC material, (B) gamma irradiated MHC material.

nanoparticles. When the acidity of the solution increases to pH 11-12, the negative ζ_n potential reaches values of about -60 mV. Values obtained in the present work are -27 mV and -31 mV for the magnetite nanocomposite and the iron carbonyl material, respectively. Upon gamma irradiation, values of the potential increase only slightly and are -21 mV and -22 mV, respectively. Such a small difference observed for the respective pairs may be assumed as supplementary proof of the chemical stability of both sorbents.

Chemical stability of the proposed sorbents was, in turn, checked by their prolonged equilibration with water and determining the removal of cobalt and iron metals. A ten-day equilibration of sorbent containing the carbonyl iron magnetite $100 \times$ magnification reveal that the magnetite MHC is formed of more compact nanoparticles than the iron carbonyl material. The surfaces of both samples viewed with magnification of $5000 \times$ exposes, however, more pronounced differences. The sorbent containing magnetite is more uniform when compared with the material formed from iron carbonyl. Agglomerates formed on the surface of the latter material in certain areas are smooth, while in the former they are frayed. The aforementioned dissimilarities may be attributed to the different magnetic materials.

The obtained nanoparticles display large surface areas in both cases of about $32.5 \text{ m}^2/\text{g}$. Such a large surface area suggests high sorption capacity of the material.



Fig. 5. SEM image of the sorbent surface: (a) $100 \times$ magnification of sorbent made with magnetite; (b) $5000 \times$ magnification of the same material, (c) $100 \times$ magnification of sorbent made with iron carbonyl, (d) $5000 \times$ magnification of the same sorbent.

showed the content of both metals in water to be about 30,000 ng/L and 350 ng/L, respectively. When the magnetite nanocomposite was used, these values were slightly lower: about 15,000 ng/L and 250 ng/L, respectively. According to the WHO directive for drinking water quality [20], the concentration of iron should be less than 0.3 mg/L ($3 \cdot 10^5$ ng/L) and there is no recommendation for cobalt. The abovementioned results clearly show that water, purified from radionuclides, should be additionally purified from heavy metals. Among the most prospective methods, nanofiltration/ultrafiltration should be considered.

Figure 5 presents images of the particles of both studied sorbents. Photographs taken with

Diffraction patterns for the iron carbonyl MHC are shown in Fig. 6A. The main reflections are recorded for the similar angles with these for the former material and confirm the presence of the $K_2Co[Fe(CN)]_6$ structure. An additional undefined reflection at 29° most likely comes from the iron carbonyl species.

Results of the X-ray diffraction analysis of the magnetite sorbent are shown in Fig. 6B and confirm the existence of the $K_2Co[Fe(CN)]_6$ structure. One may also see reflections that probably come from other structures, *e.g.* from $CoFe_2O_4$ or $K_2CoFe(CN)_6$. Nevertheless, the main reflections marked with the line indicate that $K_2Co[Fe(CN)]_6$ is the dominant component of the sample.


Fig. 6. X-ray diffraction patterns (XRD) of (A) iron carbonyl sorbent, (B) magnetite sorbent.

According to the proposition of Sadlej-Sosnowska *et al.* [21], calculation of the Pearson's correlation coefficient (c.c.) seems to be the most powerful tool for comparing two spectra. The c.c. may range from the value of -1 to 1. A zero value implies a lack of correlation between both spectra, *i.e.* they are totally different. On the contrary, values close to ± 1 indicate a similarity between the compared spectra and, consequently, between the identities of the substances being compared. The calculated correlation coefficient for the powder XRD spectra of both cobalt hexacyanoferrate inorganic--inorganic composites revealed the value of the c.c. to be 0.715, meaning that both materials, even though similar, are not the same at all. Similarity in sorption properties of the MHC nanocomposites in conjunction with dissimilarities in the structure of both sorbents allows for the conclusion that sorption properties do not depend on the magnetic material used.

In conclusion, decontamination of radioactive wastes with the use of insoluble cobalt hexacyanoferrate nanocomposites (containing the iron hexacarbonyl or magnetite), originally dedicated to the removal of caesium(I) from aqueous solutions, was found to be effective for solutions containing caesium(I), and americium(III) radionuclides. If the concentration of the chelating agents is not too high, cobalt(II) may also be effectively removed from the solution. The sorbent seems to be sufficiently stable for practical application in the treatment of radioactive wastewaters.

Sorption properties of the transition metal hexacyanoferrate nanocomposites do not depend on the magnetic material used for the synthesis of the sorbent.

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APPLICATION OF ADVANCED MEMBRANE SYSTEMS IN NUCLEAR DESALINATION AS AN EFFECTIVE USE OF COGENERATION

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Cogeneration or combined heat and power (CHP) is a highly efficient form of energy conversion and a most efficient use of fuel, since in addition to producing electricity it allows the utilization of waste heat, which can be used for various purposes. Cogeneration in nuclear power plants brings many benefits: it saves energy by recovering waste heat that can be used for numerous industrial applications, *e.g.* district heating or cooling, production of hydrogen, desalination, *etc.* Such an approach brings environmental benefits, reducing

 CO_2 emissions and the amount of radioactive waste created. The fuel in cogeneration nuclear power plants (NPPs) is used efficiently, in consequence these facilities produce cheap energy, and as a result fossil fuels valuable for the chemical industry are saved.

The successful introduction of nuclear cogeneration into industrial practice is a task set by the International Atomic Energy Agency (IAEA) and the European Nuclear Cogeneration Industrial Initiative (NC2I), in which Poland actively participates, recognizing the benefits of this approach. Commencing the nuclear programme in Poland gives an opportunity to develop completely new designs and processes, leading to the achievement of maximum reactor efficiency by the effective use of the energy and heat generated in the fission process.

One of the possible ways to simultaneously increase reactor efficiency and the safety of nuclear power plants is to furnish the future NPP with advanced desalination systems, which could be helpful in regular plant operation and in emergency situations. Such industrial application of congeneration, both in the case of energy production in pressurized water reactors (PWRs) and in the development of high temperature reactors (HTRs), increases the safety of nuclear systems.

Intensive development of desalination technologies around the world has resulted in a significant decrease in the cost of the water that is produced by desalination plants. Traditional distillation processes are being replaced by novel, more economic methods. In recent years, membrane processes have become very attractive methods of desalination. With rapid progress in the production of cheap, robust membrane materials, reverse osmosis (RO) can compete with energy-intensive distillation techniques. These membrane-based processes offer numerous advantages over the conventional evaporation and distillation technologies used for desalination. They are characterized by a lower energy to product ratio because the energy supplied to membrane installations is consumed only for pumping liquid through the system. Since the primary equipment in such installations is membrane modules and pumps, which are easy to replace, they need less maintenance than evaporators. The apparatus does not have moving parts; therefore it is reliable and easy to operate. Membrane devices use less expensive construction materials, since the processes operate at room temperature, reducing corrosion. Low process temperatures also decrease scaling, which is a common problem with evaporators.

When talking about the benefits of using membrane processes, environmental issues cannot be ignored. The emissions released by fuel-fired evaporation and distillation processes are eliminated in such cases.

In recent years, reverse osmosis has become a common desalination technique. According to the International Desalination Association, in 2011 it accounted for 66% of the installed capacity of all desalination systems. Membrane distillation (MD), however, originally developed for seawater desalination [1, 2], has not yet found full implementation in this field. MD is a separation process involving a porous hydrophobic membrane, which is the only contact surface of two phases remaining at different temperatures. The driving force of MD is a temperature gradient across the membrane that results in a vapour pressure difference.

Apart from desalination, many other applications of MD have been considered, such as wastewater treatment [3], the treatment of the water produced in oilfields [4], groundwater purification [5], milk, whey and lactose concentration, as well as the concentration of juices [6]. At the Institute of Nuclear Chemistry and Technology (INCT), membrane distillation was developed both for water purification [7] and radioactive waste processing [8-11]. Extensive research conducted with the use of different experimental systems confirmed the usefulness of membrane distillation for



Fig. 1. The module with capillary porous membranes used in MD.

liquid radioactive waste treatment. Several membrane configurations were tested, including the most applicable capillary modules (Fig. 1). The experiments have demonstrated that in addition to the desalination capacity of membranes made of hydrophobic polymers (polytetrafluoroethylene – PTFE, polypropylene – PP), their radionuclide retention was highly effective. In the MD process, radionuclides were concentrated in the retentate, and the distillate was completely purified water, with an activity at the natural background level (Figs. 2 and 3).



Fig. 2. Concentration of ¹³⁷Cs in aqueous solution.

It has been revealed by numerous experiments and analyses that the MD process could be used in a nuclear power plant in the systems that allow on-site processing of low-activity liquid waste, while also producing clean water for the needs of the power plant. The feed water for an MD plant can be seawater or water from any other suitable source.

An example scheme of a power plant combining the production of electricity with water desalination is shown in Fig. 4. It generates electricity for the needs of the electric grid in a system

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Fig. 3. Concentration of ⁸⁵Sr in aqueous solution.

consisting of a generator connected to a highpressure turbine. The low-pressure steam from the turbine transfers the heat to the water in the emergency they can facilitate the handling of any waste generated.

Both RO and MD are usable methods for water and wastewater treatment. They can be combined with nuclear reactors and used in power plants for various purposes: production of clean water and deionized water, as well as for the treatment of liquid radioactive waste. Membrane distillation that can be driven by the heat from the secondary cooling circuit seems economic for conventional power plants and in nuclear power facilities. This method is advantageous because it has the lowest ratio of energy input to the amount of produced water, high salt retention and allows easy processing and equipment servicing. The MD process can use waste heat because it works at low temperatures.



Fig. 4. Membrane distillation process applied to nuclear desalination.

condenser, which heats the seawater in the intermediate heat exchanger. This system constitutes an additional loop separating the nuclear reactor from the desalination plant and securing the desalinated stream against any radioactive impurities coming from the nuclear reactor.

A more economical desalination process associated with a nuclear reactor could be the MD/RO hybrid process, which allows for the better use of the heat generated by nuclear power plants and the recovery of energy. An additional RO unit powered by electricity could use water pre-heated with waste heat, resulting in higher product yields.

It seems that membrane units could be a very important part of the water management system of an NPP. Low temperature systems, such as RO and MD, can be used for the treatment of lowlevel radioactive effluents created in the power plant and to produce pure water for the different needs of the NPP. Such integrated water management systems can enhance the safety of NPPs, allowing them to work seamlessly, and in times of The use of membrane techniques for radioactive waste processing gives various water recycling possibilities, decreasing the impact of an NPP on the environment.

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CENTRE FOR RADIOBIOLOGY AND BIOLOGICAL DOSIMETRY

Studies carried out in 2017 focused on the implementation of new biodosimetric tools that have been developed in the frame of the strategic research project "Technologies supporting development of safe nuclear power engineering" from the National Centre for Research and Development (SP/J/6/143 339/11). Accreditation No 1577 for the retrospective radiation dose estimation by dicentric chromosome measurements in peripheral blood lymphocytes given to the Laboratory for Biological Dosimetry by the Polish Centre for Accreditation in 2016 was sustained for next two years. The Centre participated in the interlaboratory comparison of methods in biological dosimetry, organized by the operational network established in the frame of Coordination Action project RENEB founded within the 7th EU Framework Programme EURATOM – Fission. In addition, new, high throughput methods for biological dosimetry, based on gene expression analysis are being developed.

An important research topic during the last few years has been the nanotoxicology and nanomedicine. Mechanism of toxicity of silver nanoparticles was studied in rat model in respect to their influence on brain functions, including long- and short-term memory. The Centre has also two ongoing projects on radium bearing nanozeolite radiopharmaceuticals against prostate and breast cancer.



STUDIES OF THE NITROSYLATION REACTION OF A PHOSPHINE COMPLEX MODELLING THE IRON CENTRE OF PROTEINS FROM THE CUPIN FAMILY

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Dinitrosyl iron complexes (DNIC) with a general formula $Fe(NO)_2(L)_2$ have been observed in many organisms over a wide spectrum of physiological conditions. The composition of these complexes vary, as they are formed from different ligands containing imidazole or sulfhydryl groups [1, 2].

Pirin is a recently identified eukaryotic iron centre-containing protein from the cupin family, which is involved in transcriptional activation and apoptosis. The homologues of pirin are highly conserved in both prokaryotic and eukaryotic cells, but their function remains poorly understood [3]. Both bacterial and human pirins have quercetinase enzyme activity, which is inhibited by the addition of typical inhibitors of quercetin 2,3-dioxygenase. The pirin iron centre is based on a system of three histidinyl ligands, the so-called trihistidinyl facial triad (3-His facial triad) [3].

The symmetry of the metallic centre, which is referred to as the facial triad, is typical of the enzymes in the cupin family; the ligands forming the "flat" coordinating environment can be three histidine imidazole rings or two histidines and one carboxylic group [4, 5]. A number of papers have modelled the coordination system of 3-His facial triads typical of non-haem iron dioxygenases using scorpionate ligands, *e.g.* substituted tris(pyrazolyl)methane [6] or tris(imidazolyl)phosphine [7, 8]. Imidazole moieties in these types of tridentate ligands can be bound at C2 [9] or at a C4 with a blocked NH nitrogen [7, 10]. Studies analysing structures of this type are aimed at developing methods of bioinspired catalysis.

Literature data indicate that nitric oxide can interact with the iron centre of proteins, resulting in the disintegration of this centre and a change in the proteins' enzymatic activities [11]. Nitric oxide is often used as an unreactive analogue of diatomic oxygen in structural studies of metallic enzyme centres. Another advantage is its paramagnetic character, which allows researchers to identify and determine the symmetry of its complexes with transition metals, such as iron(II) [2], ruthenium(II) [12], copper(I) [13], etc. Therefore, obtaining synthetic complexes is very useful for modelling the histidinyl iron centre with nitric oxide. This will help in determining a number of steric parameters and the distribution of electron charge density in similar centres.

Synthesis of [Fe(4-TIP^{ph}) (OAc) (MeOH) |BPh₄

The synthesis was performed according to the modified method published in [7]. In a 5 mL round bottom flask equipped with a stopper and magnetic stirrer, $Fe(OAc)_2$ (48.5 mg), 4-TipPh (128 mg) and MeOH (1.3 mL) were placed. The mixture was stirred for 30 min at low speed under a nitrogen atmosphere. The mixture was then filtered into a second 5 mL flask, a Pasteur pipette packed with cotton wool being used for the filtration. To the

clear filtrate (which was a light brown colour), NaBPh₄ (96.5 mg in 0.2 mL MeOH) was added dropwise; thereupon a thick precipitate formed. The flask was left on the stirrer for 1 h and then transferred to the refrigerator. After 24 h the solution was decanted from the crystals, and the product was dried under vacuum for 5 h. The obtained dry product had a brown shade (complex 1).

Recrystallization

Twice the amount of MeOH was used relative to the total amount of dry product obtained from the reaction; this mixture was stirred for 3 h and then allowed to stand for 24 h in the refrigerator. Decantation and drying gave a light yellow powder. Despite attempts to recrystallize it from various mixtures of methanol and acetonitrile, it was not possible to obtain crystals of crystallographic quality. Spectroscopic, IR and UV-VIS measurements were made.

Nitrosylation of complex 1

NO was bubbled through $600 \ \mu L$ of a saturated solution of complex 1 in acetonitrile under anaerobic conditions. The mixture turned a brown-green colour. After 10 min the reaction was completed. The mixture was allowed to stand for the next 2 h in order to allow its complete saturation with NO.



Scheme 1. Nitrosylation of 1.

The resulting complex **2** was vacuum-dried (dark green complex) and used for analysis (Scheme 1). **Examination of the structure of complex 2**

EPR spectra were measured at 100-294 K, a modulation amplitude of 3 G and a microwave power of 1 mW. The spectral parameters were determined by fitting a curve in EPRsim32 [14].



Fig. 1. The EPR signal of **2**.

The obtained complex **2**, in contrast to the initial complex **1**, was characterized by an EPR signal in the range characteristic for nitrosyl iron complexes. The shape of the signal indicated the



Fig. 2. The IR spectrum of **1** compared to that of **2**. The band at 500 cm⁻¹ in the spectrum of **1**, disappearing for **2**, can be assigned to the v(Fe-O) between the iron and the leaving acetate ligand. New v(NO) vibrations at 1749 cm⁻¹ and 1821 cm⁻¹ can be seen for **2**; the weak bands at 447 cm⁻¹ and 585 cm⁻¹ can be attributed to (Fe-N) vibrations [15, 16].

anisotropic symmetry of the paramagnetic centre. The g-values were: 2.0655, 2.0396, 2.0130 (Fig. 1).

The absorption spectra of the compounds were recorded at room temperature with a Bruker Equinox 55 FT-IR spectrometer. Spectra in the range 400-4000 cm⁻¹ at a resolution of 1 cm⁻¹ were recorded using KBr pellets, and in the range 200-600 cm⁻¹ at a resolution of 4 cm⁻¹ using CsI pellets.

The two bands at 1749 cm⁻¹ and 1821 cm⁻¹ that appear in the spectrum of the nitrosylated 4-TipPhFe(NO)₂ (Fig. 2) were assigned to the two stretching vibrations of the non-equivalent -NO groups. The non-equivalence of the two stretching vibrations [15], along with a considerable difference in band intensity [16], are typical for most of the dinitrosyl iron complexes. The disappearing band at 500 cm⁻¹ can be assigned to the v(Fe-O) between the iron and the leaving acetate ligand, in accordance with other reported stretching bands of that type [17-19]. In turn, very weak bands at 448 cm⁻¹ and 585 cm⁻¹ can be attributed to the stretching and bending vibrations of the Fe-N bond within the Fe-NO moiety [20]. Conclusions

The obtained 3-His facial triad-mimicking complex was nitrosylated to form a paramagnetic iron(II) dinitrosyl complex. Spectroscopic spectra confirmed the anisotropic nature of the paramagnetic centre and indicated the non-equivalence of the two nitrosyl ligands. The obtained spectroscopic data can be used to analyse the nitrosylation reaction of 3-His facial triad iron centres.

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IMPACT OF NANOPARTICLES ON TOXIC PROPERTIES OF OXIDIZED LOW DENSITY LIPOPROTEIN

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Nanoparticles (NPs) and nanomaterials are defined as substances with at least one dimension less than 100 nm. Due to their high surface area and small volume, coupled to other physicochemical properties, NPs possess properties substantially different from bulk materials of the same composition. The increasing use of nanomaterials of various physicochemical properties in different industrial applications has greatly enhanced human exposure to NPs. Thus, it seems necessary to duly investigate the potential interactions of NPs with living organisms in order to identify potential threats that NPs may pose for human health and to reliably estimate the risk of their use [1, 2].

The aim of the present study was to investigate the combined toxicity of NPs and oxidized low-density lipoprotein (oxLDL) on HepG2 cells. OxLDL has been shown to play a major role in atherosclerosis [3]. Macrophages in the arterial wall are able to accumulate oxLDL, forming foam cells. OxLDL increases the activity of the foam cells, while also stimulating the replication of macrophages and their migration into the atherosclerotic plaque. OxLDL is also a toxic and proinflammatory agent [4, 5].

OxLDL is eliminated from the blood stream by hepatocytes in the liver by means of the oxLDL--specific receptor CD36. However, NPs can absorb proteins forming an NP-protein corona, a process that may change the conformation of the adsorbed protein and its biological properties [6]. Thus, in the context of the increasing exposure of humans to NPs, it seems interesting to examine the interaction of NPs and oxLDL in terms of their potential toxicity to human hepatocytes. In this study we examined the impact of selected NPs on the toxic properties of oxLDL to HepG2 cells. We used two types of NPs (approximately 20 nm in size) widely used in industry:

- silver nanoparticles (AgNPs), which are the most commonly used in commercial products. Because of their well-known antimicrobial properties, AgNPs are present in many consumer goods and are able to penetrate the human body *via* multiple paths [7, 8].
- titanium dioxide nanoparticles (TiO₂NPs), which are widely used in industrial and consumer products due to their strong catalytic activity (such as antibiotics and antifungal agents, aerospace materials or optical filters) [9, 10].

Experiments were performed on human hepatoma HepG2 cells purchased from the American Type Culture Collection (ATCC, Rockville, MD, USA). HepG2 cells were cultured in DMEM high glucose (Gibco, Thermo Fischer Scientific, USA) supplemented with 10% fetal calf serum (FCS, Biological Industries, Israel). The cells were incubated in a 5% CO_2 atmosphere at 37°C. To assess the effect of NPs on oxLDL toxicity, an MTT assay was used. Cells were seeded in 96-well microplates (TPP, Switzerland) at a density of $1 \cdot 10^4$ cells/well in 100 µL of culture medium. Twenty four hours after cell seeding, the cells were treated with AgNPs (10 μ g/mL, 25 μ g/mL) or TiO₂NPs (10 µg/mL, 50 µg/mL) for 2 h. The medium containing the nanoparticles was then removed, and the cells were washed with PBS. The oxLDL solution (50 μ g/mL) was added to the culture medium with 1% FCS; the cells were incubated for 24 h in the resulting medium. The oxLDL-containing

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medium was then removed, the cells were washed with 150 μ L/well of PBS and incubated for 3 h with 100 μ L/well of MTT solution (3 mg/mL) at 37°C. After this time, the dye solution was reNPs can potentially be used in the future in the treatment of atherosclerosis, which is one of the main causes of death in Poland and other industrialized countries.



Fig. 1. Metabolic activity of HepG2 cells treated with the mixture of AgNPs (A) or TiO₂NPs (B) and oxLDL. The cells were treated with AgNPs (10 μ g/mL or 25 μ g/mL) or TiO₂NPs (10 μ g/mL or 50 μ g/mL) for 2 h at 37°C and then with oxLDL (50 μ g/mL) for another 24 h at 37°C. Data are expressed as a percentage of the control value ±SD from three independent experiments.

moved, and the remaining insoluble formazan crystals were dissolved in 100 μ L/well of DMSO. The absorbance was measured at 570 nm in an Infinite M200 plate reader spectrophotometer (Te-can, Austria). At least three independent experiments in six technical replicates were conducted for each experimental point. Significant differences between the treated samples and the control sample were evaluated by a Student's t-test using GraphPad Prism 5.0 software (GraphPad Software Inc., USA).

As shown in Fig. 1A, pretreatment with AgNPs caused a significant decrease in the toxicity of oxLDL. Treatment with AgNPs (25 µg/mL) prior to the treatment with oxLDL (50 μ g/mL) reduced the metabolic activity (viability) of the HepG2 cells to 84% of the control value, while incubation of the cells with oxLDL alone reduced their metabolic activity to 52% of the control value. As presented in Fig. 1B, preincubation with TiO₂NPs prior oxLDL also resulted in a significant reduction of the toxicity of oxLDL. Treatment of the cells with TiO₂NPs (50 μ g/mL) and oxLDL (50 μ g/mL) significantly decreased the metabolic activity of HepG2 cells to 82% of the control value, in comparison to incubation of the cells with oxLDL alone, which reduced their metabolic activity to 52% of the control value.

To conclude, the results of this study clearly show that NPs significantly altered the toxicity of oxLDL towards HepG2 cells. Therefore, a systematic study of the effects of NPs on the uptake and biological properties of oxLDL is needed to provide more information about the nature and mechanisms of the interactions between NPs and oxLDL. The study will help to determine whether This work was supported by the statutory grant from the Ministry of Science and Higher Education to the INCT (020-606).

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LABORATORY OF NUCLEAR ANALYTICAL METHODS

The research programme of the Laboratory of Nuclear Analytical Methods is focused on the development of nuclear and nuclear-related analytical methods for application in nuclear chemical engineering, radiobiology and environmental problems associated with the use of nuclear power and other specific fields of high technology. New procedures of chemical analysis for various types of materials are also being developed. The main areas of activity of the Laboratory include inorganic trace analysis, as well as analytical and radiochemical separation methods. The Laboratory cooperates with the centres and laboratories of the INCT and provides analytical services for them and for the outside institutions. The Laboratory produces certified reference materials (CRMs) for the purpose of inorganic trace analysis and provides proficiency testing schemes on determination of radionuclides and trace elements in food and environmental samples.

The main analytical techniques employed in the Laboratory comprise: neutron activation analysis with the use of a nuclear reactor (instrumental and radiochemical modes), inductively coupled plasma mass spectrometry (together with laser ablation and HPLC), atomic absorption spectrometry, HPLC including ion chromatography, as well as gamma-ray spectrometry and alpha- and beta-ray counting.

In 2017, the Laboratory of Nuclear Analytical Methods conducted a proficiency test (PT) regarding the determination of ³H, ²²⁶Ra, ²⁴¹Am and ²³⁹Pu contents in waters, food and environmental samples. PT was provided at the request of the National Atomic Energy Agency (PAA), Poland. Nine laboratories took part in the PT, including six laboratories forming a radiation monitoring network in Poland (at the request of the PAA) and three other laboratories. The proficiency tests were provided in accordance with the requirements of ISO/IEC 17043:2010, ISO 13528:2015 and the IUPAC International Harmonized Protocol (2006).

In 2017, the Food and Environmental Laboratory (FEL) provided services to municipal and private customers. The Laboratory specializes in the determination of radionuclides in waters and foodstuffs, as well as in environmental samples. Applied analysis methods have been approved by the State Sanitary Inspection.



APPLICATION OF IONIZING RADIATION FOR DECOMPOSITION OF BISPHENOL A IN ENVIRONMENTAL SAMPLES

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Bisphenol A (BPA) (Fig.1) is a well-known hazardous endocrine disruptor that exhibits hormone-like properties. It has also been found that BPA shows mutagenic effects and acute toxicity to various organisms [1].



Fig. 1. Structure of bisphenol A (4,4'-(propane-2,2-diyl) diphenol).

It is widely used in many polymers, epoxy resins and paper products. The worldwide annual production of BPA is estimated at about 3 million tons, and is still increasing due to the strong demand for polymers and epoxy resins [2, 3]. BPA is water soluble (about 300 mg/L), and therefore BPA-containing waste can readily release BPA into the aquatic environment. It has already been proved that BPA is ubiquitously distributed in the natural environment and surface waters [4].

The aim of these studies was further investigation of the decomposition of BPA, and the formation of by-products, during the application of ionizing radiation for the treatment of its solution in deionized water, as well as in real samples of river water. Earlier results obtained from studies on the application of ionizing radiation to BPA for environmental purposes have been promising [5-9].

Generally, it is known that BPA's percentage decomposition increases with an increase in the absorbed dose, and decreases with increasing initial concentration, for both gamma and electron beam (EB) irradiation. It was also found that for aerated solutions containing BPA at an initial concentration of 50 mg/L and pH 4.0, the chemical oxygen demand (COD) was decreased after gamma irradiation by 40% for an absorbed dose of 600 Gy, and by 60% for an absorbed dose of 1.0 kGy [6]. Greater BPA removal was observed, however, in more acidic conditions [8]. It means that BPA removal by radiolytic processes was mainly controlled by OH radical-induced oxidation. The second-order reaction rate constants with e_{aq}^{-} and 'OH were determined when EB irradiation was applied to decompose BPA [7]. Their values were found to be $1.85\cdot 10^{10}~M^{-1}~s^{-1}$ for BPA's reaction with 'OH, and $1.8\cdot 10^9~M^{\text{--}1}\,\text{s}^{\text{--}1}$ for its reaction with e_{aq} . The rate constant of BPA's reaction with 'OH is about ten times higher than that with e_{aq}^{-} , which indicates that although both •OH and e_{aq}^{-} can react with BPA, •OH radicals are more efficient reagents for the radiolytic decomposition of BPA.

In the conducted experiments, aqueous solutions of BPA were gamma-irradiated using a ⁶⁰Co source Gamma Chamber 5000 with a dose rate 3.85 kGy/h. The samples were irradiated in closed 10 mL conical glass flasks, and dosimetry was carried out with Fricke solution. The monitoring of the decomposition of BPA and the identification of its products were carried out with the use of liquid chromatography-mass spectrometry (LC-MS) analysis, employing an Agilent 1290 Infinity HPLC instrument with an Agilent 6530 Q-TOF mass spectrometer with electrospray ionization (ESI). HPLC separations were carried out using an Agilent Bonus-RP column $(2.1 \times 50 \text{ mm})$ 1.8 µm) and gradient elution, with eluent A being 5 mM ammonium acetate and eluent B methanol; the flow rate was 0.4 mL/min, the concentration of B was increased from 0 to 95% between 0 and 8 min, and at 10 min the concentration of A was 100%. The MS conditions were as follows: dual AJS ESI ion polarity - negative, drying gas - 8 L/min, sheath gas temperature - 200°C, sheath gas flow – 11 L/min, fragmentor voltage – 110 V, skimmer voltage - 65 V, and examined mass range - 100-1700 m/z. The solid-phase preconcentration of the analytes was carried out with Oasis HLB columns (Waters), which were conditioned by flushing with 3 mL methanol and 3 mL water at 5 mL/min; 500 mL of sample was then loaded. The bed was next rinsed with 1 mL 5% methanol, and the analytes were eluted with 2 mL methanol.

Generally, the radiolysis of water in deaerated solutions leads to the formation of various products according to the scheme [10]:

 $\begin{array}{l} H_{2}O \xrightarrow{\gamma \text{ or EB}} \bullet OH \ (0.28), \ e^{-}_{aq} \ (0.28), \ \bullet H \ (0.062), \\ H_{2} \ (0.047), \ H_{2}O_{2} \ (0.073), \ H_{3}O^{+} \ (0.28) \end{array}$

In air-saturated solutions, however, the reactive species are the 'OH radicals, as hydrated electrons and 'H are scavenged by dissolved oxygen. The numbers in parentheses in the above equation indicate the radiation chemical yields (G-values) expressed in μ M J⁻¹ units. The G-values also correspond to the number of molecules consumed or produced in a given reaction per 100 eV of energy absorbed as a result of radiation.



Fig. 2. The yield of BPA removal in gamma irradiation process from 10 mg/L BPA aerated solution of pH 7.0.



Fig. 3. Extracted ion chromatograms (EIC) obtained for 10 mg/L BPA aerated solution of pH 7.0 after gamma irradiation with absorbed dose 1.5 kGy. For identification of measured species, see Table 1.

After gamma irradiation of aqueous aerated solutions of BPA at an initial concentration of 10 mg/L, chromatographic analysis with quadrupole time of flight (Q-TOF) detection was carried out for solutions irradiated with different doses. It was found that practically complete decomposition of this initial amount of BPA was achieved after the absorption of a 0.5 kGy irradiation dose (Fig. 2). These results are in agreement with our earlier studies [5]. The decomposition products of BPA at dose range up to 1 kGy were identified using LC-Q-TOF measurements (Fig. 3). The available literature data on radiolytic degradation of BPA indicate that the main degradation products are monohydroxylated BPA and dihydroxylated BPA

[6, 8]. It was postulated that the main chemical process in the decomposition of BPA was hydroxylation, which results in the formation of different aromatic hydroxylated derivatives. The formation of these species can be explained based on the stabilization of the aromatic radical generated after the loss of an **'H** promoted by **'HO**. Peller *et al.* [9] suggested that subsequent formation of the hydroxylated product takes place only in pure water or very highly treated wastewater. In our experiment, monohydroxylated BPA (m/z = 243.1026) and a small amount of dihydroxylated BPA (m/z = 259.0976) were also found after irradiation with the 1 kGy dose. Additionally, the following species were identified in solution after irradia



Fig. 4. Mass spectra of products identified in 10 mg/L BPA solution of pH 7.0 after gamma irradiation with absorbed dose 0.75 kGy. For identification of measured species with m/z values indicated in circles, see Table 1.

Compound	Molecular structure	m/z (exp.)	m/z (calc.)	Error [ppm]	Found in another AOPs [Reference]
BPA	но-СН ₃ Он	227.1071	227.1078	0.7	photolytic [11] ozone/persulphate [12] H ₂ O ₂ /persulphate [13]
p-Hydroxyacetophenone	он	135.0448	135.0451	0.3	H ₂ O ₂ /persulphate [13] UV-C/peroxymonosulphate [14]
4-(1-Hydroxypropan-2-yl) catechol		151.0760	151.0764	0.4	ozone/persulphate [12] photo-Fenton [15]
Quinone of monoxydroxylated BPA		241.0875	241.0870	-0.5	photolytic [11] H ₂ O ₂ /persulphate [13] TiO ₂ photocatalytic/photolytic [16] UV-C/peroxymonosulphate [14]
Monohydroxylated BPA	но-СН3 ОН	243.1028	243.1026	-0.2	gamma irradiation [6, 8] photolytic [11] H ₂ O ₂ /persulphate [13] UV-C/peroxymonosulphate [14]
Dihydroxylated BPA	HO CH ₃ OH	259.0974	259.0976	0.2	gamma irradiation [6, 8] photolytic [11]
Quinone of dihydroxylated BPA	$O \longrightarrow \begin{array}{c} CH_3 \\ CH_3 $	257.0813	257.0819	0.6	H ₂ O ₂ /persulphate [13] UV-C/peroxymonosulphate [16]
Coupling product		319.1334	319.1339	0.5	UV-C/peroxymonosulphate [16]

Table 1. Products of radiolytic decomposition of BPA identified by LC/MS with indicated m/z values measured (exp.) and theoretical (calc.).

tion: a quinone of monohydroxylated BPA (m/z =241.0870), p-hydroxyacetophenone (m/z = 135.0451), 4-(1-hydroxypropan-2-yl)catechol (m/z = 151.0764), a quinone of dihydroxylated BPA (m/z = 257.0819) and also a coupling product with m/z = 319.1339 (Figs. 3 and 4). The concentration of these compounds decreased with an increase in the absorbed dose. Other compounds identified by us (Table 1) were previously found as products of other advanced oxidation processes (AOPs), such as photodegradation [11], an ozone/ persulphate process [12] or photooxidation with hydrogen peroxide and sodium persulphate [13]. The main route of BPA decomposition in these processes was also postulated to be hydroxylation followed by dehydration, coupling and ring-opening reactions.

The radiolytic decomposition of BPA was also investigated in real environmental matrices made up of river water from various regions of Poland. The pH values and the content of organic compounds, expressed by total organic carbon (TOC) values, for the examined samples are presented in Table 2. The 250 mL river water samples used for irradiation were spiked with 1.0 μ g/L (4.4 pM) BPA prior to irradiation with doses up to 100 Gy. The irradiated samples were analysed, employing the solid-phase extraction pre-treatment that had been developed earlier [5]. As shown in Fig. 5, the dose needed for practically complete decomposition of 1 μ g/L BPA in deionized water was 30 Gy. The effect of the matrices of river water on the decomposition yield, in spite of their low loading with organic substances as shown by TOC values,

Table 2. Characteristics of river water samples used as natural matrices for radiolytic decomposition of BPA.

River (place of sampling)	pН	Total organic carbon [mg/L]
Vistula (Cracow)	7.82	5.43
Odra (Opole)	7.64	5.26
Brda (Bydgoszcz)	7.66	4.97

was quite substantial. For samples of water from the Vistula and Brda rivers, about 90% decomposition of 1 μ g/L BPA required an absorbed dose of 100 Gy, while in the case of samples from the

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Fig. 5. The yield of radiolytic decomposition of BPA at initial level 1 μ g/L (4.4 pM) in aerated distilled water and in spiked river water.

Odra river, for an absorbed dose of 100 Gy about 70% decomposition was observed. These data indicate a quite substantial scavenging effect of other matrix components or other organic pollutants present in treated river water samples. In Table 3 there are listed G-values calculated for the smallest applied dose and the values of rate constants for pseudo-first order decomposition reactions of BPA under the applied conditions for

Table 3. The values of pseudo-first order reaction rate constants, k, and G-values calculated from experimental data obtained from gamma irradiation of 1 μ g/L BPA solution in deionized water and in river water samples spiked with 1 μ g/L BPA. G-values were calculated for the smallest applied dose – 10 kGy.

Sample	G-value [µM J ⁻¹]	k [s ⁻¹]
Solution in deionized water	0.00033	0.15
Vistula river	0.00017	0.40
Odra river	0.00010	0.23
Brda river	0.00024	0.40

radiolytic decomposition of samples containing 1.0 μ g/L BPA. The obtained pseudo-first order rate constant for radiolytic decomposition of BPA indicates that it is a much faster process compared to many other investigated AOPs. For instance, for photolytic decomposition the corresponding rate constants were 0.010 min⁻¹ [11] and 0.0186 min⁻¹ [14], for photolysis carried out in the presence of persulphate – 0.0118 min⁻¹ [13], with persulphate and ozone – 0.0149 min⁻¹ [12], or with peroxymonosulphate – 0.025 min⁻¹ [14], while for photocatalytic processes with TiO₂ – 0.032 min⁻¹ [11] and 0.0973 min⁻¹ [16], and then for the photo-Fenton process – 0.0087 s⁻¹ [15].

Compared to many other environmental pollutants, the decomposition of which has been examined with the use of ionizing radiation [17], BPA can be very efficiently and practically removed from aerated natural waters due to its high reactivity with 'OH radicals, without any additional chemical adjustment of the treated samples.

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PROFICIENCY TEST ON THE DETERMINATION OF ACTIVITY CONCENTRATIONS OF ²²⁶Ra, ²⁴¹Am, ²³⁹Pu AND ³H IN FOOD AND ENVIRONMENTAL SAMPLES

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The participation of laboratories in interlaboratory comparisons (ILCs) and proficiency tests (PTs) is a unique way to demonstrate the laboratories' abilities to conduct specific measurements in an objective and externally comparable manner. The determination of radioactive isotopes in various food and environmental samples by the radiochemical laboratories forming the radiation monitoring network in Poland is required by a regulation of the Polish government [1]. Following a request by the President of the National Atomic Energy Agency (PAA), in 2017 the Institute of Nuclear Chemistry and Technology (INCT) con-ducted PT on the determination of ²²⁶Ra, ²⁴¹Am, ²³⁹Pu and ³H in drinking water, ²⁴¹Am and ²³⁹Pu in wheat flour and ²³⁹Pu in soil. The PT was carried out according to ISO/IEC 17043:2010 [2], the **IUPAC** International Harmonized Protocol (2006) [3] and ISO 13528:2015 [4].

Test materials

Test materials and activity concentrations of radionuclides requested by the PAA are shown in Table 1.

Six laboratories from the radiation monitoring network in Poland and three laboratories from outside the network took part in the PT. The activity of raw material candidates for test materials was measured, and materials of appropriate low initial activity concentrations of the aforementioned radionuclides were chosen (Table 1). The test materials were prepared by spiking the chosen raw materials with a certified standard solution of only one radionuclide. All the water test materials and the wheat flour spiked with ²⁴¹Am were prepared in this way, ensuring their homogeneity. The wheat flour and soil samples spiked with ²³⁹Pu were not homogeneous, being prepared individually for each participant. In these cases, work-

Table 1. Test materials to be prepared.

Test material	Nuclide	Activity concentration requested by the PAA
Drinking water 1H1/17	³ H	0.5-5 Bq/L
Drinking water 1H2/17	³ H	15-30 Bq/L
Drinking water 1R1/17	²²⁶ Ra	0.5-1 Bq/L
Drinking water 1R2/17	²²⁶ Ra	2-3 Bq/L
Drinking water 1P1/17	²³⁹ Pu	1-2 Bq/L
Drinking water 1A1/17	²⁴¹ Am	0.5-2 Bq/L
Wheat flour 2A1/17	²⁴¹ Am	5-10 Bq/kg
Wheat flour 2P1/17	²³⁹ Pu	0.5-1 Bq/kg
Sand 3P1/17	²³⁹ Pu	0.5-1 Bq/kg

ing solutions of ²³⁹Pu were weighed and added to samples of raw materials. Participants were informed that the test samples containing ²³⁹Pu were not homogeneous and the total mass of the sample should be taken to analysis. The details of the preparation of test materials have been published earlier [5, 6].

Reference values of the activity concentrations of ³H, ²²⁶Ra, ²⁴¹Am and ²³⁹ Pu in drinking water, wheat flour and soil were determined from the formulation details and from the certified activity values given in the certificates, considering the successive dilution steps, the mass of the spiking solutions and the mass of the test material being spiked [3, 4]. The expanded combined standard uncertainty U (k = 2, which corresponds to 95% probability) was evaluated in accordance with GUM [7]. The following sources of uncertainty were taken into account: the uncertainty of

Table 2. Reference values of activity concentration of 3 H, 226 Ra, 241 Am and 239 Pu in drinking water and 241 Am in wheat flour.

Test material	Nuclide	$X_{ref} \pm U \ (k = 2),$ reference date
Drinking water 1H1/17	³ H	4.11 ± 0.25 Bq/L on 23 June 2017
Drinking water 1H2/17	³ H	21.02 ± 0.60 Bq/L on 23 January 2017
Drinking water 1R1/17	²²⁶ Ra	0.837 ± 0.041 Bq/L on 5 February 1998
Drinking water 1R2/17	²²⁶ Ra	2.48 ± 0.12 Bq/L on 5 February 1998
Drinking water 1P1/17	²³⁹ Pu	1.01 ± 0.10 Bq/L on 1 May 2015
Drinking water 1A1/17	²⁴¹ Am	1.640 ± 0.049 Bq/L on 11 June 2001
Wheat flour 2A1/17	²⁴¹ Am	9.260 ± 0.444 Bq/kg on 11 June 2001

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Wheat flour 2P1/17	$X \pm U (k = 2) [Bq/kg]$ on 1 May 2015	Soil 3P1/17	$X \pm U (k = 2) [Bq/kg]$ on 1 May 2015
2P1/17/A1	0.897 ± 0.045	3P1/17/A1	0.616 ± 0.031
2P1/17/A2	0.922 ± 0.046	3P1/17/A2	0.698 ± 0.035
2P1/17/A3	0.923 ± 0.046	3P1/17/A3	0.836 ± 0.042
2P1/17/C1	0.918 ± 0.046	3P1/17/C1	0.738 ± 0.037
2P1/17/F1	1.014 ± 0.051	3P1/17/F1	0.725 ± 0.036
2P1/17/J1	0.756 ± 0.038	3P1/17/J1	0.756 ± 0.038

Table 3. Reference values of activity concentration of ²³⁹Pu in wheat flour and soil.

the certified values of activity concentration of the certified standard solutions provided in the certificates, weighing of the standard solutions and raw materials, and (in the case of solid materials) uncertainty due to dry mass determination. The assigned values, together with their expanded uncertainties (k = 2), are shown in Tables 2 and 3. **Evaluation of results**

The performance of each laboratory has been expressed in terms of *z* and *zeta* scores [2, 3]:

$$z = \frac{X_{\text{lab}} - X_{\text{pt}}}{\sigma_{\text{pt}}} \tag{1}$$

$$zeta = \frac{X_{lab} - X_{pt}}{\sqrt{u_{lab}^{2} + u(X_{pt})^{2}}}$$
(2)

where: X_{lab} – result reported by the participant, X_{pt} – assigned value, $u(X_{pt})$ – standard uncertainty of assigned values, u_{lab} – standard uncertainty reported by the participant, σ_{pt} – standard deviation for proficiency assessment.

 \bar{X}_{lab} is the mean of the individual measurement results as reported by the participant or as calculated by the PT organizer when no mean was reported. The *z*-score value |z| < 3 has been set as an acceptance level for this PT [2-4].

The *zeta*-score provides information on whether the laboratory uncertainty estimation is consistent with its deviation from the reference value. The *zeta*-score value should be analysed together with the *z*-score value calculated for the same laboratory result. The interpretation of the *zeta*-score value is similar to that of the *z*-score value [2, 3]. The results obtained by the participants were also evaluated according to the criteria for trueness and precision proposed by the International Atomic Energy Agency (IAEA) [8, 9]. The criteria are as follows:

1. Result passes trueness criterion if:

$$|\mathbf{x}_{ref} - \mathbf{x}_{lab}| \le 2.58 \sqrt{u_{ref}^2 + u_{lab}^2}$$
(3)
2. Result passes precision criterion if:

$$\sqrt{\left(\frac{u_{ref}}{x_{ref}}\right)^2 + \left(\frac{u_{lab}}{x_{lab}}\right)^2} \cdot 100\% \le \sigma_{pt}$$
(4)

where σ_{pt} (in %) denotes the standard deviation for proficiency assessment used in the calculation of the *z*-score value or the maximum value of uncertainty that can be accepted.

Table 4. Value (percent of assigned value) of standard deviation for proficiency assessment σ_{pt} .

Test motorial	³ H	²²⁶ Ra	²⁴¹ Am	²³⁹ Pu		
lest material	σ _{pt} [%]					
Drinking water	1H1/17 – 25 1H2/17 – 5	8	8	20		
Wheat flour	-	-	8	12		
Soil	-	-	-	12		

Measurement results reported as smaller than the limit of detection of the analytical procedure applied by the laboratory (\leq LOD) were assumed to be acceptable if the activity concentration was

Table 5. Summary of the scoring of the results provided by the participants – tests z and zeta.

Test material	Number of results provided by the participants	Number of results accepted/unaccepted by test z	Number of results accepted/unaccepted by test <i>zeta</i>	Number of qualitative (≤ LOD) results/number of accepted ones
Drinking water 1H1/17	9	3/3	3/3	3/3
Drinking water 1H2/17	10	6/4	7/3	-
Drinking water 1R1/17	7	6/1	6/1	-
Drinking water 1R2/17	7	7/0	7/0	-
Drinking water 1P1/17	5	4/1	2/3	-
Drinking water 1A1/17	8	8/8	7/1	-
Wheat flour 2A1/17	6	6/6	5/1	-
Wheat flour 2P1/17	3	3/3	3/3	-
Soil 3P1/17	3	3/3	3/3	-

Test material	Number of results provided by the participants	Number of results passed criteria for trueness	Number of results passed criteria for precision	Number of accepted results (passed both criteria)
Drinking water 1H1/17	6	3	5	2
Drinking water 1H2/17	10	7	9	6
Drinking water 1R1/17	7	6	7	6
Drinking water 1R2/17	7	7	7	7
Drinking water 1P1/17	5	1	5	1
Drinking water 1A1/17	8	6	8	6
Wheat flour 2A1/17	6	5	6	5
Wheat flour 2P1/17	3	3	3	3
Soil 3P1/17	3	3	3	3

Table 6. Summary of the scoring of the results provided by the participants - the IAEA approach.

below the detection limit of the analytical method applied by the laboratory.

The standard deviation for the proficiency assessment, σ_{pt} , was calculated as the standard deviation of reproducibility equal to the standard deviation of the arithmetic mean of all the laboratory means calculated after rejection of the outliers. The established values for σ_{pt} are shown in Table 4.

The scoring of the results provided by the participants is summarized in Tables 5 and 6. The results of the scoring show good performances of all the laboratories participating in the PT, especially taking into account that low activity concentrations were measured. The results supplied by the laboratories forming the radiation monitoring network in Poland confirm their competency. Even so, the participants have had the opportunity to compare their results with those of other laboratories and improve the quality of their routine analyses.

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RECERTIFICATION OF THE POLISH REFERENCE MATERIALS: MODAS-2 BOTTOM SEDIMENT (M-2 BotSed), MODAS-3 HERRING TISSUE (M-3 HerTis), MODAS-4 CORMORANT TISSUE (M-4 CormTis) AND MODAS-5 COD TISSUE (M-5 CodTis)

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The certified reference materials (CRMs) – MODAS-2 Bottom Sediment (M-2 BotSed), MODAS-3 Herring Tissue (M-3 HerTis), MODAS-4 Cormorant Tissue (M-4 CormTis) and MODAS-5 Cod Tissue (M-5 CodTis) – were prepared and certified in 2012-2015 within the scope of the MODAS consortium, consisting of seven Polish universities and institutes, as well as LGC Standards (Polish branch). The elemental contents of the above-mentioned CRMs were certified by the Institute of Nuclear Chemistry and Technology (INCT) on the basis of a worldwide interlaboratory comparison in which 46 to 54 laboratories from 13 countries (Poland, Argentina, Austria, Bul-

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Fig. 1. Frequency of use of analytical methods in the interlaboratory comparison.

garia, Brazil, Hungary, Indonesia, Italy, Norway, Pakistan, Romania, Russia and Slovenia) participated. The participating laboratories provided the results of the analyses of the candidate CRMs, which were performed mainly by atomic absorption spectrometry (AAS), inductively coupled plasma mass spectrometry (ICP-MS), inductively coupled plasma optical emission spectrometry (ICP-OES) and neutron activation analysis (NAA) methods. The frequency of their use is shown in Fig. 1. As can be seen, mass spectrometry with ionization in inductively coupled plasma was the most frequently applied method. However, comparing the values of the uncertainty of the results obtained



Fig. 2. Uncertainty of the mean results obtained by different analytical methods.

by the individual methods, it can be noted that the uncertainty of the ICP-MS result was, as a rule, the highest among all the results (Fig. 2). It can be seen that the lowest value of uncertainty was seen with the NAA results. This is one of the reasons why NAA plays so important role in the certification of RMs for the purpose of inorganic trace analysis [1, 2].

So, when the participating laboratories provided some new results obtained by NAA after the deadline (April 15, 2014) for supplying analytical results, it was decided to create new databases from all the obtained results and to subject them to statistical analysis in order to establish new certified values. The INCT approach of certification was applied [3]. This approach is based on the outlier rejection procedure, which uses four statistical criteria concurrently, namely those of Dixon (D), Grubbs (G), Skewness (S) and Kurtosis (K), at $\alpha = 0.05$, followed by calculations of the overall means of the results (remaining after the outlier rejection), standard deviations, standard errors, confidence intervals, *etc*.

The criteria used to decide whether the overall mean can be given the status of the certified value were as follows: 1. The ratio of the one-sided confidence interval and the overall mean:

$$\frac{\text{SD} \cdot t_{0.05}}{\text{SD}} \begin{cases} \leq 20\% \text{ (trace elements)} \\ 1 \end{cases}$$
(1)

 $\overline{\mathbf{X}} \cdot \sqrt{\mathbf{N}} \mid \le 10\%$ (major elements) or relative standard deviation:

SD
$$\int \leq 25\%$$
 (trace elements)

 $\left\{ \begin{array}{c} \\ \\ \end{array} \right\} \le 15\% \text{ (major elements)} \right\}$

For the purpose of this work, elements with concentrations exceeding 5000 mg kg⁻¹ (ppm) were considered to be the major elements.

- 2. The overall mean was calculated on the basis of at least four "accepted" laboratory means $(N \ge 4)$ obtained by more than one analytical technique. If results obtained by the use of only one analytical technique are available, the number of "accepted" laboratory averages used for the calculation of the overall mean cannot be smaller than five $(N \ge 5)$.
- 3. If the conditions (1) and (2) are fulfilled, but the number of outliers exceeds 50%, an additional procedure is activated which repeats the process of outlier rejection from the beginning, simultaneously checking the changes in the mean and standard deviation accompanying successive rejections. The process of rejecting of outliers is then stopped when the successive change in both the mean and standard deviation becomes lower or equal to 15%. The condition (1) is then rechecked.
- 4. If the above criteria are met, but there are indications that after outlier rejection performed on the whole population, the remaining populations of results obtained by various analytical techniques differ significantly, the assignment of certified value is suspended, and its status is described as "information" only.

"Information" values were assigned to those elements for which the results, while not simultaneously fulfilling the conditions (1)-(4), still fulfilled the following condition:

$$\frac{\text{SD} \cdot t_{0.05}}{\text{SD} \cdot \text{C}} \begin{cases} \le 50\% \text{ (trace elements)} \\ \le 20\% \text{ (constrained on the second of the seco$$

 $\overline{\mathbf{X}} \cdot \sqrt{\mathbf{N}} \mid \leq 30\%$ (major elements) calculated on the basis of at least three "accepted" laboratory averages; such values are quoted as numbers only, *i.e.* without confidence intervals. The elements for which the obtained values did not fulfil the above criterion were considered to be out of any classification. In the case of some elements (As, Cd, Co, Cr, Fe, Se and U), the correctness of certification was additionally confirmed by definitive methods (primary reference measurement procedures) based on radiochemical neutron activation analysis (RNAA) [4-8] and (in the case of Hg) by isotope dilution mass spectrometry (IDMS) [9, 10]. Traceability to the SI system was maintained because the laboratories, along with the analysis of MODAS samples, also analysed CRMs with a similar matrix. The new certified, as well as the information values, are presented in Tables 1-4

As additional results obtained by NAA have been taken into account, the uncertainties of the

(2)

	Certified values for M-2 BotSed								
ma	crocon	stituents			trace ele	ements			
element	conce	entration [wt%]	element	conce	ntration [mg kg ⁻¹]	element	conc	entration [ng g ⁻¹]	
Al	3	3.77 ± 0.39	As		7.07 ± 0.67	Bi*		363 ± 94	
Са	6	5.41 ± 0.56	Ва		266 ± 37	Eu		855 ± 85	
Mn	0.	102 ± 0.005	Cd		2.17 ± 0.13	Hg		884 ± 53	
Р	0.	175 ± 0.015	Ce		63.1 ± 5.7	Ho*		706 ± 131	
Ti	0.	325 ± 0.044	Со		7.85 ± 0.72	Lu		337 ± 64	
			Cs		4.21 ± 0.29	Sb		955 ± 167	
			Cu		32.9 ± 1.8	Tb		693 ± 53	
			Dy*		3.66 ± 0.62				
			Er*		2.06 ± 0.40				
			Ga		8.96 ± 1.25				
			Gd*		5.06 ± 0.39				
			La		28.6 ± 3.5				
			Li		27.3 ± 3.7				
			Мо		1.01 ± 0.15				
			Nb		11.2 ± 1.69				
			Nd		27.1 ± 3.3				
			Ni		35.4 ± 2.5				
			Pb		35.9 ± 2.9				
			Pr*		7.00 ± 0.81				
			Rb		71.5 ± 4.1				
			Sc		7.08 ± 0.71				
			Sm		5.23 ± 0.37				
			Sn		3.17 ± 0.65				
			Sr		197 ± 17				
			Th		10.3 ± 0.9				
			U		2.58 ± 0.48				
			V		80.4 ± 10.3				
			Y		20.9 ± 4.0				
			Yb		2.37 ± 0.43				
			Zn		275 ± 14				
			Informati	on valu	es for M-2 BotSed				
eleme	nt	value	uni	t	element	value		unit	
Ag		1.14	mg k	g ⁻¹	Na	4273		mg kg ⁻¹	
Be		1.23	mg k	g ⁻¹	S	2606		mg kg ⁻¹	
Br		13.9	mg k	g ⁻¹	Se	1.57		mg kg ⁻¹	
Cr		68.3	mg k	g ⁻¹	Та	1.01		mg kg ⁻¹	
Fe		2.598	wt%	⁄0	Tl	0.621		mg kg ⁻¹	
Hf		11.6	mg k	g ⁻¹	Tm	0.294		mg kg ⁻¹	
K		1.106	wt%	⁄0	W	2.43		mg kg ⁻¹	
Mg		5695	mg k	g^{-1}	Zr	434		mg kg ⁻¹	

Table 1. Certified and information values for M-2 SedBot.

 \ast Certified on the basis of results obtained by a single analytical method.

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Certified values for M-3 HerTis								
macroconstituents				trace elements				
element	conce	entration [wt%]	element	concer	ntration [mg kg ⁻¹]	element	conc	entration [ng g ⁻¹]
Cl	2	2.68 ± 0.46	As		9.22 ± 0.79	Ag		36 ± 5
K	1	17 ± 0.12	Ba		2.71 ± 0.28	Cd		325 ± 30
Mg	C	0.30 ± 0.02	Br		111 ± 11	Co		69 ± 6
Na	1	92 ± 0.17	Cu		3.19 ± 0.22	Cr		919 ± 105
Р	2	2.35 ± 0.39	Fe	190 ± 13		Cs		83.3 ± 7.5
S	0	0.93 ± 0.10	Mn	5.78 ± 0.61		Hg		227 ± 21
			Rb	2.32 ± 0.19		Li		896 ± 113
			Se	2.62 ± 0.24		Мо		127 ± 20
			Sr	192 ± 15		Ni		316 ± 49
			Zn		111 ± 6	Pb		104 ± 13
						Sb		15.8 ± 3.8
						Sc*		3.2 ± 0.4
						U		75.2 ± 8.2
						V^*		782 ± 112
			Informati	ion value	es for M–3 HerTis			
eleme	nt	value	uni	it	element	value		unit
Ca		3.73	wt	%	Y	9.6		ng g ⁻¹
Sm		1.8	ng g	g ⁻¹				

Table 2. Certified and information values for M-3 HerTis.

* Certified on the basis of results obtained by a single analytical method.

Certified values for M-4 CormTis								
ma	crocon	stituents	trace elements					
element	conce	entration [wt%]	element	concer	ntration [mg kg ⁻¹]	element	conc	entration [ng g ⁻¹]
К	1	$.19 \pm 0.08$	Br		11.1 ± 0.9	As		121 ± 12
Mg	0	0.10 ± 0.01	Cu		19.5 ± 1.2	Cd		17.2 ± 2.1
Na	0	0.22 ± 0.02	Fe		281 ± 16	Co		40.4 ± 3.9
Р	0	0.89 ± 0.14	Hg	Hg 2.20 ± 0.14		Cs		35.4 ± 4.6
S	1	$.04 \pm 0.11$	Mn	2.12 ± 0.18		Sb		66.7 ± 12.2
			Pb		2.33 ± 0.28	V*		7.8 ± 1.2
			Rb		13.3 ± 0.8			
			Se		1.27 ± 0.12			
			Sr		0.24 ± 0.03			
			Zn		63.4 ± 3.5			
			Informati	on value	s for M-4 CormTis			
eleme	nt	value	uni	it	element	value		unit
Ag		3.8	ng g	g ⁻¹	Cr	160		ng g ⁻¹
Ca		258	mg k	g ⁻¹	Мо	91		ng g ⁻¹

Table 3. Certified and information values for M-4 CormTis.

* Certified on the basis of results obtained by a single analytical method.

Certified values for M-5 CodTis									
macroconstituents			trace elements						
element	conce	entration [wt%]	element	concentration [mg kg ⁻¹]		element	conc	concentration [ng g ⁻¹]	
Cl	(0.41 ± 0.04	As		1.65 ± 0.26	Ва	162 ± 28		
K	1	1.92 ± 0.12	Br		24.8 ± 3.3	Cs		59 ± 5	
Mg	(0.12 ± 0.02	Cu		1.38 ± 0.09	Hg		310 ± 22	
Na	(0.34 ± 0.02	Fe	13.3 ± 1.1		Mn	921 ± 75		
Р	(0.96 ± 0.12	Rb	4.54 ± 0.31					
S	1.05 ± 0.16		Se	1.33 ± 0.10					
			Sr		4.07 ± 0.36				
			Zn		20.2 ± 1.1				
Information values for M-5 CodTis									
elemer	nt	value	unit		element	value		unit	
Bi		7.0	ng g ⁻¹		Cr	205		ng g ⁻¹	
Ca		0.11	wt%		Li	26		ng g ⁻¹	
Cd		5.0 ng g ⁻¹ Ni 136			ng g ⁻¹				
Со		19	ng g	-1	Pb	45		ng g ⁻¹	

Table 4. Certification and information values for M-5 CodTis

corrected values are lower than before. Analysis of the CRMs by RNAA primary reference measurement procedures (definitive methods) allowed confirmation of the correctness of the certification procedure for the elements for which such procedures were available. In the case of Cr, it was also possible to establish tentative certified values for M-4 CormTis and M-5 CodTis. These values were as follows: for M-4 CormTis 0.147 \pm 0.010 mg kg⁻¹ and for M-5 CodTis 0.205 \pm 0.018 mg kg⁻¹.

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LABORATORY OF MATERIAL RESEARCH

The activities of the Laboratory are concentrated on:

- studies of coordination polymers built of s block metals and azine carboxylate ligands,
- synthesis of nanoscale porous metal organic framework (nanoMOF) materials,
- synthesis of functional materials silver-modified cotton and cellulose fibres using radiation beam techniques,
- improvement of the usable surface properties of special materials applied in nuclear energy technologies (zirconium alloys, steels) using high intensity pulsed plasma beams (HIPPB),
- characterization of art objects. Porous coordination polymers, also called metal organic framework materials, have be-

come a topical subject in recent years. They exhibit unique pore architecture and a broad range of potential applications. The latter include greenhouse gas removal, storage of gases and the selective separation of components of gaseous mixtures that are of interest for the development of modern energy technologies. In the case of MOFs, the pores' structure and the host-guest molecules' interaction can be tailored relatively easily for a potential application by carefully combining the ligand and the type of metal ion. At present, many potential applications of MOFs require them to be obtained at a nanometre scale. Nanoscopic dimensions are essential to provide MOFs with high surface areas, and for tuning their properties (catalytic, separation, sensing and sorption). One application requiring such dimensions is mixed matrix membrane synthesis, where MOF's particles are used as fillers in a polymer matrix. Others include MOFs with size-dependent properties (optical, electrical and magnetic) and biocompatible materials for biomedical applications, e.g. encapsulation and transport of drugs. The integration of nanoscale MOFs onto porous supports would be advantageous for creating thin layer membranes. Studies performed recently in the Laboratory of Material Research concerning the synthesis of nanoscale MOFs are reported. The applied methods include template synthesis in the pores of track-etched membranes with well-defined cylindrical pores, synthesis in microfluidic flow reactors and the synthesis of MOFs on the surface of porous alumina substrates and also on composites with graphene oxide.

Zirconium, due to its good water corrosion and radiation resistance at the normal working conditions of nuclear reactors, is commonly used as cladding material for fuel elements. However, in the case of LOCA (loss-of-coolant accident) conditions, the possible extremely fast oxidation of zirconium in a steam atmosphere, or in an air/steam mixture, at temperatures above 800°C results in intense hydrogen generation and a possible hydrogen-oxygen mixture explosion. These events, despite being very rare, negatively influence the public acceptance for nuclear energy and result in high restoration costs for the accompanying damages. The development of methods to minimize the risk in the case of design-basis and beyond design-basis accidents is urgently needed. Materials with enhanced tolerance to high temperature oxidation have already been proposed for this purpose, such as silicon carbide, Mo-Zr, FeCrAl claddings, MAX phases and multilayer zirconium silicide coatings.

Zirconium silicide or zirconium silicate coatings are known for good resistance in hightemperature conditions and for that reason have been considered for application as environmental barrier coatings for high-temperature gas-turbine components. Up to now, their application as corrosion protective coatings for nuclear fuel pellets has been less explored. However, a review of the existing literature and an analysis of thermodynamic data indicate that silicon-based coatings may offer excellent prospects in this field. In particular, they may provide a more protective barrier than the native ZrO_2 films formed on alloy cladding during routine nuclear reactor operation. Our work in the last year has focused on the development of silicon-based coatings on zirconium alloys claddings and the evaluation of their properties during accident scenarios, as well as during the regular operation of reactors. Two processes have been considered for coating preparation. The first one is based on the deposition of layers containing zirconium oxide and silicon oxide onto zirconium alloys tubes (and also onto flat samples), followed by densification of the deposited layers. The second one is based on the deposition of gradient layers of zirconium and silicon (and also possibly their oxides) by the physical vapour deposition (PVD) method.

For the deposition of coatings precursors, three methods have been proposed: a dip coating method using mixed zirconium oxide and silicon oxide sols prepared by the sol-gel method, plasma electrolytic oxidation in silicate containing solutions, and electrophoretic deposition from zirconium oxide and silicon oxide containing suspensions, or directly from $ZrSiO_4$ suspension.

For the densification of the prepared porous layers, the unique technique of high intensity pulsed plasma beams will be applied or, alternatively, electron beams operating in scanning mode.

In order to examine, characterize and analyse cultural heritage artefacts or art objects and their component materials, a conservation scientist requires a palette of non-destructive and non-invasive techniques. This type of research improves knowledge concerning the elaboration, evolution and degradation of art objects over time, and provides a basis for their restoration and conservation. Among the various methods used, nuclear techniques are crucial due to their high sensitivity and reproducibility.

The purpose of last year's studies within this cultural heritage framework was the dating and determination of the provenance of a few groups of early medieval lead seals from Czermno, Gródek, Ruthenian and Russia. Ores from selected sources (Cracow-Silesian and Świętokrzyskie Mountains ore bodies, Poland) were also analysed to determine the provenance of the lead used. SEM-EDS (scanning electron microscopy–energy-dispersive X-ray spectroscopy), X-ray fluorescence analysis (EDXRF, TXRF), PIXE (particle-induced X-ray emission spectroscopy), PGAA (prompt-gamma activation analysis) and INAA (instrumental neutron activation analysis) as non-destructive methods were chosen to study a large number of samples. In addition to the study of the composition of the seals, analyses of the ratios of the lead isotopes were performed by means of a LA-ICP-MS (laser ablation inductively coupled plasma mass spectrometry) method. Interpretation of the results of the statistical methods enabled differentiation of the artefacts according to their various production centres, recipes and raw materials and the methods of purification used. Unique documentation and a database for the different types of seals is the result of these studies.

The investigation into the chemical composition and technologies employed in the manufacture of historical glass using micro-sample techniques was continued in 2017.

STUDIES ON HIGH TEMPERATURE OXIDATION RESISTANCE OF ZIRCONIUM SILICIDE COATINGS ON A ZIRCONIUM ALLOY

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Zirconium alloys are commonly used in pressurized water reactors (PWR) as fuel claddings for the hermetic enclosure of fuel pellets and fission products generated in the course of reactor operation. Zirconium alloys have been selected for their unique properties in reactor environment conditions, particularly for their low thermal neutron absorption, sufficiently good corrosion resistance at working conditions (360°C, 195 bar) and the sufficient stability of their mechanical properties under neutron irradiation. However, the main drawback of zirconium alloys is their completely unsatisfactory resistance to high temperature oxidation in steam atmosphere. Such conditions are expected in the case of a loss-of-coolant accident (LOCA). In this case the exothermic oxidation reaction of zirconium leads to a fast temperature rise and to the generation of large amounts of explosive gaseous hydrogen that may lead to the destruction of the nuclear core. As the safety of nuclear installations is of primary importance, intensive research work is being conducted worldwide on the development of so-called accident--tolerant fuels (ATFs), which will be much less prone to oxidation. Various concepts have been proposed for ATFs. The most revolutionary one is based on replacing zirconium alloys with composites of silicon carbide, another on applying an external oxidation-resistant coating on the surface of the zirconium alloy tubes. Before introducing a new type of material to the market, extensive tests are required in order to fulfil existing regulations, so the somewhat more conservative concept of external coatings seems to have the potential to be implemented in a shorter time. The results of the research work published on this subject in the open literature up to the end of 2016 have been recently reviewed [1].

Recently, a renewed interest in nuclear power has been observed in Poland. The strategic research programme called "Technologies supporting development of safe nuclear power engineering" was established in a response to the expected introduction of nuclear power engineering to Poland. The Laboratory of Materials Research at the Institute of Nuclear Chemistry and Technology joined this programme, focusing their research work on zirconium oxidation mechanisms under normal working conditions and during cases of emergency, as well as on countermeasures that would help to mitigate the catastrophic oxidation of zirconium in the case of an LOCA accident. The technique of high intensity pulsed plasma and ion beam treatment has been applied for the modification of the surface layer of zirconium. Using this

method, the melting of the surface layer and doping with eroded metallic electrode materials, such as Y, Cr and Al, into the molten surface layer was possible. However, the concentration of doped elements was usually at the level of a few percent, which was not high enough for the creation of a protective oxide layer on the surface of the fuel rod in the case of a large increase in temperature. Good results in autoclave tests and in an oxidation test in air at high temperature were obtained in the case of a chromium layer of 3.5 µm deposited by arc plasma discharge [2]. Based on that experience, we finally switched to physical deposition methods as flexible methods of coating synthesis and selected zirconium silicides as possible protective coatings. There are a number of reasons for this choice. Firstly, zirconium and silicon are preferred to chromium due to their lower cross--sections for thermal neutron absorption in comparison to chromium. Other ones are related to the material properties of silicides, particularly to their high melting points and resistance to oxidation. Phase diagrams for the Zr-Si system clearly show the existence of intermetallic compounds with different Zr/Si ratios and stability regions. For example, $ZrSi_2$ is stable up to $1620^{\circ}C$ and ZrSi is stable up to $2210^{\circ}C$, *etc*. Both zirconium and silicon readily form their respective oxides at high temperatures. According to its Ellingham diagram, ZrO₂'s formation enthalpy is higher than the enthalpy of silicon dioxide formation, and zirconium dioxide should be formed preferentially according to thermodynamic laws. However, kinetic factors also need to be taken into account. In the course of zirconium silicide oxidation, depletion of zirconium should lead to silicon's activity increasing, and the formation of silicon oxide is possible. As a result, ZrO₂, SiO₂ or even ZrSiO₄ may form, depending on the stoichiometry of the zirconium silicide. Moreover, in the case of ZrSi₂, layer depletion of silicon should lead to the formation of ZrSi, which possesses a higher melting temperature. Thus, it seems that zirconium silicide coatings with a stoichiometry of ZrSi₂ offer good prospects for their application as protective layers against oxidation at high temperatures. To check this hypothesis the relevant studies described here have been undertaken.

The commercially available Zr705 grade zirconium alloy supplied by Stanford Advanced Materials was used for sample preparation. Slices of 5 mm thickness were cut from a 25.4 mm diameter rod. The samples were polished with sandpaper and cleaned in an ultrasonic bath. Furthermore, before coating deposition their surfaces were etch-

Time [h]	Zr alloy – raw sample [g]	Sample coated from $ZrSi_2$ target [g]	Sample coated from Zr and Si targets [g]
1	0.0264	0.0096	0.0072
2	0.0325	0.0134	0.0088
3	0.0383	0.0156	0.0126
4	0.0472	0.0195	0.0143
5	0.0585	0.0241	0.0184

Table 1. The mass gain of samples due to oxidation in air at 700°C.

ed in a low-pressure glow discharge in argon gas. Zirconium- and silicon-containing coatings were sputter-deposited onto polished samples to a nominal thickness of about 3 µm by the DC magnetron sputtering technique from pure Zr and Si targets or a ZrSi2-sintered target with a diameter of 100 mm and a thickness of 8 mm, adhesively bonded on a copper washer. Before deposition, the chamber was evacuated to $3 \cdot 10^{-3}$ Pa, and the samples were heated up to about 300°C. The working pressure during deposition was 0.5 Pa, and the distance from the samples to the plasma source was 150 mm. During the process, the temperature was constantly monitored using a pyrometric temperature measurement system. The negative bias of the substrate was -100 V.

Two series of samples were prepared using different combinations of targets. The first one was prepared using two independent zirconium and silicon targets. This allowed for the synthesis of coatings with different stoichiometry by changing the relative sputtering rates of the targets. The second series was prepared using a single target made of the ZrSi₂ compound.

The as-deposited samples were amorphous, as was confirmed by X-ray diffraction (XRD) studies. For the development of crystallinity, thermal treatment in a tube furnace under an argon flow was applied.

For determining the oxidation resistance of the coatings, two types of tests were performed. One of them was aimed at determining the oxidation kinetics and the other at determining the performance under simulated accident conditions. For the oxidation kinetics study, the weight increase of the sample was determined after its oxidation in air at a temperature of 700°C. The sample, which was coated on both faces, was periodically placed into a furnace and removed from there at one-hour intervals up to 5 h maximum. After cooling in air, the weight of the sample was determined using a precision balance. The weight increment data were fitted to a power function. Independently, four samples coated on one side were placed into a furnace warmed to 700°C and were taken out one by one at one-hour intervals for scanning electron microscopy (SEM) studies on their morphologies and the thickness evolution of their coated layers in the course of oxidation

For the determination of the performance in the case of uncontrolled temperature growth during an LOCA, samples coated on one side were placed inside a furnace, warmed up to 1100°C for 1000 s in an air atmosphere and then taken out and dropped immediately into water.

At each step of the research, SEM (Ultra plus, Zeiss), XRD (D8 Advance, Bruker) and EDX (energy-dispersive X-ray spectroscopy; Quantax, Bruker) methods were applied for morphological, structural and elemental composition studies.

The results of weight increase from the oxidation in air at a temperature of 700°C are given in Table 1 for the two types of coated samples and for the raw sample. The data were fitted to the power function

$$\begin{split} w^k &= \alpha \cdot t \\ \text{or in the logarithmic form} \\ \log(w) &= 1/k \cdot \log(t) + \log(\alpha)/k \end{split}$$

where: w - mass gain, t - time.

The results of the linear regression fitting are presented in Table 2.

Table 2. The functional form of linear regression fitting to the mass gain of samples due to oxidation in air at 700°C.

Zr alloy – raw sample	$\log(w) = 0.4738 \cdot \log(t) - 1.6505$
Sample coated from ZrSi ₂ target	$\log(w) = 0.5474 \cdot \log(t) - 2.0327$
Sample coated from Zr and Si targets	$\log(w) = 0.5768 \cdot \log(t) - 2.1754$

An analysis of the data obtained clearly showed smaller mass gain in the case of the coated samples. For the samples coated from two targets, the mass gain for the 5-hour oxidation in air at 700°C was 3.17 times smaller than in the case of the raw sample. Analysis of the regression data confirmed the protective nature of the coatings. The values of exponential factor k of 1.73 (in the case of the two targets) and 1.83 (in the case of one target) obtained are slightly smaller than the ideal value of 2, which corresponds to the case when diffusion of oxygen through the layer is the main mechanism responsible for the mass gain during oxidation. It cannot be excluded that a small amount of porosity exists in the coatings, which is directly related to the columnar microstructure of coatings obtained by magnetron sputtering. It is worth mentioning that the coatings made from the two targets exhibited smaller values of mass increase over the oxidation time. It was also found that the samples prepared from two targets, after treatment at 700°C in an air at-



Fig. 1. The XRD spectra of the coating deposited from the two independent targets (Zr, Si) after thermal treatment at a temperature of 800°C in argon neutral atmosphere in normal geometry (A), at a glancing angle of 10° (B) and a glancing angle of 5° (C). The positions of zirconium alloy reflections are shown by the first row and the ZrSi₂ reflections by the second row of vertical markers below the spectra.

mosphere for 4 h, exhibited smooth surfaces, free of visible cracks, while in the case of coatings prepared with the application of the $ZrSi_2$ compound target, a network of cracks was clearly visible.

Experiments performed at the higher temperature of 1100°C with fast cooling in water again showed the better performance of the coated samples. The mass gain for the uncoated sample was equal to 0.3039 g, while that for the sample coated from two targets was 0.2216 g and that for the sample coated from the one compound target was 0.2159 g. As mentioned already, the as-deposited samples were amorphous. The thermal treatment of the samples in argon neutral atmosphere enabled the development of crystallinity. This was confirmed by the XRD measurements presented in Fig. 1, showing the spectra measured at normal Bragg-Brentano geometry and at glancing angles of 5° and 10°.

It can be seen that in the case of normal diffraction geometry, the zirconium substrate reflections are dominant in the spectrum, while in the other two cases, especially at a glancing angle of



Fig. 2. The SEM view of a cross-section of the coating made from the two independent targets (Zr, Si) (A) and elemental distribution across the coating after treatment in argon neutral atmosphere at a temperature of 800° C for 4 h (B).

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 5° , the reflections belonging to the ZrSi₂ phase are clearly visible. The Rietveld refinement of the XRD spectra based on the model of two contributing phases, Zr and ZrSi₂, converged successfully, confirming the validity of the model.

The SEM studies of cross-sections of thermally treated, coated samples allowed interesting observations of the morphology of the coatings to be made. After treatment in a non-oxidizing argon atmosphere, a layered structure inside the coating was developed. Three distinct layers could be distinguished, as shown in Fig. 2. For each of the layers, different elemental contents of zirconium, silicon and oxygen were found using the EDX method.

Starting from the outer region (Fig. 2B) we can observe:

- the thin external layer containing the elements Zr, Si and O. The presence of monoclinic ZrO₂ has already been confirmed by the XRD spectra and this phase is probably present mainly here. The structural form of Si is at present unknown. The XRD diffraction does not give any evidence for the presence of crystalline forms of silicon. The presence of amorphous silicon oxides is possible and need to be confirmed.
- the middle layer with near ZrSi₂ stoichiometry. The XRD studies confirm the presence of crystalline ZrSi₂.
- the wider, near to the bulk zirconium alloy layer with the stoichiometry ZrSi.

The safety of a nuclear installation in the case of beyond design-basis accidents strongly depends on the materials utilized. To avoid catastrophic accidents, such as the one in Fukushima, occurring in the future, high performance materials that can withstand the severe oxidation conditions present in the case of accidents need to be developed. In the studies presented in this paper, a zirconium silicide material was proposed as a protective coating for the zirconium alloy fuel tubes. Preliminary studies confirmed the protective character of this coatings at the temperature ranges investigated. The method of coating deposition from two independent targets allows for the preparation of thin coatings in the range of few micrometres with different stoichiometry. It seems that this variant is preferable over the deposition using one target with definite stoichiometric composition, as usually stoichiometry is not preserved during deposition. Moreover, no cracks were observed in the case of deposition using two elemental targets. The interesting observation of the in situ formation of a layered structure with different composition inside the coatings during thermal treatment in argon and in air could be utilized for the development of coatings that are protective against oxidation at high temperatures.

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ACCIDENT TOLERANT MATERIALS: IDEAS AND RESEARCH DIRECTIONS

Bożena Sartowska, Wojciech Starosta, Lech Waliś

Introduction

Zirconium, due to its good water corrosion and radiation resistance under the normal working conditions of nuclear reactors, is commonly used as a cladding material for fuel elements. A schematic of a light water reactor (LWR) fuel rod is presented in Fig. 1. The fuel pellets' cladding is a very important part of the rod. The elemental composition of the zirconium alloys used in nuclear reactors is presented in Table 1.

In the case of normal working conditions (a cooling water temperature of 360°C for pressurized water reactors (PWRs)), a slow zirconium oxidation occurs due to the presence of oxygen absorbed at the cladding's surface or that from water radiolysis reactions.

In the case of LOCA (loss-of-coolant accident) conditions, the extremely fast oxidation of zirconium in a steam atmosphere and/or an air-steam



Fig. 1. Schematic of a light water reactor fuel rod [1].

Zinconium allou	Producer	Alloying element [wt%]						
		Sn	Nb	Fe	Cr	Ni	0	
Zry-2	commonly used	1.50	-	0.13	0.1	0.06	0.12	
Zry-4	commonly used	1.50	-	0.21	0.1	0.007	0.12	
M5	AREVA		0.8-1.2					
ZIRLO	Westinghouse	0.7-1.0	1.0					
E110	Russia	-	0.9-1.1					

Table 1. Concentration of alloying elements in zirconium alloys used for claddings.

mixture at temperatures above 800°C results in intense hydrogen generation and a possible hydrogen-oxygen mixture explosion. The evolution of a fuel rod under LOCA and SBO (station blackout) conditions is connected with: steam oxidation of cladding, cladding ballooning and bursting, cladding internal oxidation, fuel relocation and dispersal, oxide eutectic formation and fuel rod melt [2]. These events, although very rare, negatively influence the public acceptance of nuclear energy. The development of a solution to this problem that will minimize the risk of such accidents is urgently needed. The concept of accident-tolerant fuels (ATFs) and accident-tolerant materials (ATMs) has been developed recently for this purpose [2, 3].

There have been many improvements to the original design and materials used. However, the basic concept of uranium oxide fuel pellets clad with zirconium alloy tubes has reminded the choice for the vast majority of commercial nuclear power plants. Development of a method to minimize the risk related to the design and unforeseeable situations is urgently needed. Four key performance features of the ATF and ATM concepts for improved safety margins proposed by Zinkle et al. [2] are: (i) minimize core enthalpy input (reduced steam-cladding oxidation rate and reduced heat of oxidation of cladding), (ii) minimize combustible hydrogen generation (reduced steam-cladding oxidation rate, enhanced hydrogen sequestration and chemical conversion), (iii) improved cladding to maintain core-cooling ability and to retain fission products (improved high--temperature cladding strength and fracture resistance, improved burst margins, thermal shock resistance, increased melting temperature and resistance to hydrogen embrittlement), (iv) improved fuel containment of fission products (enhanced retention of fission products, minimize fuel relocation/dispersal, lower operating temperatures, inhibit cladding's internal oxidation and increased fuel melting safety margin).

The approaches can be grouped into three general categories:

- non-zirconium cladding with high strength and oxidation resistance;
- improved high-temperature oxidation resistance and/or strength of zirconium alloy cladding;
- alternative forms of fuel with improved performance and fission product retention compared to monolithic UO₂.

These are the main research directions in the ATM area.

Non-zirconium cladding with high strength and oxidation resistance

The development of new alloys with special compositions and microstructures, along with the application of new materials resistant to water corrosion, are the main topics here. Challenges and issues to be resolved include: the effect of higher thermal neutron absorption cross-section, the impact of radiation hardening and embrittlement on cladding, pellet-cladding mechanical interaction behaviour, oxidation behaviour in steam at high temperatures, potential enhanced susceptibility to stress corrosion cracking, and potential reductions in the cost of tube fabrication.

Structural alloys

The thermal neutron cross-section of stainless steels is higher than that for zirconium alloys. But this problem with the use of stainless steel cladding can be partially overcome by thinner walled advanced stainless claddings because they are stronger than zirconium alloys. Investigations of this kind of material have covered: austenitic stainless steels (AISI 304, AISI 316 and AISI 347), austenitic nickel-based alloys (Inconel 600 and Incoloy 800) and ferritic steel with chromium (AISI 430). The replacement of the zirconium alloy by a ferritic material containing chromium and aluminium appears to be the nearest to implementation for accident-tolerant fuels [4, 5]. *Silicon carbide*

Silicon carbide (SiC) composite is used in industrial applications such as turbine components. Silicon carbide fibre reinforced SiC matrix ceramic composites (SiC/SiC) have been proposed as a potential cladding material due to: low thermal neutron absorption cross section, retention of strength up to very high temperatures, good radiation resistance and good oxidation resistance in air and steam up to 1600°C [6, 7].

FeCrAl alloys

FeCrAl alloys are used in industries where high-temperature oxidation resistance is needed (combustion and fossil fuel energy plants) [8]. FeCrAl nuclear grade alloys have important features for nuclear application: good mechanical properties for extended periods at high temperature, oxidation resistance, radiation tolerance, long-term water corrosion resistance and compatibility with the prototypically used UO_2 fuel. Nuclear grade FeCrAl alloys should be formable 66

as thin-walled tubes. The nominal composition of the model FeCrAl alloys are: Fe – 10-20 wt%, Cr – 3-5 wt% and Al – 0-0.15 wt%. The composition problem is complicated because the elemental composition has to be in a very narrow range. For example, more Cr is better for corrosion and oxidation resistance, but it would increase the potential embrittlement of the materials at relatively lower temperatures, and more Al gives better oxidation resistance at elevated temperatures, but it may raise the ductile-brittle transition temperature [5].

Refractory alloys

Refractory alloys based on Nb, Ta, Mo or W offer the potential for significant improvements in high-temperature strength as compared to zirconium alloys. Refractory alloys exhibit poor behaviour in high-temperature oxidizing environments, but research has identified some alloys (for example Mo_2Si -based) with adequate short-term oxidation resistance. It has been noted that high-temperature oxidation can be significantly different for steam and air [9]. Potential cladding designs for refractory alloys include monolithic and multilayered "sandwich" designs where thin oxidation-resistant layers are applied to the inner and outer surfaces of the refractory alloy [5].

Highly adherent oxidation-resistant coatings

Highly adherent oxidation-resistant coatings on the zirconium alloys can be an alternative to the known bulk materials for cladding. Challenges and issues to be resolved are the following: the coating should be easily regenerated if a small portion is spalled off during fuel assembly handling or during normal reactor operation, the need for careful matching of the coefficients of thermal expansion in order to minimize interfacial stresses and delamination during cycling from room temperature to 300°C (normal operation) or from 300°C up to high temperatures (abnormal operation) and the coating must be compliant enough to withstand the diametrical compression that results from reactor pressurization. The ability of coatings to continuously protect the exposed cladding to a sufficient extend after a burst also needs to be considered, because at this point the inner uncoated surface of the cladding is exposed to steam that causes rapid oxidation.

Chromium coatings

Chromium coatings serve as a protective oxidation barrier by providing the following properties: significantly improved resistance to corrosion under normal conditions in the 360°C water test, reduced corrosion rates in high-temperature steam and improved post-quench mechanical behaviour due to a reduced ingress of oxygen within the metal to form the alpha (O)-Zr phase [5]. *MAX phase*

MAX phases are a group of ternary ceramic compounds composed of early transition metals (Sc, Ti, V, Cr, Zr, Cr, Nb, Mo, Hf and Ta) as M, of elements from the A group (mainly Al, Si, P, S, Ga, Ge, As, In, Cd, Sn, Tl and Pb) as A, and carbon or nitrogen as X. MAX phases have good properties such as: thermal and electrical conductivity, corrosion resistance and mechanical properties that are maintained at high temperatures [5, 10]. *Coatings based on silicon*

Zirconium silicide or zirconium silicate coatings are known for good resistance in high temperature conditions and are applied as environmental barrier coatings for high-temperature gas-turbine components. Silicon-based coatings may provide a more protective barrier than the native ZrO_2 films formed on alloy cladding during routine nuclear plant operations and may provide a protective barrier during high-temperature accident scenarios. Phase diagrams for zirconium-silicon systems show the existence of intermetallic compounds with different Zr/Siratios and stability regions. For example, $ZrSi_2$ is stable up to 1620°C and ZrSi is stable up to 2210°C [11].

Institute of Nuclear Chemistry and Technology activity

The Institute of Nuclear Chemistry and Technology (INCT) is a partner in the International Atomic Energy Agency (IAEA) coordinated re-search project (CRP) "Analysis of options and experimental examination of fuels for water-cooled reactors with increased accident tolerance -ACTOF" in the framework of the IAEA programme "Nuclear fuel cycle and materials technologies". Globally, there is a great deal of experience with the performance of reactor fuel in abnormal conditions. Theoretical studies and experiments have been performed, and there have been excursions forming abnormal operating conditions in a few power reactors. This CRP will explore the potential to design and operate advanced fuel types that are intended to be more tolerant of severe accident conditions, whilst retaining the capability of current fuel designs for safe operation under normal operation and anticipated transient conditions. The participants of the ACTOF project are: Argentina, Brazil, China, Czech Republic, Finland, Germany, India, Italy, Korea, Poland, the Russian Federation and the United States of America.

The INCT is involved in this programme with the subject "Silicide/silicate coatings on zirconium alloys for improving the high temperature corrosion resistance".

Conclusions

Several potential approaches for development of ATMs exist and could lead to improved accident tolerance in LWR fuel systems. The various options in this paper explore and offer basic guidelines on the attributes of ATM concepts. Accident progression is dominated by the integral system response during such events. ATMs can affect the sequence and rate of this progression, thereby providing enhanced coping time during such events; the safety margins for nuclear power systems can therefore be enhanced.

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THE USE OF TRACE ELEMENT ANALYSIS ON IRON ARTEFACTS FROM THE PRZEWORSK CULTURE

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The main purpose of the studies presented here is to investigate the La Tène culture's impact on the cultures of ancient Central Europe, especially the Przeworsk culture. The provenance of prehistoric iron is one of the key questions in the archaeology of the Iron Age.

In the literature, it is accepted that three older cultural substrates contributed to the Przeworsk culture: the Cloche Grave, the La Tène, and the Jastorf cultures. Of these three, the La Tène culture had the largest impact on the Przeworsk culture. The influence of the La Tène culture is seen in the economy and crafts of the Przeworsk culture and principally in the spiritual culture that is manifested through their burial sites. The Przeworsk culture was the first culture in this part of the European Barbaricum and commonly made use of iron in their tools and weapons. Since the pre-Roman period, the Mazovia region functioned as a hub for metallurgical mass production with its local bog iron ores. Researchers have also discovered iron weapons from this period that were imported from the Celtic territories (the finds from Legionowo, Kraszewo, Kamieńczyk, Dobrzankowo, Oblin, Łęgonice Małe and Dzierzążnia Nowa). These objects, stylistically and structurally, are identical to objects made in the Western Celtic territories (e.g. the finds from the Celtic sanctuary in Gournay-sur-Aronde). For this reason, it is essential to conduct the latest physico-chemical testing methods on objects from the Western Barbaricum region as well as the Celtic territories. Such testing would allow an examination of intercultural relations with respect to iron weapons. It is worth emphasizing that this research will be the first wide-scale interdisciplinary exploration of this type involving objects found in the Western *Barbaricum* region as well as the Celtic territories. Through this research, it will be possible to confirm that objects found in the areas inhabited by the Przeworsk culture are potential imports and will simultaneously provide a new perspective on intercultural relations in Europe. Moreover, it will allow further assessment on the significance of the La Tène culture in shaping the Przeworsk culture.

A prehistoric burial ground of the Przeworsk culture dated between the 1st century BC and 2nd century AD was evacuated near Legionowo. This paper is the first part of our work, being concerned only with iron artefacts from the collection of the Historical Museum at Legionowo.

Samples for analysis were collected from 27 iron artefacts. Table 1 includes a description of the collected samples.

Trace and major elements in the iron objects were analysed by instrumental neutron activation analysis (INAA) without chemical separation, using standards of the analysed elements. The samples were packed together with standards of the following elements: Na, K, Sc, Cr, Mn, Fe, Co, Ni, Cu, Zn, Ga, Ge, As, Se, Br, Rb, Sr, Zr, Mo, Ru, Ag, Cd, Sn, Sb, Te, Cs, Ba, La, Ce, Pr, Nd, Sm, Eu, Tb, Dy, Ho, Er, Yb, Lu, Hf, Ta, W, Ir, Au, Hg, Th and ²³⁸U. Also attached were Au and Sc standards evaporated onto a piece of aluminium foil and a sample of the CTA-FFA-1 standard (Institute of Nuclear Chemistry and Technology, INCT).

Number of sample	Description of iron items	Catalogue number	Number of sample	Description of iron items	Catalogue number
P.1	iron arrow-head.1	w.325/2009	P.15	iron umbo-2	
P.2	iron arrow-head.2	w.325/2009	P.16	iron arrow-head.1	w.346/2009
P.3	iron sword.1	w.324/2009	P.17	iron arrow-head.2	w.346/2009
P.4	iron sword.2	w.324/2009	P.18	iron penknife	w.446/2009
P.5	iron sword.1	w.342/2009	P.19	iron arrow-head.1	
P.6	iron sword.2	w.342/2009	P.20	iron arrow-head.2	
P.7	iron ring-1		P.21	iron arrow-head.1	w.519/2009
P.8	iron ring-2		P.22	iron arrow-head.2	w.519/2009
P.9	iron ring-3		P.23	iron sword.1	w.658/2009
P.10	iron ring-4		P.24	iron sword.2	w.658/2009
P.11	iron ring-5		P.25	iron sword-pin pivot	w.212/35
P.12	iron ring-6		P.26	iron sword-pommel	w.212/35
P.13	iron buckle	w.343	P.28	iron object 'wolf gun'	w.189/2009
P.14	iron umbo-1				

Table 1. Description of the investigated Przeworsk culture iron artefacts from the collection of the Historical Museum at Legionowo.

They played the role of the thermal neutron flux monitor [1].

Irradiation of the samples was carried out in the MARIA reactor in Świerk, in a channel with $8 \cdot 10^{13}$ n/cm² s thermal neutron flux. The irradiation time was 2 h with a subsequent 8-hour cooling. The irradiated samples were then unpacked; they were washed in 1 M hydrochloric acid solution and rinsed in alcohol to remove surface contamination. Measurements of the activity of samples and standards prepared in such a way were carried out using an HP germanium detector with an active volume of 80 cm³ and an energy resolving power of 1.95 keV for a 1333 keV ⁶⁰Co source. The analysis of the complex gamma radiation spectra was carried out using Genie 2000 software. The measurements were repeated six times within three months after irradiation, while the measurement times varied between 300 and 10,000 s.



Fig. 1. Principal component analysis of 27 Przeworsk culture iron artefacts from the collection of the Historical Museum at Legionowo carried out on a set defined by 13 variables (Co, Cr, Cu, Eu, La, Mn, Ni, Th, U, Sb, Sm, Zn and Ta). Ellipses are defined by Gaussian distribution, confidence level 0.95.



Fig. 2. Cluster analysis of 27 Przeworsk culture iron artefacts from the collection of the Historical Museum at Legionowo describing 13 features (number of features was determined by INAA elements: Co, Cr, Cu, Eu, La, Mn, Ni, Th, U, Sb, Sm, Zn and Ta); standardized and logarithmic variables.

47 elements were identified and determined in the analysed samples. Out of the 47 determined elements, only the elements characteristic for tested samples (Co, Cr, Cu, Eu, La, Mn, Ni, U Th, Sb, Sm, Zn and Ta) were selected for multi-parameter statistical analysis aimed at identifying the degree of similarity of the analysed samples.

Principal component analysis (PCA) and cluster analysis were carried out with the use of STATISTICA (StatSoft) software [2] to identify the degree of similarity of the analysed objects. These multivariate analyses were performed for standardized and logarithmic variables. The results of principal component analysis are presented in Fig. 1, which clearly shows a division into groups closely related to the origin of the tested samples.

The output dendrogram of the cluster analysis is shown in Fig. 2, where the division between the two groups is very similar to the results from the PCA. Such a division suggests that the studied iron artefacts belonging to these defined groups originated from different sources.

The present study confirms the effectiveness of combined analyses of major and trace elements to characterize iron artefacts.

Our work illustrates the usefulness of multivariate statistical analyses in approaching a problem of this type. The obtained data show that it is not easy to completely and correctly identify or reproduce the materials used in the past and their deterioration processes.

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ANALYSIS OF HISTORIC GLASS USING MICROSAMPLING TECHNIQUES: LA-ICP-MS AND EPMA

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Historical glass constitutes a part of our material cultural inheritance and can be considered as an important witness of various human activities, such as trade, economy, culture or even politics. However, our understanding of glass as a material is still severely limited. The main objective of the project is to determine the provenance of various central European glass vessels dated to the mid-late 17th and 18th centuries. Differences between the compositions of glasses made in different locations are to be anticipated.

153 samples were taken from the selected glass vessels of interest. Most of them were goblets of various forms, made of colourless glass. With a few exceptions, they belonged to the Polish museums' collections. All the samples had already been analysed by the use of electron probe microanalysis (EPMA). Analyses by wavelength dispersive spectrometry in an EPMA system were carried out using a Cameca SX-100 with three simultaneously working spectrometers (PET, LiF, TAP crystals and PC2 for boron) at the Electron Microprobe Laboratory, Faculty of Geology, University of Warsaw, Poland. Corning Museum of Glass reference glasses "A", "B", "C" and "D"; NIST SRMs 610 and 612; as well as CRMs CZ4001, CZ4002 and CZ4003 from the Czech Metrology Institute were used for calibration and/or as controls. All the samples and standards were embedded in resin blocks of 25 mm diameter.

Many recent papers have shown that trace element analysis is even more effective in discriminating between the types and sources of some raw materials [1-2]. For this goal, inductively coupled plasma mass spectrometry with sampling using laser ablation (LA-ICP-MS) was used. The technique is a relatively new tool in glass archaeometry; however, it is of very high importance in such studies [1-2]. The method offers direct microsampling and fast multi-elemental capabilities (including Li and B) and is used for the accurate and precise determination of minor and trace elements with very low detection limits down to ng/L [2-6].

In the present study, a subset of these samples was analysed quantitatively, utilizing two LA-ICP--MS systems:

- LSX-213 G2+ Nd:YAG laser at 213 nm (Teledyne/CETAC/Photon Machines, Chromium 2.2b Software, HelEx sample cell) with aurora Elite ICP-MS (Bruker/Analytik Jena, Quantum v3.2b1633 software) at the Corning Research and Development Corporation, Sullivan Park campus, Corning, N.Y., USA;
- Analyte G2 excimer laser at 193 nm (Teledyne/ CETAC/Photon Machines, Chromium V2011.3.31 Software), laminar-flow "Frames" cell, with Varian 820 ICP-MS (Varian/Analytik Jena) at the Rensselaer Polytechnic Institute, Troy, N.Y., USA.

We reused the samples prepared for the EPMA analysis. A narrow laser beam was used for the sampling of a small amount of substance from the surface. This technique is increasingly being used in the direct analysis of solid samples.

The lack of suitable matrix-matched glass samples is still recognized as an important limitation of LA-ICP-MS in data interpretation. The LA-ICP-MS testing of the same glass samples and standards was carried out at both facilities with similar lasing parameters (with the obvious exception of the wavelength). The effects of time resolved analysis (TRA) with either ablation spot or line scan were evaluated and compared with the results from EPMA testing as well as literature data, when applicable.

Data processing was carried out utilizing Quantum software as well as Iolite software's "X_Trace_Elements_IS" data reduction scheme (version 3.5, Iolite Software).

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POLLUTION CONTROL TECHNOLOGIES LABORATORY

The research activities of the Pollution Control Technologies Laboratory concern the concepts and methods of process engineering applicable to the environmental area. In particular, we participate in research on the application of electron accelerators to such environmental technologies as flue gas and water treatment, wastewater purification, the processing of various types of industrial waste, *etc*.

- The main aims of the Laboratory's activity are:
- · development of new processes and technologies of environmental engineering,
- development of environmental applications of radiation technologies,
- promotion of nuclear methods in the field of environmental applications.

The activities of our group cover both basic and applied research. Among others, the most important research fields are:

- development of electron beam flue gas treatment (EBFGT) technology,
- support for the industrial implementation of the EBFGT process,
- investigation of chemical reaction mechanisms and kinetics in gas phases and aqueous solutions irradiated by electron beams,
- study of the mechanism of removal of volatile organic compounds (VOCs) and other pollutants from flue gas by electron beam irradiation,
- process modelling. The Laboratory is equipped with research tools such as:
- a laboratory installation for electron beam flue gas treatment;
- Model 40 UV pulsed fluorescent SO₂ analysers and Model 10 A/R chemiluminescent NO/NO_x analysers with molybdenum converters, manufactured by Thermo Electron Corporation (USA);
- GC-17A gas chromatograph with a GCMS-QP5050 mass spectrometer, manufactured by Shimadzu Corporation (Japan);
- Lancom II portable gas analyser type, manufactured by Land Combustion (UK) (NO_x, SO₂, CO, O₂, *etc.*).

The Laboratory is open for any form of cooperation. In particular, we offer such activities as:

- laboratory research on environmental applications of electron accelerators,
- theoretical modelling of chemical processes under electron beam irradiation,
- concept design of electron beam technology implementation,
- process equipment design with the use of CFD methods.
- In recent years, the Laboratory cooperated with such institutions as:
- Faculty of Chemical and Process Engineering, Warsaw University of Technology (Poland);
- International Atomic Energy Agency;
- Saudi ARAMCO (Saudi Arabia);
- EB Tech Co., Ltd. (Republic of Korea);
- Technology Centre of Western Pomerania (Germany);
- Leibniz Institute for Plasma Science and Technology (Germany);
- Risø National Laboratory for Sustainble Energy, Technical University of Denmark (Denmark);
- Uppsala University, The Ångström Laboratory (Sweden);
- Kaunas University of Technology (Lithuania);
- Vilnius Gediminas Technical University (Lithuania);
- Robert Szewalski Institute of Fluid-Flow Machinery, Polish Academy of Sciences (Poland);
- West Pomeranian University of Technology (Poland);
- National Centre for Nuclear Research, Otwock-Świerk (Poland);
- Ukrainian Engineering Pedagogics Academy (Ukraine); ٠
- Tsinghua University (China);
- Xi'an Jiaotong University (China);
- Joint Institute for Power and Nuclear Research Sosny, National Academy of Sciences of Belarus (Belarus);
- University of Palermo (Italy);
- Institute for Polymers, Composites and Biomaterials (IPCB), National Research Council (CNR) (Italy);
- Hacettepe University (Turkey);
- Institute of Macromolecular Chemistry "Petru Poni" Iasi (Romania);
 University of Reims Champagne-Ardenne (France);
- University Politehnica of Bucharest (Romania);
- Texas A&M AgriLife, Texas A&M University (USA).

NUMERICAL SIMULATION OF NO_x REDUCTION IN AQUEOUS SOLUTION

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In the process of burning high-sulphur fuels in marine diesel engines used in transoceanic ships, high concentrations of NO_x and SO₂ are emitted to the atmosphere, which significantly exceed the emission limits of both pollutants. Therefore, the application of emission control methods is necessary.

A new hybrid method of SO₂ and NO_x removal from flue gases designed for application in marine conditions was elaborated at the Institute of Nuclear Chemistry and Technology (INCT). The removal of SO₂ and NO_x from flue gases takes place in two stages: in the first stage the gases are irradiated by an electron beam from an accelerator, while in the second stage the off-gas after irradiation is absorbed in a water scrubber.

The results of preliminary laboratory research with the use of a 0.6 dm³ scrubber to treat a gas stream with a volumetric flow rate of 150 $dm^{\overline{3}}/h$ showed high removal efficiency of SO₂ and NO_x. However, the research showed that NO_x removal efficiency strongly depends on the irradiation dose and initial NO_x concentration. As the radiation--induced process had been modelled elsewhere [1], a mathematical model of NO_x removal in the water absorption process was constructed to enable analysis of the process mechanism.

The following reactions in the gas and liquid phases were considered [2]: ase nhase

$$2NO_{(g)} + O_{2(g)} \xrightarrow{k_1} 2NO_{2(g)}$$
(1)
$$2NO_{2(g)} \xleftarrow{K_2} N_4 O_{2(g)}$$
(2)

$$NO_{(g)} + NO_{2(g)} \xleftarrow{K_3}{K_3} N_2O_{3(g)}$$
(3)

$$N_2O_{3(g)} + H_2O_{(1)} \xleftarrow{K4}{2} 2HNO_{2(1)}$$
 (4)

$$N_2O_{4(g)} + H_2O_{(1)} \xleftarrow{K_5} HNO_{3(1)} + HNO_{2(1)}$$
 (5)

• liquid phase $2NO_{2(2)} + H_2O_{(1)} \xrightarrow{k_6} HNO_{2(1)} + HNO_{2(1)}$ (6)

$$N_{2}O_{3(g)} + H_{2}O_{(l)} \xrightarrow{k7} 2HNO_{2(l)}$$
(7)

$$N_2O_{4(r)} + H_2O_{(1)} \xrightarrow{k_8} HNO_{3(1)} + HNO_{2(1)}$$
 (8)

$$3HNO_{2(1)} \xrightarrow{k9} HNO_{3(1)} + H_2O_{(1)} + 2NO_{(g)} (9)$$

The chemical reaction rates are as given by Loutet *et al.* [2]: $k1 = 0.0102 \text{ m}^6 \text{ kmol}^{-2} \text{ s}^{-1}, k2 = 10^9 \text{ m}^3 \text{ kmol}^{-1} \text{ s}^{-1}, k3 = 10^9 \text{ m}^3 \text{ kmol}^{-1} \text{ s}^{-1}, k4 = 41,000 \text{ m}^3 \text{ kmol}^{-1} \text{ s}^{-1}, k5 = 250 \text{ m}^3 \text{ kmol}^{-1} \text{ s}^{-1}, k6 = 46,999 \text{ m}^3 \text{ kmol}^{-1} \text{ s}^{-1}, k7 = 17,000 \text{ s}^{-1}, k8 = 1000 \text{ s}^{-1}, k7 = 17,000 \text{ s}^{-1}, k8 = 1000 \text{$ 93.32 s⁻¹, k9 = 0.0468 atm² m⁹ kmol⁻³ s⁻¹, k2' = $k2 \cdot K2^{-1}$, $K2 = 0.439 \text{ m}^3 \text{ kmol}^{-1}$, $k3' = k3 \cdot K3^{-1}$, $K_3 = 13,699 \text{ m}^3 \text{ kmol}^{-1}, \text{ k4}' = \text{k4} \cdot \text{K4}^{-1}, \text{ K4} = 0.2763, \text{ k5}' = \text{k5} \cdot \text{K5}^{-1}, \text{ K5} = 0.00002191.$ For the gas phase the temperature was assumed to be 90°C, while for the liquid phase the temperature was assumed to be 15°C.

The absorption kinetics were described by Eq. (10):

$$r_{i} = k_{L}a \cdot (p_{i} - p_{i}^{*})$$
(10)
= NO NO, NO, or NO, k.a. - the

where: i = NO, NO_2 , N_2O_3 or N_2O_4 ; $k_La - the mass transfer coefficient [s⁻¹], in this work the value of <math>k_La$ was set as 0.01 s⁻¹ [3]; p_i – the partial pressure of component i in the bulk; and p_i^* – the interface pressure of component i in the liquid.

The system of ordinary differential equations, being the mathematical model of the considered process, was solved with the use of the Runge--Kutta procedure in the MATLAB programming language. Simulations were carried out for the following concentrations:

• initial NO concentrations: 200, 500, 1000 and 1271 ppmv;

• NO₂ concentrations: 0, 20 and 50 ppmv.

The following irradiation doses were considered: 3.5, 10.9 and 44.1 kGy. The concentrations of NO and NO₂ at the scrubber inlet were taken from the literature [1, 4]. The calculated results for the NO and NO₂ concentrations are shown in Table 1. The efficiency of NO_x removal in the electron beam process (without absorption) and in the hybrid electron beam process (with absorption) is shown in Table 2.

The obtained modelling results show that application of the hybrid electron beam process in comparison to the electron beam process is the most profitable method for moderate doses of irradiation and high inlet concentration of NO_x. These results make the hybrid electron beam process at-

Table 1. NO and NO₂ concentrations in the hybrid electron beam process.

No.	Dose [kGy]	NO initial concentration [ppmv]	NO ₂ initial concentration [ppmv]	NO at scrubber inlet [ppmv]	NO ₂ at scrubber inlet [ppmv]	NO at scrubber outlet [ppmv]	NO ₂ at scrubber outlet [ppmv]
1	3.5	200.0	0.0	163.0	19.0	161.1	15.1
2	3.5	1000.0	0.0	902.0	78.0	872.2	41.4
3	10.9	1271.0	52.0	940.0	318.0	822.8	204.5
4	10.9	500.0	20.0	200.0	120.0	186.0	107.0
5	44.1	200.0	0.0	82.0	8.0	81.6	6.9
6	44.1	1000.0	0.0	621.0	89.0	594.6	56.0

No.	Efficiency of NO_x removal in the electron beam process [%]	Efficiency of NO _x removal in the hybrid electron beam process [%]		
1	9.0	11.9		
2	2.0	8.7		
3	4.9	22.4		
4	38.5	43.7		
5	55.0	55.8		
6	29.0	34. 9		

Table 2. Efficiency of NO_x removal in the electron beam process and hybrid electron beam process.

tractive for potential practical application in marine flue gas treatment.

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STABLE ISOTOPE LABORATORY

Basic activities of the Stable Isotope Laboratory concern the techniques and methods of stable isotope measurements (H, C, N, O, S) by the use of an isotope ratio mass spectrometer (IRMS). Our activity area also concerns the application of IRMS to the environmental area, including determination of the stable isotope composition of hydrogeological, environmental, and food samples.

The main aims of the activity of the Laboratory are:

- preparation and measurement of the stable isotope composition of food and environmental samples;
- establishing new areas of the application of stable isotope compositions for food authenticity control, environmental protection, and origin identification. The Laboratory is equipped with the following instruments:
- mass spectrometer DELTA^{plus} (FinniganMAT, Germany);
- elemental analyser Flash 1112NC (ThermoFinnigan, Italy);
- GasBench II (ThermoQuest, Germany);
- H/Device (ThermoQuest, Germany);
- gas chromatograph (Shimadzu, Japan);
- gas chromatograph with a mass spectrometer (Shimadzu, Japan);
- liquid scintillation counter (for ¹⁴C and tritium environmental samples) 1414-003 Guardian (Wallac-Oy, Finland);
- freeze dryer Alpha 1-2 LD plus (Christ, Germany). The Laboratory research staff is involved in the following projects:
- "The study of the influence of the environmental factors on the isotopic compositions of dairy products",
- accreditation process (isotopic method for food authenticity control),
- interlaboratory proficiency test FIT-PTS (food analysis using isotopic techniques proficiency testing scheme).

The Stable Isotope Laboratory is open for any form of cooperation. We are prepared to undertake any research and development task within the scope of our activity. In particular, we offer our measurement experience, precision, and proficiency in the field of stable isotope composition to aid the research of others. In addition, we are open to perform any service regarding the control of food authenticity by stable isotope methods supported by gas chromatography (GC) and gas chromatography-mass spectrometry (GC-MS) methods.

Our Laboratory cooperates with the following national partners:

- Agricultural and Food Quality Inspection,
- Polish Association of Juice Producers,
- customs inspections,
- food export-import company,
- food control laboratories,
- private customers

and foreign partners:

- Eurofins Scientific Analytics (France),
- International Atomic Energy Agency (IAEA),
- Joint Research Centre (Ispra, Italy).

FIT.PTS Toffciency testing scheme 2011 Round 1 water samples ref: 11/1/F

ret : 11/1/F

FIT-PTS Proficiency testing scher 2010 Round 3 Honey ref : 10/3/C FIT-PTS feiency testing not 2011 Round 1 casein ref: 11/2/C FIT-PTS *acy testin 311 Round

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ISOTOPIC AND TRACE ELEMENT COMPOSITION FOR DAIRY PRODUCTS' AUTHENTICITY CONTROL

Ryszard Wierzchnicki, Renata Adamska, Zbigniew Samczyński

The different compositions of cow fodder (natural and processed) and drinking water (tap water) are reasons for the seasonal and regional variation of materials an elemental analyser coupled with a mass spectrometer was used. The measurements of trace element composition (Fig. 3) were performed



Fig. 1. Sample preparation and measurement by the use of the IRMS technique: EA – elemental analyser, GB – GasBench II, HD – H/Device, CF – ConFlo III, IRMS – isotope ratio mass spectrometer DELTA^{plus}.

the isotopic and trace element composition of fresh milk and, finally, dairy products. The stable isotopic composition of food is strictly related to environmental conditions, such as climate, geographical region and season.

The aim of this study was to demonstrate the regional and seasonal variations of the isotopic and trace element composition of milk. Samples of fresh and commercial milk were obtained from the main regions of milk production in Poland and measured using an isotope ratio mass spectrometer. The measurement of the stable isotope composition (carbon, hydrogen, oxygen, nitrogen and sulphur) of food is a very sensitive method for controlling its origin and authenticity [1-5].

Isotope ratio mass spectrometry (IRMS) is the main method used for the measurement of the stable isotopic content of food and beverages (Fig. 1). A DELTA^{plus} mass spectrometer connected with an H/Device instrument was used to determine the hydrogen isotope ratio. The oxygen isotope ratio was determined in water samples with the use of a GasBench instrument connected with a mass spectrometer. For the determination of carbon, nitrogen (Fig. 2) and sulphur in solid



Fig. 2. Carbon ¹³C and nitrogen ¹⁵N isotopic composition of defatted milk.

using inductively coupled plasma mass spectrometry (ICP-MS) for a limited number of dairy products.



Fig. 3. Concentration of trace elements Sr, Se and Mo in defatted milk.

Finally, application of the multi-element (isotopic and trace element composition) method for the origin control of dairy products requires testing more samples of such products, from both organic and conventional farms. After the construction of a large database of isotopes and trace elements, the method could be used as a standard to control the origin and authenticity of dairy products.

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ISOTOPIC STUDY OF APPLE DRINKS

Ryszard Wierzchnicki, Renata Adamska

Stable isotope analyses have been useful tool for food authenticity control. An important limitation of applying the isotopic method for food authenticity control is a lack of a database comprising the stable isotope compositions of different origin foods. For many years, the Stable Isotope Laboratory of the Institute of Nuclear Chemistry and Technology (INCT) has carried out studies on the isotopic composition of food for elaboration and implementation in new isotope ratio mass spectrometry (IRMS) methods, and also creates a database of isotopic composition of chosen food from the Polish market. Our recent studies concerned the intramolecular isotopic distribution pattern between the components of ciders.

Cider is one of the popular drinks produced by apple juice fermentation (Table 1). The main chemical components of cider are: water, ethanol, sugars, flavours and CO_2 . The subject of the study is determination of the stable isotope composition of water, ethanol and CO_2 included in ciders. Accordingly, the study aim is to investigate the stable isotope compositions of these components, as well as identifying their source, in an effort to control the authenticity of the drinks [1-8].

The addition of sugar to the production of ciders is allowed in some countries but forbidden in the others (*e.g.* France). During the fermentation of sugars in juice, ethanol and CO_2 are produced. The addition of beet sugar (C3 plants – Calvin cycle) or cane sugar and corn syrup (C4 plans – Hatch-Slack pathway) results in different isotopic



Fig. 1. The measured δ^{13} C values of ethanol for tested ciders.

composition of CO_2 and ethanol (Fig. 1). Artificial carbonating ciders, typically using CO_2 from an industrial source, result in a $\delta^{13}C$ value of CO_2 lower than -40‰ (Fig. 2).

Our method is to look for the range of values of the isotopic composition for authentic ciders. In the study, the stable isotope method for measurement of ethanol and CO_2 bubbles (natural or exogenous carbonation) will be elaborated to control the quality of ciders and their compliance

Table 1. Names of popular apple drinks and concentration of alcohol.

Apple drink	Alcohol [vol%]	Polish market	French market	English market	German market
Juice	0	sok jabłkowy	pomme jus	apple juice	apfelsaft
Cider	1.3-8.0	cydr	cidre	cider	cidre
"Wine"	9-18	"wino jabłkowe"	cidre	cider	apfelwein
Liqueur	18-35	nalewka, likier jabłkowy	eau-de-vie de pommes	apple liqueur	Apfelkorn
Vodka	35-45	wódka jabłkowa	calvados (only region Calvados)	applejack (apple brandy)	Obstwasser

Parameter Component		Composition	Method	
δ^{13} C ethanol, CO ₂		$^{13}C/^{12}C$	IRMS	
$\delta^{18}O$	water	¹⁸ O/ ¹⁶ O	IRMS	
δ^{18} O solid residue		¹⁸ O/ ¹⁶ O	IRMS	
δ^{87} Sr	solid residue	⁸⁷ Sr/ ⁸⁶ Sr	MC-ICP-MS, TIMS	
Elements solid residue		trace elements	ICP-MS	
Sugars solid residue		fructose, glucose, sucrose	HPLC	

Table. 2. Analytical methods used for control of cider (apple drinks) authenticity.

with labelling (authenticity). The basic problem surrounding CO_2 is: Is the CO_2 gas in ciders from a natural source (natural fermentation) or from an industrial (technical) source? For control of cider authenticity, the basic information is the compliance oxygen isotopic composition in cider (water) with the corresponding composition for apple juices from the same geographical region.



Fig. 2. The measured δ^{13} C values of ethanol *vs*. δ^{13} C of CO₂ for the Polish commercial ciders.

Samples of 34 commercial ciders were purchased from the Polish market. We examined their isotopic composition using IRMS methods. Specially, our study concerned the isotopic composition of: carbon δ^{13} C in ethanol, oxygen δ^{18} O in water and carbon δ^{13} C in CO₂.

The isotopic composition of δ^{13} C in CO₂ and ethanol is finally expressed by the following equation:



The examples of measured isotopic composition values of cider components are presented in Figs. 1 and 2.

The isotopic compositions of water and CO_2 were determined using GasBench (ThermoQuest, Germany) connected in the continuous flow mode to a DELTA^{plus} (FinniganMat, Germany) mass spectrometer. The isotopic composition of ethanol was measured using the same mass spectrometer with the elemental analyser Flash EA1112 (ThermoQuest, Italy). Every sample was measured six times for both the carbon and oxygen isotopic composition. The standard deviation of the values obtained from measurements was 0.2% for $\delta^{13}C$ (ethanol and CO_2) and 0.15% for $\delta^{18}O$ (water). In conclusion, the work will be continued to characterize components from the bigger population of commercial products (ciders). The main result of the studies will be the formation of a database containing the compositions (Table 2) for all the basic components of apple drinks from different origins. By utilizing the database, employing the methods for the origin control of drinks can be implemented in everyday practice. It will still be necessary to test the sensitivity of the method for each component of apple drinks. Finally, on the basis of the study, the correlations between components of apple drinks will be elaborated.

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LABORATORY FOR MEASUREMENTS OF TECHNOLOGICAL DOSES

The Laboratory for Measurements of Technological Doses (LMTD) was created in 1998 and accredited as a testing laboratory in February 2004 (Polish Centre of Accreditation, accreditation number: AB 461). The actual accreditation range is:

- gamma radiation dose measurement by means of a Fricke dosimeter (20-400 Gy),
- gamma radiation dose measurement by means of a CTA film dosimeter (10-80 kGy),
- electron radiation dose measurement by means of a CTA film dosimeter (15-40 kGy),
- electron radiation dose measurement by means of graphite and polystyrene calorimeters (1.5-40 kGy),
- irradiation of dosimeters or other small objects with ⁶⁰Co gamma radiation to strictly define doses,
- irradiation of dosimeters or other small objects with 10 MeV electron beams to strictly define doses.

The secondary standard of the dose rate utilized by the LMTD is a 60 Co gamma source Issledovatel and a Gamma Chamber 5000. The sources were calibrated in January 2016 and in March 2012, respectively, according to the NPL (National Physical Laboratory, Teddington, UK) primary standard. The uncertainty of the dose rate was estimated to be 3.0% and 3.1% (U, k = 2).

In accordance with the recommendations of the standard PN-EN ISO/IAC 17025:2005, a laboratory establishes traceability of its own measurement standards and measuring instruments to the SI by means of an unbroken chain of calibrations or comparisons that links them to the relevant primary standards of the SI units of measurement. The link with SI units may be achieved by referencing to the national measurement standards. These may be primary standards, which are primary realizations of the SI units or agreed representations of SI units based on fundamental physical constants, or they may be secondary standards calibrated by another national metrology institute. The calibration of all types of dosimeter systems that are used routinely should be checked by comparison with a reference standard or a transfer standard dosimeter.

In 2017, the LPDT performed measurements for its PVC, B3 and Amber Perspex dosimeters. These consisted in determining calibration curves for electron beam irradiation and comparing them with the LPDT calorimeters. The dose range of 5-40 kGy corresponded to the routine radiation sterilization process for medical devices, transplants and pharmaceuticals. For the above-mentioned dosimeters, the following calibration curves (D = f(Abs)) were obtained:

- for the PVC dosimeter: $Y = -26.236x^2 + 71.513x 4.6068$, $R^2 = 0.9971$, uncertainty = 4.62%;
- for the B3 dosimeter: $Y = -22.386x^2 + 96.345x 3.7356$, $R^2 = 0.9698$, uncertainty = 4.39%;
- for the Amber Perspex dosimeter: $Y = 32.211x^2 + 12.759x + 1.4207$, $R^2 = 0.9944$, uncertainty = 5.39%, and $Y = 77.847x^2 + 21.088x + 1.5026$, $R^2 = 0.9937$, uncertainty = 4.24%.

LABORATORY FOR DETECTION OF IRRADIATED FOOD

The Laboratory for Detection of Irradiated Food was created at the Institute of Nuclear Chemistry and Technology in 1994. The adoption of the quality assurance system resulted in accreditation of the Laboratory in 1999 by the Polish Centre for Accreditation (PCA). Since that time, the Laboratory for Detection of Irradiated Food has constantly maintained the status of an accredited R&D unit and is authorized to examine food samples to classify them as being either irradiated or not irradiated. Every four years, the Laboratory accreditation certificate must be renewed after successfully passing the PCA expert audit. The current certificate, which is the 5th, was received on 30th September 2014 and is valid until 24th October 2018.

Professional and well-experienced staff members are working to improve irradiation detection methods adapted in the Laboratory to make them more sensitive and reliable for an extended group of food articles. The Laboratory offers analytical services in this field to domestic and foreign customers for an extended assortment of food articles *via* the use of five appropriate and normalized analytical methods. The *Scope of Accreditation* – an integral part of the accreditation certificate, offers customers five methods suitable to detect radiation treatment in almost all foodstuffs available in the open market.

Nowadays, many food items such as herbal pharmaceuticals, diet supplements, and food extracts are received by the Laboratory for examination of irradiation from our domestic and foreign customers.

The Laboratory implemented the following detection methods:

- A method for the detection of irradiated food from which silicate minerals can be isolated using a thermoluminescence (TL) reader; this method is based on analytical procedures recommended by the CEN European standard EN 1788.
- A method for the detection of irradiated food containing bone *via* the use of electron paramagnetic spectroscopy (EPR/ESR) based on an analytical procedure described by the CEN European standard EN 1786.
- A method for the detection of irradiated food containing cellulose *via* the use of EPR spectroscopy based on an analytical procedure described by the CEN European standard EN 1787.
- A method for the detection of irradiated food containing crystalline sugars with EPR spectroscopy based on analytical procedures described by the CEN European standard EN 13708.
- A method for the detection of irradiated food using a photostimulated luminescence (PSL) reader; this method is based on analytical procedures recommended by the CEN European standard EN 13751.

The application of the aforementioned five standardized detection methods to specified groups of foods that have been validated in the Laboratory guarantees the accurate analysis and reliable classification of food samples delivered to the Laboratory for testing.

The Laboratory is actively and effectively implementing improved analytical and measurement procedures that are suitable for detection of irradiation in complex food articles containing low or very low concentrations of irradiated ingredients. These are typically aromatic herbs and spices admixed to the product.

It has been experimentally proven that modification of the mineral isolation procedure, the determination of isolated mineral content, and the effectiveness of mineral thermoluminescence are the important factors that influence the detection ability of the employed analytical method.

In 2017, the Laboratory reported a large increase in the number of samples delivered by domestic customers to be analysed; samples were also delivered by foreign customers from Germany, Italy, Denmark, Latvia, Hungary and Spain. The assortment of samples received comprised food extracts, herbal pharmaceuticals, diet supplements, spices, dried vegetables and red fermented rice. In total, 1426 samples were examined. Of these, 1419 samples were examined by the TL method, while the EPR based analytical procedures were applied only three times and the PSL – four times.

From 19th June 2012, the Laboratory has held the status of the reference laboratory in the field of the detection of irradiated food in Poland under the nomination of the Ministry of Health (National Reference Laboratory No. 5). As such, the Laboratory is responsible for organizing control and monitoring irradiated food around the country.

INVESTIGATION OF LOW DOSE IRRADIATED DRIED FRUITS BY A STANDARDIZED ELECTRON PARAMAGNETIC RESONANCE DETECTION METHOD

Grzegorz Piotr Guzik, Magdalena Miłkowska

The dried fruit components of diet supplements and fruit extracts are supposed to positively influence human condition and health. These components are usually dried in a traditional manner and contain many impurities and microbial contaminants, including dangerous pests. For this reason, dried fruits undergo disinfection leading to microbial decontamination.

One of the most effective methods of microbial decontamination of dried and fresh fruits is irradiation [1, 2]. As recommended by a FAO/WHO expert group, the safe dose of ionizing radiation as applied to food is 0.2-10 kGy [3], while in the case of dried spices and fruits it is 5-10 kGy, depending on the content of microbial contaminants. Recently, thermal or high pressure treatment combined with irradiation have been applied for the decontamination of these products. In such combined disinfection processes markedly lower doses of ionizing radiation than those recommended are required.

The aim of the present study was to evaluate the detection limits of such low dose irradiation applied to dried fruits examined after a long period of storage. In other words, the determination of the possibility of the detection of irradiation in dried fruits irradiated with low doses of ionizing radiation and stored for a prolonged period. An analytical procedure based on the CEN European standard EN 13708 was used [4].

The subject of the investigation were ten species of dried fruits used in pharmacy as available on the market. These were: plum, apricot, strawberry, cherry, cranberry, date, raisin, mulberry, fig, and blackcurrant. All the fruits were irradiated with doses of 0.3 kGy and 0.5 kGy (the lowest recommended technological doses in use). For comparison, non-irradiated batches of the tested fruits were investigated in parallel.



Depending on the species examined by the EPR (electron paramagnetic resonance) method, the fruit components tested were the peels, stones, skins or pulp/flesh. The samples taken for investigation were dried fruits purchased from the market or fresh fruits dried for 48 h at 40°C in a dryer machine under a flow of dry air. The samples



Fig.2. EPR signal of dried cherry irradiated with a 0.5 kGy dose of ⁶⁰Co gamma radiation and measured 20 months after irradiation. The multiline broad signal is typical of radiation-treated food products containing crystalline sugar.

were irradiated in a ⁶⁰Co gamma source (Gamma Chamber 5000) with two doses, 0.3 kGy and 0.5 kGy. A suitable portion of each fruit was placed in a signal-free glass ampoule 4 mm in diameter and subsequently measured with a Bruker EMXplus EPR spectrometer equipped with Xenon software. Representative EPR signals obtained with the investigated fruits are shown in Figs. 1-4, respectively.

The EPR measurements of dried samples were carried out 1 day after the irradiation to avoid the interference of short-lived radicals produced by radiation. The specific signal characteristic for irradiated sugars (Fig. 1) appeared for all fruits, with the exception of plum and apricot (Table 1). However, in the irradiated, dried strawberry's EPR signal only a central line with two weak satellite lines on both sides was noted, proving that radiation-evoked cellulose radicals were involved.



Fig. 1. EPR signals of dried cherry and strawberry irradiated with a 0.5 kGy dose of ⁶⁰Co gamma radiation and measured 1 day after irradiation at room temperature. The multiline broad signal of dried cherry resembles the signals observed with radiation-treated food products containing crystalline sugar. The characteristic signal of cellulose radicals can be recognized in the dried strawberry's EPR trace.

Fig.3. EPR signals of dried mulberry irradiated with 0.3 kGy and 0.5 kGy doses of ⁶⁰Co gamma radiation, measured 20 months after irradiation.



344 346 348 350 352 354 356 358 Magnetic field [mT]

Fig.4. EPR signals of dried cranberries. The multiline, broad signal recorded is like a weak sugar signal. The whole fruit was irradiated with 0.3 kGy and 0.5 kGy doses of ⁶⁰Co gamma radiation and the signal was measured 20 months after irradiation. Two weak satellite lines on both sides of central line prove that cellulose radiation-evoked radicals were involved.

The samples were then stored in the dark for 20 months to determine the stability of the EPR signals involved. The results of the second EPR examination are shown in Table 2.

diation were markedly higher than those registered after 20 months of storage.

The EPR signal recorded with irradiated cherry was a broad (5.8 mT), multiline spectrum derived from stable radicals trapped in crystalline sugars (Fig.2). This kind of stable EPR signal was recorded with most commercially available dried fruits. However, after 20 months of storage this EPR signal was only seen for three irradiated fruits: cherry, cranberry and mulberry (all treated with a 0.5 kGy dose and also for mulberry treated with a 0.3 kGy dose) [5, 6].

The EPR signal recorded with irradiated mulberry represented a different multiline signal (7.4 mT broad), which could be assigned to a sugarderived signal (Fig.3).

A different EPR signal was recorded with the sample of irradiated dried cranberries (Fig. 4). In this signal a triplet with a strong central line was seen. The distance between the two weak satellite lines was about 6 mT with g = 2.004, evidence that cellulose radicals were involved [7].

The multiline, broad signal that was recorded was like a weak sugar signal. The whole fruit was

examination are shown in Table 2.			ke a weal	x sugar signal. The whole fruit w
Table 1. The results of the EPR study of dried fruits 1 day after irradiation at room temperature.				
Dried fruit	Dose [kGy]	EPR result 1 day after irradiation	Dose [kGy]	EPR result 1 day after irradiation
Plum	0.3	no signal	0.5	no signal
Apricot	0.3	no signal	0.5	no signal
Strawberry	0.3	weak cellulose-derived signal	0.5	weak cellulose-derived signal
Cherry	0.3	specific sugar-derived signal	0.5	specific sugar-derived signal
Cranberry	0.3	specific sugar-derived signal	0.5	specific sugar-derived signal
Date	0.3	specific sugar-derived signal	0.5	specific sugar-derived signal
Raisin	0.3	specific sugar-derived signal	0.5	specific sugar-derived signal
Mulberry	0.3	specific sugar-derived signal	0.5	specific sugar-derived signal
Fig	0.3	specific sugar-derived signal	0.5	specific sugar-derived signal

The intensities of the central EPR signals recorded for dried samples immediately after irra-

0.3

Blackcurrant

irradiated with 0.3 kGy and 0.5 kGy doses of ⁶⁰Co gamma radiation and measured 20 months after

specific sugar-derived signal

0.5

Table 2. The results of the EPR study of ten fruits after 20 months of storage at room temperature.

specific sugar-derived signal

Dried fruit	Dose [kGy]	EPR result after 20 months of storage	Dose [kGy]	EPR result after 20 months of storage
Plum 0.3 no signal		no signal	0.5	no signal
Apricot	Apricot 0.3 no signal		0.5	no signal
Strawberry	0.3	no characteristic signal – only central line	0.5	no characteristic signal – only central line
Cherry	0.3	no characteristic signal – only central line	0.5	sugar-derived signal
Cranberry	0.3	no characteristic signal – only central line	0.5	weak sugar-derived signal
Date	0.3	no characteristic signal – only central line	0.5	no characteristic signal – only central line
Raisin	0.3	no satellite lines	0.5	no satellite lines
Mulberry	0.3	weak sugar-derived signal	0.5	weak sugar-derived signal
Fig	0.3	no characteristic signal – only central line	0.5	no characteristic signal – only central line
Blackcurrant	0.3	no characteristic signal – only central line	0.5	no characteristic signal – only central line

irradiation. Two weak satellite lines on both sides of the central line proved that cellulose radiationevoked radicals were involved.

The EPR spectra of seven species of dried fruits irradiated with 0.3 kGy and 0.5 kGy doses and investigated 1 day after treatment gave wellresolved multiline signals specific for irradiated sugars. No EPR signal was found with the plum or apricot. The characteristic signal of the cellulose radical could be recognized with dried strawberry.

After prolonged storage of the samples for 20 months, the radiation-induced EPR signals survived in the samples of cherry, cranberry and mulberry, giving evidence of the unique stability at room temperature of the EPR signals in these fruits. The remaining seven species of investigated fruits did not give any characteristic signal after prolonged storage, but only a strong single line in the central part of the spectrum. Only for dried cherry, cranberry and mulberry treated with 0.3 kGy or 0.5 kGy doses it was possible to detect irradiation after 20 months of storage at room temperature.

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LABORATORY OF NUCLEAR CONTROL SYSTEMS AND METHODS

The primary focus of the Laboratory activity in 2017 was the development of methods and equipment, based generally on the application of ionizing radiation and process engineering for both measurements and diagnostic purposes. The research programme of the Laboratory was focused on the following topics:

- development of measuring devices and systems for industry and for protection of the environment;
- development of a new leakage control method for testing industrial installations during their operation;
- identification and optimization of industrial processes using the tracer and radiotracer methods;
- application of membrane processes of biogas separation and their enrichment in methane;
- elaboration and industrial scale implementation of new methods and technology of biogas production by fermentation of agriculture substrates and by-products such as wastewater sediments obtained during wastewater clarification;
- development of new radiotracer and radiometric methods for optimization of hydrometallurgical processes.

In the field of elaboration and construction of nuclear instrumentation, the works were directed towards the detection of radioactive contamination and measurements of the concentration of radon daughter radioisotopes in air.

In the above-mentioned areas of activity, the Laboratory cooperated with the International Atomic Energy Agency (IAEA).



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ARTICLES

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Organized by the Institute of Nuclear Chemistry and Technology; Centre for Innovation and Technology Transfer Management, Warsaw University of Technology

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Organized by the Institute of Nuclear Chemistry and Technology

Organizing Committee: Agnieszka Miśkiewicz, Ph.D., Prof. Grażyna Zakrzewska-Kołtuniewicz, Ph.D., Dorota Gajda, M.Sc.

7. NUTECH-2017 INTERNATIONAL CONFERENCE ON DEVELOPMENTS AND APPLICA-TIONS OF NUCLEAR TECHNOLOGIES, 10-13 SEPTEMBER 2017, KRAKÓW, POLAND

Organized by the Faculty of Energy and Fuels, AGH University of Science and Technology; Faculty of Physics and Applied Computer Science, AGH University of Science and Technology; Fundation for AGH University of Science and Technology; Institute of Nuclear Chemistry and Technology; Ministry of Energy; Ministry of Science and Higher Education; National Atomic Energy Agency; Polish Nuclear Society; International Atomic Energy Agency

Organizing Committee: Prof. Marek Lankosz, Ph.D., D.Sc., Prof. Andrzej G. Chmielewski, Ph.D., D.Sc., Prof. Kazimierz Różański, Ph.D., D.Sc., Jakub Nowak, Ph.D., Leszek Furman, Ph.D., Lucyna Samek, Ph.D., Marek Ciechanowski, Ph.D., Joanna Dudała, Ph.D., Pawel Wrobel, Ph.D., Jerzy Cetnar, Ph.D., D.Sc., professor in AGH, Mikołaj Oettingen, Ph.D., Paweł Gajda, Ph.D., Przemysław Stanisz, Ph.D., Prof. Grażyna Zakrzewska-Kołtuniewicz, Ph.D., D.Sc., Wojciech Migdał, Ph.D., D.Sc., professor in INCT

8. XIV SZKOŁA STERYLIZACJI I MIKROBIOLOGICZNEJ DEKONTAMINACJI RADIACYJNEJ (XIV TRAINING COURSE ON RADIATION STERILIZATION AND HYGIENIZATION), 19-20 OCTOBER 2017, WARSZAWA, POLAND

Organized by the Institute of Nuclear Chemistry and Technology

Organizing Committee: Zbigniew Zimek, Ph.D., Andrzej Rafalski, Ph.D., Wojciech Głuszewski, Ph.D., Magdalena Rzepna, M.Sc.

9. KONFERENCJA NAUKOWA "WYBRANE ZAGADNIENIA ANALIZY ŚLADOWEJ" Z OKAZJI JUBILEUSZU 60-LECIA PRACY W IBJ-IChTJ PROF. DR. HAB. RAJMUNDA S. DYBCZYŃ-SKIEGO (SCIENTIFIC CONFERENCE "SELECTED PROBLEMS OF TRACE ANALYSIS" ON THE OCCASION OF THE 60th ANNIVERSARY OF WORK OF PROF. RAJMUND S. DYBCZYŃSKI AT THE INR-INCT), 5 DECEMBER 2017, WARSZAWA, POLAND

Organized by the Institute of Nuclear Chemistry and Technology

Organizing Committee: Halina Polkowska-Motrenko, Ph.D., D.Sc., professor in INCT, Anna Bojanowska-Czajka, Ph.D., Jakub Dudek, Ph.D.

Ph.D./D.Sc. THESES IN 2017

Ph.D. THESES

1.	Iwona Bartosiewicz, M.Sc. (INCT Ph.D. student) Biogeochemiczne zachowania uranu i pierwiastków towarzyszących na terenach poeksploatacyjnych rud uranu w bloku karkonosko-izerskim (Biogeochemical behaviour of uranium and associated elements in areas of uranium post-mining wastes in the Karkonosze-Izera block) supervisor: Halina Polkowska-Motrenko, Ph.D., D.Sc., professor in INCT Institute of Nuclear Chemistry and Technology, 31.03.2017
2.	Grzegorz Guzik, M.Sc. (Institute of Nuclear Chemistry and Technology, Warszawa, Poland) Badania trwałych efektów radiacyjnych w cukrach prostych pod kątem identyfikacji napromieniowanej żywności (Stable radiation effects in simple sugars related to detection of irradiated food) supervisor: Prof. Jacek Michalik, Ph.D., D.Sc. Institute of Nuclear Chemistry and Technology, 31.03.2017
3.	Liang Zhao, M.Sc. (INCT Ph.D. student) Magnetic sorbent for the removal of cesium from aqueous solutions supervisor: Prof. Andrzej G. Chmielewski, Ph.D., D.Sc. Institute of Nuclear Chemistry and Technology, 31.03.2017
4.	Łukasz Steczek, M.Sc. (Co-tutelle, INCT Ph.D. student) Complexation of actinides Am(III), Th(IV), Pu(IV) and U(VI) with poly-N-dentate ligands SO ₃ -Ph-BTP and SO ₃ -Ph-BTBP supervisors: Philippe Moisy, Ph.D., Prof. Jerzy Ostyk-Narbutt, Ph.D., D.Sc. Institute of Nuclear Chemistry and Technology, 5.07.2017
5.	Katarzyna Łuczyńska, M.Sc. (Institute of Nuclear Chemistry and Technology, Warszawa, Poland) Synthesis and complementary structural spectroscopy study of selected organic complexes of donor-ac- ceptor type supervisor: Prof. Jan Cz. Dobrowolski, Ph.D., D.Sc. Institute of Nuclear Chemistry and Technology, 15.12.2017
6.	Ewa Anna Zwolińska, M.Sc. (Institute of Nuclear Chemistry and Technology)

The removal of NO_x and SO_2 from exhaust gases using a hybrid electron beam method supervisor: Yongxia Sun, Ph.D., D.Sc., professor in INCT Institute of Nuclear Chemistry and Technology, 15.12.2017

D.Sc. THESES

 Marek Danilczuk, Ph.D. (Institute of Nuclear Chemistry and Technology, Warszawa, Poland) Badania spektroskopowe membran jonowymiennych stosowanych w ogniwach paliwowych (Spectroscopic studies of ion exchange membranes for fuel cells applications) Institute of Nuclear Chemistry and Technology, 15.12.2017

EDUCATION

Ph.D. PROGRAMME IN CHEMISTRY

The Institute of Nuclear Chemistry and Technology (INCT) holds a four-year Ph.D. degree programme for graduates of chemical, physical and biological departments of universities, for graduates of medical universities and to engineers in chemical technology and material science.

- The main areas of the studies are:
- chemical aspects of nuclear energy,
- radiation chemistry and biochemistry,
- · chemistry of radioelements,
- isotopic effects,
- radiopharmaceutical chemistry,
- analytical methods,
- chemistry of radicals,
- application of nuclear methods in chemical and environmental research, material science and protection of historical heritage.

The candidates can apply for a doctoral scholarship. The INCT offers accommodation in 10 rooms in the guesthouse for Ph.D. students not living in Warsaw.

During the four-year Ph.D. programme, the students participate in lectures given by senior staff from the INCT, University of Warsaw and the Polish Academy of Sciences. In the third year, the Ph.D. students are obliged to prepare a seminar related to the various aspects of nuclear energy. Each year the Ph.D. students are obliged to deliver a lecture on topic of his/her dissertation at a seminar. The final requirements for the Ph.D. programme graduates, consistent with the regulation of the Ministry of Science and Higher Education, are:

- submission of a formal dissertation, summarizing original research contributions suitable for publication;
- final examination and public defence of the dissertation thesis.
- In 2017, the following lecture series and lectures were organized:
- Selected problems in nanomaterials chemistry Prof. Adam Proń, Ph.D., D.Sc. (Warsaw University of Technology, Faculty of Chemistry, Poland);
- Drug chemistry Piotr Jakub Lipiński, Ph.D. (Mossakowski Medical Research Centre, Polish Academy of Sciences, Warszawa, Poland);
- Radioactive waste Prof. Grażyna Zakrzewska-Kołtuniewicz, Ph.D., D.Sc. (Institute of Nuclear Chemistry and Technology, Warszawa, Poland);
- (1) Spent fuel: reprocessing vs. direct disposal; characterization, inventory verification and disposal. The reasoning for the CHANCE project WP4; (2) Calorimetric characterization of rad-waste and the reasoning for the CHANCE project, WP3 Prof. Holger Tietze-Jaensch (Forschungszentrum Jülich, Germany).

The qualification interview for the Ph.D. programme takes place in the mid of September. Detailed information can be obtained from:

• head: Prof. Aleksander Bilewicz, Ph.D., D.Sc.

- (phone: +48 22 504 13 57, e-mail: A.Bilewicz@ichtj.waw.pl);
- secretary: Ewa Gniazdowska, Ph.D., D.Sc., professor in INCT (phone: +48 22 504 11 78, e-mail: E.Gniazdowska@ichtj.waw.pl).

TRAINING OF STUDENTS

Institution	Country	Number of participants	Period
Gdańsk University of Technology, Faculty of Applied Physics and Mathematics	Poland	1	5 months
International Atomic Energy Agency	Myanmar	1	1 week

EDUCATION

Institution	Country	Number of participants	Period
International Atomic Energy Agency	Myanmar	1	2 months
University of Gdańsk, Faculty of Chemistry	Poland	1	1 month
ilitary University of Technology	Poland	1	1 month
		1	1.5 months
	Poland	1	2 weeks
		1	2.5 weeks
University of Warsaw, Faculty of Chemistry		3	3 weeks
		2	1 month
		2	1.5 months
niversity of Warsaw, Faculty of Physics	Poland	1	2 months
		1	6 months
Warsaw University of Life Sciences – SGGW	Poland	2	1 month
arsaw University of Technology, aculty of Chemical and Process Engineering	Poland	1	1 month
		1	1 year
	Poland	2	3.5 weeks
Warson University of Technology Feculty of Chamistan		1	3 weeks
arsaw University of Technology, Faculty of Chemistry		2	1 month
		1	1 year
Warsaw University of Technology, Faculty of Physics	Poland	24	one-day course

MASTER'S, BACHELOR'S AND ENGINEER'S THESES

1. Emilia Balcer

Master's thesis: Synteza i badanie właściwości fizykochemicznych ⁶⁸Ga-NODA-GA-Tocilizumab jako potencjalnego radiofarmaceutyku do diagnostyki reumatoidalnego zapalenia stawów (Synthesis and physicochemical properties of the ⁶⁸Ga-NODA-GA-Tocilizumab, a potential radiopharmaceutical for the diagnosis of rheumatoid arthritis)

supervisors: Przemysław Koźmiński, Ph.D., Michał Grdeń, Ph.D., D.Sc. University of Warsaw, Faculty of Chemistry

2. Marta Jolanta Kania

Engineer's thesis: Badanie wpływu parametrów procesowych na efektywność adsorpcji radionuklidów ¹³⁴Cs i ⁶⁰Co z roztworów wodnych przez sorbent SiEA-KNiFe (Investigation of influence of operating parameters on effectiveness of adsorption process of radionuclides ¹³⁴Cs and ⁶⁰Co in water solutions using SiEA-KNiFe)

supervisors: Marek Henczka, Ph.D., D.Sc., WUT professor

supervisor in INCT: Dagmara Chmielewska-Śmietanko, M.Sc.

Warsaw University of Technology, Faculty of Chemical and Process Engineering

3. Aneta Piasecka

Master's thesis: Trikarbonylkowe kompleksy technetu(I) i renu(I) z ligandami bidentnymi (Tricarbonyl technetium(I) and rhenium(I) complexes with bidentate ligands) supervisors: Maciej Mazur, Ph.D., D.Sc., Monika Łyczko, Ph.D.

University of Warsaw, Faculty of Chemistry

4. Magdalena Rodak

Master's thesis: Nanociało HER2 znakowane ¹⁷⁷Lu jako potencjalny radiofarmaceutyk terapeutyczny (¹⁷⁷Lu labelled HER2 nanobody as a potential therapeutic radiopharmaceutical) supervisors: Maciej Jan Kamiński, Ph.D., D.Sc., Marek Pruszyński, Ph.D. University of Warsaw, Faculty of Physics

5. Ilona Ściśniak

Master's thesis: Analiza ekspresji genów związanych z odpowiedzią na uszkodzenia DNA jako potencjalny biomarker wrażliwości komórek na promieniowanie (Analysis of gene expression related to DNA damage response as a potential biomarker of intrinsic radiosensitivity) supervisors: Beata Brzozowska, Ph.D., Kamil Brzóska, Ph.D.

University of Warsaw, Faculty of Physics

6. Piotr Żmijewski

Engineer's thesis: Badanie kinetyki procesu adsorpcji radionuklidów ¹³⁴Cs i ⁶⁰Co z roztworów wodnych przez sorbent SiEA-KNiFe (Investigaton of kinetics of adsorption process of radionuclides ¹³⁴Cs and ⁶⁰Co from aqueous solution onto SiEA-KNiFe sorbent)

supervisor: Marek Henczka, Ph.D., D.Sc., WUT professor

supervisor in INCT: Dagmara Chmielewska-Śmietanko, M.Sc.

Warsaw University of Technology, Faculty of Chemical and Process Engineering

RESEARCH PROJECTS AND CONTRACTS

RESEARCH PROJECTS GRANTED BY THE NATIONAL SCIENCE CENTRE IN 2017

1. Chiral cores/monomers of drugs and conducting polymers: from calculations to experimental characteristics.

supervisor: Prof. Jan Cz. Dobrowolski, Ph.D., D.Sc.

2. Nanobodies labelled with alpha emitters as potential radiopharmaceuticals in targeted radioimmunotheraphy.

supervisor: Marek Pruszyński, Ph.D.

- Nanoparticles of gold, gold-gold sulphide and titanium dioxide modified with tellurium as carriers for At-211 for targeted alpha theraphy.
 supervisor: Prof. Aleksander Bilewicz, Ph.D., D.Sc.
- 4. Studies on the phenomena occurring in the membrane boundary layer during the filtration of aqueous solutions and suspensions proceeding in membrane apparatuses with different configurations. supervisor: Agnieszka Miśkiewicz, Ph.D.
- 5. The influence of nanoparticles on beta-amyloid removal by microglia cells. supervisor: Katarzyna Sikorska, M.Sc.
- 6. Impact of nanoparticles on cellular signalling activated by tumour necrosis factor. supervisor: Kamil Brzóska, Ph.D.
- 7. Analytical, kinetic and toxicological study of degradation selected perfluorinated compounds using ionizing radiation.

supervisor: Prof. Marek Trojanowicz, Ph.D., D.Sc.

- 8. New analytical procedures based on neutron activation analysis for the determination of chosen Se, As and Fe chemical formulae in infant alimentation. supervisor: Halina Polkowska-Motrenko, Ph.D., D.Sc., professor in INCT
- **9.** Radiation-induced radical processes involving amino acids and quinoxalin-2-one derivatives relevant to their pharmacological applications. supervisor: Konrad Skotnicki, M.Sc.
- **10.** Bioconjugates of multimodal nanoparticles for targeted alpha and hyperthermia therapy. Synthesis, retention studies of recoiling daughters radionuclides and preliminary cell. supervisor: Edyta Cedrowska, M.Sc.
- **11.** In vitro and in vivo preclinical studies of NaA nanozeolite funcionalized with antibodies anti-PSMA and labelled with radium radioisotope for targeted prostate cancer therapy. supervisor: Prof. Anna Lankoff, Ph.D., D.Sc.
- 12. In vitro studies of NaA nanozeolite functionalized with integrins inhibitor cilengitide and labelled with radium radioisotope for targeted breast cancer therapy. supervisor: Sylwia Męczyńska-Wielgosz, Ph.D.
- **13.** Studies on cefepime labelling with Ga-68. supervisor: Przemysław Koźmiński, Ph.D.
- 14. Nanostructured lipidic liquid-cystalline carriers for chemotherapeutics and corpuscular radiation emitters in targeted cancer therapy.

supervisor: Agnieszka Majkowska-Pilip, Ph.D.

15. Radiopharmaceuticals based on neurokinin-1 receptor antagonists for the diagnosis and therapy of brain tumour-glioblastoma multiforme. supervisor: Ewa Gniazdowska, Ph.D., D.Sc., professor in INCT 16. 6xS for chiral semiconducting naphtalene diimides: synthesis, simulations, structure, spectroscopy, spectroelectrochemistry, sensors.

supervisor: Prof. Jan Cz. Dobrowolski, Ph.D., D.Sc.

PROJECTS GRANTED BY THE NATIONAL CENTRE FOR RESEARCH AND DEVELOPMENT IN 2017

- Plasma technology to remove NO_x from off-gases. (common undertaking of the National Science Centre, and the National Centre for Research and Development "TANGO2") supervisor: Prof. Andrzej G. Chmielewski, Ph.D., D.Sc.
- 2. The integrated system of sewage treatment, biogas production and its enrichment in the methane. supervisor: Jacek Palige, Ph.D.
- Syntheses of radiopharmaceuticals based on scandium radionuclides for positron emission tomography (Petscand).
 supervisor: Prof. Aleksander Bilewicz, Ph.D., D.Sc.
- 4. The development of innovative advanced therapy with use of a biological dressing of the human race in the treatment of *epidermolysis bullosa* and other chronic wounds (STRATEGMED). supervisor: Zbigniew Zimek, Ph.D.

IAEA RESEARCH CONTRACTS IN 2017

- Based on starch-PVA system and cellulose reinforced active packaging materials for food prepared using of radiation modification (PackRad). No. 17493 principal investigator: Krystyna Cieśla, Ph.D., D.Sc., professor in INCT.
- 2. The study of the influence of the environmental factors on the isotopic compositions of dairy products.
 - No. 18056

principal investigator: Ryszard Wierzchnicki, Ph.D.

- Application of advanced membrane systems in nuclear desalination. No. 18539 principal investigator: Prof. Grażyna Zakrzewska-Kołtuniewicz, Ph.D., D.Sc.
- 4. Studying the social and socio-economic effects of the iomplementation of the Polish nuclear programme using new methodology.

No. 18541

principal investigator: Agnieszka Miśkiewicz, Ph.D.

- 5. Application of low energy electron beam for microbiological control of food and agricultural products.
 - No. 19000

principal investigator: Urszula Gryczka, M.Sc.

6. Radiometric methods applied in hydrometallurgical processes development and optimization. No. 18945

principal investigator: Prof. Andrzej G. Chmielewski, Ph.D., D.Sc.

- Silicide/silicate coatings on zirconium alloys for improving the high temperature corrosion resistance. No. 19026
 - principal investigator: Bożena Sartowska, Ph.D.
- 8. Recovery of uranium and accompanying metals from various types of industrial wastes. No. 18542

principal investigator: Katarzyna Kiegiel, Ph.D.

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9.	Electron beam for preservation of biodeteriorated cultural heritage paper-based objects.		
	No. 18493		
	supervisor: Dagmara Chmielewska-Śmietanko, M.Sc.		
10			

- 10. Synthesis of 50 g synthesized nanotracer material by sol-gel process including a detailed report with the methodology and characterization of the material. No. 201506067-VG supervisor: Tomasz Smoliński, M.Sc.
- New cyclotron method for ⁴⁷Sc production and conjugation of ⁴⁷Sc to monoclonal antibodies. No. 20488

supervisor: Prof. Aleksander Bilewicz, Ph.D., D.Sc.

 Cyclic ^{99m}Tc isolation from the gamma irradiated ¹⁰⁰Mo target. No. 22521 supervisor: Przemysław Koźmiński, Ph.D.

PROJECTS WITHIN THE FRAME OF EUROPEAN UNION PROGRAMME HORIZON 2020

1. Accelerator research and innovation for European science and society (ARIES). GA 730871 principal investigator: Prof. Andreai C. Chroiolauski, Ph.D. D. So

principal investigator: Prof. Andrzej G. Chmielewski, Ph.D., D.Sc.

2. Characterization of conditioned nuclear waste for its safe disposal in Europe (CHANCE). GA 755371

principal investigator: Prof. Grażyna Zakrzewska-Kołtuniewicz, Ph.D., D.Sc.

- **3. GEN IV integrated oxide fuels recycling strategies (GENIORS).** GA 755171 principal investigator: Prof. Jerzy Narbutt, Ph.D., D.Sc.
- European tools and methodologies for an efficient ageing management of nuclear power plant cables (TeamCABLES).
 GA 755187

principal investigator: Grażyna Przybytniak, Ph.D., D.Sc., professor in INCT

OTHER INTERNATIONAL RESEARCH PROGRAMMES IN 2017

 Nanostructured porous materials with tailored properties (with Joint Institute for Nuclear Research, Dubna, Russia).
 Contract No. 04-4-1131-2017/2021

supervisor: Bożena Sartowska, Ph.D.

- Studies on oxidation resistance in water and on radiation resistance of zirconium silicide coatings on zirconium alloys (with Joint Institute for Nuclear Research, Dubna, Russia). Contract No. 04-5-1131-2017/2021 supervisor: Wojciech Starosta, Ph.D.
- New nanocomposite sorbent for different radionuclides removal (with Joint Institute for Nuclear Research, Dubna, Russia).
 Contract No. 04-4-1131-2017/2021
 supervisor: Dagmara Chmielewska-Śmietanko, M.Sc.
- 4. Measurement of the depth profiling of the cesium atom on the surface of the adsorbent for its recovery from the spent nuclear waste by time-of-flight elastic recoil detection analysis (TOF-ERDA). No. 20152054 (Central European Research Infrastructure Consortium project) supervisor: Danuta Wawszczak, Ph.D.

ERASMUS+ PROGRAMME

- Joint innovative training and teaching/learning program in enhancing development and transfer knowledge of application of ionizing radiation in materials processing. No. 2014-1-PL01-KA203-003611
- **2. Mobility for learners and staff higher education student and staff mobility.** Key action 103
- 3. Inter-institutional agreement 2015-2017 between institutions from programme and partner countries (China).

Key action 107

4. Inter-institutional agreement 2017-2019 between institutions from programme and partner countries (USA).

Key action 107

ANALYSES FOR THE MINISTRY OF ENERGY

1. Analysis "Treatment of nuclear waste and burnt nuclear fuel in Poland – present situation and perspectives".

Contract No. 156/II/P/15004/4390/17/DEJ-244/062/2017 (together with ZUOP Radioactive Waste Management Plant)

2. Analysis of non-nuclear applications of ionizing radiation and recommendations for domestic industrial companies.

Contract No. 149/II/15004/4390/17/DEJ

LIST OF VISITORS TO THE INCT IN 2017

- 1. D'Angelantonio Mila, Institute of Polymers, Composites and Biomaterials (IPCB), National Research Council (CNR), Bologna, Italy, *18-22.04.2017*
- 2. Dzeba Iwa, Ruđer Bošković Institute, Zagreb, Croatia, 26.03-01.04.2017
- 3. Gryzłov Anatoly, TORIJ, Russia, 13-22.03.2017
- 4. Günal Aytaç, Anadolu University, Turkey, 24.07-29.09.2017
- 5. Kyaw Myat, International Atomic Energy Agency, Myanmar, 31.05-1.08.2017
- 6. Nadarayah Manivannan, Brunel University London, the United Kingdom, 14-15.03.2017
- 7. Orelovitch Oleg, Joint Institute for Nuclear Research, Dubna, Russia, 4-10.09.2017, 13-16.09.2017
- 8. Pesnekhonov Vladimir, Przedsiębiorstwo Naukowo-Produkcyjne TORIJ, Russia, 13-22.03.2017
- 9. Pillai Surech D., Texas A&M University/Texas A&M AgriLife Research, Texas, USA, 26-30.06.2017
- 10. Tietze-Jaensch Holger, Forschungszentrum, Jülich, Germany, 13-14.12.2017
- 11. Wamadera Balachandran, Brunel University London, the United Kingdom, 14-15.03.2017
- 12. Zaw Min Min, International Atomic Energy Agency, Myanmar, 17-25.06.2017

THE INCT SEMINARS IN 2017

- Marek Danilczuk, Ph.D. (Institute of Nuclear Chemistry and Technology, Warszawa, Poland; Bowling Green State University, Ohio, USA)
 Badania spektroskopowe membran jonowymiennych stosowanych w ogniwach paliwowych (Spectroscopic studies of ion exchange membranes for fuel cells applications)
- Łucja Dziawer, M.Sc. (Institute of Nuclear Chemistry and Technology, Warszawa, Poland) Biokoniugaty nanocząstek złota jako nośniki ²¹¹At w celowanej terapii promieniowaniem alfa (Bioconjugates of gold nanoparticles as ²¹¹At carriers for targeted alpha therapy)
- **3.** Ireneusz Janik, Ph.D. (Notre Dame Radiation Laboratory, University of Notre Dame, USA) Termochemia wiązań połowicznych i jej związek z mechanizmem przekształceń wolnorodnikowych (Thermochemistry of odd-electron bonds in relation to free radical transformation)
- 4. Prof. Anna Lankoff, Ph.D., D.Sc. (Institute of Nuclear Chemistry and Technology, Warszawa, Poland) Biopaliwa I i II generacji – korzyści i zagrożenia (First and second generation biofuels – adavantages and disadavantages)
- Prof. Janusz Lipkowski, Ph.D., D.Sc. (Cardinal Stefan Wyszyński University in Warsaw, Poland) Chemia supramolekularna – w pół wieku od prapoczątków (Supramolecular chemistry – half a century from the origin)
- 6. Nadarajah Manivannan, Ph.D. (Brunel University London, London, the United Kingdom) Non-thermal plasma technologies for the marine diesel engine exhaust emission control
- 7. Andrzej Pawlukojć, Ph.D., D.Sc., professor in INCT (Institute of Nuclear Chemistry and Technology, Warszawa, Poland; Joint Institute for Nuclear Research, Dubna, Russia) Zastosowanie neutronowej spektroskopii oscylacyjnej do badania oddziaływań w ciele stałym (Application of the neutron oscillation spectroscopy in the studies of interaction in solid state)
- 8. Prof. Suresh D. Pillai (College of Agriculture & Life Sciences, Texas A&M University/Texas A&M AgriLife Research, Texas, USA) The status of electron beam irradiation technology in N. America and the role the National Center for Electron Beam Research plays in commercializing the technology
- 9. Prof. Suresh D. Pillai (College of Agriculture & Life Sciences, Texas A&M University/Texas A&M AgriLife Research, Texas, USA) The research and outreach program at the National Center for Electron Beam Research as it relates to environmental quality
- 10. Prof. Suresh D. Pillai (College of Agriculture & Life Sciences, Texas A&M University/Texas A&M AgriLife Research, Texas, USA) The research and outreach program at the National Center for Electron Beam Research as it relates to foods
- 11. Prof. Suresh D. Pillai (College of Agriculture & Life Sciences, Texas A&M University/Texas A&M AgriLife Research, Texas, USA)

The research and outreach program at the National Center for Electron Beam Research as it relates to vaccines, advanced materials including packaging materials

12. Rafał Walczak, M.Sc. (Institute of Nuclear Chemistry and Technology, Warszawa, Poland) Radionuklidy skandu dla radiofarmaceutyków teranostycznych (Scandium radionuclides for teranostic radiopharmaceuticals)

13. Iga Zuba, M.Sc. (Institute of Nuclear Chemistry and Technology, Warszawa, Poland)

Podstawowa procedura oznaczania chromu metodą radiochemicznej neutronowej analizy aktywacyjnej (RNAA) i jej zastosowanie w procesie certyfikacji materiałów odniesienia dla nieorganicznej analizy śladowej (Primary procedure for the determination of chromium by radiochemical neutron activation analysis (RNAA) and its application in certification of reference materials for inorganic trace analysis)

LECTURES AND SEMINARS DELIVERED OUT OF THE INCT IN 2017

LECTURES

1. Bobrowski K.

Chemia radiacyjna cieczy (Radiation chemistry of liquids).

Dissemination of the textbook "Applications of ionizing radiation in materials processing" in the frame of the project entitled "Joint innovative training and teaching/learning program in enhancing development and transfer knowledge of application of ionizing radiation in materials processing" of Erasmus+KA2 programme, Warszawa, Poland, 25.05.2017.

2. Brykała M.

Synteza ziaren ditlenku uranu dotowanych wybranymi pierwiastkami przy zastosowaniu kompleksowej metody zol-żel (CSGP) (Synthesis of spherical grains of uranium dioxide doped with different elements by complex sol-gel process (CSGP)).

Konferencja naukowa "Polska nukleonika w drugiej dekadzie XXI wieku", Warszawa, Poland, 27.05.2017.

3. Brykała M., Wawszczak D., Olczak T.

Sol-gel for radiochemistry and nuclear medicine.

1st Joint CNRS/PAN Workshop: Nuclear Medicine & Radiochemistry, Gif-sur-Yvette, France, 11-12.12.2017.

4. Chmielewski A.G.

IChTJ – atom dla społeczeństwa (The INCT – atom for society). Konferencja "Promieniujemy na całą gospodarkę – polski przemysł dla elektrowni jądrowej", Warszawa, Poland, 30.01.2017.

5. Chmielewski A.G.

Hybrid biogas – an electron beam system for electricity and bio-fertilizer production. EuCARD-2 (Enhanced European Coordination for Accelerator Research and Development) Annual Meeting, Glasgow, UK, 28-30.03.2017.

6. Chmielewski A.G.

Science and education in the nuclear project in Poland. Seminar "Nuclear energy: educational policy", Warszawa, Poland, 16.05.2017.

7. Chmielewski A.G.

Environmental applications of electron accelerators – gaseous pollutants. Summer School "Application of accelerators for radiation processing", Warszawa. Poland, 19-23.06.2017.

8. Chmielewski A.G.

Rola chemii radiacyjnej i radiochemii w rozwoju bezpiecznej energetyki jądrowej (The role of radiation chemistry and radiochemistry in the development of safe nuclear power).

Jubileuszowa Sesja Naukowa Polskiego Towarzystwa Badań Radiacyjnych im. Marii Skłodowskiej--Curie "Promieniowanie w nauce, technologii, medycynie i środowisku naturalnym", Warszawa, Poland, 29.06.2017.

9. Chmielewski A.G.

Fostering industrial development through radiation technology. International Conference on the IAEA Technical Cooperation Programme: Sixty years and beyond – contributing to development, Vienna, Austria, 30.05.-01.06.2017.

10. Chmielewski A.G.

INCT at ARIES WP3. ARIES WP3 Kick-off Meeting, Kraków, Poland, 13-14.09.2017.

11. Chmielewski A.G.

New applications of low energy electron beams. ARIES WP3 Kick-off Meeting, Kraków, Poland, 13-14.09.2017.

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32. Zimek Z.

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SEMINARS

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Reaktory energetyczne (Energy reactors).

Warsaw University of Technology, Faculty of Chemical and Process Engineering, Warszawa, Poland, 21.04.2017.

2. Brzóska Kamil

Promieniowrażliwość wewnątrzkomórkowa i jej potencjalne markery (Intrinsic radiosensitivity and its potential markers).

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3. Chmielewski Andrzej G.

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4. Chmielewski Andrzej G.

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5. Gajda Dorota

Analiza możliwości pozyskiwania uranu dla energetyki jądrowej z zasobów krajowych (Analysis of the possibility of uranium supply from domestic resources). University of Szczecin, Szczecin, Poland, 11.01.2017.

6. Kołacińska Kamila

Analiza kosztów i korzyści wdrożenia energetyki jądrowej w Polsce (Cost and benefit analysis of nuclear power energy in Poland).

SGH Warsaw School of Economics, Collegium of Business Administration, Warszawa, Poland, 3.11.2017.

7. Lankoff Anna

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Siedlce University of Natural Sciences and Humanities, Faculty of Sciences, Siedlce, Poland, 20.10.2017.

8. Pruszyński Marek

Nowe metody znakowania przeciwciał monoklonalnych radionuklidami jodu (New methods of monoclonal antibodies labelling with iodine radionuclides).

National Centre for Nuclear Research, Otwock-Świerk, Poland, 29.09.2017.

9. Pruszyński Marek

Nanociała – nowe wektory jako nośniki dla radionuklidów diagnostycznych i terapeutycznych (Nanobodies – new vectors for diagnostic and therapeutic radionuclides). National Centre for Nuclear Research, Otwock-Świerk, Poland, 18.10.2017.

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AWARDS IN 2017

- Synthesis method of radiotracer and a radiotracer Gold medal with mention at the International Warsaw Invention Show IWIS 2017, Warszawa, Poland, 9-11.10.2017
 Tomasz Smoliński, Partyk Wojtowicz, Andrzej G. Chmielewski
- 2. A method of reducing high concentrations of nitrogen oxides present in the exhaust gases from diesel engines

Gold medal at the International Warsaw Invention Show IWIS 2017, Warszawa, Poland, 9-11.10.2017 Andrzej G. Chmielewski, Janusz Licki, Yongxia Sun, Ewa Zwolińska, Zbigniew Zimek, Aleksandra Błocka

- Method and system for the transport and mixing of biomass in hydrolyzer and in the fermenter Gold medal at the International Warsaw Invention Show IWIS 2017, Warszawa, Poland, 9-11.10.2017 Adam Kryłowicz, Janusz Usidus, Andrzej G. Chmielewski, Kazimierz Chrzanowski
- A method for hygienization Gold medal at the International Warsaw Invention Show IWIS 2017, Warszawa, Poland, 9-11.10.2017 Andrzej G. Chmielewski, Jacek Palige, Otton Roubinek, Zbigniew Zimek, Urszula Gyczka, Janusz Usidus, Krzysztof Pietrzak, Rob Edgecock
- Method for obtaining diagnostic amounts of ⁴³Sc radionuclide Gold medal at the International Warsaw Invention Show IWIS 2017, Warszawa, Poland, 9-11.10.2017 Aleksander Bilewicz, Rafał Walczak, Agnieszka Majkowska-Pilip
- A process for the preparation of diagnostic amounts of ^{99m}Tc radionuclide Gold medal at the International Warsaw Invention Show IWIS 2017, Warszawa, Poland, 9-11.10.2017 Aleksander Bilewicz, Magdalena Gumiela
- Wysokowydajna polska technologia uzyskiwania biometanu z wytwarzaniem nawozu organicznego (High efficient Polish technology of obtaining biomethane with organic fertilizer production) Gold Laurel of Innovation in the 7th edition of Stanisław Staszic competition for the best innovative products organized by the Polish Federation of Engineering Associations, Warszawa, Poland, 14.11.2017 Institute of Nuclear Chemistry and Technology
- A method of reducing high concentrations of nitrogen oxides in the exhaust gases from diesel engines Bronze medal at the International Trade Fair "Ideas – Inventions – New Products" iENA 2017, Nuremberg, Germany, 2-5.11.2017
 Andrzej G. Chmielewski, Janusz Licki, Yongxia Sun, Ewa Zwolińska, Zbigniew Zimek, Aleksandra Błocka
- 9. Andrzej Waksmundzki medal awarded by the Committee of Analytical Chemistry, Polish Academy of Sciences for the achievements in chromatography **Marek Trojanowicz**
- Bioconjugates of gold nanoparticles with trastuzumab labelled with ²¹¹At for internal alpha theraphy Young Scientist Award for oral presentation at the Wiley Award Session of the 22nd International Symposium on Radiopharmaceutical Sciences, Dresden, Germany, 14-19.05.2017 Łucja Dziawer
- 11. Pre-treatment of radioactive waste using destructive processes of organic compounds followed by concentration of radionuclides

iia scientific award in recognition for contributing to the advancement of the science of ionizing radiation at the NUTECH-2017 International Conference on Developments and Applications of Nuclear Technologies, Kraków, Poland, 10-13.09.2017

Dorota Gajda

12. Irradiation of wastewater from Solvay process

iia scientific award in recognition for contributing to the advancement of the science of ionizing radiation at the NUTECH-2017 International Conference on Developments and Applications of Nuclear Technologies, Kraków, Poland, 10-13.09.2017

Marcin Sudlitz

- The removal of NO_x and SO₂ from exhaust gases using a hybrid electron beam method The first prize in the category of best oral presentation at the NUTECH-2017 International Conference on Developments and Applications of Nuclear Technologies, Kraków, Poland, 10-13.09.2017 Ewa Zwolińska
- Electron beam for preservation of water-damaged paper The second prize for the best poster presentation at the NUTECH-2017 International Conference on Developments and Applications of Nuclear Technologies, Kraków, Poland, 10-13.09.2017 Marta Wójcik
- 15. Uzasadnienie ekonomiczne wdrożenia Programu Polskiej Energetyki Jądrowej (Economical aspects of nuclear power programme in Poland)

Second degree award of the Polish Nuclear Society for the best master's thesis in 2016 concerning nuclear sciences

Kamila Kołacińska

16. Generowanie rodników utleniających i ich reaktywność w wytypowanej grupie cieczy jonowych (Generation of oxidizing radicals and their reactivity in ionic liquids) Distinction of the Polish Nuclear Society for the best doctoral thesis in 2016-2017 concerning nuclear sciences

Małgorzata Nyga (INCT Ph.D. student)

17. First degree team award of Director of the Institute of Nuclear Chemistry and Technology in 2017 for publications concerning the application of flow measurements and modern nanotechnology to improve methods of chemical analysis

Marek Trojanowicz, Kamila Kołacińska

18. Second degree team award of Director of the Institute of Nuclear Chemistry and Technology in 2017 for a series of original scientific publications concerning the fuel cycle and the search for alternative sources of fissile materials

Grażyna Zakrzewska-Kołtuniewicz, Katarzyna Kiegiel, Agnieszka Miśkiewicz, Dorota Gajda, Ewelina Chajduk

- Second degree team award of Director of the Institute of Nuclear Chemistry and Technology in 2017 for publications on the current problems of air pollution, presenting the practical solutions for reduction of sulphur and nitrogen oxides emissions in the air
 - Andrzej G. Chmielewski, Yongxia Sun, Ewa Zwolińska
- Third degree team award of Director of the Institute of Nuclear Chemistry and Technology in 2017 for two publications on cable aging processes in nuclear power plants Grażyna Przybytniak, Jacek Boguski
- 21. First degree team award of Director of the Institute of Nuclear Chemistry and Technology in 2017 for the application achievements in 2015-2016 Technetium-99m labelled antibiotics for healing bacterial infections

Ewa Gniazdowska, Przemysław Koźmiński

22. Second degree team award of Director of the Institute of Nuclear Chemistry and Technology in 2017 for the application achievements in 2015-2016 – Analytical procedures for radionuclides and heavy metals in inorganic fertilizers

Halina Polkowska-Motrenko, Ewelina Chajduk, Jakub Dudek, Zbigniew Samczyński, Paweł Kalbarczyk, Iga Zuba

- Distinction of the first degree of Director of the Institute of Nuclear Chemistry and Technology in 2017 for the achieved progress in the preparation of Ph.D. thesis and professional activity
 Pawet Halik
- 24. Distinction of the second degree of Director of the Institute of Nuclear Chemistry and Technology in 2017 for the achieved progress in the preparation of Ph.D. thesis and professional activity, **Weronika Gaweda**

25. Distinction of the second degree of Director of the Institute of Nuclear Chemistry and Technology in 2017 for the achieved progress in the preparation of Ph.D. thesis and professional activity, including published articles

Kamila Kołacińska

- Distinction of the second degree of Director of the Institute of Nuclear Chemistry and Technology in 2017 for the achieved progress in the preparation of Ph.D. thesis and professional activity Rafał Walczak
- 27. Award of Director of the Institute of Nuclear Chemistry and Technology in 2017 for the activity in events promoting science, among others, Science Festival, Night of Museums Marcela Grabowska
- Award of Director of the Institute of Nuclear Chemistry and Technology in 2017 for the activity in events promoting science, among others, Science Festival, Night of Museums Marcin Sudlitz
- 29. Award of Director of the Institute of Nuclear Chemistry and Technology in 2017 for the activity in events promoting science, among others, Science Festival, Night of Museums **Ewa Zwolińska**
- Distinction of Director of the Institute of Nuclear Chemistry and Technology for outstanding achievements in the field of scientific publications Marek Trojanowicz

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