

**Chemical methods of uranium separation from
unconventional resources with regard to selected aspects
of the nuclear cycle**

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Attachement 4

**SUMMARY OF PROFESSIONAL ACCOMPLISHMENTS SUBMITTED FOR
THE HABILITATION PROCEDURE**

Warsaw, April 2023

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1. Name and surname:

Katarzyna Barbara Kiegiel

2. Academic diplomas and degrees:

- PhD in chemistry (20/05/1998)

Doctoral dissertation: Examination of asymmetric induction in the addition reaction of organometallic reagents to carbonyl group of (2R)-bornane-10,2-sultam derivatives

Supervisor: prof. dr hab. Janusz Jurczak, University of Warsaw, The Faculty of Chemistry

- MsC in chemistry (21/11/1994)

Master thesis: Asymmetric addition reaction of metallorganic reagents to (2R)-glyoxyloyl and (2R)-(phenylglyoxyol)-10,2-sultam

Supervisor: prof. dr hab. Janusz Jurczak, University of Warsaw, The Faculty of Chemistry

3. Information on employment in research institutes or faculties/departments or school of arts:

- 14/02/2011-31/04/2015 specialist, Institute of Nuclear Chemistry and Technology, Warsaw
- 01/05/2015-present senior scientist, Institute of Nuclear Chemistry and Technology, Warsaw
- 01/04/2000–31/07/2001 postdoctoral position, University of Kentucky, Department of Molecular and Cellular Biochemistry, Lexington, KY USA
- 1/10/1998-03/04/2000, assistant lecturer, University of Warsaw, The Faculty of Chemistry
- 01/12/1994.-31/07/1998 PhD studies, University of Warsaw, The Faculty of Chemistry

4. Description of the achievements, set out in art. 219 para 1 point 2 of the Act of 20 July 2018 Law on Higher Education and Science (Journal of Laws of 2020, item 85, as amended):

4.1 Title of scientific achievement:

Chemical methods of uranium separation from unconventional resources with regard to selected aspects of the nuclear cycle

4.2 List of scientific publications constituting the basis for scientific achievement

The scientific achievement, which is the basis for applying for the degree of a habilitated doctor, is the series of publications listed below. The series consists of seven original papers, three review papers, and one project report.

[H1] K. Frackiewicz, **K. Kiegiel***, I. Herdzik-Koniecko, E. Chajduk, G. Zakrzewska-Trznadel, S. Wolkowicz, J. Chwastowska, I. Bartosiewicz, *Extraction of Uranium from Low-grade Polish Ores: Dictyonemic shales and Sandstones*, NUKLEONIKA, 2012, 58(4), 451-459.

IF_{2012/2021} = 0.507/1.154 MNiSW_{2012/2021} points = 15/70

My contribution to this article was the planning and developing the concept of the article and performing part of the experimental work. I planned and performed leaching of sandstones and developed the results. I participated in the formulation of the conclusions. I also took a significant part in performing the analysis of the results, in the preparation of the presented graphs. Moreover, I participated in the preparation and editing of the text of the manuscript. I also participated in its editing.

[H2] **K. Kiegiel***, L. Steczek, G. Zakrzewska-Trznadel, *Application of calixarenes as macrocyclic ligands for Uranium(VI) - a review*, JOURNAL OF CHEMISTRY, 2013, Article ID 762819, 16 pages. <https://doi.org/10.1155/2013/762819>

IF_{2013/2021} = 0.622/3.241 MNiSW_{2013/2021} points = 15/40

My contribution to this article was the planning the outline of the publication, preparing a literature review, and writing a significant part of the manuscript: Introduction, Application of Calixarenes in Separation of Uranium(VI), The Potential Industrial Application of Calix[6]arenes for Uranium(VI), Conclusions. My work also involved preparing the drawings and conducting the publication process.

[H3] D. Gajda, **K. Kiegiel***, G. Zakrzewska-Kołodziej, E. Chajduk, I. Bartosiewicz, S. Wołkiewicz, *Mineralogy and uranium leaching of ores from Triassic Peribaltic Sandstones*, JOURNAL OF RADIOANALYTICAL AND NUCLEAR CHEMISTRY 2015, 303, 521-529. <https://doi.org/10.1007/s10967-014-3362-0>

IF_{2015/2021}= 0.983/1.754 MNiSW_{2015/2021}points =20/40

My contribution to this article involved: defining the research objective, planning the research, developing the concept of the article. I performed, in consultation with the other co-authors, the analysis and the discussion of the obtained results of the study. I wrote the manuscript of the article, including the summary and conclusions, except for the part describing the geological and mineralogical characteristics and the preparation of the graphs. I conducted the publication process.

[H4] **K. Kiegiel***, G. Zakrzewska-Kołodziej, D. Gajda, A. Miskiewicz, A. Abramowska, P. Biełuszka, B. Danko, E. Chajduk, S. Wołkiewicz, *Dictyonema black shale and Triassic sandstones as potential sources of uranium*, NUKLEONIKA, 2015, 60(3), 515-522. <https://doi.org/10.1515/nuka-2015-0096>

IF_{2015/2021}=0.585/1.154 MNiSW_{2015/2021}points =15/70

My contribution to this work consisted of conducting a significant part of the research work on leaching, extraction, and precipitation of uranium oxide precursors. I coordinated the execution of the experiments and developed the concept of the article. I collected literature and wrote a substantial part of the article. I held the publication process.

[H5] **K. Kiegiel***, A. Abramowska, P. Biełuszka, G. Zakrzewska-Kołodziej, S. Wołkiewicz, *Solvent extraction of uranium from leach solutions obtained in processing of Polish low grade ores*, JOURNAL OF RADIOANALYTICAL AND NUCLEAR CHEMISTRY 2017, 311, 589-598. <https://doi.org/10.1007/s10967-016-5029-5>

IF_{2017/2021} =1.181/1.754 MNiSW_{2017/2021}points =15/40

My contribution to this work was a significant contribution to the research work. I defined the scientific goal, planned the research, and developed the concept of the article. I prepared the literature review and wrote a significant part of the manuscript, including the summary (except for the part on the geochemical analysis), prepared tables, figures, in addition to graphs. I also carried out the publication process.

[H6] A. Abramowska, D. Gajda, **K. Kiegiel***, A. Miskiewicz, P. Drzewicz, G. Zakrzewska-Kołtuniewicz: *Purification of flowback fluids after hydraulic fracturing of Polish gas shales by hybrid methods*, SEPARATION SCIENCE AND TECHNOLOGY 2018 53(8):1207-1217. <https://doi.org/10.1080/01496395.2017.1344710>

IF_{2018/2021} =1.354/2.779 MNiSW_{2018/2021}points =25/40

My contribution to this work was contribution to the research work and the development of a technological scheme for the purification of fluids after fracturing and the carrying out of alpha spectrometric measurements. I defined the scientific goal and developed the concept of the article. I conducted, in consultation with the co-authors, the analysis and the discussion of the results obtained. I prepared the manuscript, including the summary, and conducted the publication process.

[H7] F. Reitsma*, P. Woods, M. Fairclough, Y. Kim, H. Tulsidas , L. Lopez, Y. Zheng, A. Hussein, G. Brinkmann, N. Haneklaus, A. R. Kacham, T. Sreenivas, A. Sumaryanto, K. Trinopiawan, N. Al Khaledi, A. Zahari, A. El Yahyaoui, J. Ahmad, R. Reyes, **K. Kiegiel**, N. Abbas, D. Mwalongo. E.D. Greaves, *On the Sustainability and Progress of Energy Neutral Mineral Processing*, SUSTAINABILITY 2018, 10, 235. <https://doi.org/10.3390/su10010235>

IF_{2018/2021} =2.592/3.889 MNiSW_{2018/2021}points =70/100

The paper is a project report from an international project coordinated by IAEA (IAEA's Coordinated Research Project "T11006", entitled "Uranium/Thorium Fuelled High Temperature Gas Cooled Reactor Applications for Energy Neutral and Sustainable Comprehensive Extraction and Mineral Product Development Processes"). This work discusses the sustainability of energy neutral mineral processing and provides an overview of the current progress of multinational research. My contribution to this work was the preparation of the concept of the article, discussion and consultation of the text. I participated in the preparation of the fragment on phosphogypsum and copper earths.

[H8] H.Tulsidas, S.Gabriel, **K. Kiegiel**, N.Haneklaus*, *Uranium resources in EU phosphate rock imports*, RESOURCES POLICY, 2019, 61: 151–156.

<https://doi.org/10.1016/j.resourpol.2019.02.012>

IF_{2019/2021} = 3.986/8.222 MNiSW_{2019/2021}points =140/140

My contribution to this work involved discussion and consulting the text of article.

[H9] **K.Kiegiel***, D.Gajda, G. Zakrzewska-Kołtuniewicz, *Recovery of uranium and other valuable metals from substrates and waste from copper and phosphate industries*, SEPARATION SCIENCE AND TECHNOLOGY, 2020, 55 (12):2099-2107.

<https://doi.org/10.1080/01496395.2019.1642356>

IF_{2020/2021} =2.475/2.779 MNiSW_{2020/2021}points =40/40

My contribution to this work involved proposing the topic of study, planning experiments, developing the concept of the article. I took part in the performance of the research work (leaching tailings from the copper industry in an autoclave, leaching phosphate rock, leaching phosphogypsum). I collected the literature, conducted, in consultation with the other authors, the analysis and the discussion of the results obtained, prepared the manuscript (apart from the graphs), and carried out the publication process.

[H10] **K.Kiegiel***, O. Rubinek, D. Gajda, P. Kalbarczyk, G. Zakrzewska-Kołtuniewicz, A. G. Chmielewski, *The studies on uranium recovery from U-bearing Radoniów dump*, NUKLEONIKA 2021 66(4):115–119. <https://doi.org/10.2478/nuka-2021-0017>

IF₂₀₂₁ =1.154 MNiSW₂₀₂₁points =70

My contribution to this work was developing the concept of the article. I conducted, in consultation with the other authors, the analysis and the discussion of the results obtained in the study. I prepared a significant part of the manuscript, including the summary and conclusions. I also conducted the publication process.

[H11] **K. Kiegiel***, I. Herdzik-Koniecko, L. Fuks and G. Zakrzewska-Kołtuniewicz, *Management of radioactive waste from HTGR reactors including spent TRISO fuel – state of the art*, ENERGIES 2022, 15(3), 1099. <https://doi.org/10.3390/en15031099>

IF₂₀₂₁ =3.252 MNiSW₂₀₂₁points =140

My contribution in this publication involved planning the outline of the publication, conducting literature analysis, preparing a significant part of the manuscript: Introduction, The types of waste Generated in HTGR Cycle, Methods of Chemical Characterization of TRISO Spent Nuclear Fuel, TRISO spent Fuel Management, Conclusions. I prepared drawings and carried out the publication process.

4.3 Description of the scientific achievement

Research objective

The development of nuclear power around the world is associated with constant increase in demand for uranium, which is a raw material for the production of nuclear fuel. Primary raw materials will continue to be the main source for global uranium production for a long time to come. However, unconventional resources may be considered as potential sources of uranium, the importance of that may increase as conventional sources become depleted in the future. The strategic concepts related to the exploitation of natural resources depend on the current state policy and its economic situation. In countries with only poor uranium resources, the analysis of the possibility of uranium extraction from unconventional sources is fully justified.

The nuclear fuel cycle is the series of processes and operations that consists of three stages: the production of nuclear fuel (called front-end), the use of fuel for the production of electricity (burn-up), and the final stage of the cycle which takes place after discharging the fuel from the reactor, i.e. storage and reprocessing or final storage (called back-end). The first stage of the cycle (front-end) includes both the extraction of ore and its chemical processing.

The main objective of the presented study was to analyze the possibility of uranium recovery from selected domestic deposits and industrial waste of various origins, including copper industry and phosphorus fertilizers production. The procedure of uranium extraction should be fitted to the specific composition of the raw material: uranium concentration, chemical forms of uranium, the type of uranium mineralization, uranium accompanying substances, etc. However, there are some general processes that are similar for both conventional and unconventional resources. The essential steps of the processing of uranium ores are: crushing, then leaching, concentration and purification of uranium solutions by solvent extraction or ion exchange chromatography, and precipitation to obtain final product “yellow cake” as polyuranate or uranyl peroxide followed by calcination step in which U_3O_8 is formed.

The solid-liquid extraction (leaching) is one of the crucial steps in the technology of uranium production from the uranium ores. It is well known that many factors, including pressure, particle size, the type of leaching solution (acidic or alkaline) and its concentration, liquid to solid mass ratio, pressure and temperature have a significant influence on the extraction

of uranium and others metals from raw material. The main aim of this study was to develop a suitable method for uranium recovery from scarce domestic resources and secondary resources. Uranium and all the metals accompanying uranium in the raw material could be present in the post-leaching solution. The uranium purification and concentration could be achieved by physical and chemical methods such as liquid-liquid extraction, ion exchange, or integrated processes ion exchange/liquid-liquid extraction. The precipitation of „yellow cake” (e.g. ammonium diuranate or uranium peroxide forms) is the most crucial step in forming triuranium octoxide used in production of the nuclear fuel. The studies performed in the laboratory scale provided the data allowing to prepare the technological scheme and then document titled "Process project of the installation for obtaining uranium from uranium ores" ending the POIG project [1].

In Poland, the construction of a nuclear power plant is intended to be one of the ways to reduce carbon dioxide emissions and meet the growing demand for electricity [2]. The motivation for the assessment of the possibilities of meeting the demand for uranium from domestic sources was the intended implementation of the nuclear energy and the high-temperature reactor technology to supply Polish industry with heat [3]. Nuclear fuel issues, presented in the Polish Nuclear Energy Program, are not limited only to the first stage of the fuel cycle. An important issue of the nuclear cycle is the management of the spent fuels and radioactive waste from the nuclear reactors. The first stage of the fuel cycle (front end) is closely related to its last stage ("back end"). Handling the fuel after being discharged from the reactor should be considered already at the stage of fuel preparation. The method of spent fuel management strictly depends on its initial composition and characteristics. In this regard, an analysis of spent fuel management was reviewed.

Introduction

The development of nuclear energy is influenced by economic, environmental, and social factors. Uranium is the main fuel in the nuclear reactors currently being used and is mainly obtained from natural resources. They are classified as conventional and unconventional (Fig. 1). Resources for which uranium recovery is economically viable, i.e. production costs being less than USD 130/kg of U_3O_8 , are considered conventional [4]. According to the OECD/NEA Red Book 2020, there are 6,147,800 tonnes of uranium resources worldwide. Rocks and materials with very low uranium content, in which uranium accompanies other valuable elements and it is

recovered from them as a minor-product in the extraction of the most important raw material, are referred to as the unconventional resources. Industrial waste containing uranium is also belongs to this group.

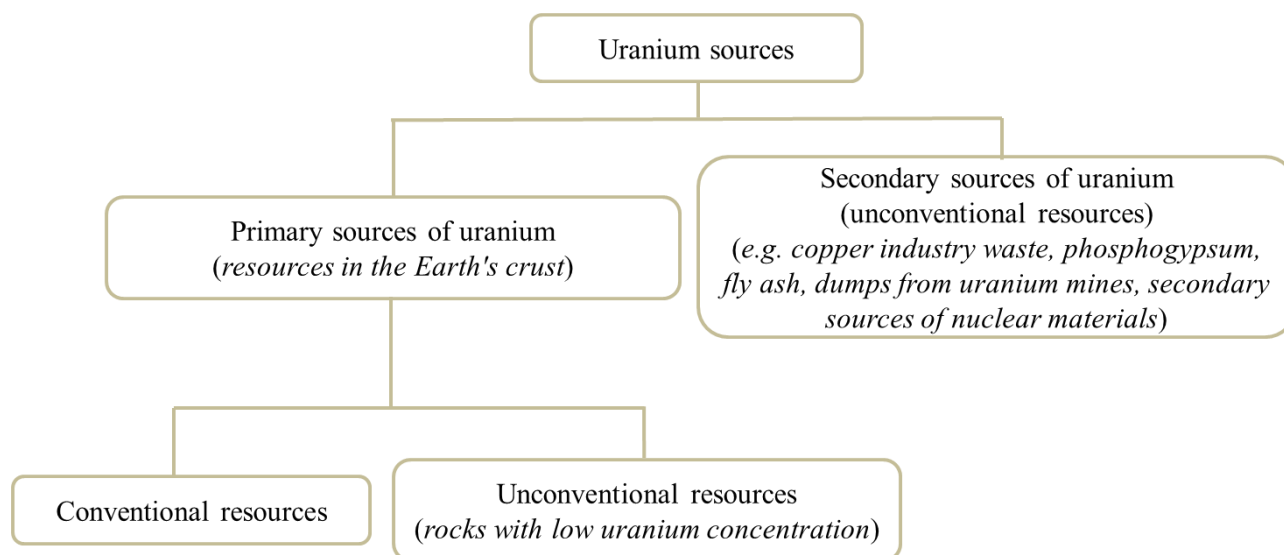


Fig. 1 Uranium sources

The unconventional resources are potential sources of uranium, the importance of which may increase with the depletion of the conventional sources of uranium in the future. The growing interest in the unconventional resources and the search for effective technologies that could be used in the exploitation of them is related to the expected increase in the demand for uranium in future. The International Atomic Energy Agency (IAEA) regularly holds conferences where each member state presents the volume of uranium deposits in the countries, including unconventional resources. It is assessed that unconventional resources cover about 39 million tonnes of uranium [4].

Furthermore, uranium recovery from unconventional sources is interesting in the context of research challenges to develop the technologies for obtaining uranium from the low-grade ores and secondary raw materials. Secondary raw materials, such as the above-mentioned copper industry waste, phosphoric acid, and fly ash are gaining more and more interest as a potential source of uranium. Many research laboratories around the world have conducted research on the extraction of uranium from secondary raw materials. Research on this topic carried out at the Institute of Nuclear Chemistry and Technology follows the world trends. recovery of heavy

metals from industrial waste extremely important for the environment, industry and for the public health.

The characteristics of the material originating from various ores vary significantly from deposit to deposit. The effect of ore mineralogy on uranium release is not well-understood. The procedure of uranium extraction must be designed to fit specific characteristics of the ore. The basic steps of processing the uranium ores, that can be adapted to the processing of industrial waste, are crushing and grinding, leaching, solid-liquid separation, ion exchange or/and solvent extraction, and finally the precipitation of the uranium oxide precursor (U_3O_8) (Fig. 2) [5,6]. Leaching with sulphuric acid is the predominant process for recovery of uranium from the rocks [7,8]. The acid consumption depends on the ore composition. In the case of limestone rocks, the major consumers of acid are carbonates and this method is not efficient for this kind of materials. The advantage of alkaline leaching is its uranium selectivity and less corrosivity than acid leaching [9,10]. Bioleaching that is an economical and environmentally friendly process, can be successfully used to leach low-grade uranium ores [11].

Uranium in the 6+ oxidation state is more soluble in aqueous solutions, therefore it will leach from the ore more easily than uranium in the 4+ oxidation state. Therefore, the first step in uranium leaching process is the oxidation of uranium to uranium (VI) form.

The scope of the carried out research covered stages related to both the first (front-end) and the final stage of the nuclear fuel cycle (back-end):

- selection and analysis of materials using in the studies,
- leaching of uranium and accompanied metals from the tested raw materials (solid-liquid extraction),
- separation of uranium from other metals present in the post-leaching solution,
- precipitation of uranium oxide precursors, and
- management of spent fuel.

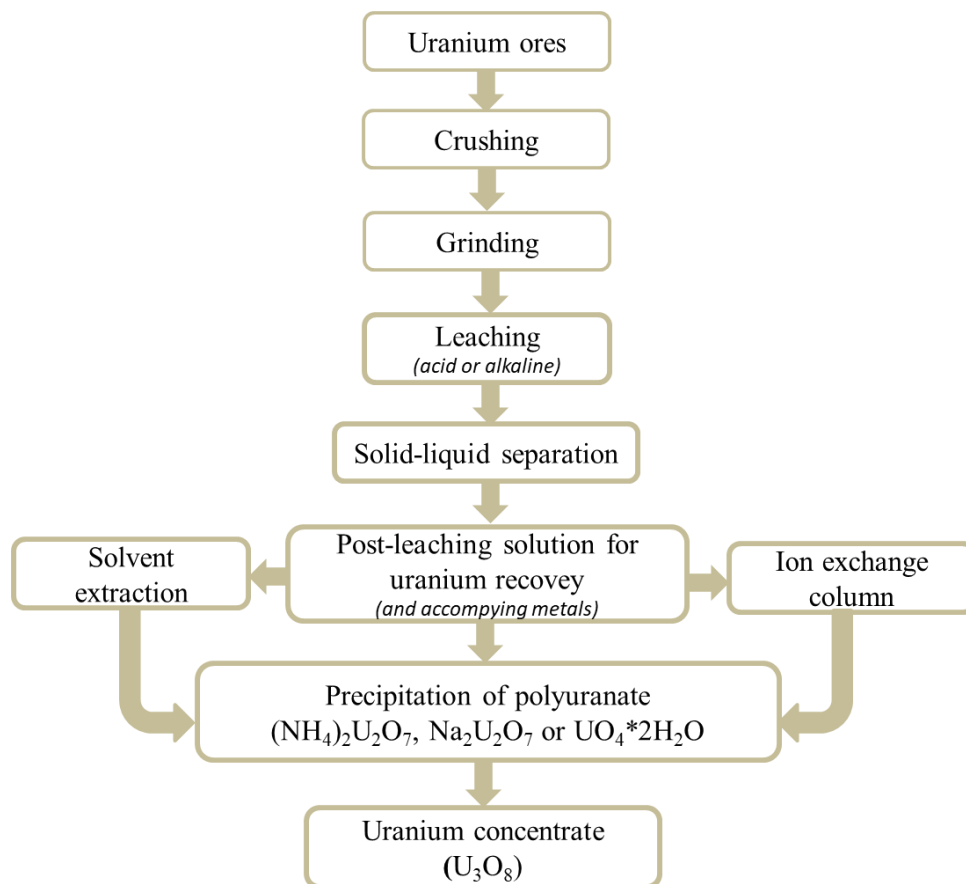


Fig.2 Brief scheme of treatments of uraniu ores

Selection of investigated materials and their chemical analysis

In Poland, there are only low-grade uraniu ores. However, it is known that a very profitable ore mines operate on the content of 300 ppm (e.g., Rossing in Namibia) and 126 ppm (Trekkopje in Namibia). According to assessments done by Polish Geological Institute – National Research Institute, the most prospective uraniu deposits are the Lower Ordovician Dictyonema shale of the Podlasie Depression with uraniu concentration of 41-215 ppm (Table 1) and Lower and Middle Triassic rocks of the central parts of the Peribaltic Syncline, where the uraniu concentration is within a very broad range of 4-1316 ppm (Table 1) [H4]. In the black shale beside uraniu, there are vanadium and molybdenum with relatively high concentrations, 100-2000 ppm and from several to 500 ppm, respectively. Shales are characterized by a high content of the organic matter (from a few to over 10% of TOC). Low uraniu content with high organic matter content causes technological difficulties in recovery of uraniu from these ores

[H1]. In sandstones, uranium is accompanied by vanadium (with the concentration from 33 ppm to 0.46%), selenium (with the concentration from <1 ppm to 0.43%), and lead (with the concentration from 6.8 ppm to 0.62%). Organic matter is also present in uranium mineralized rocks, but its content is low, rarely exceeding 0.1% [1].

Most European countries import phosphate rocks for the production of phosphorus mineral fertilizers [H8]. Phosphate rocks can contain significant amounts of uranium and rare earth metals. The metal content varies depending on the region of origin of the particular raw material, e.g. the average uranium content in phosphate rock from Morocco is 97 ppm, while the average uranium content in magmatic phosphate rocks from Russia is lower (~26,5 ppm). In 2017, the countries of the European Union required ~17,537 tons of uranium to operate nuclear reactors. Approximately 91.7% of the total required uranium was imported, 2.6% originated from EU countries and 5.7% can be imputed to savings from the use MOX fuel. The assessments show that about 2% of the required uranium could be obtained from imported phosphate rocks to the European Union countries in 2017. The benefits of uranium recovery from phosphates rocks used in the production of phosphoric acid are presented in the review article H8. The potential importance of uranium recovery from these minerals could be estimated based on the results of the leaching samples from phosphate rocks used at the Police plant to produce phosphoric acid. Furthermore, the composition and leaching of phosphogypsum collected from the Wiślinka heap, waste material from the production of phosphoric acid were analysed (Table 1) [H9].

Uranium is recovered in one of the largest copper mines in the world, Olympic Dam in Australia. According to the data from 2011, reserves of 146 million tons of ores containing 1.98%wt. Cu, 0.58kg/t U₃O₈, 0.69g/t Au and 4.01g/t Ag [12] could be found there. In Poland, the main center of copper mining is the Legnica-Głogow region, situated in the Lower Silesia Voivodeship. The content of uranium in the ore from this province ranges from 12.8 ppm to 360 ppm, with the average concentration around 60 ppm, with copper content 2% [13].

Table1. The content of selected metals in the examined raw materials determined by Inductively Plasma Mass Spectrometry (ICP MS)

Metal	Polish uranium ores			
	Dictyonema shales		Triassic sandstones	
	[ppm]		[ppm]	
U	41 - 215		4.2 – 1316	
Th	6.6 – 15		2 - 82	
Cu	142 - 620		14 - 111	
Co	<10 - 80		3.6 - 163	
La	31 - 62		4 - 53	
Yb	0 – 7		0,3 – 5.3	
V	805 – 2050		37 - 770	
Mo	10 - 270		0 – 9	
Fe	11500 – 78900		3700 – 180000	
	Phosphate rocks (<i>imported product</i>)			Phosphogypsum (Wiślinka)
	Location			
	Syria	Tunisia	Morocco	
	[ppm]	[ppm]	[ppm]	[ppm]
U	61	32	117	2
Th	1	5	4	4
Cu	13	5	39	5
Co	13	14	13	6
La	26	64	90	40
Yb	3	5	13	4
V	134	53	250	5
Fe	2911	3398	3880	2758
	Cooper ores (Legnicko-Głogów District)	Cooper flotation tailing (Lubinski District)	Cooper tailing pond (Gilów)	The material from the post-mining Small Dump (Radoniowo)
	[ppm]	[ppm]	[ppm]	[ppm]
U	27	15	5	747-801
Th	8	5	2	12-14
Cu	44975	27500	12225	12-14
Co	427	558	79	18-21
La	26	21	8	24-29
Yb	no data	no data	no data	3
V	1370	788	55	47-52
Fe	24500	no data	4750	23200-25200

In **H9**, the study of leaching of copper ores originated from the deposit in the Legnicko-Głogów District and flotation tailing from the copper industry as well as waste from the Gilów tailing pond (Table 1) was discussed. The processes involved in recovery of uranium and production of phosphoric acid and copper are very similar. Some of the stages are common for the extraction of major product and uranium (ore mining, crushing, leaching, solid-liquid separation). This could reduce the overall cost of obtaining both of the above-mentioned materials.

In first years after the second World War, there was intensive mining of Polish deposits of uranium, mainly in the Sudets. Material containing uranium concentration of less than 0.2% was dumped in heaps, many of which still exist, which is a serious ecological problem. Securing and monitoring heaps generates significant costs. Recovery of uranium and other valuable metals would support finding solution to the problem. The material investigated in this study originated from the Radoniów Small Dump (the heap formed close to the mine operating in 1954-1960). The chemical analysis of the collected material showed uranium content of 747-800 ppm (Table 1). The article **H10** shows the possibility of using this ore as a secondary source of uranium.

The world literature provides information on uranium recovery from oil shale [14]. Studies on the possibility of exploiting shale gas have been conducted in Poland in 2010-2017. The shale gas is released from the rock by using special technology, that is hydraulic fracturing, which is also capable to extract various rock components, including valued metals e.g. uranium. The analysis of the uranium content in randomly selected flowback fluids from fracturing of Polish rocks showed that the maximum uranium concentration was 3,5 ppm [15]. Similarly to seawater (the concentration of uranium about 3.3 ppb), flowback fluids can be considered as a potential source of uranium, especially in countries where shale gas is exploited on a large scale. The quantity of leached uranium depends not only on the geological properties of the rocks but but also on the composition of the fracking fluid. The fluid contains organic matter and is also characterized by high salinity (even 100 g/L). This can be the reason for difficulties of the chemical analysis of such samples. It is necessary to dilute saline samples for the measurement of metals using ICP-MS. The accuracy of the analysis diminishes and the required concentration of the sample for the detection of the individual elements increases. Therefore, alpha and gamma spectrometry were employed for characterization of the sample of the fluid. (Table 2) [**H6**]. Uranium was determined by alpha spectrometry, other radioactive

elements were determined by gamma spectrometry. The conducted analysis of various tailing materials involved two methods: alpha spectrometry and ICP MS. The results were compared. They were presented in the post-conference materials of the NORM2020 conference [16].

Table 2. The content of radionuclides in flowback fluid

Radionuclide	A/A_{tho}
U-238*	41
U-234*	2.8
Ra-226**	8
Pb-214**	11.5
Bi-214**	6
Pb-212**	6
Tl-208**	2
K-40**	1

* Radioactivity in the relation to underground and groundwater $A_{tho}= 14 \text{ Bq/m}^3$ (U-238) and $A_{tho} = 150 \text{ Bq/m}^3$ (U-234)

** Radioactivity in the relation to typical ground water natural background around the well pad $A_{tho}= 0,04 \text{ Bq/L}$.

The first stage of purification of the flowback after hydraulic fracturing of gas shales is filtration by using mechanical filters which remove solid particles from waste water. Then the organic soluble matter is removed. The flowback fluid free of organics can be directed into the cascade of ion-exchangers, a case which has been presented in the technological diagram in the article **H6**. Anion and cation beds are used for removal of selected anions and cations. At this stage some of the metals selected for recovery could be separated and among them, uranium.

Energy neutral mineral processing ores, such as phosphate rocks, cooper ores, and rare earth elements ores, are the sources of recovery of unconventional uranium as a minor-product in the extraction of the most important raw material. Then, uranium is enriched and used to produce nuclear reactor fuel (Fig. 3). This issue was discussed in **H7**.

Energy neutrality is reached if the extracted uncenvontional uranium is used to generate energy equivalent to or larger than the amount of energy required for mineral processing of the primary ore and uranium extraction, conversion, enrichment, and fuel processing.

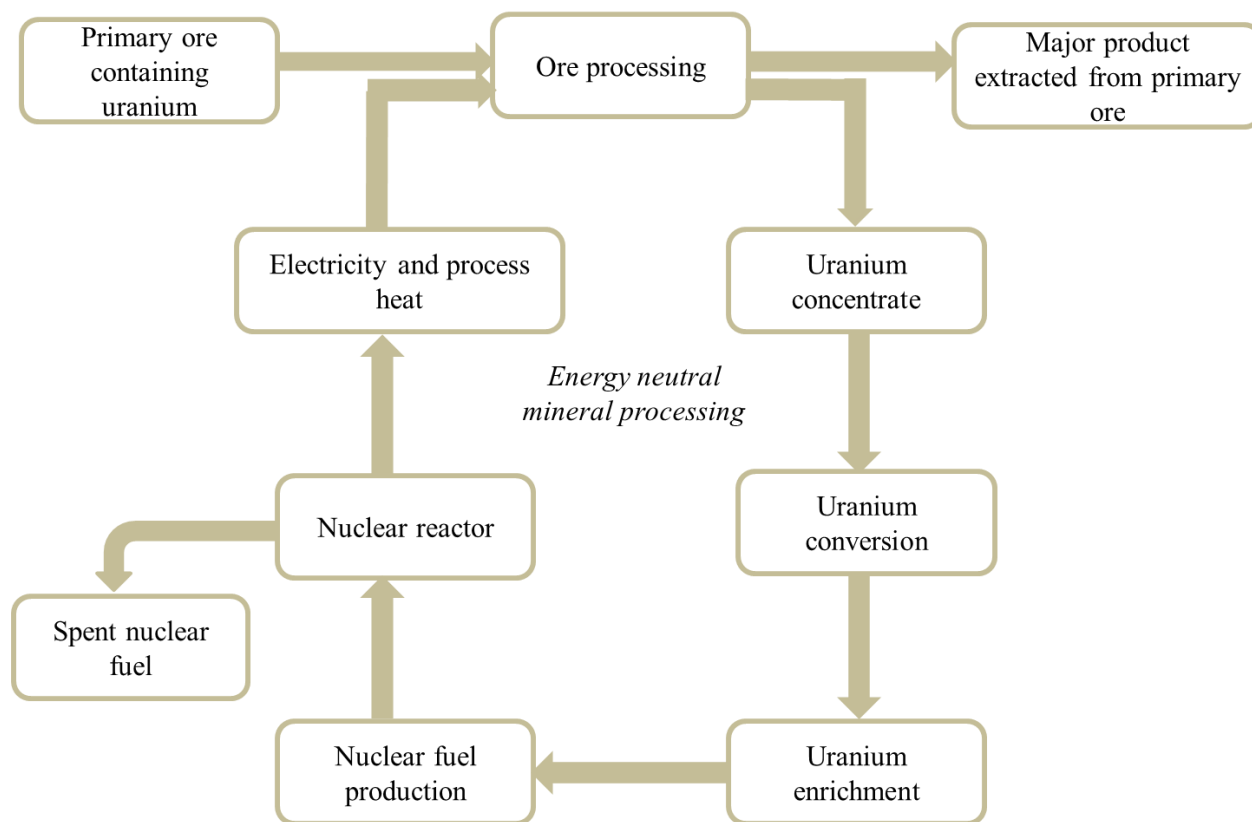


Fig. 3 Brief overview of energy neutral mineral processing

At the moment, the major part of mineral processing operations are powered by burning fossil fuel. In addition to renewable energy sources nuclear power may be a viable option to provide the large amounts of electricity and process heat required for mineral processing operations. Currently, nuclear power plants use uranium fuel (and possibly thorium fuel in the future), whereas relevant amounts of rare earths (REE) are required for the production of renewable energy sources. The demand for these elements may be partly or - in the case of energy neutral mineral processing - entirely covered by unconventional resources. It is an economical solution for the processing of raw materials containing various valuable ingredients, not all of which are present in concentrations, which justify their separate production.

Uranium leaching (*solid-liquid extraction*)

The solid-liquid extraction is a very important stage in the technology of uranium production from the uranium ores. Many factors such as temperature, pressure, the particle size, type of the leaching solution and its concentration have a significant effect on the extraction of

uranium and the others metals. The influence of all these factors on the uranium leaching efficiency from dictyonema shales and triassic sandstones was examined in **H1** and **H3**. The aim of **H9** was to develop a method of uranium recovery and other valuable metals from secondary resources-industrial waste from drom phosphoric acid and copper industries. Five different materials were examined as a potential source of uranium: phosphate rocks, phosphogypsum, copper ore, flotation tailing from the copper industry and waste from Gilów, tailing pond (Poland). The **H10** reports the possibility of uranium recovery from the post-mining uranium ore dump in Radoniów (Poland) by bioleaching method.

Prior to leaching, the samples of the investigated material were crushed and ground in the mill. Preliminary experiments were performed for dictyonema shales fractions of various particle sizes: 0-0.2 mm, 0.2-0.4 mm, 0.4-0.63 mm, 0.63-0.8 mm and 0.8-1 mm. There was no influence of the particle size on the leaching efficiency in 0-1 mm range. Similarly, no influence of the particle size on leaching efficiency was observed in the experiments of sandstones processing [**H3**]. The Dictyonema shales are more difficult to uranium leaching than sandstones because uranium is closely associated with organic matter. For this reason, it was necessary to conduct the calcination of dictyonema shales at 550°C during 4 hours before the leaching process. The non- calcined sample leaching with 10% H₂SO₄ at 80°C proceeded with 35% efficiency and leaching prior calcined sample proceeded with 64% [**H1**]. The samples of sandstones that contained less organic matter (below 0.1%) were not pre-treated in the oven.

Temperature plays a significant role in the leaching process. In the present study the efficiency of uranium leaching increased slightly with the range of 30-80°C. [**H1,H3**]. The optimal uranium leaching temperature from dictyonemic shales was 80°C and for sandstones 60°C. The selection of proper solid to liquid ratio (the weight of ores to volume of lixiviant) is important for optimization of the leaching proces. In this study, the optimum solid to liquid ratio was found to be 1:8 [**H3**]. The yield of uranium extraction decreased with increase of density of the slurries.

In the present work various oxidizing agents were tested. In the acid leaching none significant difference in effectiveness of the oxidizing agent was observed. In alkaline leaching, KMnO₄ and H₂O₂ were found to be the most efficient oxidizers [**H3**]. The influence of pressure in the range 1-7 bars was not significant either [**H1**]. The leaching efficiency of **uranium was in the range 59-64%** and lanthanum 58-65%.

The experimental work showed that sandstones were more readily leachable in comparison with the dictyonema shales. The best results of acid leaching of dictyonema shales were found for leaching with 10% H₂SO₄ during 8 hours at 80°C. (Fig. 4). **The average efficiency of metals leaching from different ore materials was in the range of U 64- 81%, Th 67-80%, V 25-52%, Mo 33-78%, Cu 28-52% and La 31-66% [H1].** The leaching of sandstones with 10% H₂SO₄ was carried out at 60°C during 1 hour. **Uranium was leached with the efficiency 71-100%**; efficiency of leaching other metals were: Th: 13-62%, Cu: 10-67%, Co: 8-57%, La: 24-60%, V: 28-58%, Yb: 26-67%, and Fe: 11-47% [H3]. The high uranium leaching efficiency was also observed in the process conducted with 10% HCl (Fig. 5).

It is worth noting that the alkaline leaching was more selective in respect of uranium than acidic leaching. In alkaline post-leaching solutions only three or two metallic components of the ores were detected: U, Mo and V (Dictyonema shales), or U and small amounts of V (sandstones). In the case of sandstones, 57-92% of uranium and 2-22% of vanadium were leached with a mixture of sodium carbonate and bicarbonate. In the case of calcinated samples of Dictyonema shales, the uranium was extracted with 42% efficiency, molybdenum with 24%, and vanadium with ca. 8% efficiency [H3]. It should be noted that rare earth elements (REEs) were found in the acid post-leaching solutions. REEs are on the list of the raw materials classified as critical by the European Commission in 2020 [17].

One of the stages before further processing is the solid-liquid phase separation. These two processes, leaching and separation, could be conducted in one apparatus with the use of the membrane contactor with Couette-Taylor helical flow [H4]. The experiments were performed at ambient temperature. The effectiveness of uranium leaching was compared to the results obtained from leaching process conducted in conventional manner, in a stationary reactor at 80°C. Such an approach results in reduction of total cost of the operation with no consequences to the separation efficiency.

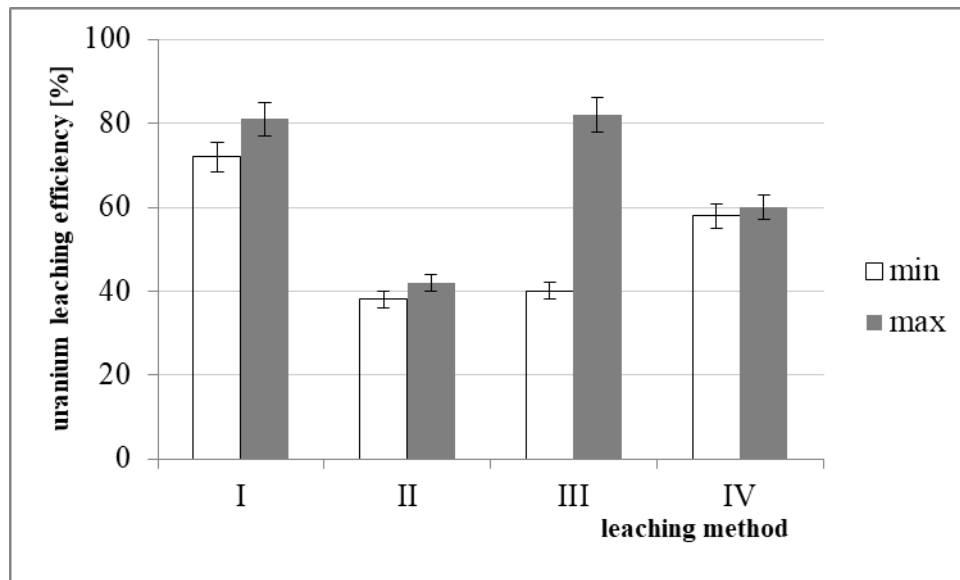


Fig. 4 Efficiency of leaching uranium from dictyonema shales using different methods: **I**-calcinated sample, lixiviant: 10% H₂SO₄, liquid/solid ratio of 8:1 (vol./wt. basis), oxidizing agent: MnO₂, 80°C, 8 h; **II**: calcinated sample, lixiviant: 5% Na₂CO₃/5% NaHCO₃, liquid/solid ratio of 8:1 (vol./wt. basis), oxidizing agent: MnO₂, 80°C, 8 h; **III**: “acid-cure”: 2 g of ground uranium ores were treated with 95% H₂SO₄ for 18 days, 25°C, 8 h; **IV**: sintering process with addition of 10% NaCl at 840°C during 3 h, than leaching with 5% H₂SO₄, MnO₂, 80°C, 8 h.

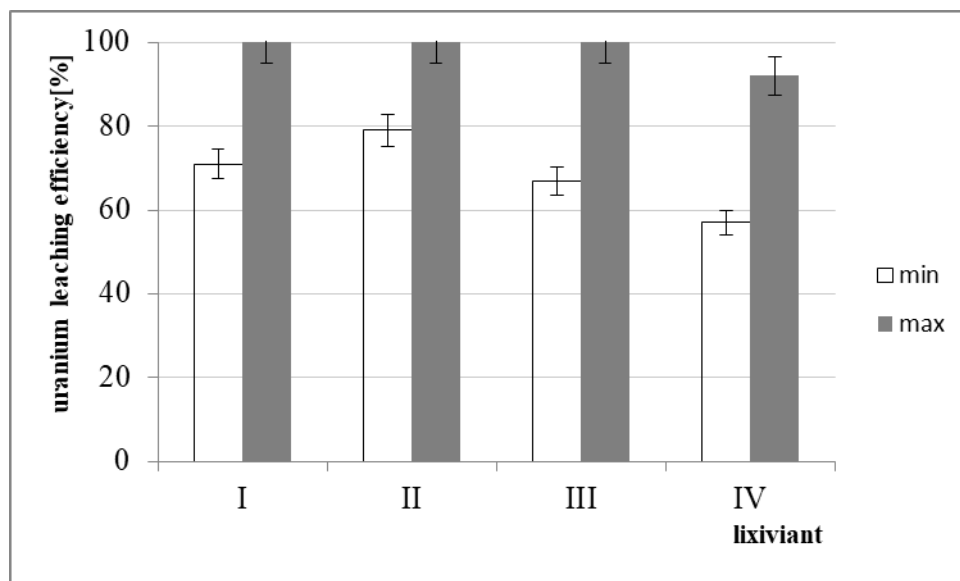


Fig. 5 Efficiency of leaching uranium from sandstones by various lixiviants, liquid/solid ratio of 8:1 (vol./wt. basis), **A**: 10% H₂SO₄, oxidizing agent: MnO₂, 60°C, 1 h; **B**: 10% HCl, oxidizing agent: 30% H₂O₂, 60°C, 1 h; **C**: 8% NaOH/ 18% Na₂CO₃, oxidizing agent: 30% H₂O₂, 60°C, 1 h; **D**: 5% Na₂CO₃/5% NaHCO₃, oxidizing agent: KMnO₄, 60°C, 1 h.

The characteristics of phosphate rocks depend vastly on the origin of the sample. The procedure of uranium extraction should be fitted to the specific composition of the rock. In this study three samples of phosphates were examined, the rocks originating from Morocco, Tunisia, and Syria. It was observed that characteristic of phosphates rocks are varying from source to source. Therefore, the procedure of uranium extraction should be design to fit to the specific characteristic of rocks. The materials used in experiments were subjected to the leaching process using alkaline and acidic lixivants. [H9]. The effect of time and temperature on the uranium leaching efficiency was investigated (Fig. 6). The acidic leaching (sulphuric acid or hydrochloric acid) of phosphates proceeded with high efficiency. In some cases it reached **even 100%**. Alkaline leaching of phosphates was also tested. It is worth noting that Syria and Tunisia phosphates were not leachable in alkaline conditions at room temperature. **Uranium from Morocco phosphate was leached with 66% yield.**

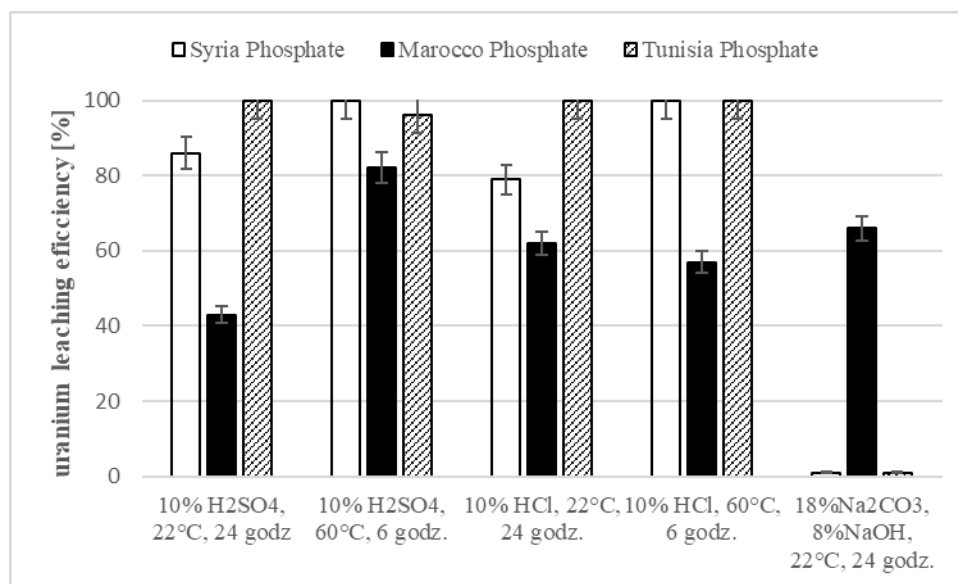


Fig. 6 Effect of various lixivants and temperature on leaching efficiency of phosphate rocks from Syria, Morocco and Tunisia. Liquid/solid ratio of 8:1 (vol/wt. basis), oxidazing agent 30% H₂O₂.

Uranium passes from the solid to the solution by acidic leaching with very high efficiency. About 80% of uranium is transferred to phosphoric acid during the sulphuric acid treatment of the phosphate rock. Thus, industrial phosphoric acid, the intermediate product in the production process of phosphoric fertilizers, could be also considered as a potential source of uranium. Apart from uranium, phosphate rocks also contain significant amounts of rare earth

elements that leach poorly with sulfuric. They are left in phosphogypsum, that is a waste from the production of phosphoric acid. The concentration of uranium in phosphogypsum in Wislinka is 2 ppm (Table 1). However, the concentration of lanthanides in this waste is significant and it reaches in some layers even 40 ppm. The main objective of **H9** was to find the optimal conditions for uranium recovery. Moreover, leaching solutions were also analyzed to estimate lanthanide recovery efficiency.

Despite the difficulties in leaching of phosphogypsum by different agents, it was found that phosphogypsum could be leached using the double-stage procedure. In the first stage phosphogypsum was treated with mixture of 30% NaOH and 30% H₂O₂ as an oxidant at 22-60°C. The solid residue was separated from the solution, washed with demineralized water, and dried at 105°C. The obtained solid was leached using 10% HCl in the presence of 30% H₂O₂ at 60°C. **Uranium** and rare earth elements **were extracted with quite high efficiency (U - 66% and La - 81%)** (Fig. 7).

The alternative method of leaching metals from phosphogypsum is the sintering leaching process. Phosphogypsum was sintered with NaOH, NaCl or Na₂CO₃, followed by treatment with concentrated H₂SO₄ (Table 4). In these conditions, **uranium leaching efficiency reached 66%** (Table 3, entry 4). Lanthanides were leached with low efficiency that did not exceed 15%.

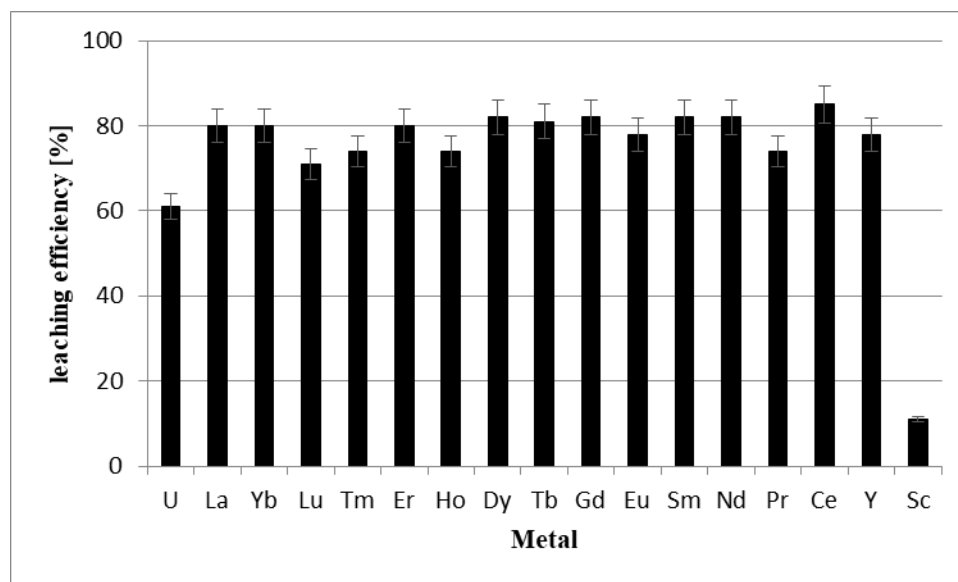


Fig. 7 The leaching efficiency of uranium and rare earth elements from phosphogypsum. Process conditions: 1st step: 30% NaOH, 30% H₂O₂, 22°C-60°C, 2nd step: 10% HCl, 30% H₂O₂, 60°C.

Table 3 The recovery of metals from phosphogypsum (PG) by sintering with NaOH, NaCl or Na₂CO₃ followed by acid leaching with concentrated H₂SO₄.

Lp	Process condition	Sintering leaching efficiency [%]															
		U	Th	La	Ce	Pr	Nd	Sm	Eu	Gd	Tb	Dy	Hp	Er	Tm	Yb	Lu
1	PG/NaOH 1:7 ^{a)}	9	3	10	1	1	1	1	1	1	1	1	1	1	1	1	1
2	PG/NaOH 1:5 ^{b)}	43	9	10	2	3	3	2	2	3	3	2	2	2	2	2	2
3	PG/NaOH 1:3 ^{c)}	13	9	3	3	3	3	3	3	5	3	3	3	4	4	4	4
4	PG/NaCl 10:1 ^{d)}	66	9	13	1	1	1	1	1	1	1	1	1	1	1	1	1
5	PG/Na ₂ CO ₃ 10:1 ^{e)}	21	5	4	3	2	1	1	2	2	1	1	2	1	1	1	1

Process conditions: a) 1st step: 500 °C, 3h; 2nd step: conc. H₂SO₄, 90°C, pH 1.8, 4h; b) 1st step: 500°C, 3h; 2nd step: conc. H₂SO₄, 100°C, pH 1.8, 4h; c) 1st step: 280°C, 3h; 2nd step: conc. H₂SO₄, 100°C, pH 1.8, 4h; d) 1st step: 700°C, 3h; 2nd step: conc. H₂SO₄, 60°C, pH 1.8, 4h; e) 1st step: 700°C, 3h; 2nd step: conc. H₂SO₄, 60°C, pH 1.8, 4h.

There are known cases in the world when uranium is recovered as a by-product from the copper extraction. Uranium is recovered in one of the largest copper mines in the world, the Olympic Dam in Australia. In Poland, the main center of copper mining is the Legnica-Głogow region, situated in the Lower Silesia Voivodeship. The content of uranium in the ore from this province is within the range 12.8-600 ppm, the average being around 60 ppm, with a copper content 2%. Polish copper deposit could only be considered as prognostic or prospective limited resources. Recovery of uranium from copper tailings could reduce the environmental impact of the production facilities.

The results of analysis of the drawn copper ore samples [**H9**] do not represent the average value of the whole deposit. Similarly, the samples of copper tailing may not represent the real average value of the whole tank.

Leaching of uranium from a copper ore sample using the double leaching method proceeded with an efficiency below 40%. There were tested also two types of the copper waste: copper flotation tailing from the Lubinski District and samples from Gilów tailing pond (Table 1). The efficiency

of leaching uranium was not satisfactory. **Uranium from copper flotation tailing** (Lubinski District) **was leached with minor efficiency, 12%**, while the leaching efficiency of **uranium from Gilów waste was higher, 67%**. (Fig. 8). Better results were obtained by leaching copper flotation tailings under elevated pressure and temperature. In such conditions, the uranium leaching efficiency increased from 12 to **53%** (Fig. 9).

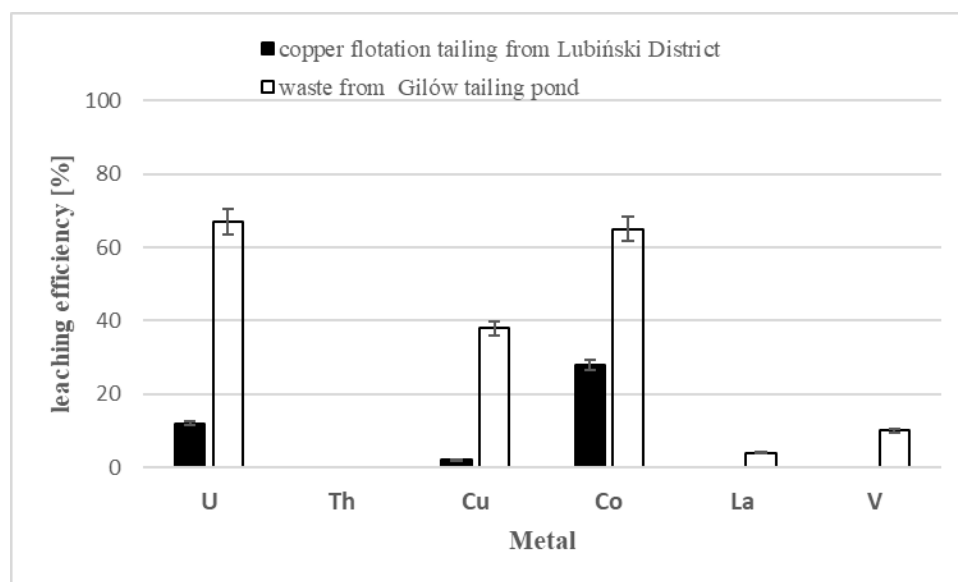


Fig. 8. The leaching efficiency of metals from copper waste: flotation tailings and waste from Gilów tailing pond. Process conditions: 1 bar, 60°C, 10% H₂SO₄, 30% H₂O₂, 2 h.

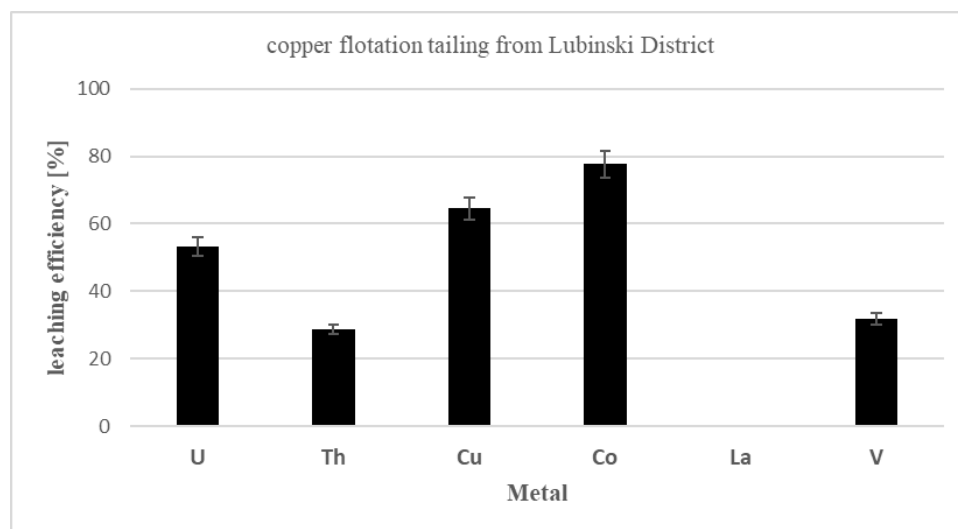


Fig. 9 The leaching efficiency of metals from copper waste: flotation tailings from Lubinski District. Process conditions: 5 bar, 60°C, 10% H₂SO₄, 30% H₂O₂, 16 h.

In addition to well-developed methods that are already applied in the technology of recovery of uranium, the bioleaching of uranium from raw materials and industrial waste containing uranium can be also technologically available. Bioleaching is an economically advantageous process that is broadly used for recovery of metals from various materials. In this study, the post-mining uranium dump from Radoniów (the Western Sudetes, Poland) was considered as a potential secondary resource of uranium. The material investigated in this study originated from the Small Dump and the uranium concentration was about 747-800 ppm [H10]. The consortium of microorganisms isolated from the former mines was used for bioleaching. It was composed of following microorganisms: Bacillus, Pseudomonas, Spingomonas, Thiobacillus, Halothiobacillus, Thiomonas and Geothrix. The composition of KO bacteria medium used in the bioleaching process was following: $(\text{NH}_4)_2\text{SO}_4$ - 5 g/dm³, KCl – 0,166 g/dm³, KH_2PO_4 – 0,083 g/dm³, $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ – 0,830 g/dm³, $\text{Ca}(\text{NO}_3)_2$ – 0,024 g/dm³, pH 2. Bioleaching of uranium ores was carried out in the bioreactor with the capacity of 100 dm³. In the experiment, about **98% of uranium was leached** after 51 days of the process. In the second test the experiment was scaled up and bioleaching was carried out in the fixed-bed deposit to simulate heap leaching process. Bed dimensions were 100x120x24 cm. The mass of charge was 570 kg. Leaching was performed for 120 days. **The efficiency of uranium leaching reached 70%**. The obtained results are a base guidelines for the preparation of technical assumptions for the design a bioleaching installation for the processing of 30 tons of charge on a dump. The resulting technology can be implemented to extract uranium from post-mining dumps in the future.

The solid-liquid extraction is the first stage of uranium production and it is of the key importance for further processing stages of ores and other uranium-containing raw materials. Appropriate selection of parameters allows to control the efficiency of the process. It is important at this stage to extract as many metals as possible has an economic value. Among others, recovery of elements such as molybdenum, vanadium, and rare earth metals is taken into account. The leaching stage can also be selective for some components by appropriate selection of reagents. This gives some flexibility in designing the process and later the flow chart. The solid-liquid extraction is followed by the following steps: purification-concentration with ion-exchange resins or solvent-solvent extraction/re-extraction, and precipitation of the final product, the "yellow-cake".

Separation of uranium from other accompanying metals in the post-leaching solution

Since in most of uranium minerals uranium is accompanied by other heavy metals, post-leaching liquors usually contain a mixture of different metal ions that should be separated from uranyl ions (UO_2^{2+}). Solvent extraction is a versatile technique for separating ionic solutes. Solvent extraction of metal ions used usually chelating reagents dissolved in an organic solvent [18]. The aim of these studies was a selection of the extracting agents and exploring extraction conditions appropriate for recovery of uranium from acidic and alkaline post-leaching liquors that were obtained by leaching of Polish ores. Raw extractants, like e.g.: tributylphosphate (TBP), di(2-ethylhexyl) phosphoric acid (D2EHPA), trioctylphosphine oxide (TOPO), triethylamine (TEA), tri-n-octylamine (TnOA) were tested (Fig. 10).

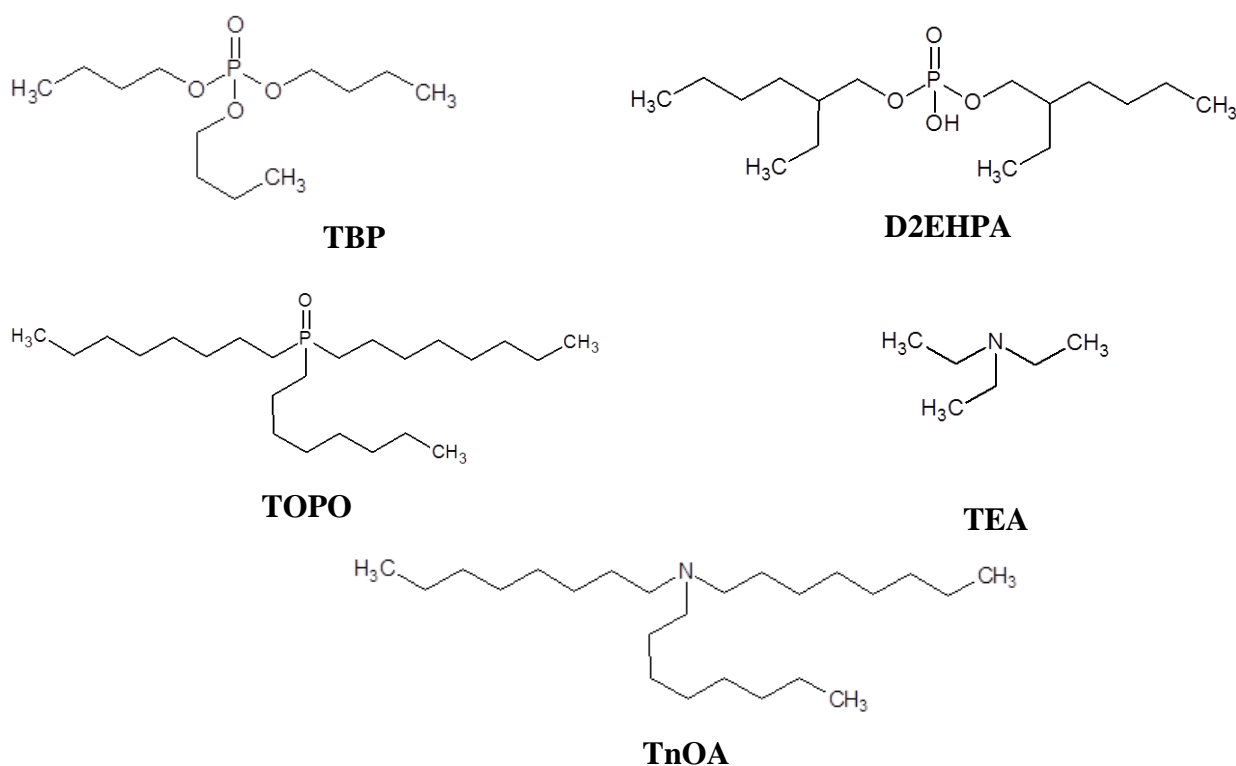


Fig. 10 The extracting agents tested for the separation of uranium from the solution

The separation of uranium from other metals present in the leach solutions and efficiency of their extraction were examined. The effect of the type of extractant, sulphuric acid, and uranium concentrations on the extraction process from model solutions was investigated.

The results of these experiments were further used for the extraction of uranium from real post-leaching liquors. The use of different reagents as strip solutions was also investigated. Stripping agents such as sodium carbonate solution, ammonium carbonate solution, sulphuric acids were tested for recovery of uranium from the organic phase [H5].

The extraction efficiency (%E) was calculated by the formula (1):

$$\%E=100 D_c/(D_c+V_{aq}/V_{org}) \quad (1)$$

where D_c is the distribution ratio, defined as the ratio of concentration of metal in the organic phase over its concentration in the aqueous phase, V_{aq} – the aqueous phase volume, V_{org} – the organic phase volume .

The stripping percentage, %S was determined by the relationship (2):

$$\%S=100 D_s/(D_s+V_{aq}/V_{org}) \quad (2)$$

where D_s is the distribution ratio of metal in the stripping phase over its concentration in the organic phase.

%R percent of recovery of uranium in the extraction/stripping process was determined by the relationship (3):

$$\%R=[\text{metal in the stripping phase}] / [\text{metal in post-leaching liquor}] \cdot 100\% \quad (3)$$

Based on the studies using a model solution, two extractants, TnOA and D2EHPA, were selected for the extraction of uranium from the post-leaching liquor. The further experiments showed that uranium from the solution obtained after ore leaching by sulphuric acid was extracted with high efficiency (99% for D2EHPA and 98% for TnOA). The yield of extraction of some metals other than uranium was also high (Th: 99%, Yb: 99% for D2EHPA and Th: 51%, V: 68% for TnOA).

Once the metal ions have been extracted by the organic phase, they should be stripped back by the aqueous phase. The experiments revealed that the stripping of uranium from organic phase containing D2EHPA with sodium carbonate was not possible because of the separation of NaD2EHP in the third phase. On the other hand, the efficiency of the stripping from the organic phase containing TnOA was not satisfactory; it was only 5-11%. Further research showed that it was possible to avoid the third phase formation when the extractions were carried out with a **synergistic mixture D2EHPA and TBP**. The obtained results were promising,

the extraction/stripping process was very efficient and recovery of uranium was more than 90% (Table 4).

Table 4 Effect of the extractant concentration and pH of initial aqueous phase on uranium extraction and stripping efficiency. ¹⁾

EXTRACTION			STRIPPING						
Concentration of synergistic extractants			0.5 M Na ₂ CO ₃		0.5 M (NH ₄) ₂ CO ₃		7 M H ₂ SO ₄		
[D2EHPA]:[TBP]	pH	%E	%S	%R	%S	%R	Lk23 oiS	%R	
0.2M:0.2M	6	99%	third phase was forming				-	-	
0.2M:0.2M	3	99%	88%	87%	97%	96%	29%	28%	
0.2M:0.2M	1	99%	93%	92%	94%	93%	-	-	
0.2M:0.07M	10	emulsion							
0.2M:0.07M	6	99%	third phase was forming				-	-	
0.2M:0.07M	1	99%	third phase was forming				34%	33%	
0.1M:0.1M	6	83%	64%	55%	82%	71%	47%	41%	
0.1M:0.1M	1	99%	99% ²⁾	98%	99% ²⁾	98%	65%	64%	

¹⁾ %E- the efficiency of the extraction experiment, %S- the efficiency of the stripping experiment, %R – the summarized efficiency of extraction and stripping experiments

²⁾ two days were needed for separation aqueous and organic phases

The addition of TBP into the organic phase was found to be essential for preventing the formation of the third phase during the alkali treatment of a solvent containing D2EHPA. The organic phase composed of 0.2 M D2EHPA and 0.2 M TBP with kerosene as a diluent is optimal for extraction of uranium at pH 1, in room temperature. The stripping of the organic phase is very efficient with 0.5 M ammonium (or sodium) carbonate solution. Recovery of uranium reached up to 98%. However, the single, one-stage, extraction of uranium from acidic post-leaching liquors is not sufficient to separate pure uranium (Fig. 11A). In the solution small amounts of other metals as lanthanum, vanadium and iron were detected. High-purity uranium is recovered from the alkaline post-leaching liquor (Fig. 11B).

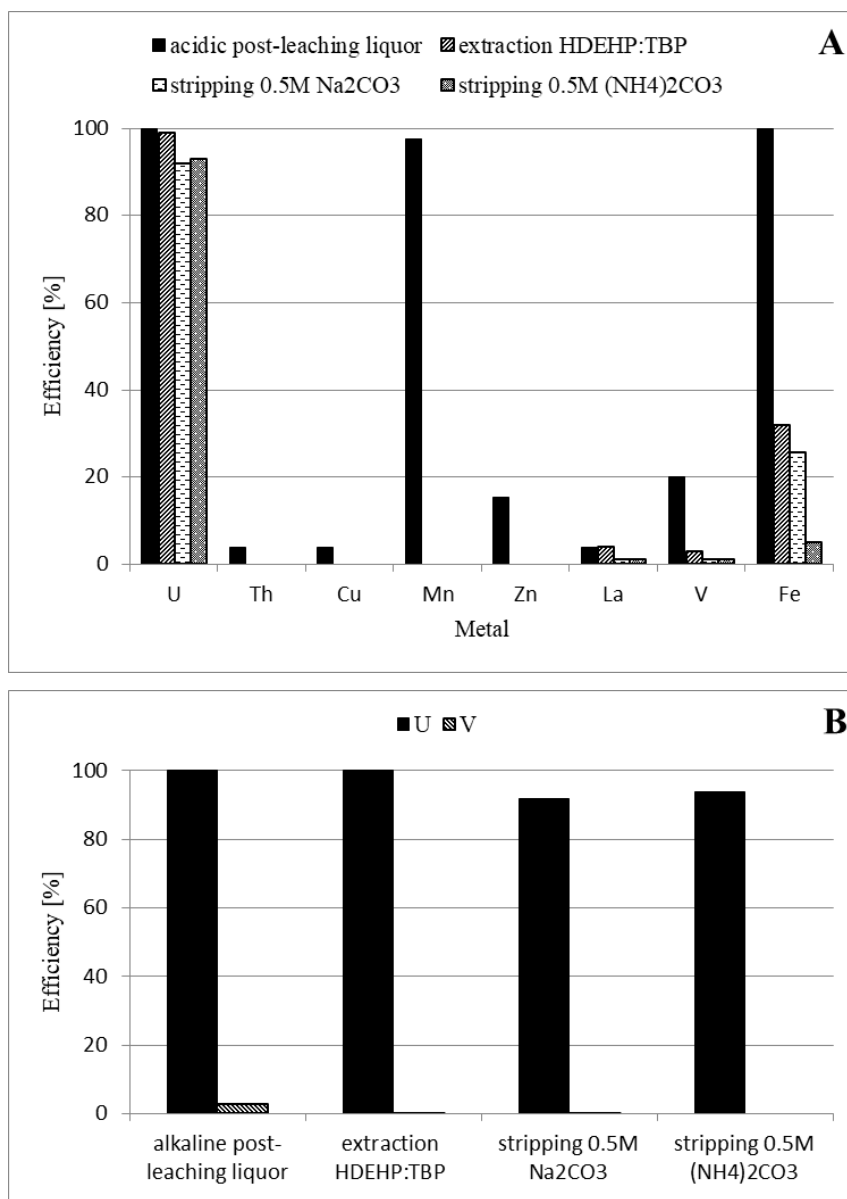


Fig. 11 Extraction and stripping efficiency of metals from: **A** - acidic post-leaching liquor and **B** - alkaline post-leaching liquor. Conditions: kerosene, V_{aq}/V_{org} 1:1, [D2EHPA]:[TBP] 0.2 M:0.2 M, $T=25^{\circ}\text{C}$.

Not much solvent extraction methods have been reported for uranium recovery from alkaline post-leaching solutions. The common problem was the third phase formation. The presented extraction of the pre-acidified alkaline post-leaching solution using a synergistic HDEHP-TBP mixture proposed in this study may contribute to solving this problem.

In the last years, increasing interest in calixarenes as potential complexing agents for metals, among them actinides, has been observed. Calixarenes are formed by *para*-phenolic units

linked by methylene bridges *ortho* to OH functions (Fig. 12). The ring takes a basket shape into which metal ions can be embedded to form a molecular complex. The conformation of calixarenes can be changed by functionalization of the hydroxyl groups at the lower (narrow) rim and introducing the groups in para-position on the upper (wide) rim. In that way, the affinity and the selectivity towards a specific molecule or ion can be changed. The review article [H2] covers the use of calixarenes as complexing agents for uranium(VI). The high effectiveness of calix[6]arenes in uranium(VI) separation process such as liquid-liquid extraction (LLE), liquid membrane (LM) separation and ion exchange was presented. They can be used for the extraction of uranyl ions from post-leaching solutions uranium ore, seawater, radioactive waste, or natural soil. The extraction study of lanthanides and actinides showed that the calixarenes bearing ligands including P=O groups were more efficient than TBP (tributyl phosphate), TOPO (trioctylphosphine oxide), CMPO (carbamoylphosphonate). The ligand concentration necessary to reach a given extraction yield was 10 to 100 times lower with the calixarenes than with the classical extractants.

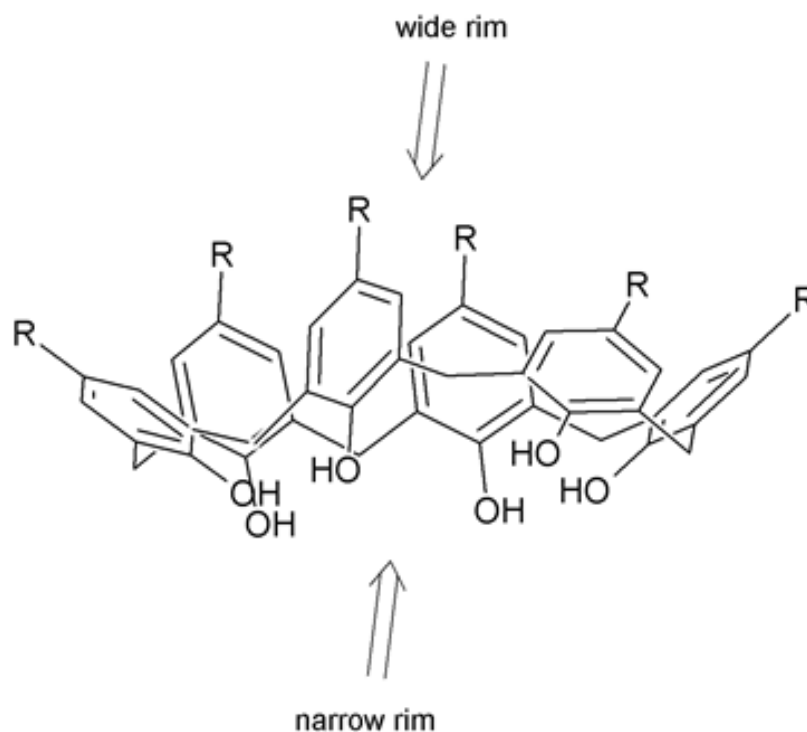


Fig.12. Illustration of the structure of calixarenes

An alternative approach to separation of uranium from other metals is the ion-ion exchange chromatography. In the presented study, the ion ion-exchange column filled with a basic anion exchanger (Dowex1 X8) was used. The complexes of uranium were eluted with 1 M sulfuric acid. **The efficiency of uranium recovery was almost quantitative 95-98%.** The molybdenum complexes are strongly fixed in anion exchange resin. They can be eluted only in part by 1 M sulfuric acid. Thus, the uranium fraction can be contaminated with molybdenum. It is worth noting that the purification of the acid pregnant solution from leaching of sandstones that does not contain the molybdenum provided a pure uranium fraction [H5]. In the process of uranium recovery from the post-mining uranium ore dump in Radoniów (Poland) by bioleaching method, significant amounts of iron ions were co-leached. Unfortunately, they did not separate completely on the ion exchange column [H10]. The presence of iron in the solution impeded precipitation of uranium salts, precursors of yellow cake - U_3O_8 , in next step.

Precipitation of uranium yellow cake

The production of yellow cake is the most crucial step in the preparation of fuel for use in nuclear power plants. This is because it is a substrate in the production of the pastilles filling fuel rod. Uranium present in acidic or alkaline solution produced by the ion exchange chromatography or solvent-extraction processes is typically precipitated as a polyuranate. The aim of preliminary studies was a selection of the precipitating agents and reaction conditions appropriate for recovery of uranium. The precipitation of three different forms of yellow cakes, $(NH_4)_2U_2O_7$, $Na_2U_2O_7$ and $UO_4 \cdot 2H_2O$ was conducted from the model uranium solution. The influence of temperature and concentration of uranyl ions in the solution were examined. The results were presented in the Annual Report 2014 [19]. The representative results were issued in H5. It is significant that ammonium diuranate was precipitated **from solutions containing low concentration of uranium (0.3-0.5 mg/mL)** with high efficiency **88-98%**. The efficiency of uranium peroxide precipitation was also high 99%, but the concentration of uranium in the starting solution was 1 mg/L. Ammonium diuranate and uranium peroxide can be easy to transform into uranium oxide (U_3O_8) by calcination. The precipitation of uranium salts is the subject of Polish Patent “Method for producing ammonium diuranate from the solutions with low uranium content” [20], that I am a co-author. The main advantage of the method of

precipitating ammonium diuranate from alkaline solutions with low uranium concentration proposed in the patent is the simplicity of the process in comparison to other methods that require much higher concentration of uranium.

The ammonium diuranate precipitation method developed in model solution studies was applied for the precipitation of uranium from the solution after the bioleaching of material from the post-mining uranium ore dump in Radoniów [H10]. The solution obtained after the purification uranium on ion exchange column filled with Dowex1 X8 contained iron ions. Therefore, the method for precipitating ammonium diuranate has been modified. In the first step of precipitation iron ions were removed from the solution with using 25% $\text{NH}_{3\text{aq}}$ at pH 4-5. The solution was agitated at 60°C during 1h. Then the obtained suspension was filtered. After that, 25% ammonia solution was added to the solution containing uranium up to first crystals formed (pH 8-10). The obtained mixture was heated for 4h at 60°C. After that, the suspension was cooled over the night. **Ammonium diuranate** was obtained with **a yield of 60%** and **iron separation from uranium** was conducted with **a yield of 99%**.

Spent nuclear fuel management

The series of processes and operations necessary to generate electricity from nuclear fuel, including its production, burn-up in a nuclear reactor, storage and reprocessing, or final disposal, is called the nuclear fuel cycle. The nuclear fuel cycle starts with uranium mining and ends with nuclear waste disposal. Reprocessing of spent fuel is an option for recovery material for nuclear power. Spent nuclear fuel, after depletion from the reactor, is submitted to further stages, such as temporary storage, reprocessing (recycling) or final disposal. These stages are known as the "back end" of the fuel cycle. Currently, there are two leading nuclear fuel cycle strategies: an open cycle (once-through cycle) and a closed cycle. In the closed cycle strategy, the spent nuclear fuel undergoes a sequence of chemical processes called reprocessing. Then, the recovered uranium and plutonium are reused in nuclear reactors. The advantages of the closed cycle is the significant reduction in the volume of high-level waste. In the view of the increasing demand for uranium and the depletion of the world's uranium resources, research into the use of a closed cycle is of great importance.

H11 is a review of radioactive waste management methods, based on the example of waste from high-temperature gas-cooled nuclear reactors (HTGR), including TRISO spent fuel. The construction of a HTGR reactor is planned in Poland [3]. HTGRs are characterized by efficient production of energy and providing process heat at high temperature used in many industrial processes. One of advantages of HTGRs over PWRs (pressurized water reactors) is the reduction of high-level radioactive waste (HLW) formation. Moreover, the burn-up of TRISO fuel (used in HTGR reactors) is higher than the fuel for PWR reactors. However, the higher U-enrichments are required in the production of TRISO fuel. The amounts of discharged heavy metal and plutonium elements in the fuel are much smaller for HTGR and these reactors require less frequent refuelling.

The most reasonable option for spent TRISO fuel management seems to be the open cycle, i.e. disposal of the spent fuel in a deep geological repository. It could be change in future in the context of increasing uranium demand, the depletion of the world resources, and perspectives of the global uranium market. Therefore, both nuclear fuel cycle strategies has been discussed in relation to TRISO spent fuel, **11**. Numerous methods used to ensure a sustainable, feasible management and long-term storage of HTGR nuclear waste for the protection of the environment and society were reviewed.

Summary and conclusions

The presented scientific achievement concerns chemical methods of uranium separation from unconventional resources, which are potential sources of raw material for the production of fuel used in nuclear power plants that do not emit CO₂. The recovery of uranium from from unconventional resources is also interesting research in the context of research challenges that aim to achieve energy neutrality in the processing of such raw materials.

The presented studies allowed preparing the set of methods and technology that could be implemented to extract uranium from poor ores and other unconventional raw materials. The solid-liquid leaching efficiencies of uranium and accompanying metals from the studied materials vary in a broad range, however for some samples they reached almost 100%. Very interesting results, in terms of selectivity and efficiencies of leaching were obtained in the process of leaching with alkaline solutions. It was showed that uranium and vanadium can be leached

selectively. However, in the process of acid leaching, all the elements associated with uranium in the raw material are obtained. The purification of uranium from accompanying metals was achieved by the ion-exchange chromatography or solvent-solvent extraction with satisfied results. In particular noteworthy is the method of liquid-liquid extraction, which works well in uranium recovery processes, also from solutions after alkaline leaching. effective methods of precipitating uranium compounds, U_3O_8 precursors, from solutions with a low percentage of uranium content. The conducted studies showed the feasibility of the proposed technology; however, its cost-effectiveness in current uranium supplies may be questioned. Nevertheless, **it is a significant contribution to the assessment of national uranium resources.**

The laboratory-scale studies allowed to develop a technological scheme for the production of “yellow cake” from Polish uranium ores. On the basis of the obtained data, **process project of the installation for uranium extraction from uranium ores** was prepared with a capacity of 100 kg of uranium ore per day [1].

In my opinion, **the studies described in this scientific achievement are an relevant element of scientific novelty and make a significant contribution to research on uranium extraction from unconventional sources.** The articles that are part of this achievement have been cited many times, what proves their impact on the world science.

It is worth noting that already at the stage of fuel preparation, it is necessary to consider the way of handling the spent fuel after it has been discharged from the reactor. However, the way of management of spent fuel after being discharged from the reactor should be considering already at the stage of fuel preparation. Therefore, there has been also included a reference to the end stage of the nuclear cycle, spent fuel storage and reprocessing or final disposal

In the case of low-grade uranium ores it is important to accomplish a detailed geo-economic analysis, the purpose of which is to accurately estimate the cost of the ore extraction. The analysis was performed for the "Rajsk" deposit and was presented in Chapter 4 of the monograph „Uranium - Safety, Resources, Separations and Thermodynamic Calculations” [21], which is not part of the cycle constituting my scientific achievement. The cost of exploitation of the deposit and extraction of uranium ore from Lower Ordovician dictyonema shale (the Podlasie Depression), estimated on the basis of the developed model, justifies

the statement that a unprofitable uranium extraction from this formation in a long-term perspective.

All the studied materials are generally considered as a potential source of uranium and other valuable metals. Nevertheless, the process of extraction of these metals is still difficult and costly. If these are to be ever considered as materials for the production of fuel for nuclear reactors, it is very likely that the process scheme will need to include the recovery of other valuable metals to improve the economics of such projects. The possibility of their practical use should be considered not only in the context of technological feasibility, but also in reference to the global situation on the uranium market. It is worth to note, that the main component of the cost of ore enrichment is the cost of electricity. The grinding stage shows high energy consumption. Industrial waste has the advantage that are already crushed.

The sum up, the most important achievement resulting directly from the presented studies is the development of a number of chemical processes consisting the scheme of uranium recovery from poor ores and secondary raw materials, what is a significant contribution to research on recovery uranium as a raw material for the production of nuclear fuel from unconventional sources.

Recovery of heavy metals and other useful materials from industrial waste is crucial as it reduces the environmental impact of the production facilities. One of the goals of the circular economy is to reduce the waste to the minimum. The uranium content of phosphogypsum proved to be very low. However, significant amounts of rare rare earth elements were found. I am currently conducting further research into recovery of rare earth metals from this material as a part of the ERAMIN-3 project Phosphogypsum Processing to Critical Raw Materials.

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5. Presentation of significant scientific or artistic activity carried out at more than one university, scientific or cultural institution, especially at foreign institutions:

5.1 International scientific cooperation

After the award the PhD degree

In 2000-2001, I worked as postdoctoral fellowship in the group of prof. H Peter Spielmann at the Department of Biochemistry at the University of Kentucky in Lexington, USA. I joined to the group realizing a project on the study of biological activity of farnesyl pyrophosphate analogues. The results of these works are two articles published in 2002 (Journal of the American Chemical Society) and 2004 (Bioorganic and Medicinal Chemistry Letters), in that I am a co-author.

In 2017, I started the scientific cooperation with Dr. Nilsem Haneklaus, currently employed by Freiberg University of Mining and Technology (TU-BAF), Germany. I am a co-author of three common publications, we submitted Horizon 2020 grant applications twice. In 2022, the ERAMIN-3 Phosphogypsum Processing to Critical Raw Materials project was positively evaluated and we are currently implementing it. I am also taking part in the mobility project *BeeWatch/Using Honeybees, Honey and Other Hive Products for Biomonitoring of Low-radioactive Phosphogypsum Tailings* coordinated by Dr. Nils Haneklaus, funded by the German Federal Ministry of Education and Research (BMBF).

In 2013-2016, I was the main contractor from side of INCT in the 7th EU framework project, Arcadia - Assessment of regional capabilities for new reactors development through an integrated. The aim of the project was to support the development of nuclear research programs in the New Member States of the European Union. The project was implemented by a consortium of 26 institutions from various EU countries.

In 2015-2019, I participated in the implementation of the research project coordinated by the International Atomic Energy Agency (IAEA) - IAEA's Coordinate Research Project T11006 entitled "Uranium/Torium Fueled High Temperature Gas Cooled Reactor Applications for Energy Neutral and Sustainable End-To-End Processes for Mining and Development of Mineral Products", as a team leader at IChTJ implementing the scientific project "Recovery of Uranium and Accompanying Metals from Various Types of Industrial Wastes" One of the results of the international cooperation is an article published in *Sustainability* in 2018, which is part of the cycle constituting the basis for my scientific achievement.

I participated in few meetings organized by IAEA, where I presented the results of research conducted at INCT

- First Research Coordination Meeting (RCM) on Uranium/Thorium fuelled High Temperature Gas Cooled Reactor Applications for Energy Neutral and Sustainable Comprehensive Extraction and Mineral Product Development Processes, 2-5 November 2015, IAEA, Vienna, Austria
- Second Research Coordination Meeting (RCM) on Uranium/Thorium fuelled High Temperature Gas Cooled Reactor Applications for Energy Neutral and Sustainable Comprehensive Extraction and Mineral Product Development Processes, 3-6 July 2017, IAEA Vienna, Austria-
- Third Research Coordination Meeting (RCM) on Uranium/Thorium fuelled High Temperature Gas Cooled Reactor Applications for Energy Neutral and Sustainable Comprehensive Extraction and Mineral Product Development Processes, 2-6 July 2018, IAEA Vienna, Austria
- Third Research Coordination Meeting on “Management of Wastes Containing Long-lived Alpha Emitters: Characterization, Processing and Storage” 12-15 October 2021, Vienna, Austria
- Technical Meeting on Advanced Fuel Cycles to Improve the Sustainability of Nuclear Power through the Minimization of High Level Waste, 17–19 October 2017, Vienna, Austria
- Technical Meeting on Back End of the Fuel Cycle Considerations for Small Modular Reactors 19-24.09.2022, IAEA Vienna, Austria

In 2019, I was at one month- scientific internship at the JRC.G.III.8 - Waste Management unit, Karlsruhe, Germany as part of the project Modern Spent Fuel Disolution and Chemistry in Failed Container Conditions, (DisCo - JRC In-situ Individual Training 2019).

5.2 National scientific cooperation

Prior to the award the PhD degree

In 1995 I was at three-months scientific internship at the Institute of Organic Chemistry in Warsaw. It was the beginning of cooperation with dr Piotr Propkopowicz, which resulted in the article published in Synthetic Communications in 1999.

My PhD disertation i prepared at the Faculty of Chemistry, University of Warsaw (1994-1998). During this time, I was the auxilary supervisor of 2 master's theses carried out in Stereocontrolled Organic Synthesis Lab

After the award the PhD degree

In the years 1998-2000 I was employed at the Faculty of Chemistry, University of Warsaw as an assistant. I am a co-author of 6 publications with the affiliation of this unit.

In 2019, I was at one-week scientific internship at the Laboratory of Radioactivity Analyses, the Henryk Niewodniczański, Institute of Nuclear Physics Polish Academy of Sciences.

In 2018, I was the scientific supervisor of the engineering thesis of Monika Milaniuk, a student of the Warsaw University of Technology, Faculty of Chemical and Process Engineering, carrying out the experimental part of the work at the Institute of Nuclear Chemistry and Technology.

6. Presentation of teaching and organizational achievements as well as achievements in popularization of science or art

Prior to the award the PhD degree

- auxiliary supervisor of of master's thesis Krzysztof Raszplewicz thesis "Asymmetric additions of organometallic compounds to N-glyoxyloyl-, N-pyruvinyl and N-phenylglyoxyloyl-(2R)-bornane-10,2-sultam and glyoxalan (R)-menthyl", under supervision of dr Tomasz Bauer, University of Warsaw, Faculty of Chemistry, 1994,
- auxiliary supervisor of master's thesis of Anna Czapla, "Badania porównawcze addycji nukleofilowej do glioksalanu 10- dicykloheksylosulfamoilo- (R)-izoborneolu i N-glioksyloilo-(2R)-bornano-10,2-sultamu", under supervision of dr Zbigniew Wielogórski, University of Warsaw, Faculty of Chemistry, 1997,
- the didactic assistant during classes at the Faculty of Chemistry in 1994-1998, Laboratory of Organic Chemistry for second year students.

After the award the PhD degree

- teacher at the Faculty of Chemistry in 1998-2000, Laboratory of Organic Chemistry for second-year students,
- participation in the Museum Night in 2013-2014 as a representative of the Institute of Nuclear Chemistry and Technology in Warsaw,
- tutor of 4 students carrying out internships at INCT,
- auxiliary supervisor of Monika Milaniuk engineering thesis, "Analiza metod efektywnego odzysku metali ziem rzadkich" (Analysis of methods for effective recovery of rare earth elements) supervisor: dr hab. Ewa Dłuska, prof. PW, scientific supervisor at INCT: dr Katarzyna

Kiegiel, thesis defended at the Warsaw University of Technology, Faculty of Chemical and Process Engineering, 2019,

- auxiliary supervisor of the doctoral dissertation of auxiliary supervisor of doctoral dissertation of mgr inż. Dorota Gajda, „Odzysk uranu i wybranych metali ciężkich z rud i surowców odpadowych” (Recovery of uranium and selected heavy metals from ores and waste materials) carried out at the Institute of Nuclear Chemistry and Technology in Warsaw under the supervision of prof. dr hab. inż. Grażyna Zakrzewska-Kołtuniewicz,

- scientific tutor of a student of the IChTJ-NCBJ Doctoral School (since 2022).

7. Other information about his/her professional career, which he/she deems important

7.1 Other articles/chapters in scientific monographs related to topic of the presented achievement, not included in the cycle

1. K. Kiegiel*, P. Kalbarczyk, G. Zakrzewska-Kołtuniewicz, #266 The determination and recovery of radioactive metals from industrial waste stored in Poland, IAEA, MANAGEMENT OF NATURALLY OCCURRING RADIOACTIVE MATERIAL (NORM) IN INDUSTRY, Proceedings Series, International Atomic Energy Agency, IAEA, Vienna (2022), <http://www-pub.iaea.org/MTCD/Publications/PDF/SupplementaryMaterials/PUB1998.zip> (accessed on 25 February 2023).
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3. G. Zakrzewska-Kołtuniewicz*, K. Kiegiel, S. Wołkowicz, *Uranium from domestic resources in Poland*, URANIUM RAW MATERIAL FOR THE NUCLEAR FUEL CYCLE: EXPLORATION, MINING, PRODUCTION, SUPPLY AND DEMAND, ECONOMICS AND ENVIRONMENTAL ISSUES (URAM-2018), IAEA, 2020.
4. K. Kiegiel*, D. Gajda, G. Zakrzewska-Kołtuniewicz, *Secondary Raw Materials as a Potential Source of Uranium*, JOURNAL OF NUCLEAR RESEARCH AND DEVELOPMENT 15 5, 2018.

5. K. Kiegiel*, G. Zakrzewska-Kołtuniewicz, *Zasoby uranu w Polsce – możliwości pozyskiwania uranu ze źródeł niekonwencjonalnych*, POSTĘPY TECHNIKI JĄDROWEJ, Instytut Chemii i Techniki Jądrowej, PTN, (*in Polish*), Vol. 61. Z.2, 2018, 17-22.
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9. G. Zakrzewska, K. Kiegiel*, D. Gajda, A. Miśkiewicz, P. Bieluszka, K. Frąckiewicz, I. Herdzik, B. Zielińska, A. Jaworska, K. Szczygłó, A. Abramowska, W. Olszewska, R. Dybczyński, H. Polkowska-Motrenko, B. Danko, Z. Samczyński, E. Chajduk, J. Chwastowska, I. Bartosiewicz, S. Wołkowicz, J. B. Miecznik, *Analysis of the possibility of uranium supply from domestic resources*, str. 212-219, PROCEEDINGS OF THE MINERAL ENGINEERING CONFERENCE MEC2014, Red. M. Lutyński i T. Suponik, Stowarzyszenie Inżynierów i Techników Górnictwa Gliwice, Polska 2014. ISBN 978-83-60837-83-2.
10. G. Zakrzewska-Trznadel, K. Kiegiel*, A. Abramowska, B. Zielinska, P. Bieluszka, Ł. Steczek, E. Chajduk, S. Wołkowicz, *Recovery of uranium from post-leaching solutions by solvent extraction: state of the art and new technological possibilities*, Proceedings of NUCLEAR 2013 The 6th Annual International Conference on Sustainable Development through Nuclear Research and Education. Part 2/3, (p. 161-167). Romania: Institute for Nuclear Research – Pitesti, 2013

7.2 The received patent related to the topic of the presented achievement

1. G. Zakrzewska-Kołtuniewicz, K. Kiegiel, A. Abramowska, D.K. Gajda; W. Łada, Instytut Chemii i Techniki Jądrowej, *Sposób wytwarzania oktatlenku triuranu z roztworów o niskiej zawartości uranu, (Method for producing ammonium diuranate from the solutions with low uranium content)* **236018**, Wiadomości Urzędu Patentowego (Patent Office News), 19/2020, Patent Office of the Republic of Poland, November 30, 2020

7.3 Information about the expert opinions concerning the topic of the presented achievement

1. G. Zakrzewska-Kołtuniewicz, K. Kiegiel, A. Miśkiewicz, S. Sommer, O. Roubinek, D. Gajda, A. Abramowska, P. Kalbarczyk, I. Bartosiewicz, *Niekonwencjonalne źródła uranu w Polsce (Unconventional sources of uranium in Poland)*, Expert opinion prepared under contract no. IV/758/P/15004/43902/DEJ, Warsaw 2015. Analysis commissioned by DEJ, Ministry of Economy

2. A. Bojanowska, M. Brykała, D. Chmielewska-Śmietanko, L. Fuks, D. Gajda, P.-Kalbarczyk, I. Herdzik-Koniecko, K. Kiegiel, A. Miśkiewicz, J. Narbutt, H. Polkowska-Motrenko, M. Siwek, W. Starosta, T. Smoliński, K. Skotnicki, S. Sommer, D. Wawszczak, G. Zakrzewska-Kołtuniewicz, D. Miernicka, M. Banach, A. Grzegorzówka, G. Kuciel, M. Kwaśniewski, *Postępowanie z odpadami promieniotwórczymi i wypalonym paliwem jądrowym w Polsce – stan obecny i perspektywy (Management of radioactive waste and spent nuclear fuel in Poland - current status and prospects)*, opinion prepared under contract no. 156/II/P/15004/4390/17/DEJ as. " Analysis - management of radioactive waste and spent nuclear fuel in Poland - current status and prospects", Warsaw, December 2017, Analysis commissioned by DEJ, Ministry of Economy.

7.4 Other scientific articles/chapters in scientific monographs

(not mentioned in section 4.2 and 7.1).

1. A. Boettcher, A. Celińska, M. Frelek-Kozak, M. Migdal, M. P. Dąbrowski, P. Sęktas, A. Sidło, G. Zakrzewska-Kołtuniewicz, D. Wawszczak, M. Brykała, K. Kiegiel, A. Miśkiewicz, L. Fuks, I. Herdzik-Koniecko, E. Chajduk, W. Starosta, T. Smoliński, M. Rogowski, A. Pawelec, *Projekt Gospostrateg-HTR: Rezultaty Projektu (The Gospostrateg-HTR project results)*, POSTĘPY

TECHNIKI JĄDROWEJ, Instytut Chemii i Techniki Jądrowej, PTN, (*in Polish*), Vol. 66. Z.1, 2023, 9-19.

2. N. Haneklaus*, S. Barbossa, M. D. Basallote, M. Bertau, E. Bilal, E. Chajduk, Y. Chernysh, V. Chubur, J. Cruz, K. Dziarczykowski, P. Fröhlich, P. Grosseau, H. Mazouz, K. Kiegiel, J.M. Nieto, S. Pavón, S. Pessanha, A. Pryzowicz, H. Roubík, C.R. Cánovas, H. Schmidt, R. Seeling, G. Zakrzewska-Kołtuniewicz, *Closing the upcoming EU gypsum gap with phosphogypsum*, RESOURCES, CONSERVATION AND RECYCLING 2022, 182, 106328. <https://doi.org/10.1016/j.resconrec.2022.106328>

3. L. Fuks*, I. Herdzik-Koniecko, K. Kiegiel*, A. Miskiewicz, G. Zakrzewska-Kołtuniewicz, *Methods of thermal treatment of radioactive waste*, ENERGIES 2022, 15(1), 375. <https://doi.org/10.3390/en15010375>

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5. K. Kiegiel*, A. Miśkiewicz, I. Herdzik-Koniecko, D. Gajda, G. Zakrzewska-Kołtuniewicz, Rozdział 2 : *Perspective of obtaining Rare Earth Elements in Poland*, LANTHANIDES, eds. Nasser S Awwad i Ahmed T. Mubarak IntechOpen, London, United Kingdom, 2019 .ISBN 978-953-51-5792-2. <http://dx.doi.org/10.5772/intechopen.76488>

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8. K. Kiegiel*, G. Zakrzewska-Kołtuniewicz, K. Wołoszczuk, P. Krajewski, M. Poumander, C. Mays, D. Diaconu, *Assessment of Regional Capabilities for New Reactors Development Through an Integrated Approach – Evaluation of Polish Contribution to Alfred Demonstrator*, PROCEEDINGS - ENC 2016, European Nuclear Society, Bruksela 2016, s. 408-414, ISBN 978-92-95064-27-0.

9. K. Kiegiel, W. Starosta and J. Leciejewicz*, *Pyrimidine-4-carboxylic acid*, ACTA CRYSTALLOGRAPHICA SECTION E: STRUCTURE REPORTS ONLINE 2013, 69(6), o885. <https://doi.org/10.1107/S1600536813012610>
10. W. Starosta, J. Leciejewicz*, K. Kiegiel, *Tetraaqua(pyrimidine-4,6-dicarboxylato- κ^2 N 1,O 6)magnesium monohydrate*, ACTA CRYSTALLOGRAPHICA SECTION E: STRUCTURE REPORTS ONLINE 2013 Volume 69(4): m189. <https://doi.org/10.1107/S1600536813005850>
11. K. Raszplewicz, L. Sikorska, K. Kiegiel, T. Bałakier, J. Jurczak*, *Diastereoselective addition of grignard reagents to chiral α -ketoimides derived from Oppolzer's sultam*, POLISH JOURNAL OF CHEMISTRY, 2005, 79(12): 1901 – 1907.
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7.5 Received awards and distinctions

1. Team Award of the Director of INCT 2017 for a series of five original and valuable scientific publications on the issues of the fuel cycle and the search for alternative sources of fissile materials.
2. 1st prize in the PGE EJ 1 competition, 2016, for scientific publications on nuclear energy published in 2013-2015
3. Team Award of the Director of IChTJ, 2015, for a series of four publications dedicated to the issues of obtaining uranium from uranium ores for the production of nuclear fuel.