

Properties and application of diketones and their derivatives

Włodzimierz URBANIAK, Katarzyna JUREK, Katarzyna WITT, Andrzej GORAŹCZO - Faculty of Chemical Technology and Engineering, University of Technology and Life Sciences, Bydgoszcz; Bartosz STANISZEWSKI - Faculty of Chemistry, Adam Mickiewicz University, Poznań

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β -diketone compounds, whose simplest and the most widely known member is pentane-2,4-dione (informally referred to as acetylacetone), have a number of very interesting and specific properties due to their structure (the presence of two carbonyl groups separated with one carbon atom). Their crucial feature is keto-enol tautomerism, the presence of the ketone and the enol forms in equilibrium. The equilibrium in the case of β -diketones is strongly shifted towards the enol form due to the formation of the distinct resonance structure as a six-membered ring. Keto-enol equilibrium is affected by a number of other factors with the most important being solvent polarity and the presence and properties of substituents (both terminal ones and those in the methylene group). The capacity to form stable complexes with most metals is a direct consequence of the occurrence of such compounds in the enol form.

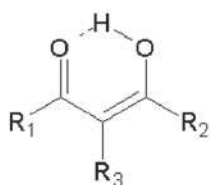


Fig. 1. General structure of β -diketones and the complex compounds they form

Due to the presence of two carbonyl groups, β -diketones are valuable substrates in many chemical syntheses. They can be used e.g. for the preparation of ketoimines (by condensation with amines), thioketones and various heterocyclic compounds (e.g. pyrimidine derivatives). Modifications of the methylene group in a β -diketone molecule which involve the incorporation of a substituent usually do not impact the activity of the resulting compound in further reactions. Therefore, it is possible to incorporate substituents which may be

further modified, e.g. by the polymerisation of unsaturated bonds, which opens up many possibilities for the application of the compounds.

A general scheme of the possibilities of using diketones is shown in Figure 2.

Owing to their properties, β -diketones and their complexes have been used both in science and in industry. The compounds are frequently used in polymer technology e.g. as substrates for the manufacture of homogeneous and heterogeneous catalysts, as polymerisation catalysts (metal complexes) and substances which modify the properties of resulting polymers (UV resistance, oxygen resistance). β -diketone complexes (especially with transition metals) are often used as catalysts of reactions, such as olefin oxidation and epoxidation or oligomerisation [1, 2].

They have also been widely used in healthcare, both as active pharmaceutical ingredients (or substrates for the manufacture of medicines) and cosmetic additives which reduce the detrimental effects of UV radiation on the skin. Furthermore, they are important for chemical analysis in which they are used for sample concentration (owing to their complexing activity), for air pollution monitoring (formaldehyde) or as stationary phases in gas chromatography (olein analysis). They are also employed as fuel additives [3], antiulcer and gastroprotective drugs [4a], antiasthmatic and lung disease drugs [4b], carcinogenic agents [4c] and antidiabetic agents [4d], fillers which improve polymer properties [5], substrates for the preparation of hydrophobic polymers [6], luminescent compounds [7], etc.

Owing to their complexing properties, they have also been used in environmental protection, e.g. for metal chelation in sewage [8].

Because of their extensive research and application potential, they have been investigated for a number of years within research projects carried out in the Department of Coordination Chemistry of the University of Technology and Life Science; owing to the development

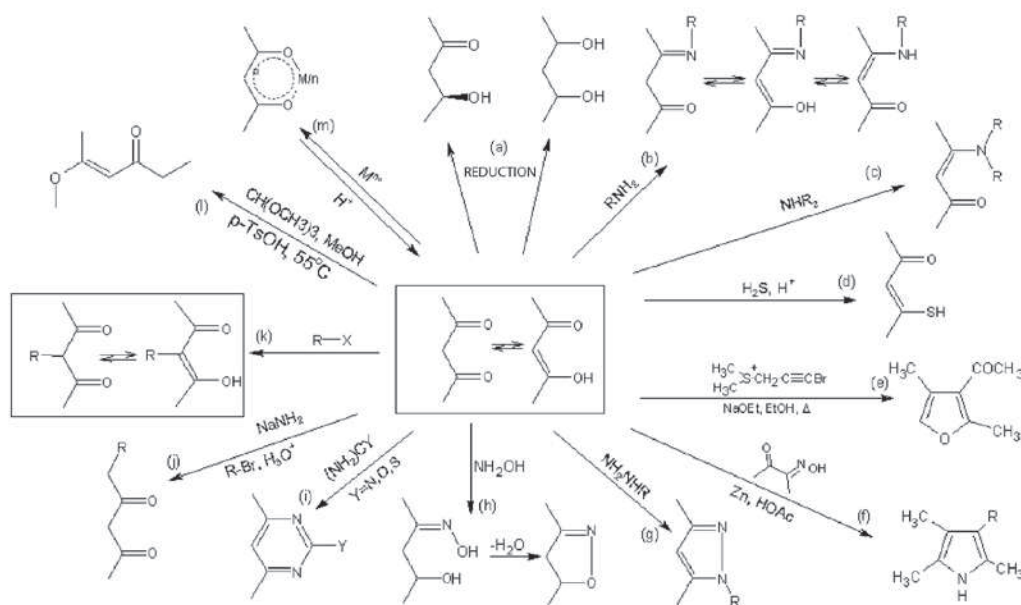


Fig. 2. Scheme of β -diketone application potential

of efficient methods for the synthesis and purification of new 3-substituted diketone derivatives [9, 10], their research and application potential has been significantly increased.

In particular, research is being carried out into the complexing properties of β -diketone derivatives with various substituents in the methylene group. Such derivatives were previously reported by Martin and Ferneliuss [11]. They discuss compounds, such as 3-n-butyl-2,4-pentanedione or 3-allyl-2,4-pentanedione and their various metal complexes and list deprotonation constant and stability constant values.

Even though the class of compounds has much application potential, there are no current literature data on their complexing capability and the stability of resulting compounds. The latest foreign publications discuss only compounds which contain acetylacetonate in their molecules, a mere part of the vast coordination sphere of more extensive complexes. The general lack of researchers interest in 3-substituted β -diketonates, caused mostly by the difficulties in the isolation of pure products of hydrogen substitution in the acetylacetonate methylene group, is the main reason for the initiation of studies of these compounds in the Department of Coordination Chemistry. Our research focuses on the synthesis of new 3-substituted β -diketones and their complexes, analysis of their properties and search for their industrial applications [10–13].

One of the directions of metal β -diketonate studies is the evaluation of their stability using potentiometry and UV-Vis spectrophotometry. Within the research, acetylacetonate derivatives have been synthesised and tested, such as: 3-allyl-2,4-pentanedione, 3-benzyl-2,4-pentanedione, 3-butyl-2,4-pentanedione, benzoylacetone, dibenzoylmethane, 3-methyl-2,4-pentanedione, 3-trimethylsilylpropyl-2,4-pentanedione, etc. The analysis of their stability using UV-Vis spectrophotometry involves the measurement of absorbance of a series of solutions of varying concentrations of the complex being formed. The solutions were prepared so that the concentration of one component, e.g. a metal ion, was constant, while that of the other component (ligand) increased. Water-methanol solutions were analysed due to the need for dissolving the test ligands in methanol. The resulting UV spectra showed gradual complex formation. Examples of UV-Vis spectra prepared for the test compounds are presented below.

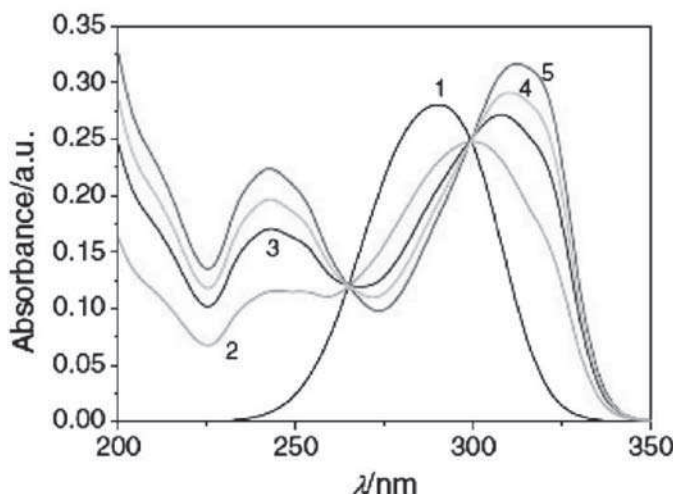


Fig. 3. UV-Vis spectrum of the Cu(II) complex with 3-allylacetylacetonate being formed at various Cu/L molar ratios: 1:0 (1), 1:0.2 (2), 1:0.6 (3), 1:0.8 (4), 1:1 (5)

Based on the resulting spectra, stability constants were calculated for complexes with the 1:1 metal/ligand molar ratio, using a method discussed in our earlier paper. Example values of resulting stability constants for various complexes are shown in Table I. The results are well correlated with literature data [12].

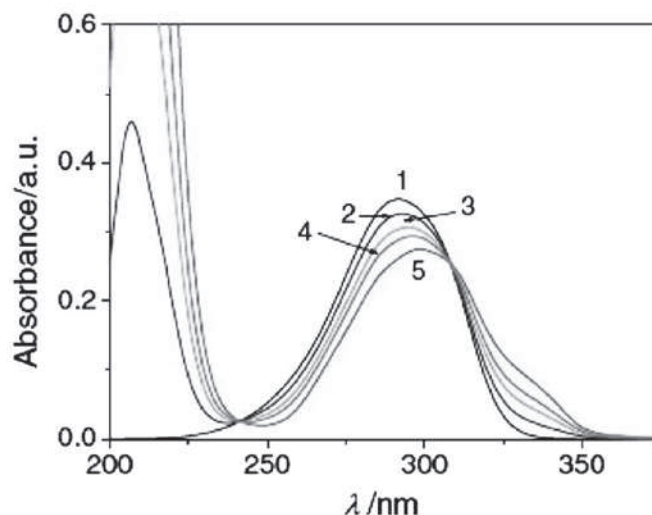


Fig. 4. UV-Vis spectrum of the Co(II) complex with 3-allylacetylacetonate being formed at various Co/L molar ratios: 1:0 (1), 1:0.2 (2), 1:0.4 (3), 1:0.6 (4), 1:1 (5)

Table I

Stability constant values for complexes with 1:1 metal/ligand molar ratio

ligand	log K	
	Cu(II)	Co(II)
2,4-pentanedione	8.24	5.40
3-benzyl-2,4-pentanedione	6.90	4.04
3-allyl-2,4-pentanedione	8.10	5.55

Stary and Liljenzin give the value of the first stability constant for the Cu(acac)₂ complex of $\log K_1 = 8.25 \pm 0.05$ [14]; the result of Izatt et al. for the complex is very similar, $\log K_1 = 8.22$ [15]. The results confirm the high stability of the test compounds and raise hopes for their numerous industrial applications.

Diketone complexes have also been structurally tested using mass spectrometry. For example, EI spectra of 2,4-pentanedione and its alkyl derivatives and also complexes of the following formulae were tested: M(I)(acac) and M(II)(acac)₂ (where: M(I)=Li, Na, K, Rb, Cs, M(II)=Be, Mg, Ca, Sr, acac=2,4-pentanedione). In all cases a band corresponding to the protonated ligand form was seen in the spectra. The band corresponding to the metal cation was present in the spectra only for complex compounds with first group metals. Furthermore, a peak was observed in the mass spectrum which corresponded to the polymer complex molecule of formula $[M_x(acac)_y]^{(+)}$; it was concluded based on this that I and II group metals form complex compounds with polymeric structures in gas phase [16].

Furthermore, mass spectra of acetylacetonate complexes with various cations were studied, including:

- bis-acetylacetonates: beryllium, zinc(II), cobalt(II), magnesium, manganese(II), copper(II), nickel(II), calcium, iron(II), molybdenyl, titanyl, vanadyl
- tris-acetylacetonates: chromium(III), aluminium, indium, iridium, cobalt(III), lanthanum(III), manganese(III), osmium, rhodium, ruthenium, titanium, vanadium(III), iron(III)
- tetrakis-acetylacetonates: zirconium and thorium.

The spectra belong to the collection of organometallic and coordination compound spectra, based mainly on NIST spectra databases [17–19]. They were recorded using electron irradiation as an ionisation method. Common features of the spectra were determined based on them. The regularities seen concern fragmentation ions specific

for acetylacetonate decay. The initial fragmentation of acetylacetonate molecules usually leads to the elimination of a single ligand molecule, $M(\text{MeL}_n) \leftrightarrow \text{MeL}_{n-1} + \text{L}$. The process is sometimes preceded by CH_3 group elimination. If so, the elimination of the other part of the ligand molecule follows. Further disintegration of the resulting fragmentation ion usually involves the carbon backbone which proves the stabilisation of the MeL_{n-1} ion. It seems that the elimination of one ligand molecule particularly stabilises the carbon-metal bond. This is seen for bis- and tris-acetylacetonates. It is difficult to state whether it occurs also for tetrakis-acetylacetonates, because a representative number of such complexes could not be obtained. Signals at $m/z = 100$ are observed in many spectra, corresponding to the protonated acetylacetonate ion, eliminated in whole from the complex molecule. This confirms that the elimination of one ligand molecule is the first stage of molecule fragmentation. Ions with $m/z = 85$ Da are seen in the spectrum in many cases. This occurs for acetylacetonate complexes in which the elimination of the first ligand molecule is preceded by the elimination of the methyl group from the molecule. The ion with $m/z = 43$ seen in the spectra of all the acetylacetonates tested may be assigned to the $\text{C}_2\text{H}_3\text{O}^+$ ion which most likely forms due to the extensive destruction of the ligand.

Owing to the quality of the simple acetylacetonate spectra obtained, it is possible to develop detailed fragmentation mechanisms and look for the correlation between complex molecular structures and their fragmentation pathways. In previous studies, model calculations using polynomial methods were used [20, 21], based on the natural decay of stable isotopes in nature [22].

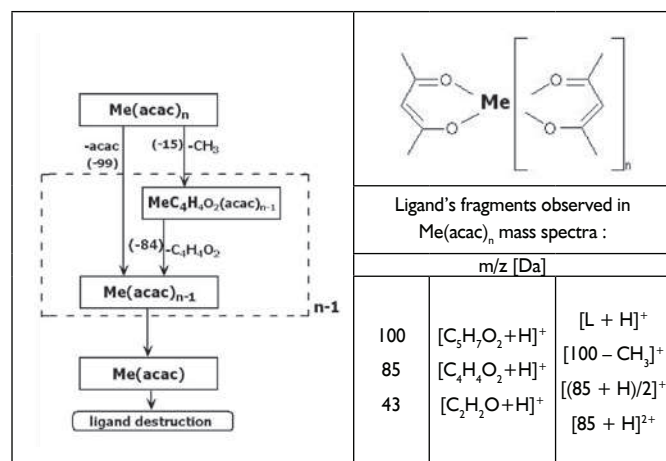


Fig. 5. Fragmentation of metal β -diketonates with general formula $\text{Me}(\text{acac})_n$ ($n = 2 - 4$)

Hypotheses developed based on this may be verified by the interpretation of mass spectra of complex compounds of acetylacetonate derivatives with metal cations. Mass spectra of approx. 100 such compounds were collected which will be used to develop detailed fragmentation mechanisms of the group of complexes.

The other research field being intensely investigated in the Department of Coordination Chemistry is the possibility of using the resulting metal β -diketonate derivatives as precursors for inorganic materials prepared by the sol-gel method.

The properties of metal alkoxides and acetylacetonates and their application in various areas are extensively covered and widely used, especially in the sol-gel method [23]. The main precursors for the sol-gel method are: inorganic salts and metal β -diketonates and alkoxides. The first group, especially chlorides, contaminate final products, being difficult to remove. The other are far more expensive due to difficulties in the isolation of pure products from the reaction mixture [11, 24].

The application of in particular complex compounds of metals with an oxygen-carbon bond in non-aqueous media opens up new

possibilities for the synthesis of inorganic nanomaterials [25] with, however, many research difficulties involved. These include, among other things, the determination of the role of organic compounds with a C-O group used as ligands in the formation of porous gel structures of metal oxides.

It was found that the gelling process for SiO_2 -based gels can be easily controlled by changing the pH of the mixture. The preparation process for oxides of other metals using the sol-gel method is more complex and it depends on a number of factors, such as the precursor structure [26].

Among the number of ongoing studies on the preparation and use of metal oxides and oxide systems [27], a few only concern the application of mixed, polyligand complex compounds used as oxide phase precursors for preparative purposes. It follows from the available literature data that the use in the controlled hydrolysis of e.g. bis(2-methylbutan-2-oxy)di(pentane-2,4-dione)tin leads to materials stable in lower temperatures, with higher specific surface areas and lower pore sizes; therefore, such powders could be used in the manufacture of photoelectrochemical elements [28].

The preliminary research into the synthesis of dihydroxy-bis(pentane-2,4-dione)tin(IV) acetate and the determination of its application potential in binary gel synthesis confirms our suggestions. The resulting materials have modified surface acid-base properties and better cross-linked internal structures, which leads to the preparation of materials with lower pore sizes [29].

The incorporation of ligands with distinct properties and structure and similar hydrolysis potential yields potential oxide phase precursors used in the sol-gel method. Furthermore, it enables selective and specific control of the process for the preparation of materials with distinct catalytic, optical, adsorption and absorption properties. The use of as inexpensive substrates and energy saving synthetic technology as possible may lead to practical applications of such systems. Particularly promising is the use of the resulting gels in heterogeneous catalysis and as coating with specific absorption properties.

A tin(IV) compound with 2,4-pentanedion in the coordination sphere as one precursor was used. Binary $\text{Al}_2\text{O}_3 - \text{SnO}_2$ systems were prepared in the following molar ratios: 1:0.1; 1:0.25; 1:0.5 and 1:1. The resulting products were analysed using FT-IR spectroscopy, XRD and low-temperature liquid nitrogen adsorption with BJH method used for calculation. Based on the results, the shape of the isotherm type can be classified as IV and the type of the hysteresis loop as the H1/H2 mixture type [30]. The texture analysis of modified aluminium oxide using a tin(IV) complex compound as the precursor showed that the resulting material had a much more narrow pore size range (2-7 nm) than pure Al_2O_3 (2-15 nm), (Fig. 6).

The narrow pore distribution shows that interactions between gel molecules are stronger and cross-linking is higher than in pure aluminium-based gel. As expected, this has also an effect on surface properties. Changes in surface properties were confirmed based on FT-IR spectra; the analysis of hydroxyl groups using band positions in the spectra proved it clearly that only type III groups occur in such binary systems (related to three Al_O atoms according to Knozinger, contrary to pure Al_2O_3 (IIa) [31]. XRD studies proved the amorphous structure of the binary system, (Fig. 7)

With the increasing contribution of tin oxide in the binary system, the cassiterite crystal structure contribution increases, proved by higher intensities and occurrence of peaks related to the presence of the rutile structure of cassiterite.

The use of the tin(IV) β -diketonate complex as the oxide phase precursor changed the oxide system hydrolysis rate compared to the use of other tin precursors [32]. The resulting gel has an amorphous nature which changes with the increasing tin content in the system. The incorporation of the tin component as a complex compound led to the shift of pore diameters towards lower values with respect to

pure aluminium oxide and, simultaneously, narrow pore distribution which confirms higher homogeneity of the resulting material. As expected, the tin additive also influenced the properties of surface hydroxyl groups, not seen for pure aluminium oxide.

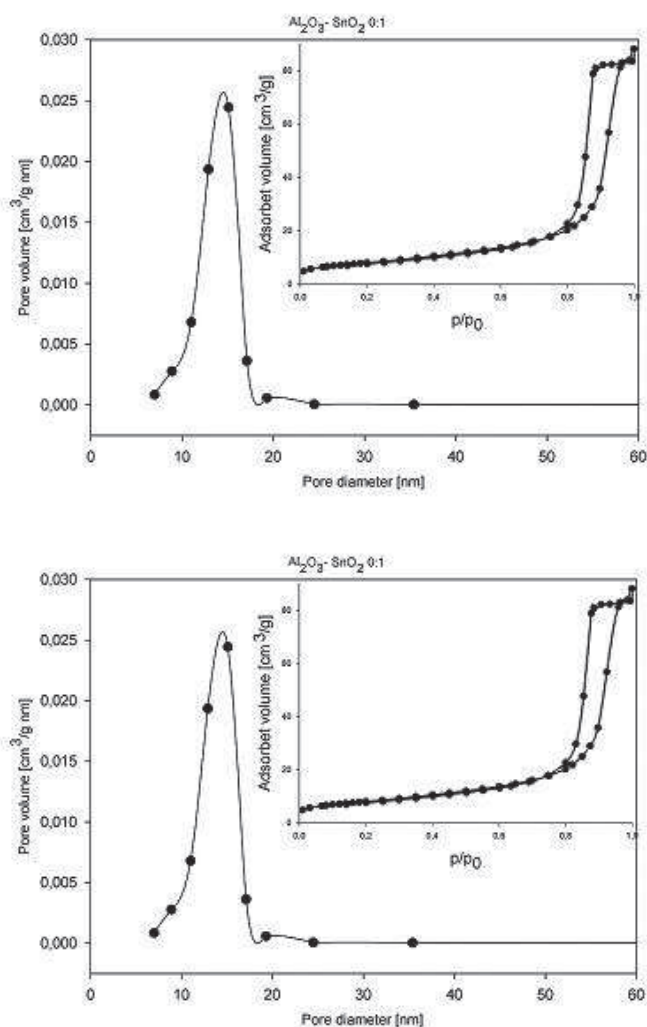


Fig. 6. Texture analysis of modified aluminium oxide

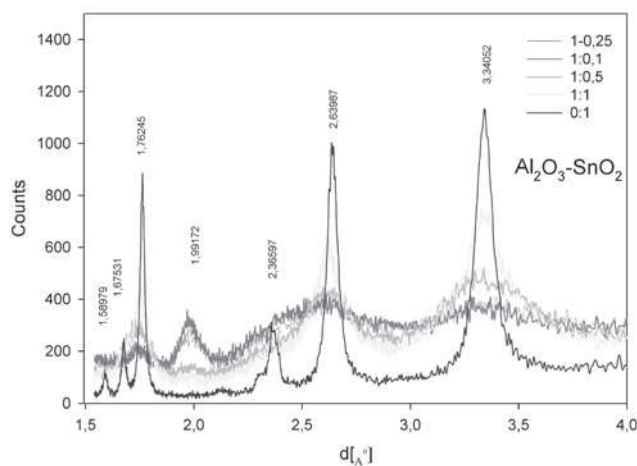


Fig. 7. $\text{Al}_2\text{O}_3\text{-SnO}_2$ binary system diffractograms with component proportions of 1:0, 1:1, 1:0.5, 1:0.25 and 1:0.1 as an intensity - interplanar distance relationship

The results of our research confirmed our proposal; therefore, it seems justified to continue our investigation and look for further possible applications for the resulting groups of compounds.

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Translation into English by the Author

Włodzimierz URBANIAK (Ph.D.) Associate Professor at the Faculty of Chemistry, Adam Mickiewicz University in Poznan and the at the Faculty of Chemical Technology and Engineering, University of Technology and Life Sciences in Bydgoszcz. Education: PhD - 1985 (Chemistry) Faculty of Chemistry, Adam Mickiewicz University in Poznan. Associate Professor – 2001 Faculty of Chemistry Adam Mickiewicz University in Poznan. In this study developed a number of effective methods for the synthesis of organosilane compounds (mostly described for the first time), among others β-diketonate and β-ketoimine groups and their derivatives as well. He is the author of their applicability in various fields of science and practice. The current research interests are environmental monitoring, waste management technologies, non-waste technology, hazardous waste technologies, production of new materials, and legal aspects of waste management. The author or co-author of more than 170 publications in foreign magazines and national and more than 50 patents and patent applications. The author or co-author of more than 90 communications presented at national and international conferences.

Katarzyna JUREK Ph.D. is the assistant professor at the Faculty of Chemical Technology and Engineering, University of Technology and Life Sciences in Bydgoszcz, Poland. Education: 1993 – M. Sc. (Chemical Technology), University of Technology and Life Sciences, Bydgoszcz, Poland; 2003 – Ph.D. (Chemistry), Poznań University of Technology, Poznań, Poland. Current research interests are focused on the areas of synthesis and processing of binary oxide system using the sol-gel process. In particular, her research has emphasized processing studies on the conditions for forming gels for optical applications, porous membranes, and electrochemical devices, such as fuel cells and solar cells. Her research is focused on exploring the dynamics and structure of unique sol-gel materials. She is co-organized 8 international conferences, and is the co-author of six scientific papers and ten papers in Polish and International Conferences materials.

Katarzyna WITT M.Sc. Eng- graduated from the University of Technology and Life Sciences in Bydgoszcz, Faculty of Chemical Technology and Engineering (2008). At present she is an assistant in Institute of Coordination Chemistry on University of Technology and Life Sciences in Bydgoszcz. Scientific interests: inorganic chemistry, specially coordination chemistry. She is the co-author of 1 chapter of monograph, 1 article in scientific press, 2 articles in conference materials, 2 polish patent applications and 4 polish and international conference reports and posters.

Andrzej GORĄCZKO Ph.D. is the assistant professor at the Faculty of Chemical Technology and Engineering, University of Technology and Life Sciences in Bydgoszcz, Poland. Education: 1969 – M. Sc. Faculty of Chemistry, Technical University, Gdańsk, Bydgoszcz, Poland; 1979 – Ph.D. (Chemistry), Faculty of Chemical Technology, Technical University, Szczecin. Current research interests are: mass spectrometry, modelling of mass spectra. He was awarded of a Cross of Merit (Silver), Medal of the National Education Commission and Gold Decoration of the Polish Teachers Society. He is co-organized 8 international conferences. He is the autor and co-author of 32 monographs and chapters of monograph He is the co-author of 22 scientific papers and 35 papers in Polish and International Conferences materials.

Bartosz STANISZEWSKI Ph.D. Education: 2004 - MSc. Faculty of Chemistry Adam Mickiewicz University in Poznan, 2011- PhD. (Chemistry) Faculty of Chemistry Adam Mickiewicz University in Poznan in 2011. Winner of Nowicki Foundation. for the best graduates in the field of environmental protection. In 2006, took an internship in Bundesamt Seeschiffahrt fuer und Hydrographie, institution in charge of monitoring contamination of the North Sea. Co-author of 10 publications, 8 patent applications and communications presented at several national and international conferences.