Polish J. Chem., **79**, 441–443 (2005)

Professor Przemysław Mastalerz

Przemysław Mastalerz, Professor emeritus of Wrocław University of Technology, was born at November 8th 1925, in Częstochowa (Poland). In 1947–1951 he had completed studies at the Department of Mathematics, Physics and Chemistry at the University and Polytechnics of Wrocław. In 1951–1959 was appointed as research fellow at Wrocław Medical Academy, then at Technical University of Wrocław and finally at Institute of Immunology and Experimental Therapy of Polish Academy of Sciences. In 1959 he received PhD degree on the basis of Doctoral Thesis entitled „*Inhibitors of glutamine biosynthesis*" (under the supervision of Prof. Tadeusz Baranowski). In 1961, after a year post-doctoral stay at the University of California in San Francisco, he was appointed as Assistant Professor at the Technical University of Wrocław, where in 1967 received DSc title on the basis of monographic paper entitled „*Studies on phosphinic acids*" and simultaneously received position of Associate Professor. In 1977 he received a title of Professor from Polish Government. In 1980/1981 he was appointed as Visiting Professor at Southern Illinois University in Carbondale (USA) where he was giving lectures in organic chemistry. Professor Przemysław Mastalerz also actively participated in academic life as a vice-Dean of the Department of Chemistry of Wrocław University of Technology, Director of Institute of Organic and Physical Chemistry of the same University and six years as a member of Central Qualification Commission.

Professor Przemysław Mastalerz is a non-standard personality in Polish science. It results from his unusual curiosity in the entire world, great spirit and enormous ability to create personalities of his pupils. Without any doubt he has created a scientific school, which is still alive and active at Wrocław Technical University and University of Opole. Professor had formed the school non-typical according to Polish standards because it attracted mostly such persons, who done PhD degrees in other laboratories. He has promoted 10 Philosophy Doctors; two of them later became Professors while four immigrated to USA. Professor Przemysław Mastalerz created worldwide known scientific group characteristic in that it undertook challenges resulting from the studies on the borders of chemistry and biology. Professor Roman Tyka was the second major scientist of the group (his former PhD student, Dr. Józef Oleksyszyn acts now as a Professor of the Technical University of Wrocław). Activity initiated in Prof. Mastalerz's group is continued by his coworkers, now Professors of Wrocław University of Technology, namely by Prof. Barbara Lejczak (has promoted 6 PhD students), Prof. Paweł Kafarski (has promoted 9 PhD students), Prof. Mirosław Soroka, Prof. Roman Gancarz (has promoted one PhD student) and Prof. Bogdan Boduszek. The scientific school created by Professor Przemysław Mastalerz might be called School of Bioorganic Chemistry. He had soon realized that the studies done on the borders of chemistry and biology create new dimension in research. Such studies were initiated in 1959 when designed and evaluated inhibitors of glutamine synthetase in hope that they would act as antiepileptic agents. The designed phosphonic analogues of glutamic acid were then the most potent inhibitors of this important enzyme. He also promoted pioneering studies on the antiviral activity of phosphonates and studies on biodegradative cleavage of carbon-to-phosphorus bond. In 1971 he had created

Laboratory of Bioorganic Chemistry and stimulated studies on the rational design of inhibitors of chosen proteolytic enzymes, on antibacterial phosphono peptides, and stereocontrolled synthesis of aminophosphonates. These works had resulted that he was named “father of bioorganic chemistry of phosphorus”.

At the beginning of eighties he had started to organize the program of studies and research basis for introduction of biotechnology to Wrocław University of Technology. He had created the research laboratory, which after his retirement was called Laboratory of Biotechnology and is well known in Poland. Prof. Przemysław Mastalerz also created the first syllabus for biotechnology.

During last two years he has published in *Wiadomości Chemiczne* a series of three critical reviews (totally over 200 pages) devoted to facts and myths in environmental chemical pollution. First article presents history of DDT, second considers polychlorinated biphenyls, while third dioxins. These papers present real results and consequences of ecological propaganda.

Professor Przemysław Mastalerz is authoring and co-authoring over 120 original papers published in worldwide-acknowledged journals. His works have been cited over 1,500 times. He is also a single author of three organic chemistry handbooks and one inorganic chemistry handbook.

Paweł Kafarski

Polish J. Chem., **79**, 445–466 (2005) **REVIEW ARTICLE**

Role of the Side Chain Modifications for the Solution Behavior and the Complex-Forming Abilities of Some Aminomethane-1,1-diphosphonic Acids. Relevance to Biological Activity

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This paper is a highlight of the recent advances made in the studies of the N-substituted aminomethane-1,1-diphosphonic acids, which are an interesting class of bisphosphonates with a nitrogen atom attached directly to the P–C–P backbone. The paper is focused on elucidating the role, which can play subtle positional and substitutional modifications for the solution behavior of the aminomethane-1,1-diphosphonic acids. In particular, the topochemically modified heterocyclic side chains are considered with respect to possible relation with the biological activities of the compounds. Of special interests are the N-(2-pyridyl)aminomethane-1,1-diphosphonic acids and a group of derivatives with a nitrogen atom involved in saturated five-, six-, seven- or eight-membered ring. Some examples demonstrating the importance of side chain modifications for the metal ion complex-forming abilities of the aminomethane-1,1-diphosphonic acids are also presented.

Key words: aminomethane-1,1-diphosphonic acids, bisphosphonates, structure-activity relationships, complex-forming abilitie

Polish J. Chem., **79**, 467–472 (2005)

Studies on the Role of DBU in the Reaction of P(III)–OAryl System with Nucleoside

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The involvement of a $(=P\text{-}N\equiv)^{+}$ cationic species has been unambiguously demonstrated in the phosphitylation of nucleosides in the presence of DBU (1,8-diazabicyclo-[5.4.0]undec-7-ene) by ^{31}P NMR spectroscopy and its reaction with benzoyl fluoride.

Key words: phosphitylation, nucleosides, nucleotides, DBU (1,8-diazabicyclo[5.4.0]undec-7-ene), ^{31}P NMR spectroscopy

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Mechanism of Decomposition of Quasiphosphonium Intermediates: Borderline S_N1 Character of Alkyl-Oxygen Fission in *sec*-Alkyloxyphosphonium Salts

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Short-lived alkoxyphosphonium intermediates have been detected in the interactions of alkyl diphenylphosphinites $ROPPh_2$ (R = Et, Pr^i, Bu^s, and 3-pentyl) with iodomethane at room temperature. Phosphorus chemical shifts for the *sec*-alkoxy(methyl)diphenylphosphonium iodides (δ_P 68.6–68.7 ppm) are at slightly higher field than for ethoxy(methyl)diphenylphosphonium iodide (δ_P 72.4 ppm), in accord with higher electron density at phosphorus in the secondary alkyl sytems. Relative rates of decomposition in $CDCl_3$ (Me > Et > Pr^i >> neopentyl) are in accord with S_N2-type cleavage of the R–O bond but within the secondary alkyl series the relative rates (Pr^i < Bu^s < 3-pentyl) are indicative of an increasing tendency towards carbonium ion character as the *sec*-alkyl group becomes more bulky. For the *sec*-alkyl derivatives, a borderline-S_N1 mechanism is proposed.

Key words: Michaelis-Arbuzov, quasiphosphonium, alkoxyphosphonium, ^{31}P NMR, kinetics, mechanism

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Polycations. Part 16. Polyphosphonium Species Containing Phosphorus–Phosphorus Bonds

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This effort has had as its focus the generation of analogues of phosphoric acid amides in which phosphorus is present in place of the usual nitrogen on an amide species. Of particular interest for us have been such phosphorus analogues of *quaternary* amides in which a phosphonium site is directly attached to the phosphorus of each parent acidic site. The procedure for the generation of such materials, along with their spectral and physico-chemical characteristics, is presented here.

Key words: phosphorus analogues of amides, phosphonium salts

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Tetrahydrofuran, One of the Most Popular Solvents in Organic Synthesis, Is Not Completely Inert

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Tetrahydrofuran undergoes fast ring cleavage in the reaction with PhI(OH)OTs/$(RO)_2P(O)H$ system, which causes the formation of O-(dialkoxyphosphoryl)-O′-tosyl-1,4-butanediol. This fact indicates that tetrahydrofuran is not suitable as a solvent for reactions with strong electrophilic reagents, such as hypervalent iodine reagents.

Key words: di*iso*propylphosphite, tetrahydrofuran, Koser's reagent

Polish J. Chem., **79**, 493–498 (2005)

1-Azidoalkylphosphonates – A Convenient Substrates for the Synthesis of *N*-Alkyl α-Aminoalkylphosphonates

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Diethyl [1-(alkylamino)alkyl]phosphonates **5** have been efficiently synthesized *via* a two-step reaction of diethyl 1-azidoalkylphosphonates **1** with triphenylphosphine, followed by *in situ* transformation of thus formed phosphazenes **2** into imines **4** by means of aldehydes and subsequent reduction of **4** with sodium borohydride in ethanol.

Key words: phosphazenes, Staudinger reaction, diethyl 1-azidoalkylphosphonates, reduction, aminophosphonates

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1-(N-Acylamino)alkanephosphonates. Part IV. N-Acylation of 1-Aminoalkanephosphonic Acids

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Practical general procedures of N-acylation of 1-aminoalkanephosphonic acids, including N-acetylation, N-benzoylation and N-pivaloylation of the representative aminophosphonic acids are described. Physico-chemical properties of synthesized derivatives are presented. Dissociation equilibria, determined for some representative 1-(N-acylamino)alkanephosphonic acids, are listed.

Key words: amino acids, aminophosphonic acids, N-acylation, 1-(N-acylamino)alkanephosphonic acids, protonation/dissociation equilibria

Polish J. Chem., **79**, 515–523 (2005)

A General One-Pot Synthesis of Vinyl-Thiiranes and Conjugated Dienes

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The first general synthesis of vinyl-thiiranes **7** and an effiecient preparation of conjugated dienes **8** and **9** is presented. Methodology described for the preparation of these compounds is based on the corresponding readily available thiophosphates **1** and selenophosphates **2**.

Key words: vinyl-thiiranes, dienes, thiophosphates, selenophosphates

Polish J. Chem., **79**, 525–528 (2005)

Ferrocene-derived Aminomethanephosphonous Acids. Synthesis of Their Esters and Amides with Polyethylene Glycols and Polyamines

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The synthesis of (ferrocenyl)-N-diphenylmethylaminomethane phosphonous acid ester with polyethylene glycols and the synthesis of its amides with polyamines in dichloromethane and in the presence of N,N′-dicyclohexylcarbodiimide (DCC) as condensing agent are presented.

Key words: ferrocene-derived aminophosphonous acids, condensation, polyethylene glycols, polyamines

Polish J. Chem., **79**, 529–539 (2005)

Oxidation of Phosphonocysteine and Phosphonohomocysteine. Synthesis of Phosphonocysteic and Phosphonohomocysteic Acids

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The bromine promoted oxidations of phosphonocysteine and phosphonohomocysteine are described. The reactions were found to occur *via* S-bromination-hydrolysis sequence. The formation of cyclic intermediate – sulfonamide derivative of phosphonohomocysteic acid is proposed. The ultimate products of oxidations – phosphonocysteic and phosphonohomocysteic acids were isolated in high yields and characterized.

Key words: amino acids, aminophosphonic acids, bromine promoted oxidation of the thiolo group, oxidation of phosphonocysteine and phosphonohomocysteine, phosphonocysteic acid, phosphonohomocysteic acid

Polish J. Chem., **79**, 541–545 (2005)

Synthesis of Diphenylphosphine Oxide and Diethyl Phosphonate with 4-Dimethylsila-2-hexen-6-ol Moiety

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Two reagents useful for the Horner-Wittig or Wadshworth-Emmons reaction: 3-(2-hydroxyethyldimethylsila)-2-propen-1-yldiphenylphosphine oxide (**5a**) and diethyl 3-(2-hydroxyethyldimethylsila)-2-propen-1-ylphosphonate (**5b**) were synthesized from propargyl chloride and dimethylchlorosilane. The usefulness of phosphine oxide was demonstrated in the olefination reactions of benzaldehyde and hexanal.

Key words: 3-(2-hydroxyethyldimethylsila)-2-propen-1-yldiphenylphosphine oxide, diethyl 3-(2-hydroxyethyldimethylsila)-2-propen-1-ylphosphonate, Arbusov reaction, hydrosilylation, Horner-Wittig reaction, phosphorus, silicon

Polish J. Chem., **79**, 547–552 (2005)

The Protonation Site in Aminophosphonates

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The protonation equilibria in aminophosphonates: protonation on nitrogen *vs* phosphoryl oxygen has been calculated. The calculated energy differences explain satisfactory experimentally observed tendency toward nonstability of aminophoshonates in acidic media.

Key words: P–C bond break, aminophosphonates, *ab-initio*, protonation

Polish J. Chem., **79**, 553–559 (2005)

Synthesis of New Imidazole Aminophosphine Oxides

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A series of imidazole aminophosphine oxides were synthesized by addition of dialkyl, diaryl, or alkyl(aryl)phosphine oxides to imidazole imines, formed from 1-benzylimidazole-5-carboxaldehyde and primary amines. The phosphine oxides were obtained by alkylation of diethyl phosphite, or ethyl phenylphosphinate with Grignard reagents. The new imidazole aminophosphine oxides were characterized by means of ^{1}H and ^{31}P NMR spectroscopy.

Key words: aldimines, phosphine oxides, aminophosphine oxides, Grignard reagents, 1-benzylimidazole-5-carboxaldehyde

Polish J. Chem., **79**, 561–566 (2005)

Lipase-Catalysed Resolution of 1-Hydroxyethane-P-phenylphosphinates

by P. Majewska, P. Kafarski and B. Lejczak

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Lipase-catalysed hydrolysis and transesterification were used to obtain α-hydroxyalkanephosphinates with two stereogenic centers, namely at α-carbon and phosphorus atoms. These compounds were chosen to check if there is a transfer of chirality from carbon to phosphorus atom during lipase catalysed reactions.

Key words: biocatalysis, kinetic resolution, enantioselectivity, chirality transfer

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$\alpha_v\beta_3$ Antagonists Based on a Central Benzoic Acid Scaffold

by D.W. Will[1], G. Breipohl[1], J.-F. Gourvest[2], J.-M. Ruxer[2], B. Doucet[2], M. Auberval[2], R. Baron[2], M. Gaillard[2], T.R. Gadek[3], J. Knolle[1], H.U. Stilz[1] and A. Peyman[1]

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A series of novel, highly potent $\alpha_v\beta_3$ antagonists based on a benzoic acid scaffold and containing an acylguanidine as an Arg-mimetic and sulfonamide side chains is described. The compounds are selective against the fibrinogen receptor $\alpha_{IIb}\beta_3$ and they are capable of inhibiting bone resorption *in vivo* in a TPTX model of osteoporosis. Therefore the compounds are promising drug substances for the treatment of osteoporosis.

Key words: peptidomimetics, structure activity relationship, RGD analog

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Solid-State CP/MAS P-31 NMR in Studies of the Aminophosphonic Acids and Esters in Marine Animals

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For the first time the ^{31}P solid-state NMR CP/MAS technique was applied to determine the amount of C-P forms in the total phosphorus in sea anemones. Signals of phosphonates (*ca* 20 ppm) and phosphates (*ca* 0 ppm) are well resolved and the phosphonate/phosphate ratios were easily calculated. Sea anemones *Anemonia sulcata* from the Adriatic Sea contain 41–54% of phosphorus as phosphonates (P-C). ^{31}P NMR analysis of lipids was also performed.

Key words: ^{31}P NMR, CP/MAS, sea anemones, phospholipids

Polish J. Chem., **79**, 583–592 (2005)

(*E*)-Cinnamic Acid Analogues as Inhibitors of Phenylalanine Ammonia-Lyase and of Anthocyanin Biosynthesis

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Several carboxylic, phosphonic, phosphinic, boronic and nitro analogues of (*E*)-cinnamic acid were synthesized. These and other compounds related to (*E*)-cinnamic acid were evaluated as potential inhibitors of both phenylalanine ammonia-lyase and of anthocyanin biosynthesis in buckwheat. The most potent inhibition was found for 3-phenylprop-2-ynoic acid (**21**), however its K_i is comparable to K_M. The molecular modelling of the interaction of (*E*)-cinnamic acid (**1**) and **21** with PAL model suggests some similarities in the binding mode of both compounds.

Key words: phenylalanine ammonia-lyase, enzyme inhibitors, molecular modelling, anthocyanin biosynthesis, phenylpropanoid pathway

Polish J. Chem., **79**, 593–602 (2005)

α-Aminoalkylphosphonates Induced Apoptosis in Human Tumor Cell Lines

by M. Drąg[1], M. Sieńczyk[1], A. Marcinkowska[2], M. Drąg-Zalesińska[3], T. Wysocka[3] and J. Oleksyszyn[1]**

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Evaluation of the *in vitro* activity of the 1-amino-3-phenylpropanephosphonic acid and diphenyl 1-(N-benzyloxycarbonylamino)-1-(4-amidinophenyl)methanephosphonate hydrochloride toward acute, human Jurkat and A549 cell lines revealed that these compounds are potent inducers of apoptosis. Their potency to inhibit proliferation of tested tumor lines is similar to bestatin, a well-known inhibitor of aminopeptidases and strong inducer of apoptosis in the number of cancer cell. In this paper preliminary experimental details as well as the possible mechanism of action of α-aminoalkylphosphonates will be discussed.

Key words: α-aminoalkylphosphonates, apoptosis, cancer, aminopeptidase, comet assay

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Specific Interactions of Divalent Metal Ions with Phosphonic Analogues of Dipeptide Inhibitors of Proteases

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Potentiometric and spectroscopic methods were used to evaluate the binding ability of two dipetides with C-terminal phosphonic group. This phopshonate moiety is quite effective in the coordination of metal ions. The two phosphinate analogues, which are very effective enzyme inhibitors, were found to be also powerful ligands for Cu^{2+} ions involving both amino and phosphinic groups in metal ion coordination.

Key words: Cu(II) complexes, Ni(II) complexes, inhibitors of proteases, phosphonic acids, phosphinic acids