BUDAPEST UNIVERSITY OF TECHNOLOGY AND ECONOMICS
FACULTY OF CHEMICAL AND BIOENGINEERING
GEORGE OLAH DOCTORAL SCHOOL

MICROWAVE-ASSISTED SYNTHESIS OF α-AMINOPHOSPHONATES AND RELATED DERIVATIVES

PhD Thesis

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1. Introduction

α-Aminophosphonates may be considered as the phosphorus-analogues of α-amino acids. Due to this similarity, they are of potential biological activity.[1] The preparation of new derivatives, and development of the known synthetic procedures are especially important in the field of organophosphorus chemistry.

I have started my PhD work under the supervision of Dr. György Keglevich and Dr. Erika Bálint at the Green Chemical and Organophosphorus Research Group at the Department of Organic Chemistry and Technology, at Budapest University of Technology and Economics.

The aim of my research work was to elaborate the synthesis of new α-aminophosphonate and aminomethylene-bisphosphonate derivatives without any catalyst and/or solvent.

Our research group have proved the advantages of applying the microwave (MW) technique in several organophosphorus transformations.[2] In most cases, the reactions were faster and more selective, as well as the products could be obtained in higher yields. Moreover, MW-assisted variations also allowed catalyst- and/or solvent-free accomplishments. Due to these benefits, we decided to carry out our reactions under MW conditions.

We also aimed at synthesizing dialkyl phosphites bearing different alkoxy groups on the phosphorus atom ("mixed" phosphites) by alcoholysis. These molecules may serve as valuable starting materials of *P*-chiral derivatives.

We also wished to develop a continuous MW system, and elaborate a few flow organophosphorus transformations.

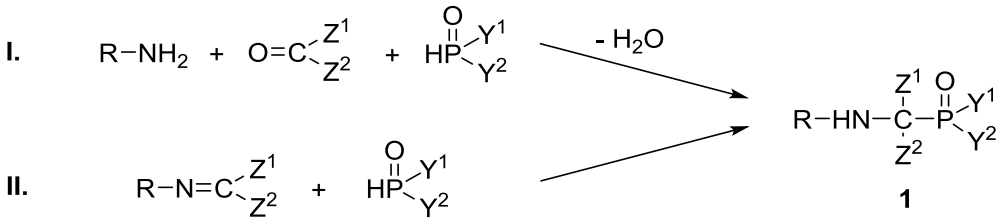
2. Background

α-Aminophosphonates (**1**) may be applied as enzyme inhibitors, and antibacterial-, antiviral- and anticancer agents as well. Beside their pharmaceutical importance, they may also serve as herbicides and fungicides.[1]

These derivatives may be prepared by the Kabachnik–Fields (or phospha-Mannich) reaction (**I**), in which a primary or a secondary amine, an oxo compound (aldehyde or ketone) and a >P(O)H reagent react, and by the Pudovik (or aza-Pudovik) reaction (**II**), in which the addition of >P(O)H

[1] Hudson, H. R.; Kukhar, V. P. *Aminophosphonic and aminophosphinic acids: chemistry and biological activity*; Wiley: Chichester, 2000.
[2] Keglevich G.; Kiss, N. Z.; Grün, A.; Bálint, E.; Kovács, T. *Synthesis* **2017**, *49*, 3069.

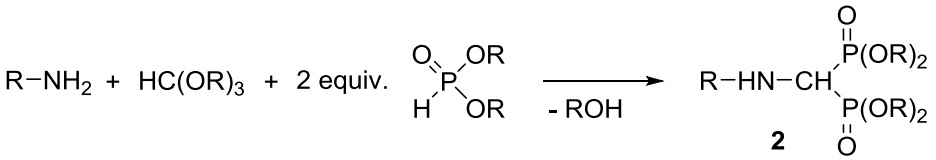
derivatives takes place to the C=N double bond of imines (or Schiff bases) (Scheme 1).[3] In most cases, these transformations were carried out in the presence of a catalyst and/or a solvent.



Scheme 1 Synthesis of α-aminophosphonates (**1**) by Kabachnik–Fields- (**I**) or by Pudovik-reaction (**II**)

In aminomethylene-bisphosphonates (**2**), an amine function and two phosphonate moiety are connected to a central carbon atom.[4] As the phosphorus-analogues of natural α-amino acids, and the amino-analogues of the substituted hydroxymethylene-bisphosphonates (dronates), they are potentially biologically active compounds.

Aminomethylene-bisphosphonates are usually prepared by the three-component condensation of amines, orthoesters and dialkyl phosphites (Scheme 2), however, in most cases, these procedures were not optimized. Moreover, the similar reaction of secondary phosphine oxides is not known in the literature.



Scheme 2 Synthesis of aminomethylene-bisphosphonates (**2**) by three-component condensation

In continuous-flow MW reactors, the flow- and MW chemistry are combined: the heating of the reaction mixture is carried out by MW irradiation (Figure 1).[5] This technique may be considered as a new field in organophosphorus chemistry.

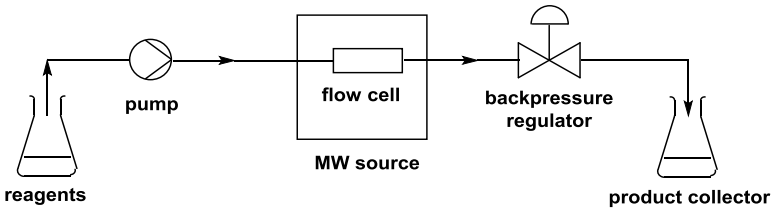


Figure 1 Schematic drawing of a continuous-flow MW system

[3] Bálint, E.; Tripolszky, A.; Tajti, Á. *Synthesis of α-aminophosphonates by the Kabachnik–Fields reaction and by the Pudovik reaction*. In: Organophosphorus Chemistry; Keglevich, G. (ed.); Walter de Gruyter: Berlin, 2018, p 108.
[4] Romanenko, V. D.; Kukhar, V. P. *Arkivoc* **2012**, 127.
[5] Bálint, E.; Keglevich, G. *The Spread of the Application of the Microwave Technique in Organic Synthesis*. In *Milestones in Microwave Chemistry*; Keglevich, G. (ed.); Springer: Switzerland, 2016, p 1.

3. Experimental methods and equipment

The MW-assisted reactions were carried out in a CEM Discover® MW reactor (300 W), equipped with a pressure device. The continuous-flow MW reactions were carried out in a system containing a CEM Flow Cell Accessory®, a CEM Discover® MW reactor (300 W), a HPLC pump, a cooler and a backpressure regulator (250 Psi).

The products were purified by column chromatography. The reactions were followed by gas chromatography (GC), high pressure liquid chromatography (HPLC) and/or thin layer chromatography (TLC). The products were identified by GC-MS and/or HPLC-MS measurements. The compounds synthesized were characterized by ^{31}P, ^{13}C, and ^{1}H NMR spectroscopy, moreover, they were identified by HRMS data. The crystal structure of a few derivatives were analyzed by X-ray diffraction measurements.

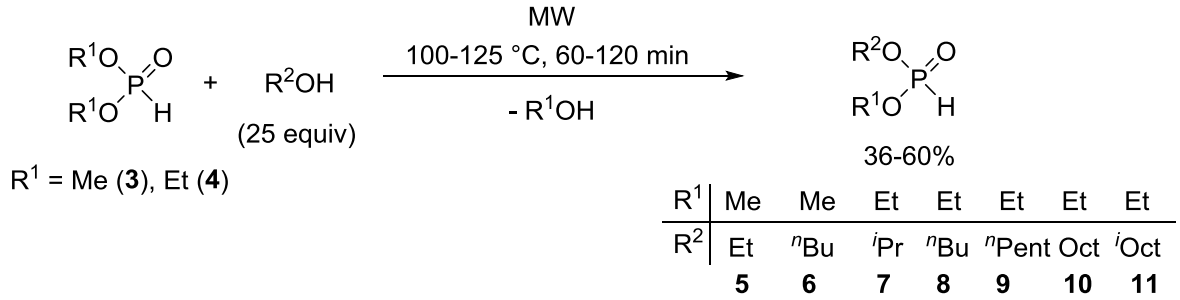
The quantum chemical calculations were carried out using B3LYP/6-31G (d,p) method.

4. New scientific results

4.1. Synthesis of α-aminophosphonate derivatives by Kabachnik–Fields reaction

4.1.1. Preparation of ethyl octyl α-aminophoshonate derivatives

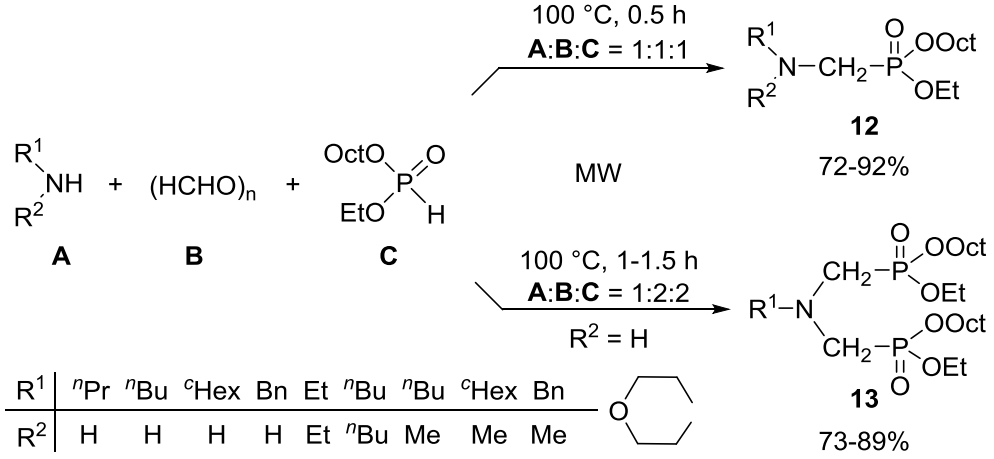
In the first part of my research work, we have elaborated the synthesis of a few dialkyl phosphites (**5-11**) bearing different alkoxy groups on the phosphorus atom including ethyl octyl phosphite (**10**) by the partial alcoholysis of dimethyl (**3**) or diethyl phosphite (**4**) (Scheme 3). These derivatives are not commercially available. Altogether seven mixed dialkyl phosphites (**5-11**) were synthesized, from which four esters (**6**, **9-11**) are new compounds [1].



Scheme 3 Preparation of mixed dialkyl phosphites (**5-11**) by partial alcoholysis

Starting from ethyl octyl phosphite (**10**), primary or secondary amines and paraformaldehyde, we have investigated the synthesis of α-aminophosphonates containing different alkoxy groups on the phosphorus atom (**12** and **13**) (Scheme 4). The single and the double

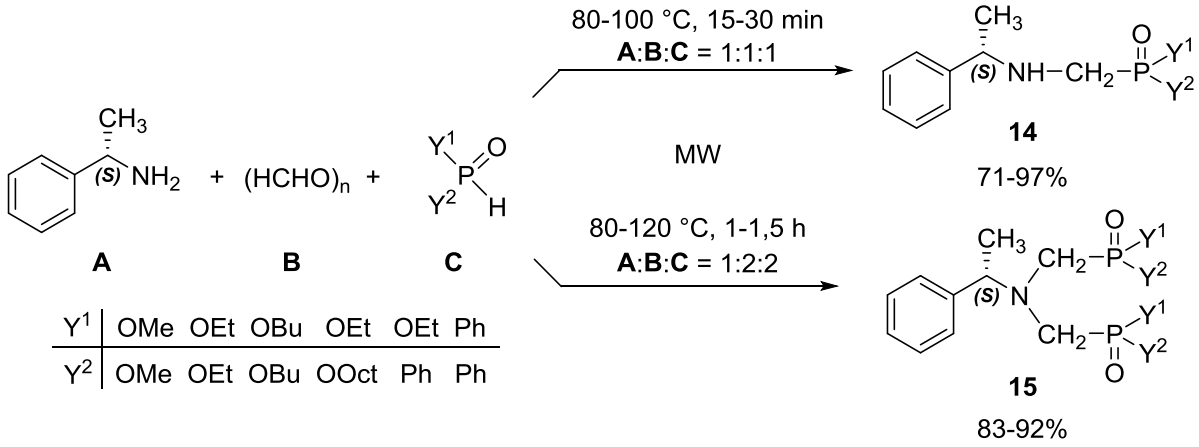
Kabachnik–Fields (phospha-Mannich) reactions afforded ten new ethyl octyl α-aminophosphonates (**12**), and four new *N*,*N*-bis(ethyloctylphosphonomethyl)amines (**13**) in yields of 72-92% [2].



Scheme 4 Synthesis of ethyl octyl α-aminophosphonate derivatives (**12** and **13**)

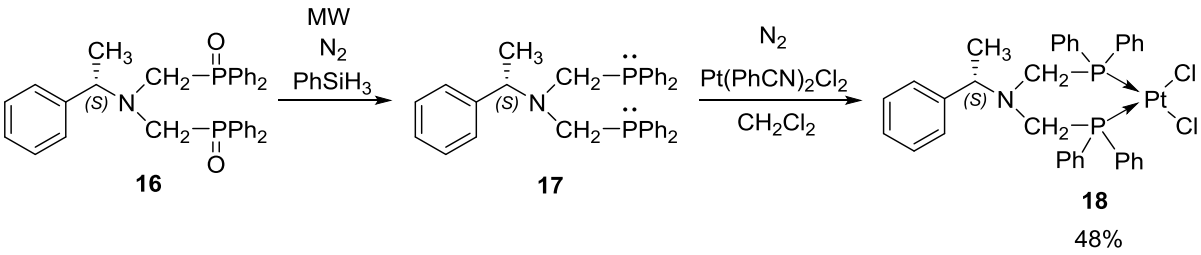
4.1.2. Preparation of (*S*)-α-phenylethylaminomethylaminophosphonates and related derivatives

We have elaborated the synthesis of optically active α-aminophosphonate derivatives (**14** and **15**) by the Kabachnik–Fields condensation of (*S*)-α-phenylethylamine, paraformaldehyde and >P(O)H reagents (Scheme 5). Altogether ten new optically active α-aminophosphonate derivatives (**14** and **15**) were synthesized [3].



Scheme 5 Kabachnik–Fields reaction of (*S*)-α-phenylethylamine, paraformaldehyde and >P(O)H reagents

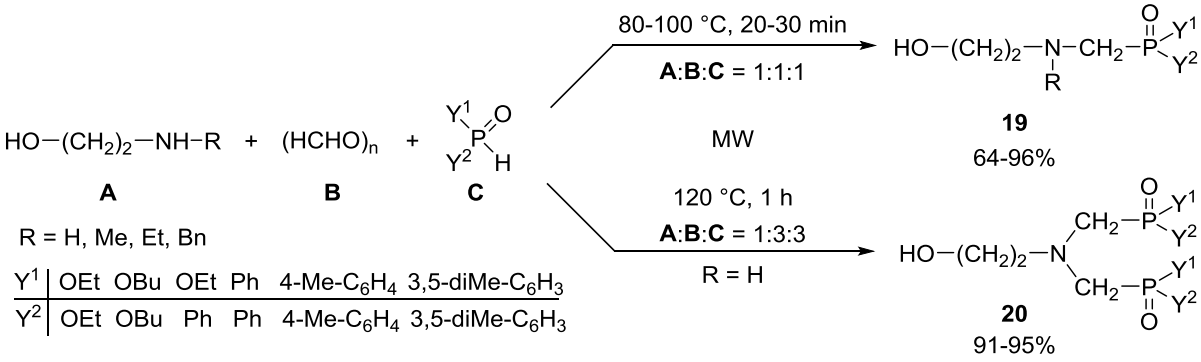
The optically active bisphosphine (**17**) obtained by the double deoxygenation of the (*S*)-*N*,*N*-bis(diphenylphosphinoylmethyl)-α-phenylethylamine (**16**) was utilized as a bidentate P-ligand in the synthesis of the corresponding platinum complex (**18**) (Scheme 6) [3].



Scheme 6 Synthesis of a platinum complex (**18**) from the optically active bisphosphine (**17**) obtained from compound **16**

4.1.3. Synthesis of 2-hydroxyethyl-α-aminophosphonate derivatives

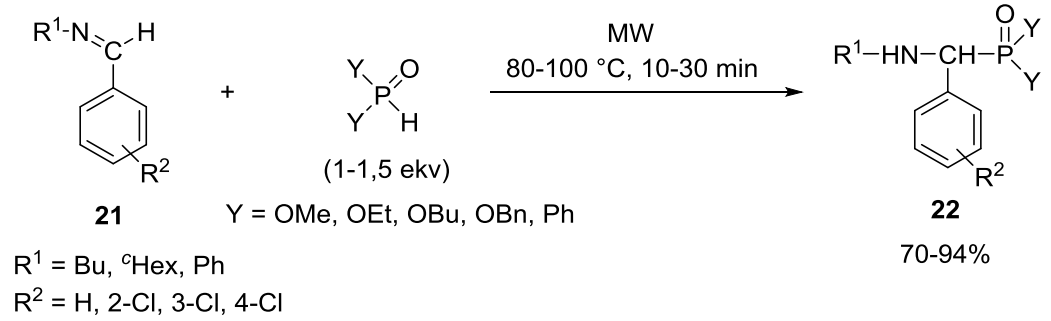
We have also studied the phospha-Mannich reaction of amino alcohols (Scheme 7). By the condensations carried out with dialkyl phosphites and alkyl phenyl-*H*-phosphinate, I have synthesized six new 2-hydroxyethyl-α-aminophosphonate derivatives (**19**, Y^1 = alkoxy, Y^2 = alkoxy or aryl). The Kabachnik–Fields reactions were also performed starting from secondary phosphine oxides, which resulted in 12 new α-aminophosphine oxides (**19**, $Y^1 = Y^2$ = aryl), and three new *N*,*N*-bis(diphenylphosphinoylmethyl)amines (**20**). The crystal structures of two derivatives were determined by X-ray diffraction measurements [4].



Scheme 7 Phospha-Mannich reaction of amino alcohols

4.2. Preparation of α-aryl-α-aminophosphonate derivatives by Pudovik reaction

A MW-assisted catalyst- and solvent-free procedure was elaborated for the addition of >P(O)H reagents to imines (**21**) (aza-Pudovik reaction) (Scheme 8). Altogether 24 α-aryl-α-aminophosphonate derivatives (**22**) were synthesized, from which 19 compounds are new. Two products were analysed by X-ray diffraction measurements. The additions were followed by *in situ* FT-IR spectroscopy. From the data obtained, we determined the concentration–time functions of the reaction components. Formation of the α-aryl-α-aminophosphonates (**22**) was also studied by quantum chemical calculations. We have evaluated the energetics of the reactions, and the reactivity of the starting materials (**21**).



Scheme 8 Addition of >P(O)H reagents to *N*-benzylidenamines (**21**)

4.3. Organophosphorus transformations in a continuous-flow microwave reactor

4.3.1. Development and testing of the continuous-flow microwave system

In the next part of my research work, we have developed a continuous flow MW system (Figure 2). The flow MW reactor has been tested in a simple model, in the direct esterification of the benzoic acid (**23**) by butanol (Scheme 9) [6].

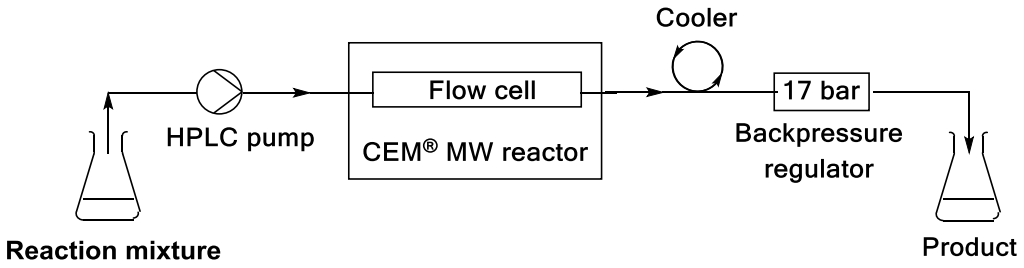
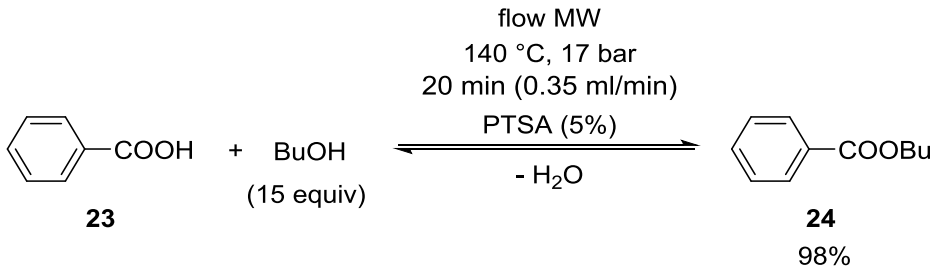


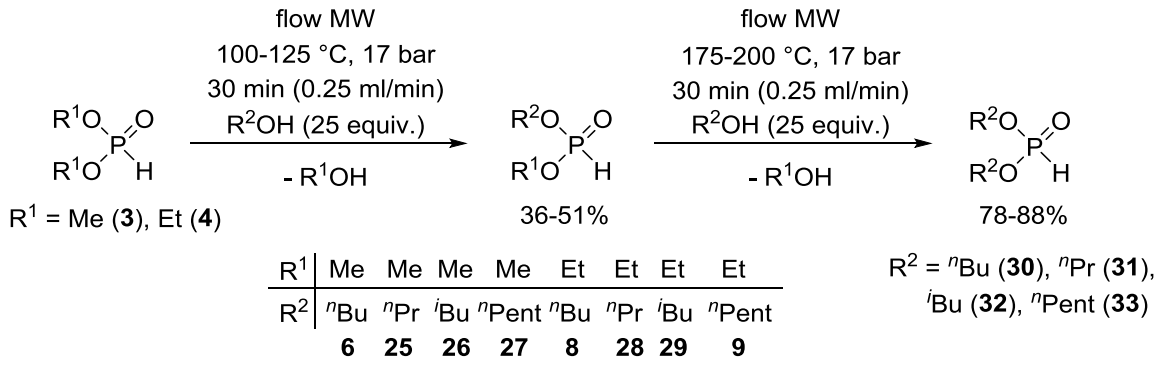
Figure 2 Schematic drawing of the continuous flow MW system developed



Scheme 9 Continuous preparation of butyl benzoate (**24**) by the direct esterification of benzoic acid (**23**)

4.3.2. Continuous alcoholysis of dialkyl phosphites in a flow microwave reactor

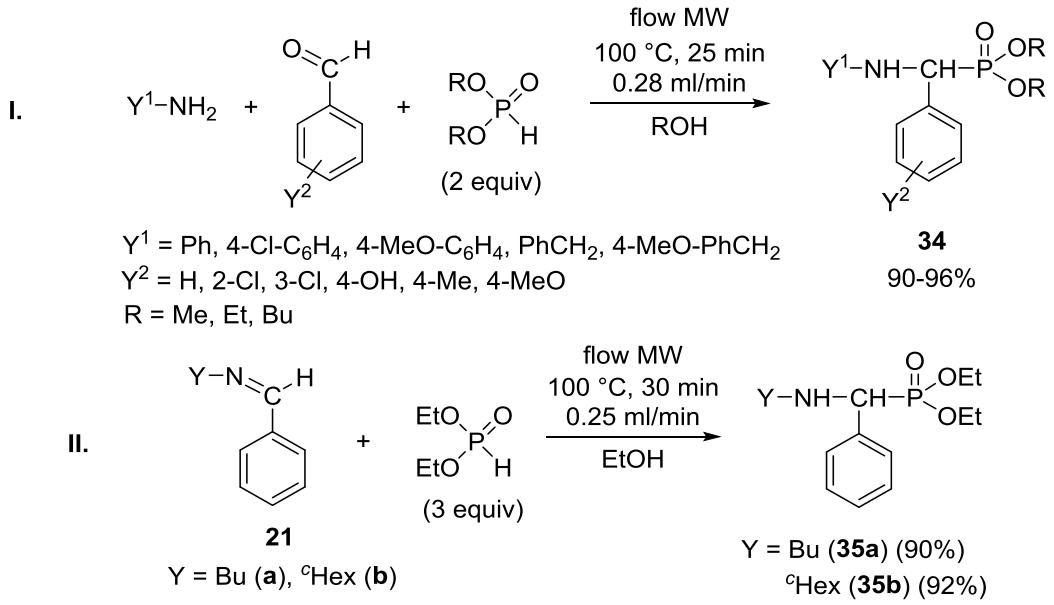
The continuous alcoholysis of dialkyl phosphites (**3** and **4**) was elaborated in the flow MW reactor (Scheme 10). At lower temperatures (100-125 °C), the mixed esters were formed in higher proportion, while applying higher temperatures (175-200 °C), the fully transesterified products dominated. At higher temperatures, increase of the alcohol–phosphite ratio has not affected the composition. From the mixed dialkyl phosphites synthesized, **25-27**, **28** and **29** are new [7].



Scheme 10 Continuous-flow alcoholysis of dialkyl phosphites (**3** and **4**) with aliphatic alcohols

4.3.3. Continuous-flow synthesis of α-aryl-α-aminophosphonates in flow microwave reactor

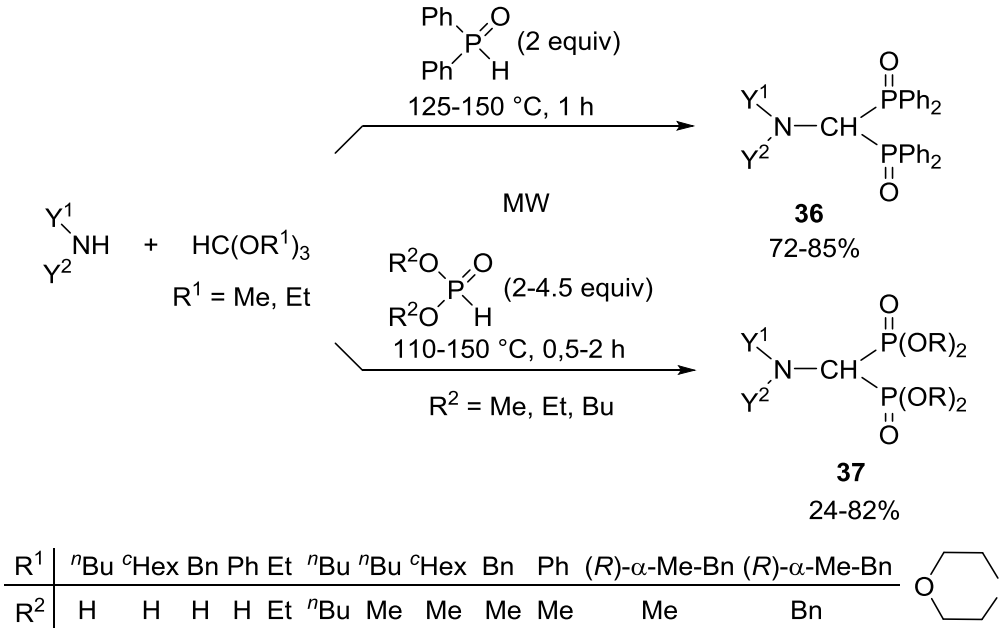
We have developed the MW-assisted continuous-flow synthesis of α-aryl-α-aminophosphonates (**34** and **35**) by the Kabachnik–Fields condensation (**I**) and Pudovik-reaction (**II**) (Scheme 11). Altogether 15 derivatives were obtained in high yields. Our method was the first synthesis of α-aryl-α-aminophoshponates (**34**) by the continuous-flow three-component Kabachnik–Fields reaction [8].



Scheme 11 Continuous-flow synthesis of α-aryl-α-aminophosphonates by Kabachnik–Fields condensation (**I**) and by Pudovik reaction (**II**)

4.4. Preparation of aminomethylene-bisphosphine oxides and aminomethylene-bisphosphonates by three-component condensation

In the last part of my work, the synthesis of aminomethylene-bisphosphine oxides (**36**) and aminomethylene-bisphosphonates (**37**) was studied by the catalyst- and solvent-free three-component condensation (Scheme 12). By the reaction of amines, orthoformates and secondary phosphine oxides, we have developed a new synthetic route towards aminomethylene-bisphosphine oxides (**37**). During the optimization of the preparation of aminomethylene-bisphosphonates (**36**), we have identified several intermediates and by-products. Altogether 11 aminomethylene-bisphosphine oxides (**36**) and 14 aminomethylene-bisphosphonates (**37**) were synthesized, from which 21 derivatives are new [9,10].



Scheme 12 Synthesis of aminomethylene-bisphosphine oxides (**36**) and aminomethylene-bisphosphonates (**37**) by three-component condensation

In conclusion, the target model reactions were successfully accomplished without any catalyst and – in most cases – without any solvent under MW conditions. Moreover, we have developed the continuous synthesis of dialkyl phosphites and α-aryl-α-aminophosphonates in a flow MW reactor. During my PhD work, I have prepared more than 100 derivatives, and most of them are new compounds.

5. Theses

1 I have accomplished the catalyst-free synthesis of the not easily accessible dialkyl phosphites bearing different alkoxy groups on the phosphorus atom under microwave (MW) conditions [1].

2. I have developed the preparation of α-aminophosphonates by the Kabachnik–Fields condensation of primary or secondary amines, paraformaldehyde and >P(O)H reagents, including ethyl octyl phosphite, without any catalyst and solvent [2,3].

3. The synthesis of optically active α-aminophosphonate derivatives was elaborated by the Kabachnik–Fields reaction of (*S*)-α-phenylethylamine, paraformaldehyde and >P(O)H reagents. The optically active bisphosphine obtained by the double deoxygenation of the (*S*)-*N*,*N*-bis(diphenylphosphinoylmethyl)-α-phenylethylamine was utilized as a bidentate P-ligand in the synthesis of the corresponding platinum complex [3].

4. I have accomplished the MW-assisted phospha-Mannich reaction of *N*-alkyl-ethanolamines, paraformaldehyde and >P(O)H derivatives. Altogether 21 new 2-hydroxyethyl-α-aminophosphonate derivatives were synthesized [4].

5. An efficient, MW-assisted, catalyst and solvent-free synthetic method was developed for the addition of >P(O)H reagents to the C=N double bond of imines. The reaction was also studied by quantum chemical calculations, which revealed that the additions are exothermic and the formation of the products occur in a single concertic step [5].

6. The continuous-flow alcoholysis of dialkyl phosphites was elaborated for the first time. Applying 25 equivalents of the alcohols, it was found that at lower temperatures (100-125 °C), the esters containing different alkoxy groups on the phosphorus atom were formed in higher proportion, while at higher temperatures (175-200 °C), the fully transesterified products dominated. At higher temperatures, increase of the alcohol–phosphite ratio has not affected the composition [7].

7. I have developed the MW-assisted continuous-flow synthesis of α-aryl-α-aminophosphonates by Kabachnik–Fields condensation and by Pudovik reaction. The preparation of the α-aryl-α-aminophosphonates by the flow three-component Kabachnik–Fields reaction is a novel procedure [8].

8. A new, environmentally friendly synthetic route was elaborated for the synthesis of aminomethylene-bisphosphine oxides by the MW-assisted, catalyst- and solvent-free

condensation of primary or secondary amines, orthoformates and secondary phosphine oxides. During the optimization of the preparation of aminomethylene-bisphosphonates, I have identified several intermediates and by-products. These procedures allowed the efficient preparation of new and known derivatives as well [9,10].

6. Application possibilities

During my PhD work, I have elaborated the environmentally friendly synthesis of various α-aminophoshonate- and aminomethylene-bisphosphonate derivatives. These procedures may be generally applied for such compounds. Moreover, aminomethylene-bisphosphine oxides became available by the new synthetic route developed under catalyst- and solvent-free conditions.

In the flow MW system, I have accomplished esterifications, additions and condensations. I have proved that flow chemistry may also be efficient in the preparation of organophosphorus compounds. Our results may be the starting point of further researches in this field.

7. Publications

7.1. Full scientific publications related to the PhD Thesis

[1] Bálint, E.; **Tajti, Á.**; Drahos, L.; Ilia, G.; Keglevich, G. *Curr. Org. Chem.* **2013**, *17*, 555. (IF 2,537, FI: 3, TÁ: 30%)

[2] **Tajti, Á.**; Bálint, E.; Keglevich, G. *Curr. Org. Synth.* **2016**, *13*, 638. (IF 2,050, FI: 2, TÁ: 100%)

[3] Bálint, E.; **Tajti, Á.**; Kalocsai, D.; Mátravölgyi, B.; Konstantin, K.; Czugler, M.; Keglevich, G. *Tetrahedron* **2017**, *73*, 5659. (IF 2,377, FI: 8, TÁ: 70%)

[4] **Tajti, Á.**; Szatmári, E.; Perdih, F.; Keglevich, G.; Bálint, E. *Molecules* **2019**, *24*, 1640. (IF(2018) 3,060, FI: 1, TÁ: 70%)

[5] Bálint, E.; **Tajti, Á.**; Ádám, A.; Csontos, I.; Karaghiosoff, K.; Czugler, M.; Ábrányi-Balogh, P.; Keglevich, G. *Beilstein J. Org. Chem.* **2017**, *13*, 76. (IF 2,330, FI: 16, TÁ: 70%)

[6] **Tajti, Á.**; Tóth, N.; Bálint, E.; Keglevich, G. *J. Flow Chem.* **2018**, *8*, 11. (IF 1,658, FI: 1, TÁ: 70%)

[7] Bálint, E.; **Tajti, Á.**; Tóth, N.; Keglevich, G. *Molecules* **2018**, *23*, 1618. (IF 3,060, FI: 1, TÁ: 60%)

[8] Bálint, E.; **Tajti, Á.**; Ladányi-Pára, K.; Tóth, N.; Mátravölgyi, B.; Keglevich, G. *Pure Appl. Chem.* **2019**, *91*, 67. (IF(2018) 2,350, FI: 0, TÁ: 60%)

[9] Bálint, E.; **Tajti, Á.**; Dzielak, A.; Hägele, G.; Keglevich, G. *Beilstein J. Org. Chem.* **2016**, *12*, 1493. (IF 2,337, FI: 15, TÁ: 100%)

[10] Amadeu, N.; Bálint, E.; Boenigk, W.; **Tajti, Á.**; Hägele, G.; Janiak, C.; Keglevich, G. *Phosphorus, Sulfur, Silicon Relat. Elem.* **2017**, *192*, 643. (IF 0,674, FI: 1, TÁ: 50%)

7.2. Short publications related to the PhD Thesis

[11] **Tajti, Á.**; Tóth, R. E.; Kalocsai, D.; Keglevich, G.; Bálint, E. *Phosphorus, Sulfur, Silicon Relat. Elem.* **2016**, *191*, 1541. (IF 0,809, FI: 2, TÁ: 80%)

[12] **Tajti, Á.**; Bálint, E.; Keglevich, G. *Phosphorus, Sulfur, Silicon Relat. Elem.* **2019**, *194*, 379. IF(2018) 0,674, FI: 0, TÁ: 100%)

7.3. Publications indirectly related to the PhD Thesis

[13] Bálint, E.; Fazekas, E.; Takács, J.; **Tajti, Á.**; Juranovič, A.; Kočevar, M.; Keglevich, G. *Phosphorus, Sulfur, Silicon Relat. Elem.* **2013**, *188*, 48. (IF 0,827, FI: 3, TÁ: 20%)

[14] Keglevich, G.; Bálint, E.; **Tajti, Á.**; Mátravölgyi, B.; Balogh, G. T.; Bálint, M.; Ilia, G. *Pure Appl. Chem.* **2014**, 86, 1723. (IF 2,492, FI: 0, TÁ: 85%)

[15] Bálint, E.; Tripolszky, A.; Ádám, A.; **Tajti, Á.**; Keglevich, G. *Phosphorus, Sulfur, Silicon Relat. Elem.* **2016**, *191*, 1539. (IF 0,809, FI: 0, TÁ: 30%)

[16] **Tajti, Á.**; Keglevich, G.; Bálint, E. *Phosphorus, Sulfur, Silicon Relat. Elem.* **2017**, *192*, 769. (IF 0,674, FI: 0, TÁ: 100%)

[17] Bálint, E.; **Tajti, Á.**; Tripolszky, A.; Keglevich, G. *Dalton Trans.* **2018**, *47*, 4755. (IF 4,099, FI: 8, TÁ: 70%)

[18] **Tajti, Á.**; Keglevich, G. *The importance of organophosphorus compounds as biologically active agents*. In: Organophosphorus Chemistry; Keglevich, G. (ed.); Walter de Gruyter: Berlin, 2018, p 53. (FI: 5, TÁ: 100%)

[19] Bálint, E.; Tripolszky, A.; **Tajti, Á.** *Synthesis of α-aminophosphonates by the Kabachnik–Fields reaction and by the Pudovik reaction*. In: Organophosphorus Chemistry; Keglevich, G. (ed.); Walter de Gruyter: Berlin, 2018, p 108. (FI: 1, TÁ: 50%)

[20] Bálint, E.; **Tajti, Á.**, Keglevich, G. *Materials*, **2019**, *12*, 788. (IF(2018) 2,972, FI: 0, TÁ: 100%)

[21] Tóth, N.; **Tajti, Á.**; Ladányi-Pára, K.; Bálint, E.; Keglevich, G. *Phosphorus, Sulfur, Silicon Relat. Elem.* **2019**, *194*, 285. IF(2018) 0,674, FI: 0, TÁ: 30%)

7.4. Additional publications

[22] Banerjee, B; **Tajti, Á.**; Keglevich, G. *Ultrasound-assisted synthesis of organophosphorus compounds*. In: Organophosphorus Chemistry; Keglevich, G. (ed.); Walter de Gruyter: Berlin, 2018, p 248. (FI: 0, TÁ: 50%)

7.5. Conference proceedings

1. **Tajti, Á.**; Bálint, E. *Tavaszi Szél 2015 Conference book* **2015**, 183. Conference place, time: Eger, Hungary, 2015.04.10-12. (ISBN 978-615-5250-11-8)
2. **Tajti Á.**; Bálint E. *IV. Interdiszciplinális Doktorandusz Konferencia Conference book* **2015**, 553. Conference place, time::Pécs, Hungary, 2015.05.14-15. (ISBN 978-963-642-830-3)
3. **Tajti, Á.**; Tóth, N.; Kalocsai, D.; Szatmári, E.; Keglevich, G.; Bálint, E. In: Ádám, A. A.; Ziegenheim, Sz. (szerk.) I. FKF Szimpózium, Fiatal Kémikusok Fóruma Konferencia kiadvány, 2019, pp. 105. Conference place, time: Debrecen, Hungary, 2019.04.3-5. (ISBN: 978-615-6018-00-7)

7.6. Oral presentations

1. **Tajti, Á.**; Bálint, E.; Keglevich, G. *H-foszfonátok alkoholízise mikrohullámú körülmények között*, Tavaszi Szél 2015 konferencia, Eger, 2015.
2. **Tajti, Á.**; Bálint, E.; Keglevich, G. *H-foszfonátok alkoholízisének tanulmányozása*, IV. Interdiszciplináris Doktorandusz Konferencia, Pécs, 2015.
3. **Tajti, Á.**; Bálint, E.; Keglevich, G. *A foszforatomon különböző alkilcsoportokkal rendelkező aminofoszfonátok és aminometilén-biszfoszfonátok előállítása*, XXXVIII. Kémiai Előadói Napok, Szeged, 2015.
4. **Tajti, Á.**; Ádám, A.; Kalocsai, D.; Keglevich, G.; Bálint, E. *Aminofoszfonát- és (aminometilén)biszfoszfonát-származékok előállítása mikrohullámú körülmények között*, Vegyészkonferencia 2017, Hajdúszoboszló, 2017.

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7.7. Poster presentations

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2. Bálint, E.; Tripolszky, A.; Ádám, A.; **Tajti, Á.**; Keglevich, G. *Synthesis and utilization of α-aminophosphine oxides and related derivatives*, 21st International Conference on Phosphorus Chemistry, Kazan, Oroszország, 2016.06.05-10.

3. **Tajti, Á.**; Tóth, R. E.; Kalocsai, D.; Keglevich, G.; Bálint, E. *Formation of compounds with P–C–N moiety by microwave-assisted condensations*, 21st International Conference on Phosphorus Chemistry, Kazan, Oroszország, 2016.06.05-10.

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