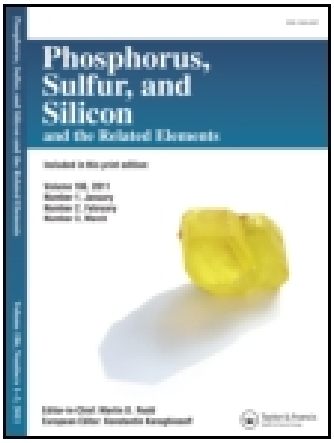

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A Study on the Reduction of 4-Chloro-1,2-Dihydrophosphinine Oxides by Transfer Hydrogenation

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A STUDY ON THE REDUCTION OF 4-CHLORO-1,2-DIHYDROPHOSPHININE OXIDES BY TRANSFER HYDROGENATION

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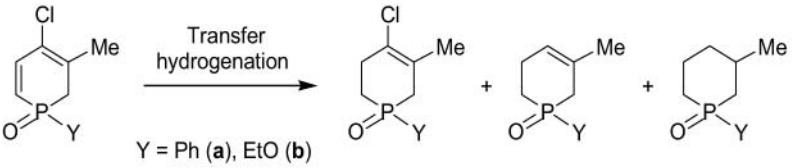
GRAPHICAL ABSTRACT



Abstract *Under the conditions of transfer hydrogenation by ammonium formate, the title compounds (**6**) were converted to a mixture of the corresponding 1,2,3,6-tetrahyidrophosphinine oxide (**7**), its dechlorinated derivative (**8**) and the respective 1,2,3,4,5,6-hexahydrophosphinine oxide (**9**). In the phenyl-substituted instance, the ratios of the components depended on the mode of heating (traditional heating or microwave irradiation), temperature, and the solvent (toluene or [bmim][BF_4]) used. In the ethoxy-substituted series, the dechlorinated 1,2-dihydrophosphinine oxide (**10b**) was detected as an intermediate.*

Keywords Transfer hydrogenation; 1,2-dihydrophosphinine oxide; 1,2,3,6-tetrahydrophosphinine oxide; 1,2,3,4,5,6-hexahydrophosphinine oxide; dechlorination; intermediate

INTRODUCTION

Six-membered P-heterocycles called phosphinine derivatives form a representative group of P-heterocycles.[1–3] An attractive approach toward dihydro-, tetrahydro-, and hexahydrophosphinine oxides is based on ring enlargement of easily available 3-phospholene

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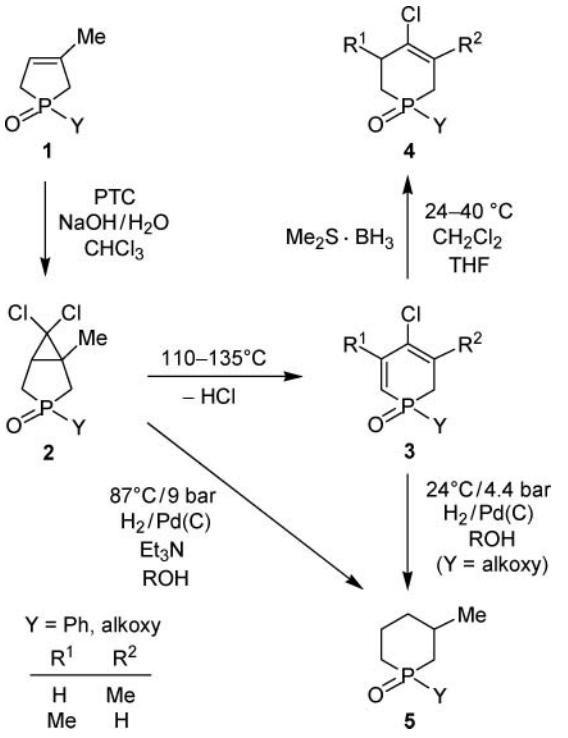
oxides and their subsequent transformations. In the first step, dichlorocarbene is added to the double-bond of 3-phospholene oxide (**1**) to afford 3-phosphabicyclo[3.1.0]hexane 3-oxide (**2**). Then the cyclopropane ring of the dichlorocarbene adduct **2** is opened up on heating, resulting in the formation of 1,2-dihydrophosphinine oxide **3** as a mixture of two double-bond isomers.[4,5] Selective reduction of the α,β-double-bond of 1,2-dihydrophosphinine oxide **3** by dimethyl sulfide-borane led to 1,2,3,6-tetrahydrophosphinine oxides **4**,[6] while catalytic hydrogenation of 1,2-dihydrophosphinine oxides **3**, or even that of 3-phosphabicyclo[3.1.0]hexane oxides **2** provided 1,2,3,4,5,6-hexahydrophosphinine oxides **5** (Scheme 1).[7–9]



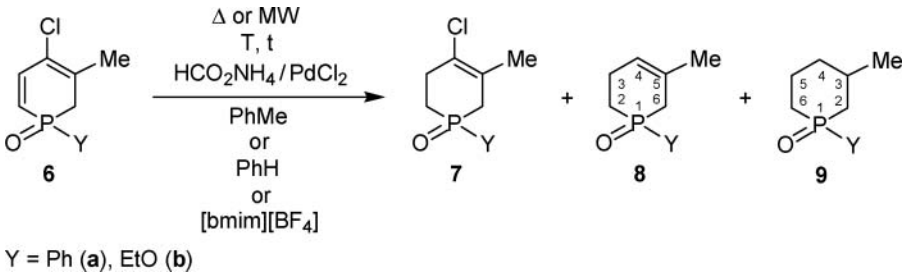
Scheme 1[4–8]

Catalytic transfer hydrogenation is a useful synthetic method for the reduction of organic compounds.[10,11] Recently, transfer hydrogenations applying ammonium formate derivatives as the hydrogen source have been carried out in ionic liquids (ILs).[12–17] Substrates with C=C and C=N groups, as well as the carbonyl group of ketones, were reduced in ILs (e.g. in [bmim][PF_6]) in the presence of Pd/C or ruthenium complex catalysts.[12–14] The unsaturation of cinnamic acid derivatives was reduced in a few imidazolium ILs using $Pd(OAc)_2$, Pd/MgLa mixed oxide or Pd/MgAl hydrotalcite catalysts.[15,16] The outcome of the rhodium catalyzed transfer hydrogenation of α,β-unsaturated ketones depended on the nature of the solvent used. In ILs only the unsaturation was reduced, but in conventional solvents the carbonyl group was reduced in addition.[17]

In this paper, the transfer hydrogenation of 1,2-dihydrophosphinine oxides under different conditions, including the use of ILs and/or microwave (MW) irradiation, is discussed.

RESULTS AND DISCUSSION

In the first approach, 1-phenyl-1,2-dihydrophosphinine oxide **6a** was reduced with a 20-fold quantity of ammonium formate in the presence of $PdCl_2$ as the catalyst in different solvents (Scheme 2). After a 4 h reflux in toluene, the crude mixture contained 22% of 4-chloro-1,2,3,6-tetrahydrophosphinine oxide **7a**,[6] 31% of 1,2,3,6-tetrahydrophosphinine oxide **8a**[9], and 47% of 1,2,3,4,5,6-hexahydrophosphinine oxide **9a**[8] (Table 1/Entry 1). The components were identified after analyzing the mixture by ^{31}P NMR and mass spectrometry, along with LC-MS liquid chromatography-mass spectrometry. Repeated column chromatography led to a 55%:45% mixture of hexahydrophosphinine oxide **9a** and tetrahydrophosphinine oxide **8a**. The ^{13}C NMR spectrum of this fraction confirmed the presence of components **8a**[9] and **9a**.[8] It is noteworthy that the hexahydrophosphinine oxide (**9a**) was formed as only one diastereomer, meaning that its formation by transfer hydrogenation was diastereoselective. The earlier described catalytic hydrogenation of dihydrophosphinine oxide **6a** afforded hexahydrophosphinine oxide **9a** as a 77:23 mixture of two diastereomers.[8]



Scheme 2

Carrying out the reduction in toluene, but under MW conditions, the dechlorinated tetrahydrophosphinine oxide (**8a**) was the major (79%) P-heterocycle formed along with 11% of **7a** and 5% of **9a**. There were also a few unidentified byproducts in a small quantity (∼5%) in the reaction mixture (Table 1/Entry 2). By replacing toluene with [bmim][BF_4], the relative quantity of the chloro-tetrahydrophosphinine oxide (**7a**) decreased, but that of the dehalogenated tetrahydrophosphinine oxide (**8a**), or the hexahydrophosphinine oxide (**9a**) increased. On heating at 110 °C for 3 h, only 10% of compound **7a** and 41%

Table 1 Transfer hydrogenation of 1,2-dihydrophosphinine oxides **6** under different conditions

Entry	Y	Mode of heating	Solvent	T (°C)	t (h)	Composition (%)[a]			
						7	**8**	**9**	Other
1	Ph (**a**)	Δ	PhMe	110	4	22	31	47	
2	Ph (**a**)	MW	PhMe	110	2	11	79	5	5
3	Ph (**a**)	Δ	[bmim][BF_4]	110	3	10	41	49	
4	Ph (**a**)	MW	[bmim][BF_4]	110	2	2	59	39	
5	Ph (**a**)	Δ	PhH	78	4	9	44	47	
6	Ph (**a**)	Δ	[bmim][BF_4]	130	1.5	3	45	52	
7	EtO (**b**)	Δ	PhMe	110	24	10	36	54	

[a]Determined on the basis of relative ^{31}P NMR intensities.

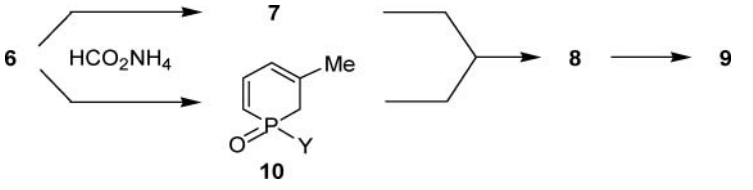
of its dechlorinated form (**8a**) were formed (Table 1/Entry 3), while MW irradiation at 110 °C for 2 h resulted in, 2% of **7a** and 39% of hexahydrophosphinine oxide **9a**. In the thermal experiment, the hexahydrophosphinine oxide **9a** (49%) dominated, while in the MW variation the dehalogenated tetrahydrophosphinine oxide **8a** (59%) was the major product (Table 1/Entry 4). Hence, under MW irradiation it was possible to carry out the reaction so that the dechlorinated tetrahydrophosphinine oxide (**8a**) was formed as the major component (Table 1/Entries 2 and 4). If the dechlorinated tetrahydrophosphinine oxide (**8a**) is the target compound, whose preparation is not easy by the other method described,[9] the transfer hydrogenation should be carried out under MW irradiation using toluene as the solvent. Tetrahydrophosphinine oxide **8a** may be a valuable intermediate in further syntheses to be explored in due course. In boiling benzene, the reduction was not more selective for the formation of the chloro-tetrahydrophosphinine oxide (**7a**) (Table 1/Entry 5). In IL at 130 °C, the reduction did not proceed to afford the hexahydrophosphinine oxide **9a** as an almost exclusive product (Table 1/Entry 6). It can be seen that variation of the temperature in the range of 80 °C –100 °C does not have much impact on the course of the transfer hydrogenation.

It is also noteworthy that a large excess, namely a 20-fold molar equivalent of the ammonium formate, must be used, otherwise, the conversion of the starting material (**6a**) to any of the products (**7a**, **8a**, or **9a**) is incomplete. In the dihydrophosphinine oxide **6a**, there are three functions that may be reduced, and the reaction sequence could not be influenced to take place with an exclusive selectivity toward any of the products. There is also a need to use the $PdCl_2$ catalyst in a quantity of 50%. In the experiments, where only 10% of the catalyst was used, there was practically no reduction.

The 1-ethoxy-1,2-dihydrophosphinine oxide (**6b**) was also subjected to transfer hydrogenation applying ammonium formate and $PdCl_2$ in a the similar reaction of the phenyl substituted starting material (**6a**) and using toluene as the solvent at the boiling point. After a 24 h of stirring at reflux, a mixture of 4-chloro-1,2,3,6-tetrahydrophosphinine oxide **7b**[6] (10%), its dechlorinated form **8b** (36%) and the 1,2,3,4,5,6-hexahydrophosphinine oxide **9b**[8] (54%) was obtained (Scheme 2, Table 1/Entry 7). Composition of the mixture was analyzed by ^{31}P NMR, mass spectrometry and LC-MS after flash column chromatography. The ^{13}C NMR spectrum obtained on this mixture was also informative as the ^{13}C NMR data of compounds **7b**[6] and **9b**[8] were known.[6,8] Repeated column chromatography afforded tetrahydrophosphinine oxide **8b** in a yield of 16% and in a purity of 96%. The structure of this new compound was proved by ^{31}P, ^{13}C, and ^{1}H NMR, as well as MS methods.

We observed that varying the reaction conditions for the transfer hydrogenation of dihydrophosphinine oxide **6b** did not influence the outcome of the reaction to a significant extent.

Tetrahydrophosphinine oxides **7** and **8**, as well as the dechlorinated 1,2-dihydrophosphinine oxide (**10**), may be intermediates toward the fully saturated hexahydrophosphinine oxide (**9**) (Scheme 3). Intermediate **10** could only be detected in the



Scheme 3

ethoxy-substituted model (Y = EtO, **10b**) after interrupting the reaction before completion and analyzing the mixture by ^{31}P NMR (δ_P ($CDCl_3$) 36.2) and MS ($[M+H]^+_{found}$ = 173.0733, $C_8H_{14}O_2P$ requires 173.0731).

In summary, it was found that the transfer hydrogenation of 4-chloro-1,2-dihydrophosphinine oxides led to a mixture of three products, a chloro-tetrahydrophosphinine oxide, its dechlorinated form, and the fully saturated hexahydrophosphinine oxide formed diastereoselectively. In the phenyl-substituted case, the relative proportion of the components depended on the conditions applied. Choosing appropriate conditions (MW/PhMe/110 °C/2 h), the dechlorinated 1-phenyl-1,2,3,6-tetrahydrophosphinine oxide (**8a**) could be obtained in a selectivity of 79%. In the ethoxy-substituted series, the dechlorinated dihydrophosphinine oxide could also be detected as an intermediate.

EXPERIMENTAL

The ^{31}P and ^{13}C NMR spectra were recorded on a Bruker DRX-500 spectrometer operating at 202.4 and 125.7 MHz, respectively. The couplings are given in Hz. Mass spectrometry was performed on a ZAB-2SEQ instrument.

LC-MS experiments were carried out on an Agilent 1200 liquid chromatography system coupled with a 6120 MSD (Agilent Technologies, Palo Alto, CA, USA). Analysis was at 40 °C on an Kinetex C_{18} column (5 cm × 2.1 mm, 2.6 μm) (Phenomenex, Torrance, CA, USA) with a mobile phase flow rate of 0.9 mL/min. Composition of eluent A was 0.1% (v/v) trifluoroacetic acid in water (pH 1.9), and eluent B was the mixture of acetonitrile and water in 95:5 (v/v) with 0.1% (v/v) trifluoroacetic acid. A fast linear gradient of 0%–100% B was applied at a range of 0–4 min, then 100% B held for 3 min. The injection volume was set at 1 μL, and the sample concentration was uniformly 1.0 mg/mL (using dimethyl sulfide as the solvent). The chromatographic profile was registered at 240 nm. The Mass spectrometry defector operating parameters were as follows: positive ionization mode, scan spectra from *m/z* 100 to 800, drying gas temperature 350 °C, nitrogen flow rate 12 L/min, nebuliser pressure 60 psi, quadrupole temperature 100 °C, capillary voltage 3000 V, and fragmentor voltage 50 V.

The MW experiments were carried out in a CEM Discover 300 W reactor equipped with a pressure control system.

The starting 1,2-dihydrophosphinine oxides (**6a** and **6b**) were prepared by our methods described earlier.[18–20]

A Typical Procedure for the Transfer Hydrogenation of 1-Phenyl-4-Chloro-3-Methyl-1,2-Dihydrophosphinine Oxide 6a (Table 1/Entry 1)

To a mixture of 0.15 g (0.63 mmol) of dihydrophosphinine oxide **6a** and 60 mg (0.34 mmol) of $PdCl_2$ in dry toluene (10 mL) was added 0.80 g (12.7 mmol) of ammonium formate, and the contents of the flask were stirred at the boiling point for 4 h. After cooling to room temperature, the solids were removed by filtration, and the solvent was evaporated. The crude product obtained was passed through a 10 cm layer of silica gel using 3% methanol in chloroform as the eluant. This mixture consisted of 22% of 4-chloro-tetrahydrophosphinine oxide **7a** (^{31}P NMR ($CDCl_3$) δ 27.7, δ_P[6] 28.2; $[M+H]^+_{found}$ = 241.0552, $C_{12}H_{15}{}^{35}ClOP$ requires 241.0549), 31% of tetrahydrophosphinine oxide **8a**, and 47% of hexahydrophosphinine oxide **9a**. Further chromatography (as described above, but

on a longer column) of this fraction provided a 55:45 mixture of products **9a** and **8a** as an oil.

8a: ^{31}P NMR ($CDCl_3$) δ 29.8, δ_P[9] 30.5; ^{13}C NMR ($CDCl_3$) δ 22.5 ($J = 5.3$, C_3), 24.2 ($J = 67.4$, C_6), 26.1 ($J = 11.1$, Me), 31.9 ($J = 66.2$, C_2), 122.9 ($J = 12.6$, C_4), (δ_C[9] 22.1 ($J = 4.9$, C_3), 23.8 ($J = 67.4$, C_6), 25.7 ($J = 11.1$, Me), 31.4 ($J = 66.2$, C_2), 122.5 ($J = 12.6$, C_4)); ^{1}H NMR ($CDCl_3$) δ 1.76 (s, 3H, Me), 5.59 (broad signal, 1H, CH=); $[M+H]^+_{found}$ = 207.0938, $C_{12}H_{16}OP$ requires 207.0939.

9a: ^{31}P NMR ($CDCl_3$) δ 32.7, δ_P[8] 33.3; ^{13}C NMR ($CDCl_3$) δ 20.9 ($J = 5.7$, C_5), 24.7 ($J = 14.9$, Me), 27.4 ($J = 66.3$, C_6), 28.5 ($J = 4.7$, C_3), 35.5 ($J = 5.1$, C_4), 36.6 ($J = 64.8$, C_2), (δ_C[8] 22.3 ($J = 3.7$, C_5), 24.1 ($J = 14.7$, Me), 26.8 ($J = 65.3$, C_6), 31.1 ($J = 3.6$, C_3), 34.5 ($J = 5.2$, C_4), 35.7 ($J = 63.3$, C_2)); ^{1}H NMR ($CDCl_3$) δ 1.07 (dd, $J^1 = 6.6$, $J^2 = 2.4$, 3H, Me), δ_H[8] 1.08 (dd, $J = 6$, 3H, Me); $[M+H]^+_{found}$ = 209.1090, $C_{12}H_{18}OP$ requires 209.1095.

A Typical Procedure for the Transfer Hydrogenation of 1-Ethoxy-4-Chloro-3-Methyl-1,2-Dihydrophosphinine Oxide (6b)

The reaction was carried out as that described for the phenyl substituted starting material **6a**. 0.38 g (1.84 mmol) of **6b** and 2.3 g (36.8 mmol) of ammonium formate were reacted in toluene (25 mL), in the presence of 0.16 g (0.92 mmol) of $PdCl_2$ by stirring the mixture at the boiling point for 24 h. Flash column chromatography (as above) furnished a mixture of 10% of 4-chloro-1,2,3,6-tetrahydrophosphinine oxide **7b**, 36% of 1,2,3,6-tetrahydrophosphinine oxide **8b**, and 54% of 1,2,3,4,5,6-hexahydrophosphinine oxide **9b** as an oil. Repeated column chromatography gave compound **8b** in a yield of 16% in a purity of 96%.

7b: ^{31}P NMR ($CDCl_3$) δ 45.1, δ_P[6] 45.6; ^{13}C NMR ($CDCl_3$) δ 16.4 ($J = 3.1$, CH_2CH_3), 22.8 ($^3J = 11.5$, C_5–Me), 23.8 ($^1J = 88.6$, C_6), 31.4 ($^1J = 86.6$, C_2), 32.0 ($^2J = 6.3$, C_3), 59.7 ($^2J = 5.4$, CH_2CH_3), 124.4 ($^3J = 4.9$, C_5), 126.3 ($J = 13.3$, C_4), (δ_C[6] 16.6 (CH_2CH_3), 23.2 ($^3J = 11.0$, C_5–Me), 24.5 ($^1J = 89.1$, C_6), 31.6 ($^1J = 89.7$, C_2), 32.3 ($^2J = 4.9$, C_3), 60.6 ($^2J = 5.4$, CH_2CH_3), 124.8 (C_5), 126.6 ($^3J = 12.9$, C_4)); $[M+H]^+_{found}$ = 209.0509, $C_8H_{15}{}^{35}ClO_2P$ requires 209.0498.

8b: ^{31}P NMR ($CDCl_3$) δ 48.7; ^{13}C NMR ($CDCl_3$) δ 16.4 ($J = 3.9$, CH_2CH_3), 21.9 ($J = 89.5$, C_6), 23.2 ($J = 5.9$, C_3), 25.7 ($J = 12.9$, C_5–Me), 30.2 ($J = 88.3$, C_2), 59.7 ($J = 3.3$, CH_2CH_3), 121.7 ($J = 13.9$, C_4), 128.9 ($J = 4.7$, C_5); ^{1}H NMR ($CDCl_3$) δ 1.34 (t, $J = 7.0$ CH_2CH_3), 1.75 (s, C_5–Me), 4.03–4.17 (m, CH_2CH_3), 5.26 (s, C_4H); $[M+H]^+_{found}$ = 175.0886, $C_8H_{16}O_2P$ requires 175.0888.

9b[8]: ^{31}P NMR ($CDCl_3$) δ 51.8, δ_P[8] 50.7; ^{13}C NMR ($CDCl_3$) δ 16.4 ($J = 5.5$, CH_2CH_3), 21.6 ($J = 3.7$, C_5), 23.8 ($J = 16.3$, C_3–Me), 26.1 ($J = 86.5$, C_6), 30.2 ($J = 3.1$, C_3), 34.7 ($J = 6.2$, C_4), 35.0 ($J = 83.9$, C_2), 59.7 ($J = 6.2$, CH_2CH_3), (δ_C[8] 15.5 ($J = 5.9$, CH_2CH_3), 21.8 ($J = 5.2$, C_5), 23.1 ($J = 16.1$, C_3–Me), 25.0 ($J = 86.4$, C_6), 30.3 ($J = 5.1$, C_3), 33.7 ($J = 6.5$, C_4), 34.3 ($J = 84.3$, C_2), 58.2 ($J = 5.8$, CH_2CH_3)); $[M+H]^+_{found}$ = 177.1042, $C_8H_{18}O_2P$ requires 177.1044.

REFERENCES

1. Mathey, F. Ed. *Phosphorus–Carbon Heterocyclic Chemistry: The Rise of a New Domain* (Pergamon, Amsterdam, 2001).

2. Keglevich, G. In *Topics in Heterocyclic Chemistry*, Gupta, R. R.; Bansal, R. K., Ed. (Springer, Dordrecht, 2009), Vol 20, pp. 65-98.
3. Keglevich, G. *Curr. Org. Chem.* **2006,** 10, 93-111.
4. Keglevich, G. *Synthesis* **1993,** 931-942.
5. Keglevich, G. *Rev. Heteroatom Chem.* **1996,** 14, 119-136.
6. Keglevich, G.; Böcskei, Z.; Újszászy, K.; Tőke, L. *Synthesis* **1997,** 1391-1393.
7. Keglevich, G.; Kovács, A.; Újszászy, K.; Tungler, A.; Tóth, G.; Tőke, L. *Phosphorus, Sulfur, Silicon* **1992,** 70, 219-227.
8. Keglevich, G.; Tungler, A.; Novák, T.; Tőke, L. *J. Chem. Res. (S)* **1996,** 528-529.
9. Keglevich, G.; Novák, T.; Tungler, A.; Hegedűs, L.; Á. Szöllősy, Ludányi, K.; Tőke, L. *J. Chem. Res. (S)* **1997,** 290-291.
10. Brieger, G.; Nestrick, T. J. *J. Chem. Rev.* **1974,** 74, 567-580.
11. Johstone, R. A.; Wilby, W. A. H.; Entwistle, I. D. *Chem. Rev.* **1985,** 85, 129-170.
12. Bertold, H.; Schotten, T.; Hönig, H. *Synthesis* **2002,** 11, 1607-1610.
13. Geldbach, T. J.; Dyson, P. J. *J. Am. Chem. Soc.* **2004,** 126, 8114-8115.
14. Kawasaki, I.; Tsunoda, K.; Tsuji, T.; Yamaguchi, T.; Shibuta, H.; Uchida, N.; Yamashita, M.; Ohta, S. *Chem. Commun.* **2005,** 2134-2136.
15. Baán, Z.; Finta, Z.; Keglevich, G.; Hermecz, I. *Tetrahedron Lett.* **2005,** 46, 6203-6204.
16. Baán, Z.; Potor, A.; Cwik, A.; Hell, Z.; Keglevich, G.; Finta, Z.; Hermecz, I. *Synth. Commun.* **2008,** 38, 1601-1609.
17. Baán, Z.; Finta, Z.; Keglevich, G.; Hermecz, I. *Green Chem.* **2009,** 11, 1937-1940.
18. Keglevich, G.; Petneházy, I.; Miklós, P.; Almásy, A.; Tóth, G.; Tőke, L.; Quin, L. D. *J. Org. Chem.* **1987,** 52, 3983-3986.
19. Keglevich, G.; Androsits, B.; Tőke, L. *J. Org. Chem.* **1988,** 53, 4106-4108.
20. Keglevich, G.; Brlik, J.; Janke, F.; Tőke, L. *Heteroat. Chem.* **1990,** 1, 419-424.