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Efficient Combination of Task-Specific Ionic Liquid and Microwave Dielectric Heating Applied to Synthesis of a Large Variety of Nitrones

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ABSTRACT: *Under mild microwave irradiation conditions and without any additional organic solvents, a large variety of nitrones were prepared in a weak Lewis base phosphinite task-specific ionic liquid (TSIL-OPPh$_2$) in excellent yields. This catalytic TSIL was also recyclable. Under optimized microwave irradiation conditions, the reaction occurred at very shorter reaction time and higher yields in comparison with conventional heating conditions.* © 2010 Wiley Periodicals, Inc. Heteroatom Chem 21:78–83, 2010; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20581

INTRODUCTION

The importance of green reactions in organic synthesis has encouraged scientists to explore the use of room-temperature ionic liquids (RTILs) as a powerful alternative to conventional molecular organic solvents or catalysts due to their particular properties, such as undetectable vapor pressure, wide liquid range, as well as the ease of recovery and reuse [1–5]. More recently, much more attention has been focused on the synthesis of functionalized ionic liquids (FILs), through incorporation of additional functional groups as a part of the cation and/or anion, so-called task-specific ionic liquids (TSILs), and their applications in chemical research. They have shown great promise not only as alternative green solvents but also as reagents or catalysts in organic transformations [1,4]. Wang and Li used TSIL as Lewis base, ligand and reaction medium for the palladium-catalyzed Heck reaction [4]. Paun et al. reported the basic ionic liquid catalyzed Knoevenagel condensation reaction [6]. Very recently, we used the TSIL-OPPh$_2$ as weak Lewis base catalyst and reaction medium for the Biginelli and Knoevenagel reactions [7,8].

Nitrones are highly valuable synthetic intermediates in organic synthesis [9,10] and are employed, for instance, in stereoselective formation of synthetically useful isoxazolidines by their 1,3-dipolar cycloaddition reaction with alkenes [11–13]. Recently, novel reactivities have been described [14,15]. For the preparation of nitrones, the most popular method is the condensation of aldehydes or ketones with *N*-monosubstituted hydroxylamines [16]. Colacino et al. reported the synthesis of various C-aryl and C-alkyl-nitrones under solvent-free conditions in a ball-mill apparatus [17]. Kim et al. prepared the α-phenyl-*N*-tert-butyl nitrones and studied the protective effects of these compounds against

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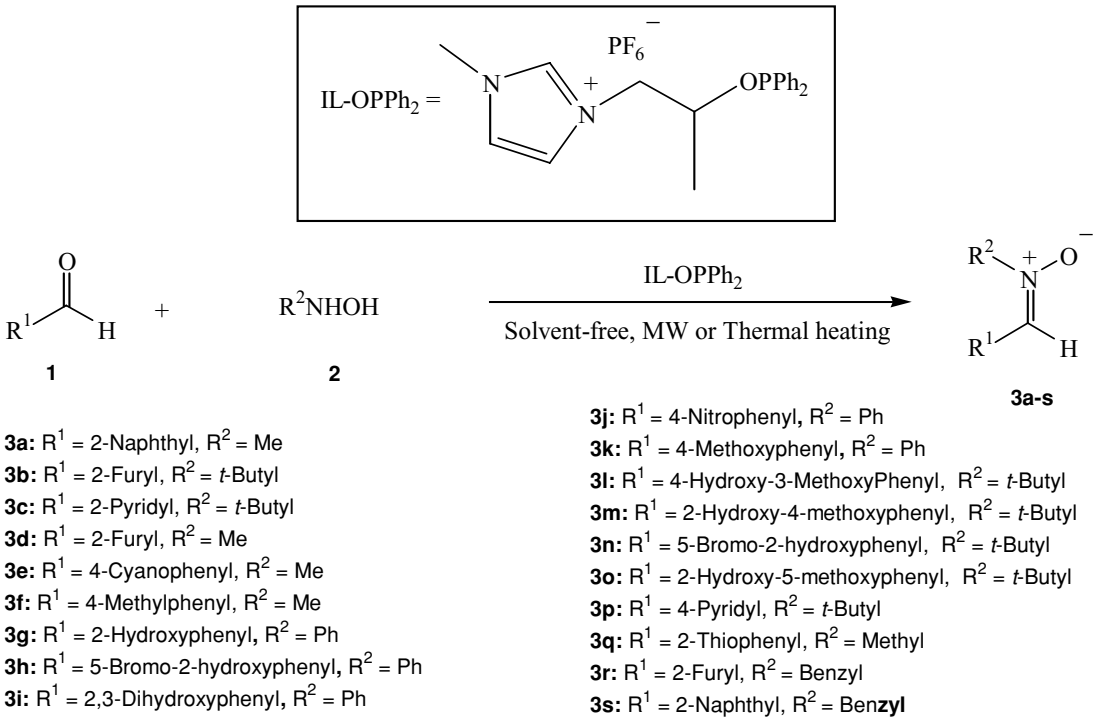
microvascular damages in schemia/reperfusion in the "hamster cheek pouch" assay [18].

The use of microwave irradiation has been employed for a number of organic syntheses to reduce the reaction time and rate enhancement, and to increase the selectivity and yields. Microwave irradiation has also been used for the synthesis of α-(5-substituted-2-hydroxyaryl)-*N*-aryl nitrone [19]. Recently, Bortolini and Maiuolo reported the synthesis of nucleoside derivatives by the microwave-assisted 1,3-cycloaddition reaction of vinyl nucleobases with nitrones under solvent-free conditions [20]. Very recently, we reported the high yield synthesis of a large variety of nitrones over MgO at room temperature under solvent-free conditions [21]. To the best of our knowledge, no reports are available using ionic liquids for the synthesis of nitrones. In general, the essential requirement for the synthesis of nitrones includes a base and a reaction medium. This promoted us to use a task-specific ionic liquid, IL-OPPh$_2$, which acts as a weak Lewis base as well as an environmentally benign reaction medium in an ion-pair unity, for the synthesis of nitrones. Very recently, we used IL-OPPh$_2$ as an efficient and recyclable basic catalyst and reaction medium [7–8]. Here, we report the development of this ionic liquid, which exhibits a very high activity and recyclability to the synthesis of nitrones. In continuation of our work in studying organic reactions in ionic liquids, water, or solventless systems as a green reaction medium [22–26], we report the synthesis of nitrones via the condensation of *N*-(aryl and alkyl)hydroxylamines with arylaldehydes in TSIL-OPPh$_2$ under microwave irradiation (Scheme 1).

EXPERIMENTAL

General Information

All reagents were purchased from Merck (Germany) and were used without further purification. ^{1}H NMR spectra were obtained in $CDCl_3$ solution from Bruker Avance AC-400 MHz (or 300 MHz) and ^{13}C NMR spectra at 100 MHz (or 75 MHz) on the aforementioned instruments. Mass spectra were recorded on a Platform II (Micromass, Manchester, UK) quadrupole mass spectrometer fitted with an electrospray interface. Elemental analyses were carried out on a Perkin-Elmer 240C elemental analyzer and are reported in percent atomic abundance. All melting points are uncorrected and are measured in an open glass-capillaries using Stuart melting point apparatus. Microwave experiments were conducted in a Milestone MicroSYNTH apparatus.



3a: R^1 = 2-Naphthyl, R^2 = Me
3b: R^1 = 2-Furyl, R^2 = *t*-Butyl
3c: R^1 = 2-Pyridyl, R^2 = *t*-Butyl
3d: R^1 = 2-Furyl, R^2 = Me
3e: R^1 = 4-Cyanophenyl, R^2 = Me
3f: R^1 = 4-Methylphenyl, R^2 = Me
3g: R^1 = 2-Hydroxyphenyl, R^2 = Ph
3h: R^1 = 5-Bromo-2-hydroxyphenyl, R^2 = Ph
3i: R^1 = 2,3-Dihydroxyphenyl, R^2 = Ph
3j: R^1 = 4-Nitrophenyl, R^2 = Ph
3k: R^1 = 4-Methoxyphenyl, R^2 = Ph
3l: R^1 = 4-Hydroxy-3-MethoxyPhenyl, R^2 = *t*-Butyl
3m: R^1 = 2-Hydroxy-4-methoxyphenyl, R^2 = *t*-Butyl
3n: R^1 = 5-Bromo-2-hydroxyphenyl, R^2 = *t*-Butyl
3o: R^1 = 2-Hydroxy-5-methoxyphenyl, R^2 = *t*-Butyl
3p: R^1 = 4-Pyridyl, R^2 = *t*-Butyl
3q: R^1 = 2-Thiophenyl, R^2 = Methyl
3r: R^1 = 2-Furyl, R^2 = Benzyl
3s: R^1 = 2-Naphthyl, R^2 = Ben**zyl**

SCHEME 1 Synthesis of nitrones in IL-OPPh$_2$ as a catalyst and reaction medium.

TABLE 1 TSIL-$OPPh_2$ Catalyzed Synthesis of Nitrones under Microwave and Thermal Heating Conditions[a]

Entry	Product[b]	Time		Yield[c] (%)		Mp (°C)
		Microwave (min)	Thermal (h)	Microwave	Thermal	Found, Lit. [Ref.]
1	**3a**	5	2.5	96	87	106–109, 108–110 [17]
2	**3b**	4.5	3	95	89	67–68, 65.5 [18]
3	**3c**	5	3	93	83	64–66, 63–65 [17]
4	**3d**	4	2.5	98	91	88–90, 90 [28]
5	**3e**	3.5	3	96	84	183–185, 187 [29]
6	**3f**	5	3.5	92	81	117–120, 118–20 [30]
7	**3g**	4	3	95	87	129–131, 129–131 [21]
8	**3h**	4	3	99	90	173–176, 173–176 [21]
9	**3i**	5	3	98	89	159–161, 159–161 [21]
10	**3j**	5	2.5	97	84	181–183, 183–184 [28]
11	**3k**	4.5	3	93	80	113–116, 115–116 [28][b]
12	**3l**	4	2.5	99	88	193–195, 196 [18]
13	**3m**	3.5	3	99	92	13–116, 115.2–116 [31]
14	**3n**	4	3	98	88	115–118, 118.7 [31]
15	**3o**	5	3.5	96	87	69–71, 70.4–70.6 [29]
16	**3p**	5	3	99	87	83–85, 84–86 [17]
17	**3q**	4.5	3.5	99	90	113–115, 115–117 [17]
18	**3r**	4	3	97	89	84–86, 85–88 [17]
19	**3s**	4	3	99	90	102–105, 104–106 [17]

[a]Reaction conditions: 10 mmol of arylaldehyde, 10 mmol of *N*-monosubstituted hydroxylamine, and 10 mmol of TSIL.
[b]Products were characterized by elemental analysis and by comparison of their spectroscopic data (^{1}H NMR, ^{13}C NMR, and IR) and melting points with those reported in the literature.
[c]Isolated yields after recrystallization.

Preparation of Nitrones in TSIL-$OPPH_2$ under Microwave Irradiation

General Procedure. In a 10-mL glass microwave vessel, arylaldehyde derivatives (10 mmol), *N*-monosubstituted hydroxylamine (10 mmol), and TSIL-$OPPh_2$ (10 mmol) were placed. The mixture was subjected to microwave irradiation at 60 W for a few minutes (depending on the reactants; see Table 1). The completion of reaction was monitored by TLC using (EtOAc/petroleum 1:8) as eluent. After completion of the reaction, the mixture was cooled to room temperature and the mixture was extracted with ether or ethyl acetate. The extracts were concentrated on a rotary evaporator, and the crude mixture was purified by recrystallization using (EtOH/EtOAc) to afford corresponding nitrones (**3a–s**).

Preparation of Nitrones in TSIL-$OPPH_2$ under Thermal Conditions

General Procedure. The selected arylaldehyde derivative (10 mmol), *N*-monosubstituted hydroxylamine (10 mmol), and TSIL-$OPPh_2$ (10 mmol) were stirred at 70°C for appropriate time (Table 1). The completion of reaction was monitored by TLC using (EtOAc/petroleum) as eluent. After completion of the reaction, the mixture was extracted with ether or ethyl acetate. The extracts were concentrated on a rotary evaporator, and the crude mixture was purified by recrystallization using (EtOH/EtOAc) to afford corresponding nitrones.

Selected Spectroscopic Data

*C-(2-Furyl)-N-(t-butyl)-nitrone (**3b**).* IR ν_{max}/cm^{-1}: 3102, 2981, 2936, 1578 (C=N), 1150 (N—O) and 1052 (C—O). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (dd, $J = 3.91$ Hz, $J = 1.7$ Hz, 1H), 7.69 (s, 1H, nitronyl H), 7.48 (dd, $J = 2.70$, $J = 1.7$ Hz, 1H), 6.60 (dd, $J = 3.91$, $J = 2.70$ Hz, 1H), 1.58 (s, 9H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 148.20, 143.55, 123.12, 115.17, 113.32, 68.50, 29.01; EI-MS $[MH]^+$ *m/z* 168; Anal. Calcd (%) for $C_9H_{13}NO_2$: C, 64.65; H, 7.84; N, 8.38. Found (%): C, 64.81; H, 7.79; N, 7.21.

*C-(2-Hydroxyphenyl)-N-phenyl-nitrone (**3g**).* IR ν_{max}/cm^{-1}: 3500–3150 (OH), 1586 (C=N), 1159 (N—O), and 1033 (C—O). ^{1}H NMR (400 MHz, $CDCl_3$): δ 12.40 (s, 1H, OH), 7.92 (s, 1H, nitronyl H), 7.60–7.39 (m, 6H, ArH), 7.23 (t, $J = 8.9$ Hz, 1H), 7.04 (d, $J = 11.15$ Hz, 1H), 6.95 (t, $J = 8.9$ Hz, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 134.72, 134.69, 129.21, 128.20, 126.26, 125.39, 125.32, 121.16, 118.99, 118.95, 117.98; EI-MS $[MH]^+$ *m/z* 214; Anal. Calcd (%) for

$C_{13}H_{11}NO_2$: C, 73.22; H, 5.20; N, 6.57. Found (%): C, 72.98; H, 5.13; N, 6.48.

*C-(2,3-Dihydroxyphenyl)-N-phenyl-nitrone (**3i**).* IR ν_{max}/cm^{-1}: 3450–3000 (OH), 1569 (C=N), 1151 (N—O), and 1035 (C—O). ^1H NMR (400 MHz, $CDCl_3$): δ 12.40 (s, 1H, OH), 8.00 (s, 1H, nitronyl H), 7.77–7.75 (m, 2H, ArH), 7.52–7.47 (m, 5H, ArH and OH), 7.30 (d, $J = 2.3$ Hz, 1H), 6.90 (d, $J = 8.88$ Hz, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 159.04, 145.92, 139.56, 137.07, 134.38, 130.82, 129.46, 122.29, 121.79, 118.70, 110.70; ESI-MS $[MH]^+$ *m/z* 230; Anal. Calcd (%) for $C_{13}H_{11}NO_3$: C, 68.11; H, 4.84; N, 6.11. Found (%): C, 68.05; H, 4.83; N, 6.09.

*C-(4-Pyridyl)-N-(t-butyl)-nitrone (**3p**).* IR ν_{max}/cm^{-1}: 1588 (C=N), 1189 (N—O), and 1099 (C—O). ^1H NMR (300 MHz, $CDCl_3$): δ 8.86 (m, 2H), 8.15 (m, 2H), 7.58 (s, 1H, nitronyl H), 1.54 (s, 9H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 148.28, 137.18, 128.47, 124.44, 74.34, 51.40, 27.35, 27.20, 27.15; ESI-MS $[MH]^+$ *m/z* 179; Anal. Calcd (%) for $C_{11}H_{15}NO$: C, 74.54; H, 8.53; N, 7.90. Found (%): C, 75.01; H, 8.41; N, 7.75.

*C-(2-Furyl)-N-benzyl-nitrone (**3r**).* IR ν_{max}/cm^{-1}: 1582 (C=N), 1137 (N—O), and 1009 (C—O). ^1H NMR (300 MHz, $CDCl_3$): δ 7.79 (m, 1H), 7.62–7.30 (m, 7H), 7.64 (m, 1H), 5.16 (s, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 145.13, 143.09, 133.51, 129.01, 128.14, 128.04, 125.14, 116.30, 113.44, 69.02; ESI-MS $[MH]^+$ *m/z* 202; Anal. Calcd (%) for $C_{12}H_{11}NO_2$: C, 71.63; H, 5.51; N, 6.96. Found (%): C, 71.72; H, 5.42; N, 6.81.

*C-(2-Naphthyl)-N-benzyl-nitrone (**3s**).* IR ν_{max}/cm^{-1}: 1572 (C=N), 1161 (N—O), and 1119 (C—O). ^1H NMR (300 MHz, $CDCl_3$): δ 9.20 (s, CH=N, 1H), 7.89–7.74 (m, 4H), 7.62–7.28 (m, 8H), 5.10 (s, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 136.13, 135.21, 132.32, 132.01, 129.16, 129.06, 129.00, 128.21, 128.40, 127.83, 127.64, 127.46, 127.30, 126.41, 125.45, 70.32; ESI-MS $[MH]^+$ *m/z* 262; Anal. Calcd (%) for $C_{18}H_{15}NO$: C, 82.73; H, 5.79; N, 5.36. Found (%): C, 82.75; H, 5.76; N, 5.32.

RESULTS AND DISCUSSION

We have recently introduced an imidazolium-based phosphinite ionic liquid for the Horner–Wadsworth–Emmons-type reaction [27]. In the present report, an equimolar quantity of the phosphinite-functionalized imidazolium salt, TSIL-$OPPh_2$, as a Lewis base, gives clean nitrones by the condensation of aldehydes with *N*-monosubstituted hydroxylamines in high yields under microwave irradiation. For comparison, the reaction has also been carried out by thermal heating conditions as shown in Table 1. The optimization of the process by varying temperature, time, and TSIL-$OPPh_2$ was done to get products in high yields and purity. The yields of the products obtained by microwave irradiation verses thermal heating are higher with remarkable reduction in reaction time due to homogeneous heating (as a result of strong agitation of reactant molecules) throughout the reaction media by microwave irradiation as compared to convection currents in thermal heating. This methodology avoids the use of any base, solvents and requires equimolar amount of the ionic liquid to promote the reaction.

To determine whether the ionic liquid was an essential factor to promote this process, the reaction of *N*-phenylhydroxylamine with 4-methoxybenzaldehyde as a model was carried out using several butylmethylimidazolium-based ionic liquids (ILs), $[b_{mim}]X$, with varying anions such as Cl^-, BF_4^-, and PF_6^- under microwave irradiation. With these ionic liquids, the reaction times were longer and the yield of product **3k** was lower than those in TSIL-$OPPh_2$ under the same conditions. Next, to improve the yields, we performed the reactions using different quantities of reagents. The best results were obtained with a 1:1:1 ratio of arylaldehyde, *N*-monosubstituted hydroxylamine, and IL-$OPPh_2$, respectively. For example, a complete conversion was obtained for the reaction of 2-hydroxy-4-methoxybenzaldehyde and 4-pyridyl aldehyde after 3.5 and 5 min, respectively, under microwave irradiation (Table 1, entries 13 and 16).

A large variety of nitrones were successfully synthesized in high yields by following the above method. The products can be separated from the ionic liquid system by simple extraction with ether and ethyl acetate in all examined cases. The results of the synthesis of different nitrones **3a–s** are presented in Table 1. To examine the versatility of this procedure, the reactions of *N*-monosubstituted hydroxylamine with arylaldehydes containing electron-rich, electron-poor, or electron-neutral substituents were examined. The results are listed in Table 1. It shows that yields (>92% yields) were satisfactory under microwave irradiation conditions. On the other hand, the relatively hindered aldehydes, i.e., 2-naphtylaldehyde, 2,3-dihydroxybenzaldehyde, and 2-hydroxy-5-methoxybenzaldehyde also led to the good yields of the related nitrones (entries 9, 15, 19). The reactions worked well with aromatic aldehydes. However, aliphatic aldehydes, such as propionaldehyde and butyraldehyde did not react efficiently with *N*-phenylhydroxylamine or *N*-methylhydroxylamine under similar conditions in the presence of task-specific ionic liquid, TSIL-$OPPh_2$, and significant

TABLE 2 Comparison of Efficiency of IL-$OPPh_2$ in Synthesis of Nitrones (**3a, 3b, 3g**, and **3r**) after Five Times Recycling **Q1**

	Yield (%)[a]							
	Microwave				Thermal			
Run	**3a**	**3b**	**3g**	**3r**	**3a**	**3b**	**3g**	**3r**
1	96	95	95	99	87	89	87	90
2	95	95	94	97	87	88	85	90
3	95	94	94	97	86	88	86	88
4	93	94	92	96	86	87	85	88
5	93	93	92	96	85	86	85	87

[a]Yields of isolated product.

amount (>45%) of unreacted reactants were isolated when the reactions were carried out for several minutes under microwave irradiation or at 70°C for several hours under thermal heating conditions. As it can be seen from Table 1, no significant differences in yields of products were observed by varying the substituent at nitrogen atom in hydroxylamines in this procedure. All products are known compounds, characterized by mp, IR, ^{1}H NMR, ^{13}C NMR spectra, and elemental analysis.

Ease of recycling is a useful feature of ionic liquids. In the present study, we investigated that IL-$OPPh_2$ can be recycled. After removal of the mixture of product, possible impurities and unreacted materials, the remaining IL was reused for consecutive five cycles. In the reactions for the preparation of nitrones (**3a, 3b, 3g**, and **3r**), no significant loss of product yields was observed when TSIL-$OPPh_2$ was used after five times recycling (Table 2). In addition, compared the IR of the reused TSIL-$OPPh_2$ with that of the new synthesized ionic liquid, the difference was small. That is to say that in the reaction, the structure of the catalyst did not change. Compared with the traditional solvents and catalysts, the easy recycling is an attractive property of the TSIL-$OPPh_2$ for the environmental protection and economic reasons.

CONCLUSION

In conclusion, it was demonstrated that a readily available, economic task-specific ionic liquid could behave as recyclable dual solvent-catalyst for the preparation of nitrones. Using the basic ionic liquid without solvent produced a homogeneous reaction system and showed high initial activity and overall conversion. In addition, TSIL can be easily recycled and reused with the same efficacies for five cycles. The short reaction time, easy synthetic procedure, free of organic solvent, simple work-up in isolation of the products in good yield with high purity, and recyclability of the catalyst are prominent features of this new procedure. Therefore, we believe that the new synthetic method reported here would greatly contribute to the environmentally greener and safer process.

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REFERENCES

[1] Gong, K.; Fang, D.; Wang, H.-L.; Zhou, X.-L.; Liu, Z.-L. Dyes Pigments 2009, 80, 30.
[2] Le, Z.-G.; Chen, Z.-C.; Hu, Y.; Zheng, Q.-G. Synthesis 2004, 2809.
[3] Yue, C.; Mao, A.; Wei, Y.; Lü, M. Catal Commun 2008, 9, 1571.
[4] Wang, L.; Li, H.; Li, P. Tetrahedron 2009, 65, 364.
[5] Macfarlane, D. R.; Pringle, J. M.; Johansson, K. M.; Forsyth, S. A.; Forsyth, M. Chem Commun 2006, 1905.
[6] Paun, C.; Barklie, J.; Goodrich, P.; Gunaratne, H. Q. N.; McKeown, A.; Pârvulescu, V. I.; Hardacre, C. J Mol Catal A: Chem 2007, 269, 64.
[7] Valizadeh, H.; Shockravi, A. Heteroatom Chem 2009, 20, 284.
[8] Valizadeh, H.; Gholipour, H. Synth Commun, in press.
[9] Hamer, J.; Macaluso, A. Chem Rev 1964, 64, 473.
[10] Delpierre, G. R.; Lamchen, M. Quart Rev 1965, 19, 329.
[11] Huisgen, R. Angew Chem, Int Ed 1963, 2, 565.
[12] Black, D. St. C.; Crozier, R. F.; Davis, V. C. Synthesis 1975, 205.
[13] Tufariello, J. J. Acc Chem Res 1979, 12, 396.
[14] Masson, G. S.; Vallée, Py. Y. Angew Chem, Int Ed 2002, 41, 1772.
[15] Cardona, F.; Goti, A. Angew Chem, Int Ed 2005, 44, 7832.
[16] Sandler, S. R.; Karo, W. In Organic Functional Group Preparations, 2nd ed.; Academic Press: San Diego, CA, 1989; Vol. 3, pp. 351–376.
[17] Colacino, E.; Nun, P.; Colacino, F. M.; Martinez, J.; Lamaty, F. Tetrahedron 2008, 64, 5569.

[18] Kim, S.; de A. Vilela, G. V. M.; Bouajila, J.; Dias, A. G.; Cyrino, F. Z. G. A.; Bouskela, E.; Costa, P. R. R.; Nepveu, F. Bioorg Med Chem 2007, 15, 3572.
[19] Sridharan, V.; Muthusubramanian, S.; Sivasubramanian, S. Ind J Chem B: Org Chem 2004, 43, 857.
[20] Bortolini, O.; D'Agostino, M.; De Nino, A.; Maiuolo, L.; Nardi, M.; Sindona, G. Tetrahedron 2008, 64, 8078.
[21] Valizadeh, H.; Dinparast, L. Heteroatom Chem 2009, 20, 177.
[22] Valizadeh, H.; Vaghefi, S. Synth Commun 2009, 39, 1666.
[23] Valizadeh, H.; Heravi, M. M.; Amiri, M. Mol Divers 2009 available online Doi:10.1007/s11030-009-9189-x.
[24] Valizadeh, H.; Amiri, M.; Gholipur, H. J Heterocycl Chem 2009, 46, 108.
[25] Valizadeh, H.; Shockravi, A. Tetrahedron Lett 2005, 46, 3501.
[26] Valizadeh, H.; Mamaghani, M.; Badrian, A. Synth Commun 2005, 35, 785.
[27] Valizadeh, H.; Shockravi, A. Synth Commun 2009, 39, 4341.
[28] Andrade, M. M.; Barros, M. T.; Pinto, R. C. Tetrahedron 2008, 64, 10521.
[29] Yijima, C.; Tsujimoto, T.; Suoa, K.; Yamauchi, M. Bull Chem Soc Jpn 1986, 59, 2165.
[30] Colacino, E.; Nun, P.; Colacino, F. M.; Martinez, J.; Lamaty, F. Tetrahedron 2008, 64, 5569.
[31] Waterbury, D. L.; Carney, J. M.; Wilcox, A. L. U.S. Patent, 2001, 6197826.