Pergamon

Tetrahedron 57 (2001) 1923–1927

TETRAHEDRON

Synthesis of functionalized 4-phenyl-pyridines via electrochemically prepared organozinc reagents

Erwan Le Gall, Corinne Gosmini,* Jean-Yves Nédélec and Jacques Périchon

Laboratoire d'Electrochimie, Catalyse et Synthèse Organique, LECSO, CNRS, UMR 7582, 2-8 rue Henri Dunant, 94320 Thiais, France

Received 5 October 2000; revised 30 November 2000; accepted 21 December 2000

Abstract—The efficient and convenient synthesis of various functionalized 4-phenyl-pyridines **2** is described. The key step of the procedure is the electrochemical formation of aromatic organozinc reagents **1** and their coupling with pyridinium salts. Intermediate 1,4-dihydropyridines are oxidized using mild conditions to provide functionalized 4-phenyl-pyridines in moderate to high overall yields.

1. Introduction

Biaryl compounds and especially 2- and 4-phenyl-pyridines are of high interest in organic chemistry due to their pharmaceutical and agrochemical activities[1] and several synthetic procedures including catalytic processes have appeared during the last years.[2] In numerous works devoted to the cross-coupling of aromatic species, the Suzuki reaction,[3] which is a powerful tool in this goal, has been widely used. For instance, Mitchell et al.[4] and Lohse et al.[5] have successfully employed this method, starting from phenylboronic acids and chlorinated heteroaromatic amines in the presence of palladium, to obtain the cross-coupling products in moderate to high yields. Other procedures avoiding the use of expensive aromatic halogenated heterocycles were developed in order to achieve the synthesis of 4-substituted pyridines. Comins et al.[6] have thus shown that Grignard reagents are reactive towards pyridinium salts and lead to 4-substituted 1,4-dihydropyridines which can be further oxidized by *ortho*-chloranil. However, the scope of this reaction is limited by the instability[7,8] and the high reactivity of Grignard reagents which discard the presence of electrophilic groups on starting compounds. This drawback can be circumvent by the use of organozinc reagents[9] as shown by Shiao et al.[10] but despite its high synthetic utility, the use of difficult to handle activated zinc[11] makes this procedure very sensitive.

Among the numerous topics developed in this laboratory, the electrochemical synthesis of organozinc reagents is of current interest.[12,13] These organometallic species are synthesized in high yields using mild reaction conditions.[14] As a part of our program devoted to the electrochemical cross-coupling of aromatic compounds our aim was to achieve the synthesis of various functionalized 4-phenyl-pyridines. Unfortunately, usual protocols involving nickel-catalysts and halogenated compounds were inefficient. We now report herein a very convenient synthesis of various 4-phenyl-pyridines from functionalized phenyl bromides and pyridine as starting compounds, and using the cobalt-catalyzed electrosynthesis of organozinc reagents as the key-step.

2. Results and discussion

2.1. Electrosynthesis of organozinc reagents

It has been previously shown that organozinc reagents are valuable precursors of 4-substituted pyridines but curiously, we could not find in the literature studies dealing with the coupling between various functionalized organozinc reagents and pyridinium salts as outlined in Scheme 1.

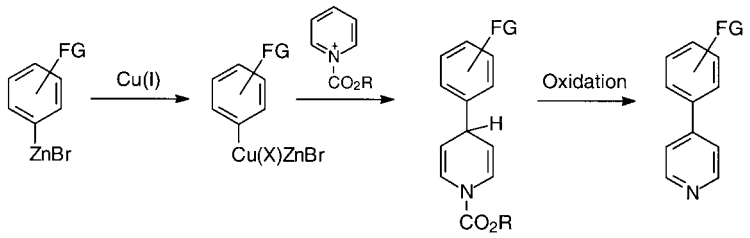
Our first investigations were thus devoted to finding the most simple and efficient conditions to achieve the electrosynthesis of functionalized aromatic organozinc reagents. A typical electrolysis medium was found as a mixture of acetonitrile, dimethylformamide and pyridine as solvents and catalytic amounts of cobalt chloride and zinc bromide. The organozinc compounds were prepared in an undivided electrochemical cell[15] flushed with argon and fitted with a zinc rod as the anode and a stainless steel grid as the cathode. The electrolyses of various phenyl bromides were carried out at ambient temperature and at constant current intensity of 0.2 A until a typical charge of 2–2.1 F per mole of the halide was passed (Scheme 2).

Yields of organozinc compounds thus obtained were estimated as follows: samples of the electrolysis solutions were

Keywords: 4-phenyl-pyridines; pyridinium.

* Corresponding author. Tel.: +1-49-781139; fax: +1-49781148; e-mail: gosmini@glvt-cnrs.fr

0040–4020/01/$ - see front matter
PII: S0040-4020(01)00015-1



Scheme 1.

exposed to iodine crystals then sodium thiosulfate, and extracted with diethyl ether. Amounts of iodinated compounds were compared to the amounts of starting phenyl bromides via an internal standard using gas chromatography. Yields of organozinc compounds are reported in Table 1.

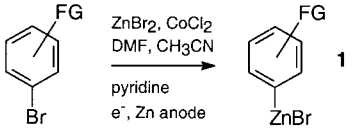
The first point to note is that with both electron-donating and -withdrawing groups satisfactory to high yields are obtained (54–85%). The electrochemical synthesis of substituted phenylzinc compounds can be considered as versatile, with only a few limitations. Thus, when FG=*o*-CO_2Et (entry 3) only homocoupling occurs resulting in the detection of the sole biaryl product. Also, the failure observed when FG=*p*-NHBOC (entry 17) is likely the consequence of the high basicity of the organozinc compound which fast deprotonates this function allowing only the detection of the reduction product (C_6H_5–NHBOC).

The numerous benefits of this convenient one-pot procedure have prompted us to investigate the coupling of these electrogenerated phenylzinc compounds with pyridinium salts.

2.2. Coupling between mixed copper–zinc organometallic species and pyridinium salts

A different approach of previously described sequences allowing the synthesis of 4-phenyl-pyridines was considered, as outlined in Scheme 3. This approach was directed towards finding the most simple conditions for all the chemical steps of the sequence. Thus, after completion of the formation of organozinc species the electrolysis solutions were cooled to 0°C. A mixture of cuprous cyanide (1 equiv.) and lithium chloride (2 equiv.) was then added and allowed to react during 2–5 min. Longer reaction times or higher temperatures proved to rise dramatically the proportions of biaryl compounds when the phenyl moiety was bearing electron-withdrawing groups and thus only low yields were obtained.

Considering the fact that this reaction sequence involves the formation of a pyridinium ion and that during the first step pyridine was present in the electrolysis medium, acting as co-solvent and ligand, we thought that simply adding methyl chloroformate to the solution of the mixed copper–zinc organometallic species would lead to the desired coupling product. It should be mentioned that in that case the use of preformed pyridinium salts of the aromatic amines is generally described in the literature. Methyl chloroformate (1 equiv.) was then added at 0°C to the solution which was allowed to warm to room temperature during 2 h. The 4-substituted-1,4-dihydropyridine thus obtained was oxidized without being isolated using silica gel and oxygen (24–72 h, rt, method A) or using hydrogen peroxide and acetic acid (12 h, rt, method B) when method A could not suit. Crude products were purified using silica gel chromatography with diethyl ether as an eluent to afford the desired 4-phenyl-pyridines. Results are reported in Table 2.



Scheme 2.

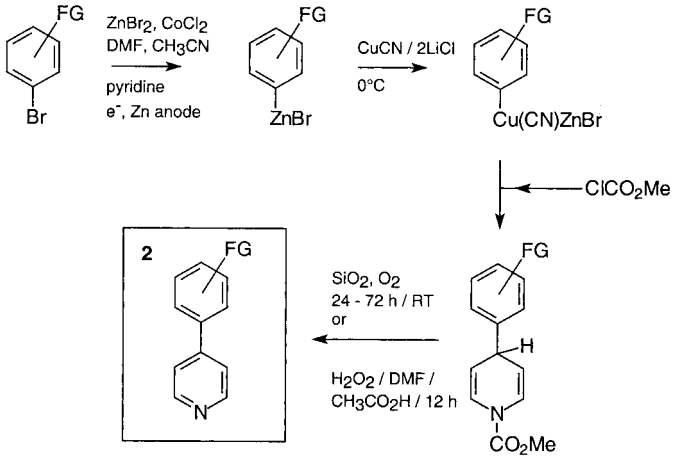
Moderate to high overall yields are obtained using this procedure which can be applied to a wide range of functionalized phenyl bromides. It can be noticed that when an amino group is involved (entry 16) no coupling products can be isolated from the reaction mixture. This result may be explained by the nucleophicity of NH_2 which may also react with methyl chloroformate, and hence numerous side-products can also be formed.

Electron-donating groups allow the smooth oxidation of intermediate dihydropyridines whereas with some electron-withdrawing groups (entries 7–10) the use of hydrogen

Table 1. Yields of electrochemically prepared organozinc reagents

Entry	ArBr (mmol)	FG	Faradic yield (%) [Charge passed (C)][a]	ArZnBr **1** (%)[b]
1	10	*p*-CO_2Et	100 [1950]	75
2	10	*m*-CO_2Et	90 [2150]	76
3	10	*o*-CO_2Et	–	–
4	10	*p*-$COCH_3$	100 [1950]	68
5	10	*m*-$COCH_3$	95 [2050]	75
6	10	*p*-CN	100 [1950]	75
7	10	*p*-CF_3	95 [2050]	73
8	10	*m*-CF_3	100 [1950]	74
9	10	*p*-F	90 [2150]	80
10	10	*m*-Cl	95 [2050]	80
11	10	*p*-OMe	90 [2050]	65
12	10	*m*-OMe	95 [2050]	85
13	10	*o*-OMe	55 [3500]	70
14	10	*p*-Me	95 [2050]	78
15	10	*p*-iPr	100 [1950]	60
16	10	*p*-NH_2	100 [1950]	54
17	10	*p*-NHBOC	–	–
18	10	3,4-	95 [2050]	83

[a] Faradic yield, based on Faraday's law: Yield=[[$nF(m/M)$]/(it)]100.
[b] Estimated using gas chromatography.



Scheme 3.

peroxide and acetic acid is required. The low overall yield observed when two electron-donating groups are on the phenyl ring (entry 18) seems paradoxical since the related organozinc compound was obtained in 83% yield. However, this result can be explained by the fact that the intermediate 1,4-dihydropyridine proved to be very sensitive to oxygen and fast darkened when adsorbed on silica gel and exposed to air.

3. Conclusion

In conclusion, the results reported in this paper show that the electrosynthesis of various functionalized aromatic organozinc reagents and their coupling with pyridine can be realized in a simple and efficient manner. The major advantage of this method relies on the use of mild and simple reaction conditions since electrolyses are conducted at room temperature and that all steps of the reaction sequence except the oxidation one are carried out one-pot.

4. Experimental

4.1. Typical procedure

The electrochemical cell fitted with a zinc rod as the anode and a stainless steel grid as the cathode was flushed with argon. Acetonitrile (40 ml), dimethylformamide (5 ml) and pyridine (5 ml) were added using a syringe. To this solution were added anhydrous zinc chloride (0.7 g, 0.3 equiv.), cobalt chloride (0.2 g, 0.13 equiv.) and tetrabutylammonium tetrafluoroborate in order to rise the conductivity of the medium. The functionalized phenyl bromide (10 mmol, 1 equiv.) and dodecane (0.2 ml) as internal standard were added, and a constant current intensity of 0.2 A was applied until complete disappearance of the starting compound (ideally 1930 C). The reaction was monitored using gas chromatography after iodination of the intermediate organozinc compound. The solution was then cooled to 0°C and a mixture of cuprous cyanide (0.9 g, 1 equiv.) and lithium chloride (0.9 g, 2 equiv.) was added under vigorous stirring. After 2–5 min, methyl chloroformiate (0.8 ml,

Table 2. Yields of functionalized 4-phenyl-pyridines

Entry	FG	Oxidation method[a]	**2** Overall isolated yield (%)[b]	Compound
1	*p*-CO_2Et	A	45	**2a**
2	*m*-CO_2Et	A	50	**2b**
4	*p*-$COCH_3$	A	39	**2c**
5	*m*-$COCH_3$	A	56	**2d**
6	*p*-CN	A	43	**2e**
7	*p*-CF_3	B	44	**2f**
8	*m*-CF_3	B	47	**2g**
9	*p*-F	B	55	**2h**
10	*m*-Cl	B	66	**2i**
11	*p*-Ome	A	45	**2j**
12	*m*-Ome	A	54	**2k**
13	*o*-Ome	A	53	**2l**
14	*p*-Me	A	69	**2m**
15	*p*-iPr	A	47	**2n**
16	*p*-NH_2	–	–	–
18	3,4-OCH_2CH_2O-	A	35	**2o**

[a] *Method A*: SiO_2, O_2, rt, 24–72 h; *method B*: H_2O_2, CH_3CO_2H, DMF, rt, 12 h.
[b] Based on starting ArBr.

1 equiv.) was added using a syringe and the reactional mixture was allowed to warm over 2 h. The mixture was then poured into 50 ml of a saturated ammonium chloride solution. After evaporation of acetonitrile under reduced pressure, sodium chloride was added to the aqueous layer which was extracted with 3×100 ml diethyl ether. The organic layer was dried over magnesium sulfate and evaporated to dryness. The resulting oil was oxidized according to either method A or B.

4.1.1. Oxidation method A. The crude oil was dissolved in 50 ml dichloromethane. Silica gel (10 g) was added to the solution, the solvent removed under atmospheric pressure and the adsorbed organic compound was placed in a large container and allowed to react with air during 24–72 h. The crude product was purified using silica gel chromatography with diethyl ether as eluent.

4.1.2. Oxidation method B. The crude oil was dissolved in 10 ml dimethylformamide. To the solution maintained at room temperature using a large water bath were added dropwise 1.5 ml acetic acid and 1.5 ml of a 30% hydrogen peroxide solution. After 12 h, 50 ml of water were added to the solution. The resulting mixture was cooled to 0°C and sodium hydroxide pellets were added under vigorous stirring until pH=12. The aqueous layer was extracted with 3×100 ml dichloromethane, the solvent dried over magnesium sulfate and evaporated to dryness. The crude product was purified using silica gel chromatography with diethyl ether as eluent.

4.2. General data and product analysis

Unless otherwise specified, materials were purchased from commercial suppliers and used without further purification. Chromatographic separations were realized using Merck 60 *ACC* (70–200 mesh) silica gel. Gas chromatography analyses were performed on a 25 m CPSIL-5CB column using a Varian 3400 CX chromatograph. Mass spectra were recorded on a Finnigan GC/MS GCQ spectrometer. ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ solutions on a Bruker AC200 spectrometer. Data are presented as follows: chemical shift (multiplicity, coupling constants, number of protons). Compounds which have been previously described in the literature fit with the structure data found in the given references, whereas new compounds labeled by asterisk (*) have been fully characterized.

4.2.1. 4-Pyridin-4-yl-benzoic acid ethyl ester 2a.[16] Pale yellow solid, ^{1}H NMR, δ (ppm): 1.30 (t, J=7.1 Hz, 3H), 4.30 (q, J=7.1 Hz, 2H), 7.40 (d, J=5.9 Hz, 2H), 7.55 (AB, J=8.4 Hz, 2H), 8.30 (AB, J=8.4 Hz, 2H), 8.55 (broad s, 2H). MS, *m/z* (relative intensity): 227 (M, 85), 199 (M-28, 100), 182 (M-45, 60), 154 (M-73, 20).

4.2.2. 3-Pyridin-4-yl-benzoic acid ethyl ester 2b.[16] Pale yellow oil, ^{1}H NMR, δ (ppm): 1.37 (t, J=7.1 Hz, 3H), 4.37 (q, J=7.1 Hz, 2H), 7.40–7.58 (m, 3H), 7.76 (d, J=7.8 Hz, 1H), 8.06 (d, J=7.8 Hz, 1H), 8.26 (t, J=1.6 Hz, 1H), 8.65 (broad s, 2H). MS, *m/z* (relative intensity): 227 (M, 65), 199 (M-28, 100), 182 (M-45, 74), 154 (M-73, 33), 127 (M-100, 15).

4.2.3. 4-Pyridin-4-yl-benzophenone 2c.[17] Light brown solid, ^{1}H NMR, δ (ppm): 2.58 (s, 3H), 7.47 (d, J=6.1 Hz, 2H), 7.66 (AB, J=8.4 Hz, 2H), 8.00 (AB, J=8.4 Hz, 2H), 8.63 (d, J=6.1 Hz, 2H). MS, *m/z* (relative intensity): 197 (M, 23), 182 (M-15, 100), 154 (M-43, 20), 127 (M-70, 9).

4.2.4. 3-Pyridin-4-yl-benzophenone 2d.* Light brown oil, ^{1}H NMR, δ (ppm): 2.59 (s, 3H), 7.40–7.58 (m, 3H), 7.76 (d, J=7.8 Hz, 1H), 7.94 (d, J=7.8 Hz, 1H), 8.15 (s, 1H), 8.60 (broad s, 2H). ^{13}C NMR, δ (ppm): 26.54, 121.45, 126.45, 128.77, 129.29, 131.25, 137.63, 138.36, 147.15, 150.58, 197.41. MS, *m/z* (relative intensity): 197 (M, 36), 182 (M-15, 100), 154 (M-43, 21), 127 (M-70, 7). HRMS, Exact calcd for $C_{13}H_{11}NO$: 197.08406; Found: 197.08460.

4.2.5. 4-Pyridin-4-yl-benzonitrile 2e.[18] Light brown solid, ^{1}H NMR, δ (ppm): 7.43 (d, J=5.9 Hz, 2H), 7.68 (AB, J=2.3 Hz, 2H), 7.71 (AB, J=2.3 Hz, 2H), 8.65 (d, J=5.9 Hz, 2H). MS, *m/z* (relative intensity): 180 (M, 100), 153 (M-27, 10).

4.2.6. 4-(4-Trifluoromethyl-phenyl)-pyridine 2f.* Pale yellow oil, ^{1}H NMR, δ (ppm): 7.58 (d, J=6.0 Hz, 2H), 7.81 (s, 4H), 8.78 (d, J=6.0 Hz, 2H). ^{13}C NMR, δ (ppm): 121.57, 125.87, 125.94, 127.26, 141.51, 146.77, 150.24. ^{19}F NMR, δ (ppm): −62.36. MS, *m/z* (relative intensity): 223 (M, 100), 154 (M-69, 22), 127 (M-96, 5). HRMS, Exact calcd for $C_{12}H_8NF_3$: 223.06088; Found: 223.06130.

4.2.7. 4-(3-Trifluoromethyl-phenyl)-pyridine 2g.* Pale yellow oil, ^{1}H NMR, δ (ppm): 7.20–7.81 (m, 6H), 8.52 (broad s, 2H). ^{13}C NMR, δ (ppm): 121.32, 123.45, 123.52, 125.36, 125.43, 129.39, 130.00, 138.61, 146.42, 150.01. ^{19}F NMR, δ (ppm): −62.61. MS, *m/z* (relative intensity): 223 (M, 100), 202 (M-21, 2), 153 (M-70, 3). HRMS, Exact calcd for $C_{12}H_8NF_3$: 223.06088; Found: 223.06110.

4.2.8. 4-(4-Fluoro-phenyl)-pyridine 2h.[19] Pale yellow solid, ^{1}H NMR, δ (ppm): 7.00–7.65 (m, 6H), 8.64 (d, J=5.6 Hz, 2H). MS, *m/z* (relative intensity): 173 (M, 100), 146 (M-27, 12), 74 (M-99, 4).

4.2.9. 4-(3-Chloro-phenyl)-pyridine 2i.[20] Pale yellow oil, ^{1}H NMR, δ (ppm): 7.30–7.60 (m, 6H), 8.59 (d, J=5.8 Hz, 2H). MS, *m/z* (relative intensity): 189 (M, 100), 154 (M-35, 31), 127 (M-62, 19).

4.2.10. 4-(4-Methoxy-phenyl)-pyridine 2j.[21] Pale yellow solid, ^{1}H NMR, δ (ppm): 3.84 (s, 3H), 7.00 (AB, J=8.5 Hz, 2H), 7.47 (d, J=6.0 Hz, 2H), 7.60 (AB, J=8.5 Hz, 2H), 8.62 (broad s, 2H). MS, *m/z* (relative intensity): 185 (M, 100), 170 (M-15, 15), 115 (M-70, 9).

4.2.11. 4-(3-Methoxy-phenyl)-pyridine 2k.[21] Pale yellow oil, ^{1}H NMR, δ (ppm): 3.80 (s, 3H), 6.85–7.45 (m, 6H), 8.58 (broad s, 2H). MS, *m/z* (relative intensity): 185 (M, 100), 155 (M-30, 21), 115 (M-70, 11).

4.2.12. 4-(2-Methoxy-phenyl)-pyridine 2l.[22] Pale yellow oil, ^{1}H NMR, δ (ppm): 3.75 (s, 3H), 6.82–7.05 (m, 6H), 8.53 (d, J=5.6 Hz, 2H). MS, *m/z* (relative intensity): 185 (M, 100), 170 (M-15, 37), 156 (M-29, 14), 115 (M-70, 38).

4.2.13. 4-(4-Tolyl)-pyridine 2m.[21] Light brown solid, ^{1}H NMR, δ (ppm): 2.46 (s, 3H), 7.35 (AB, J=9 Hz, 2H), 7.47–7.66 (m, 4H), 8.67 (broad s, 2H). MS, *m*/*z* (relative intensity): 168 (M-1, 100), 141 (M-27, 10).

4.2.14. 4-(4-Isopropyl-phenyl)-pyridine 2n.[23] Light brown solid, ^{1}H NMR, δ (ppm): 1.24 (d, J=6.9 Hz, 6H), 2.92 (hept, J=6.9 Hz, 1H), 7.30 (AB, J=8.2 Hz, 2H), 7.45 (d, J= 5.5 Hz, 2H), 7.48 (AB, J=8.2 Hz, 2H), 8.58 (broad s, 2H). MS, *m*/*z* (relative intensity): 197 (M, 91), 183 (M-14, 100), 167 (M-30, 45).

4.2.15. 4-Benzo[1,3]dioxo-5-yl-pyridine 2o.[*] Light brown solid, ^{1}H NMR, δ (ppm): 5.99 (s, 2H), 6.87 (AB, J=7.9 Hz, 1H), 7.05–7.15 (m, 2H), 7.38 (d, J=5.5 Hz, 2H), 8.56 (d, J=5.5 Hz, 2H). ^{13}C NMR, δ (ppm): 101.44, 107.11, 108.79, 120.89, 121.18, 132.05, 147.89, 148.45, 148.52, 150.02. MS, *m*/*z* (relative intensity): 199 (M, 100), 140 (M-59, 5), 114 (M-85, 6). HRMS, Exact calcd for $C_{12}H_9NO_2$: 199.06332; Found: 199.06390.

Acknowledgements

The authors thank Aventis for financial support.

References

1. (a) Micetich, R. G. In *The Chemistry of Heterocyclic Compounds*; Abramovitch, R. A., Ed.; Wiley: New York, 1974; Vol. 14 (Supplement Part 2). (b) Boyd, E. C.; Eaton, M. A. W.; Warrelow, G. J. *Chem. Abstr.* **1994**, *122*, 31544.
2. Stanforth, S. P. *Tetrahedron* **1997**, *54*, 263–303.
3. Miayaura, N.; Yamagi, T.; Suzuki, A. *Synth. Commun.* **1981**, *11*, 513–519.
4. (a) Mitchell, M. B.; Wallbank, P. J. *Tetrahedron Lett.* **1991**, *32*, 2273–2276. (b) Ali, N. M.; McKillop, A.; Mitchell, M. B.; Rebelo, R. A.; Wallbank, P. J. *Tetrahedron* **1992**, *48*, 8117–8126.
5. Lohse, O.; Thevenin, P.; Waldvogel, E. *Synlett* **1999**, 45–48.
6. Commins, D. L.; Smith, R. K.; Stroud, E. D. *Heterocycles* **1984**, *22*, 339–344.
7. Harvey, S.; Junk, P. C.; Raston, C. L.; Salem, G. *J. Org. Chem.* **1988**, *53*, 3134–3140.
8. Parham, W. E.; Jones, L. D.; Sayed, Y. A. *J. Org. Chem.* **1976**, *41*, 1184–1191.
9. For a review dealing with synthetic applications of organozinc reagents see: Knochel, P.; Singer, R. D. *Chem. Rev.* **1993**, *93*, 2117–2188.
10. (a) Chia, W. L.; Shiao, M. J. *Tetrahedron Lett.* **1991**, *32*, 2033–2034. (b) Shiao, M. J.; Chia, W. L.; Shing, T. L.; Chow, T. J. *J. Chem. Res. (s)* **1992**, 247.
11. Zhu, L.; Wehmeyer, R. H.; Rieke, R. D. *J. Org. Chem.* **1991**, *56*, 1445–1453.
12. Périchon, J.; Gosmini, C.; Rollin, Y. In *Organozinc Reagents*; Knochel, P., Jones, P., Eds.; Oxford University Press: Oxford, 1999; pp 139–156 (Chapter 8).
13. (a) Gosmini, C.; Lasry, S.; Nédélec, J. Y.; Périchon, J. *Tetrahedron* **1998**, *54*, 1289–1298. (b) Sibille, S.; Ratovelomanana, V.; Nédélec, J. Y.; Périchon, J. *Synlett* **1993**, 425–426.
14. (a) Patent application 99/08480, France, 1 July **1999** (b) Gosmini, C.; Rollin, Y.; Nédélec, J. Y.; Périchon, J. *J. Org. Chem.* **2000**, *65*, 6024–6026.
15. Troupel, M.; Rollin, Y.; Sock, O.; Meyer, G.; Périchon, J. *Nouv. J. Chem.* **1986**, *11*, 593–599.
16. Patent, Ciba Ltd, NL 6414307, 1964, *Chem. Abstr.*, 64, **1966** 713d.
17. Wright, S. W.; Harris, H. R.; Collins, R. J.; Corbett, R. L.; Green, A. M. *J. Med. Chem.* **1992**, *17* (35), 3148–3155.
18. Lin, S. T.; Yang, F. M.; Liang, D.; Shiao, M. J. *J. Chin. Chem. Soc.* **1997**, *5* (44), 527–534. *Chem. Abstr.*, 7, **1998**, *128*, 75023e
19. Shkurko, O. P.; Baram, S. G.; Mamaev, V. P. *Chem. Heterocycl. Compd.* **1983**, 60–65 (Engl. Transl.).
20. Ohkura, K.; Seki, K. I.; Terashima, M.; Kanaoka, Y. *Tetrahedron Lett.* **1989**, *30*, 3433–3436.
21. Katritzky, A. R.; Beltrami, H. *J. Chem. Soc. Perkin Trans. 1* **1980**, 2480–2484.
22. Weller, D. D.; Luellen, G. R. *Tetrahedron Lett.* **1981**, *22*, 4381–4384.
23. Schmidle, C. J.; Locke, J. E.; Mansfield, R. C. *J. Org. Chem.* **1956**, *21*, 1195–1197.