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"One-pot" Synthesis of (*Z*)-2-Aryl-1-(2-cyanoethyl)ethenylphosphonates via Hexamethylphosphoramide-Promoted Sequential Transformation

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ABSTRACT: *(Z)-2-Aryl-1-(2-cyanoethyl)ethenylphosphonates were synthesized by the hexamethylphosphoramide-promoted sequential transformation of tetra-alkyl methanediphosphonates, by the action of potassium tert-butoxide, and then by acrylonitrile and aldehydes.* © 2002 Wiley Periodicals, Inc. Heteroatom Chem 13:116–119, 2002; Published online in Wiley Interscience (www.interscience.wiley.com). DOI 10.1002/hc.10004

INTRODUCTION

Functionalized ethenylphosphonates have attracted much attention in synthetic chemistry, and their synthetic application has been widely investigated [1]. For example, they have been used for Diels–Alder reactions, Michael additions, and the synthesis of hetero- and carbocyclic compounds [1–3]. Numerous synthetic methods for their preparation have been reported [1,4–5]. However, convenient methods for the synthesis of functionalized ethenylphosphonates are still limited. Herein, we wish to report a "one-pot" synthesis of (*Z*)-2-aryl-1-(2-cyanoethyl)ethenylphosphonates via a hexamethylphosphoramide (HMPA)-promoted sequential transformation. The cyano group is an important functional group in organic transformations, and cyano-containing compounds are useful intermediates for the synthesis of pyrimidines [6], tetrazole analogues [7], thiazole derivatives [8], and *Z*-oxazolines [9]. Therefore, the development of an effective method for the preparation of the title compounds would be valuable.

RESULTS AND DISCUSSION

In connection with our interest in the synthetic application of sequential transformation of phosphonates for the synthesis of perfluoroalkylated α-fluroro-α,β-unsaturated esters [10], perfluoroalkylated 1-cyano-1,4-alkadienes [11], and perfuoroalkylated unsaturated nitriles [12], which would be difficult to prepare by presently known procedures, we attempted the following reaction (Scheme 1).

The phosphoryl stabilized carbanions **2**, generated from the corresponding phosphonates and potassium *tert*-butoxide in THF, were treated with acrylonitrile, promoted by HMPA, to give the Michael addition adducts **3** that were converted to **4** by a 1,3-proton shift. Then, **4** was reacted with aldehydes to give the products **5**. The results are summarized in Table 1.

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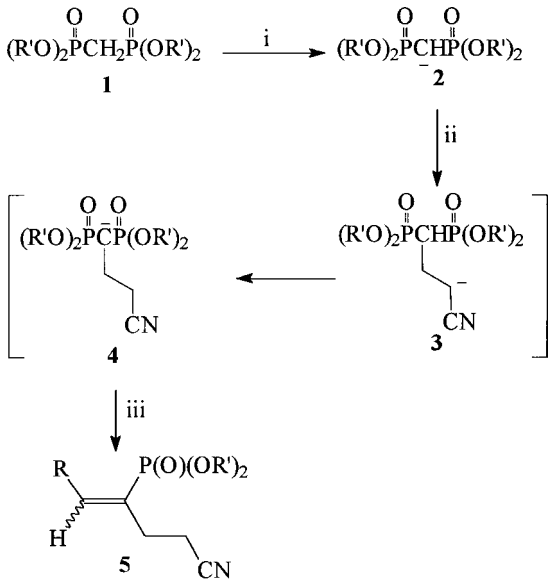

SCHEME 1 Reagents and conditions: (i) KOBu^t,THF 20°C, 0.5 h; (ii) HMPA, acrylonitile, 20°C, 2 h; and (iii) RCHO, 20°C, 3 h.

In this sequence of reactions, the addition of HMPA is necessary. In the absence of HMPA, the yields of the desired products were rather low (<10%). HMPA is a highly polar and aprotic solvent, and is frequently used to accelerate organolithium reactions [13]. In this reaction, the role of HMPA may be to promote the phosphoryl-stabilized carbanions **2** to react much more easily with acrylonitrile, and indeed the Michael addition adducts **3** were obtained in high yields. Thus, the yields of the final products were also high.

The configuration of the products was ascertained on the basis of NMR spectra. It has been reported that when the vinyl H is cis with respect to the phosphoryl group, the $^3J_{\text{cis H,P}}$ equals 21 Hz, and when the vinyl H is trans with respect to the phosphoryl group, the $^3J_{\text{trans H,P}}$ equals 41 Hz [14]. In our cases, the $^3J_{\text{H,P}}$ values equaled about 46 Hz. Thus, the configurations are ascertained to be *Z*.

It is well known that the traditional Horner–Wadsworth–Emmons (HWE) reaction results in the formation of the thermodynamically favored *E*-isomer [1]. Recently, a few *Z*-selective HWE reagents such as methanedi(trifluoroethyl)phosphonoacetate [15] and ethyl diphenyl phosphonoacetate [16] have been reported. However, *Z*-isomers were formed exclusively or predominantly in this HMPA promoted sequential transformation. Unfortunately, there is no readily apparent reasonable explanation to elucidate the observed steroeoselectivity.

In conclusion, our methodology provides a convenient and highly stereoselective synthesis of the title compounds from commercially available substances. The compounds are expected to be useful intermediates in organic synthesis. It is noteworthy that the HMPA-promoted sequential transformation of tetra-alkyl methanediphosphonates, by the action of potassium *tert*-butoxide, and then by acrylonitile and aldehydes gives *Z*-isomers exclusively or predominantly.

TABLE 1 Data for (*Z*)-2-Aryl-1-(2-cyanoethyl)ethenylphosphonates

Compound	*R′*	*R*	*Z*:*E*[a]	*Yield (%)*[b]
5a	Et	3-$CF_3OC_6H_4$	100:0	55
5b	Et	4-FC_6H_4	100:0	70
5c	Et	3-$CH_3OC_6H_4$	100:0	54
5d	Et	4-$CH_3OC_6H_4$	100:0	47
5e	Et	C_6H_5	100:0	47
5f	Et	2,4-$Cl_2C_6H_3$	100:0	64
5g	Et	4-$CH_3C_6H_4$	100:0	51
5h	Et	4-ClC_6H_4	88:12	66
5i	Et	4-$NO_2C_6H_4$	77:23	55
5j	*i*-Pr	2-$CH_3OC_6H_4$	100:0	50
5k	*i*-Pr	3-$CF_3OC_6H_4$	87:13	49
5l	*i*-Pr	4-$NO_2C_6H_4$	83:17	40

[a]The ratio of *E*- and *Z*-isomers was estimated on the basis of NMR spectrum.
[b]Isolated yields.

EXPERIMENTAL

The IR spectra of liquid products were determined as films on a Digilab FTS-20E spectrometer. ^{1}H NMR spectra were recorded on a Bruker AM-300 (300 MHz) spectrometer (values in ppm from $SiMe_4$, in $CDCl_3$; *J* values are given in Hz). Mass spectra were measured on a Finnigan GC-MS-4021 mass spectrometer.

General Procedure for the Preparation of (Z)-2-Aryl-1-(2-cyanoethyl)ethenylphosphonates

The solution of each tetra-alkyl methanediphosphonate (2 mmol) and potassium *tert*-butoxide (0.224 g, 2 mmole) in THF (20 ml) was stirred at 20°C under nitrogen for 0.5 h. Then HMPA (0.71 g, 4 mmole) and acrylonitrile (0.106 g, 2 mmol) were in turn added to the solution, and stirring was continued at 20°C for 2 h, after which an aldehyde (2 mmol) was added dropwise to the mixture. The mixture was stirred at 20°C for 3 h until the disappearance of aldehyde (monitored by TLC) was noted. It was then acidified with 1 ml of 1 M HCl and concentrated. Then water (20 ml) was added. The aqueous layer was extracted with diethyl ether (3 × 20 ml). The combined organic layer was washed with brine (3 × 10 ml)

until neutral and was dried over Na_2SO_4. Evaporation of the solvent gave a residue, which was purified by column chromatography with elution by light petroleum ether (60–90°C)–ethyl acetate (2:1) to give the product.

*(Z)-Diethyl 1-(2-Cyanoethyl)-2-(3-triflouromethoxyphenyl)ethenylphosphonate (***5a***)*

Yield: 55%, oil; IR (neat): $\nu = 2990$, 2250, 1610, 1580, 1260, 1220, 1170, 1150, 1020 cm^{-1}; ^1H NMR ($CDCl_3$/TMS): $\delta = 1.10$ (t, $J = 7.1$ Hz, 6H), 2.78–2.71 (m, 4H), 4.04–3.83 (m, 4H), 7.25–7.08 (m, 1H), 7.30 (d, $J = 47.6$ Hz,1H), 7.44–7.31 (m, 3H); MS: *m/z* 377 (M^+, 17), 376 (31), 348 (11), 337 (100), 320 (42), 267 (29), 240 (42). Anal.: Calcd. for $C_{16}H_{19}F_3NO_4P$ (377.30): C, 50.93, H, 5.08, N, 3.71. Found: C, 50.56, H, 5.28, N, 3.68.

*(Z)-Diethyl 1-(2-Cyanoethyl)-2-(4-fluorophenyl) ethenylphosphonate (***5b***)*

Yield: 70%, oil; IR (neat): $\nu = 2990$, 2250, 1600, 1510, 1240, 1050,1020, 970 cm^{-1}. ^1H NMR ($CDCl_3$/TMS): $\delta = 1.10$ (t, $J = 9.8$ Hz, 6H), 2.79–2.67 (m, 4H), 4.02–3.81 (m, 4H), 7.05–6.97 (m, 2H), 7.25 (d, $J = 46.4$ Hz, 1H), 7.51–7.45 (m, 2H); MS: *m/z* 311 (M^+, 9), 310 (9), 282 (12), 271 (43), 254 (20), 174 (16). Anal.: Calcd. for $C_{15}H_{19}FNO_3P$ (311.29): C, 57.88, H, 6.15, N, 4.50. Found: C, 57.91, H, 6.03, N, 4.72.

*(Z)-Diethyl 1-(2-Cyanoethyl)-2-(3-methoxyphenyl)ethenylphosphonate (***5c***)*

Yield: 54%, oil; IR (neat): 2980, 2250, 1620, 1600, 1490, 1250,1050, 1030, 970 cm^{-1}. ^1H NMR ($CDCl_3$/TMS): $\delta = 1.08$ (t, $J = 7.1$ Hz, 6H), 2.82–2.69 (m, 4H), 3.80 (s, 3H), 3.98–3.77 (m, 4H), 6.83 (d, $J = 8.2$ Hz, 1H), 6.91 (t, $J = 7.5$ Hz, 1H), 7.26 (d, $J = 8.1$ Hz, 1H), 7.36 (d, $J = 40$ Hz, 1H), 7.54 (d, $J = 7.6$ Hz, 1H). MS: *m/z* 324 ($M^+ + 1$, 17), 292 (54), 283 (78), 255 (26), 236 (55), 218 (16), 185 (26). Anal.: Calcd. for $C_{16}H_{22}NO_4P$ (323.32): C, 59.44, H, 6.86, N, 4.33. Found: C, 59.30, H, 6.79, N, 4.48.

*(Z)-Diethyl 1-(2-Cyanoethyl)-2-(4-methoxyphenyl)ethenylphosphonate (***5d***)*

Yield: 47%, oil; IR (neat): $\nu = 2980$, 2250, 1610, 1510, 1260, 1180, 1050, 1030 cm^{-1}; ^1H NMR ($CDCl_3$/TMS): $\delta = 1.13$ (t, $J = 7.1$ Hz, 6H), 2.78–2.68 (m, 4H), 3.80 (s, 3H), 4.00–3.83 (m, 4H), 6.85 (dd, $J_1 = 6.9$ Hz, $J_2 = 1.8$ Hz, 2H), 7.23 (d, $J = 46.7$ Hz, 1H), 7.49 (dd, $J_1 = 11.5$ Hz, $J_2 = 2.7$ Hz, 2H); MS: *m/z* 323 (M^+, 40), 294 (10), 283 (30), 266 (9), 255 (17), 227 (31), 214 (10), 185 (45), 145 (100). Anal.: Calcd. for $C_{16}H_{22}NO_4P$ (323.32): C, 59.44, H, 6.86, N, 4.33. Found: C, 59.32, H, 7.05, N, 4.60.

*(Z)-Diethyl 1-(2-Cyanoethyl)-2-phenylethenylphosphonate (***5e***)*

Yield: 47%, oil; IR (neat): $\nu = 3061$, 2990, 2250, 1620, 1490, 1450,1240, 1050, 1020, 970 cm^{-1}; ^1H NMR ($CDCl_3$/TMS): $\delta = 1.10$ (t, $J = 7.1$ Hz, 6H), 2.79–2.74 (m, 4H), 3.97–3.85 (m, 4H), 7.37–7.31 (m, 3H), 7.34 (d, $J = 46.3$ Hz,1H), 7.50–7.47 (m, 2H). MS: *m/z* 293 (M^+, 20), 264 (30), 253 (91), 236 (42), 225 (38), 115 (100). HRMS: Calcd. for $C_{15}H_{20}NO_3P$ (293.30): 293.1181. Found: 293.1152.

*(Z)-Diethyl 1-(2-Cyanoethyl)-2-(2,4-dichlorophenyl)ethenylphosphonate (***5f***)*

Yield: 64%, oil; IR (neat): $\nu = 2990$, 2250, 1630, 1590, 1470, 1240,1050, 1020, 970 cm^{-1}. ^1H NMR ($CDCl_3$/TMS): $\delta =$ 1.10 (t, $J = 7.1$ Hz, 6H), 2.79–2.62 (m, 4H), 4.03–3.78 (m, 4H), 7.17 (dd, $J_1 = 8.3$ Hz, $J_2 = 2.0$ Hz, 1H), 7.16 (d, $J = 45.6$ Hz, 1H), 7.33 (d, $J = 2.0$ Hz, 1H), 7.46 (d, $J = 8.3$ Hz, 1H); MS: *m/z* 362 (M^+, 27), 361 (100), 326 (12), 298 (9), 270 (22), 252 (7). Anal.: Calcd. for $C_{15}H_{18}Cl_2NO_3P$ (362.19): C, 49.74, H, 5.01, N, 3.87. Found: C, 49.84, H, 5.10, N, 3.97.

*(Z)-Diethyl 1-(2-Cyanoethyl)-2-(4-methylphenyl) ethenylphosphonate (***5g***)*

Yield: 51%, oil; IR (neat): $\nu = 2980$, 2250, 1610, 1510, 1240, 1050,1020, 970 cm^{-1}.^1H NMR ($CDCl_3$/TMS): $\delta = 1.15$ (t, $J = 7.1$ Hz, 6H), 2.34 (s, 3H), 2.77–2.72 (m, 4H), 3.99–3.83 (m, 4H), 7.14 (d, $J = 8.0$ Hz, 2H), 7.28 (d, $J = 46.8$ Hz, 1H), 7.39 (d, $J = 8.0$ Hz, 2H). MS: *m/z* 307 (M^+, 42), 278 (29), 267 (79), 250 (34), 239 (35), 211 (33), 198 (30), 129 (100). HRMS: Calcd. for $C_{16}H_{22}NO_3P$ (307.32): 307.1337. Found: 307.1321.

*Diethyl 1-(2-Cyanoethyl)-2-(4-chlorophenyl)ethenylphosphonate (***5h***)*

Yield: 65%, oil, $Z:E = 88:12$; IR (neat): $\nu = 2990$, 2250, 1620, 1490, 1240, 1050,1020, 970 cm^{-1}. ^1H NMR($CDCl_3$/TMS): $\delta = 1.16$ (t, $J = 7.1$ Hz, $0.88 \times$ 6H), 1.40–1.35 (m, 0.12×6H), 2.55–2.45 (m, $0.12 \times$ 4H), 2.77–2.70 (m, 0.88×4H), 4.01–3.85 (m, $0.8 \times$ 4H), 4.18–4.13 (m, 0.12×4H), 7.25 (d, $J = 46.2$ Hz, 1H), 7.44–7.28 (m, 4H). MS: *m/z* 328 (M^+, 59), 327 (42), 326 (31), 287 (100), 270 (40), 231 (24). Anal.: Calcd. for $C_{15}H_{19}ClNO_3P$ (327.74): C, 54.97, H, 5.84, N, 4.27. Found: C, 54.81, H, 5.65, N, 4.39.

*Diethyl 1-(2-Cyanoethyl)-2-(4-nitrophenyl)ethenylphosphonate (**5i**)*

Yield: 55%, oil, $Z:E = 77:23$; IR (neat): $\nu = 2990$, 2250, 1590, 1520, 1350, 1240, 1040, 1020, 970 cm^{-1}. ^1H NMR($CDCl_3$/TMS): $\delta = 1.18–1.10$ (m, $0.77 \times 6H$), 1.43–1.32 (m, $0.23 \times 6H$), 2.95–2.68 (m, 4H), 4.04–3.89 (m, $0.77 \times 4H$), 4.24–4.18 (m, $0.23 \times 4H$), 7.51 (d, $J = 58.2$ Hz, 1H),7.52 (d, $J = 8.5$ Hz, $0.23 \times 2H$), 7.65 (d, $J = 8.5$ Hz, $0.77 \times 2H$), 8.21 (d, $J = 8.8$ Hz, $0.77 \times 2H$), 8.29 (d, $J = 8.6$ Hz, $0.23 \times 2H$); MS: *m/z* 338 (M^+, 29), 321 (21), 309 (24), 298 (100), 281 (40), 265 (49), 247 (12), 228 (19). Anal.: Calcd. for $C_{15}H_{19}N_2O_5P$ (338.30): C, 53.26, H, 5.66, N, 8.28. Found: C, 52.95, H, 5.79, N, 8.19.

*(Z)-Diisopropyl 1-(2-Cyanoethyl)-2-(2-methoxyphenyl)ethenylphosphonate (**5j**)*

Yield: 50%, oil; IR (neat): $\nu = 2980$, 2250, 1620, 1600, 1490, 1250, 1240, 990 cm^{-1}. ^1H NMR ($CDCl_3$/TMS): $\delta = 1.08$ (d, $J = 6.2$ Hz, 6H), 1.16 (d, $J = 9.8$ Hz, 6H), 2.82–2.72 (m, 4H), 3.82 (s, 3H), 4.60–4.53 (m, 2H), 6.82 (d, $J = 8.3$ Hz, 1H), 6.90 (t, $J = 7.5$ Hz, 1H), 7.32–7.27 (m, 1H), 7.33 (d, $J = 40.9$ Hz, 1H), 7.61 (dd, $J_1 = 7.5$ Hz, $J_2 = 1.0$ Hz, 1H) . MS: *m/z* 351 (M^+, 7), 311 (13), 278 (15), 269 (36), 236 (100), 227 (49), 218 (12). Anal.: Calcd. for $C_{18}H_{26}NO_4P$ (351.38): C, 61.53, H, 7.46, N, 3.99. Found: C, 61.43, H, 7.71, N, 4.03.

*Diisopropyl 1-(2-Cyanoethyl)-2-(3-trifluoromethoxyphenyl)ethenylphosphonate (**5k**)*

Yield: 49%, oil, $Z:E = 87:13$; IR (neat): $\nu = 2980$, 2240, 1620, 1580, 1260, 1220, 1170, 990 cm^{-1}. ^1H NMR ($CDCl_3$/TMS): $\delta = 1.01$ (d, $J = 5.9$ Hz, $0.87 \times$ 6H), 1.09 (d, $J = 6.2$ Hz, $0.87 \times$ 6H), 1.23 (d, $J = 6.2$ Hz, $0.13 \times 6H$), 1.30 (d, $J = 6.2$ Hz, $0.13 \times 6H$), 2.68–2.62 (m, $0.13 \times$ 4H), 2.82–2.71 (m, $0.87 \times 4H$), 4.60–4.55 (m, $0.87 \times 2H$), 4.66–4.61 (m, $0.13 \times 2H$), 7.21 (d, $J = 45.5$ Hz,1H), 7.43–7.16 (m, 4H). MS: *m/z* 405 (M^+, 5), 363 (19), 348 (11), 321 (100), 304 (30), 281 (11), 268 (37), 240 (94). Anal.: Calcd. for $C_{18}H_{23}F_3NO_4P$ (405.35): 405.1317. Found: 405.1302.

*Diisopropyl 1-(2-Cyanoethyl)-2-(4-nitrophenyl) ethenylphosphonate (**5l**)*

Yield: 40%, oil, $Z:E = 83:17$; IR (neat): $\nu = 2980$, 2250, 1600, 1520, 1350, 1240, 1110, 980 cm^{-1}. ^1H NMR ($CDCl_3$/TMS): $\delta = 1.31$ (d, $J = 6.2$ Hz, $0.83 \times$ 6H), 1.24 (d, $J = 6.2$ Hz, $0.83 \times 6H$), 1.31(d, $J = 6.4$ Hz, $0.17 \times 6H$), 1.37 (d, $J = 6.2$ Hz, $0.17 \times 6H$), 2.75–2.67 (m, $0.17 \times$ 4H), 2.87–2.77 (m, $0.83 \times 4H$), 4.68–4.57 (m, $0.83 \times 2H$), 4.83–4.77 (m, $0.17 \times 2H$), 7.27 (d, $J = 45.3$ Hz, 1H), 7.47 (d, $J = 8.6$ Hz, $0.17 \times 2H$), 7.65 (d, $J = 8.7$ Hz, $0.83 \times 2H$), 8.19 (d, $J = 8.8$ Hz, $0.83 \times 2H$), 8.28 (d, $J = 8.8$ Hz, $0.17 \times 2H$); MS: *m/z* 366 (M^+, 2), 324 (13), 309 (11), 282 (100), 265 (83), 247 (10), 201 (59). Anal.: Calcd. for $C_{17}H_{23}N_2O_5P$ (366.35): C, 55.73, H, 6.33, N, 7.65. Found: C, 55.68, H, 6.23, N, 7.49.

REFERENCES

[1] Minami, T.; Motoyoshiya, J. Synthesis 1992, 333 and references cited therein.

[2] Minami, T.; Utsunomiya, T.; Nakamura, S.; Okubo, M.; Kitamura, N.; Okada, Y.; Ichikawa, J. J Org Chem 1994, 59, 6717.

[3] Okauchi, T.; Kakiuchi, T.; Kitamura, N.; Utsunomiya, T.; Ichikawa, J.; Minami, T. J Org Chem 1997, 62, 8419.

[4] (a) Taylor, E. C.; Davies, H. M. L. J Org Chem 1986, 51, 1537; (b) Midura, W. H.; Mikolajczyk, M. Tetrtahedron Lett 1995, 36, 2871 and references cited therein.

[5] (a) Han, L.-B.; Tanaka, M. J Am Chem Soc 1996, 118, 1571; (b) Afarinkia, K.; Evans, M.; Graham, J. C. H.; Jimcnez-Bueno, G. Tetrahedron Lett 1998, 39, 433; (c) Brunner, H.; de Courcy, L. C.; Genet, J.-P. Synlett 2000, 201; (d) Teulade, M. P.; Savignac, P.; Aboujaoude, E. E.; Lietge, S.; Collignon, N. J Organomet Chem 1986, 304, 283.

[6] Medwid, J. B.; Paul, R.; Baker, J. S.; Brockman, J. A.; Du, M. T.; Hallett, W. A.; Hanifin, J. W.; Hardy, R. A.; Tarrant, M. E.; Torely, L. W.; Wrenns, S. J Med Chem 1990, 33, 1230.

[7] Diana, G. D.; Cutcliffe, D.; Volkots, D. L.; Mallamo, J. P.; Bailey, T. R.; Vescio, N.; Oglesby, R. C.; Nitz, T. J.; Wetzel. J.; Giranda, V.; Pevear, D. C.; Dutko, F. J. J Med Chem 1993, 36, 3240.

[8] Chihiro, M.; Nagamoto, H.; Takemura, I.; Kitano, K.; Komatsu, H.; Sekiguchi, K.; Tabusa, F.; Mori, T.; Tominaga, M.; Yabuuchi, Y. J Med Chem 1995, 38, 353.

[9] Serrano, J. L.; Sierra, T.; Gonzalez, Y.; Bolm, C.; Weickhardt, K.; Magnus, A.; Moll, G. J Am Chem Soc 1995, 117, 8312.

[10] Shen, Y.; Ni, J. J Org Chem 1997, 62, 7260.

[11] Shen, Y.; Ni, J. J Chem Res (s) 1997, 358.

[12] Shen, Y.; Ni, J. J Fluorine Chem 1998, 89, 141.

[13] Reich, H. J.; Sikorski, W. H. J Org Chem 1999, 64, 14 and references cited therein.

[14] Nickson, T. E. J Org Chem 1988, 53, 3870.

[15] (a) Still, W. C.; Gennari, C. Tetrahedron Lett 1983, 24, 4405; (b) Davis, A. A.; Rosen, J. J.; Kiddle, J. J. Tetrahedron Lett 1998, 39, 6263; (c) Kiddle, J. J.; Rosen, J. J.; Davis, A. A. Phosphorus Sulfur and Silicon 1999, 144–146, 681; (d) Tago, K.; Kogen, H. Org Lett 2000, 2, 1975.

[16] (a) Ando, K. Tetrahedron Lett 1995, 36, 4105; (b) Kokin, K.; Motoyoshiya, J.; Hayashi, S.; Aoyama, H. Synth Commun 1997, 27, 2387; (c) Ando, K. J Org Chem 1997, 62, 1934; (d) Ando, K. J Org Chem 1998, 63, 8411; (e) Ando, K. J Org Chem 1999, 64, 8406; (f) Ando, K. J Org Chem 2000, 65, 4745.