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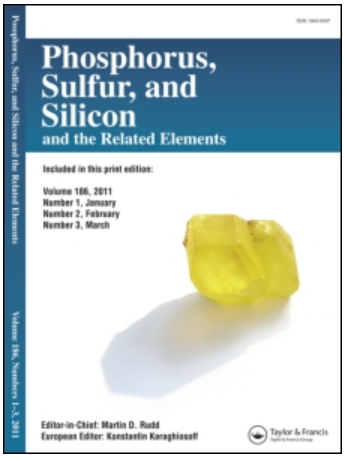

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A Novel Three Component Reaction: The Synthesis of Stable, Highly Functionalized 1,4-Diionic Nitrogen Betaines

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A Novel Three Component Reaction: The Synthesis of Stable, Highly Functionalized 1,4-Diionic Nitrogen Betaines

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The protonation of a highly reactive 1,4-dipole generated in the reaction between pyridine or isoquinoline and dialkyl acetylenedicarboxylates by the acidic C-H group of (ethoxycarbonylmethyl) triphenylphosphonium bromide leads to a vinyl pyridinium cation derivatives, which undergo a carbon-centered Michael type addition with the conjugate base of the CH-acid to produce highly functionalized stable 1,4-diionic nitrogen betaines.

Keywords 1,4-diionic nitrogen betaines; dialkyl acetylenedicarboxylates; isoquinoline; pyridine

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INTRODUCTION

The pronounced reactivity of nitrogen-containing heterocycles toward electron-deficient acetylenic compounds such as Dimethyl Acetylenedicarboxylate (DMAD) is well documented.[1] The reaction generally involves the initial addition of pyridine to DMAD to form the 1,4-zwitterionic intermediate, which undergoes a further reaction with DMAD leading to quinazoline derivatives, or it can be trapped by the various electrophiles.[2–8] Also the reaction of pyridine and DMAD has been studied in the presence of an acidic C—H group such as dimethyl malonate and ethyl cyanoacetate. In the case of dimethyl malonate, the malonate cyclohepta-1,3-diene derivatives were obtained;[9] however, the reaction of DMAD with ethyl cyanoacetate in the presence pyridine took a different course.[10]

In a continuation of our previous work on the chemistry of electron-deficient dialkyl acetylenedicarboxylates and stable 1,4-diionic nitrogen and phosphorus betaines,[11] we performed the reaction of pyridine

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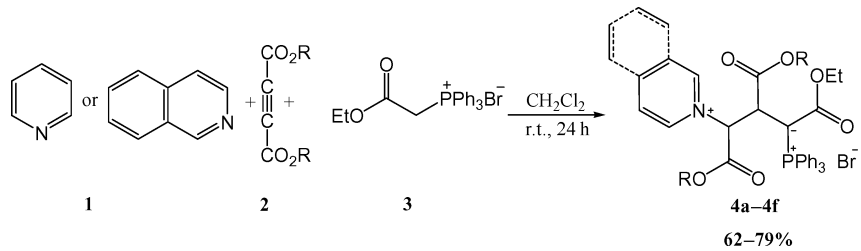
or isoquinoline and dialkyl acetylenedicarboxylates in the presence of (ethoxycarbonylmethyl) triphenylphosphonium bromide.

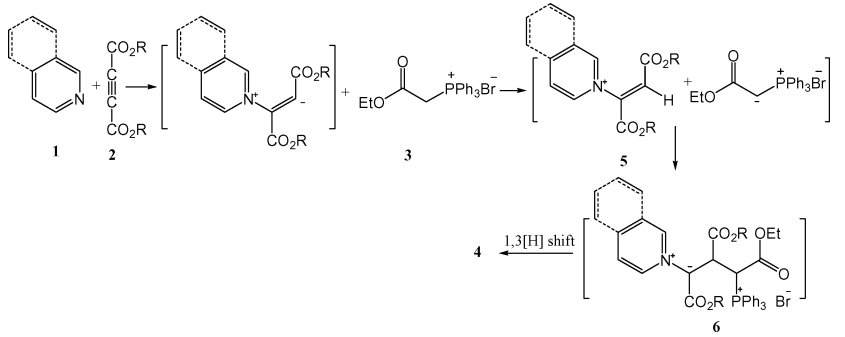
RESULTS AND DISCUSSION

As indicated in Table I, a mixture of pyridine or isoquinoline **1** and dialkyl acetylenedicarboxylate **2** when treated with (ethoxycarbonylmethyl) triphenylphosphonium bromide **3** at r.t. in the dichloromethane for 24 h affords the 1,4-diionic nitrogen betaines **4** in 62–79% yields (Scheme 1). Compounds **4a–4f** are stable solids whose structures are fully supported by IR, ^{1}H, ^{13}C, and ^{31}P NMR spectroscopy and mass spectrometric data.



SCHEME 1

On the basis of the well-established chemistry of nitrogen heterocycle nucleophiles, it is reasonable to assume that betaines **4** result from the initial addition of the pyridine or isoquinoline to the electron-deficient acetylenic ester and subsequent protonation of the 1:1 adduct by (ethoxycarbonylmethyl) triphenylphosphonium bromide. Then, the vinyl pyridinium cation **5** is attacked by the enolate anion of the CH-acid to generate the nitrogen ylide **6**, which isomerizes under the reaction conditions produced the 1,4-diionic compound **4** (Scheme 2).



SCHEME 2

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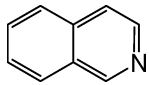
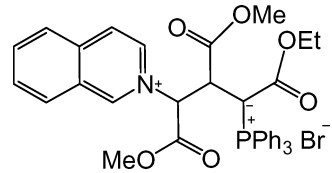
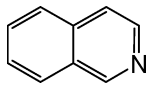
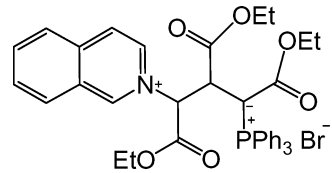
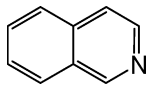
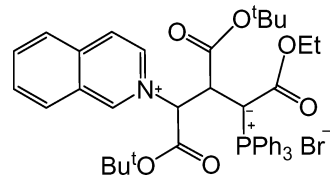
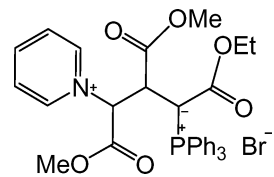
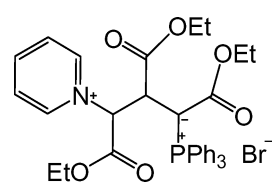
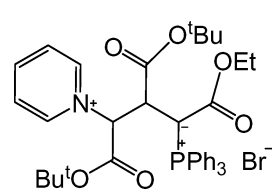
TABLE I Synthesis of Stable 1,4-Diionic Nitrogen Betaines

4	1	R	Product	Yield (%)
a		Me		79
b		Et		74
c		^{t}Bu		71
d		Me		72
e		Et		68
f		^{t}Bu		62

It is important to note that the compound of **4** has two stereogenic centers, and therefore two diastereomers are expected (Scheme 3). ^{1}H NMR spectra of the crude reaction mixture obtained from **4a–4f** were consistent with the presence of only one diastereomer.

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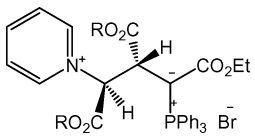

(2R,3S or 2S,3R) (2R,3R or 2S,3S)

SCHEME 3

In conclusion, we have found that the reaction of pyridine or isoquinoline with electron-deficient dialkyl acetylenedicarboxylates in the presence of a strong acidic C—H group such as (ethoxycarbonylmethyl) triphenylphosphonium bromide leads to a facile synthesis of the highly functionalized stable 1,4-diionic nitrogen betaines **4a–4f** in fairly good yields. The present method carries the advantage that the reaction is performed under mild conditions, and the substrates can be mixed without any activations and modifications.

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EXPERIMENTAL

Melting points were measured on an Electrothermal 9200 apparatus. IR spectra were recorded on a Shimadzu IR-470 spectrometer. Mass spectra were recorded on a FINNIGAN-MAT 8430 mass spectrometer operating at an ionization potential of 70 eV. ^{1}H and ^{13}C NMR spectra were recorded on a BRUKER DRX-300 AVANCE spectrometer at 300.13 and 75.47 MHz. ^{1}H and ^{13}C NMR spectra were obtained on solutions in $CDCl_3$. Pyridine, isoquinoline, and dialkyl acetylenedicarboxylates were obtained from Fluka and Merck and were used without purification.

The General Procedure for the Preparation of 4a–4f

To a magnetically stirred solution of (ethoxycarbonylmethyl) triphenylphosphonium bromide (1 mmol) and isoquinoline or pyridine (1 mmol) in dichloromethane (10 mL), a solution of dialkyl acetylenedicarboxylate (1 mmol) in dichloromethane (2 mL) was added dropwise at $-5^{\circ}C$ over 10 min. The reaction mixture was then allowed to warm up to r.t. was added dropwise and stirred for 24 h. The solvent was removed under reduced pressure, and the products were crystallized from a 2:1 ethylacetate:hexane mixture and washed with ethylacetate (3×5 mL), the products **4a–4f** were obtained.

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Selected Data for 4a

Yellow solid, yield 79%; m.p. 165–167°C (dec); IR (KBr): $\bar{\nu}$1731, 1604 cm^{-1}; MS, *m/z* (%): 491 (M^+−isoquinoline and Br, 5), 476 (5), 417 (6), 371 (8), 345 (8), 278 (25), 262 (80), 201 (25), 183 (100), 129 (75), 94 (50), 44 (50); ^{1}H NMR ($CDCl_3$): $\delta = 0.29$ (3H, t, $^3J_{HH} = 6.9$ Hz, O—CH_2—CH_3), 3.54–3.61 (3H, m, P—C—CH, O—CH_2), 3.71, 3.73 (6H, 2s, 2O—CH_3), 6.77 (H, d, $^3J_{HH} = 11.2$ Hz, CH—N), 7.13–9.57 (22H, m, H—Ar) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 13.69$ (O—CH_2—CH_3), 37.71 (d, $^1J_{PC} = 122.9$ Hz, C—P), 47.03 (d, $^2J_{PC} = 14.1$ Hz, P—C—CH), 52.39, 54.24 (2O—CH_3), 58.14 (O—CH_2), 70.70 (CH—N), 124.96 (d, $^1J_{PC} = 93.5$ Hz, C_{ipso} of P—C_6H_5), 127.52 (carbon of isoquinoline), 128.98 (d, $^3J_{PC} = 12.2$ Hz, C_{meta} of P—C_6H_5), 129.18, 129.27, 129.99, 132.38 (carbons of isoquinoline), 132.84 (C_{para} of P—C_6H_5), 132.28 (d, $^2J_{PC} = 9.3$ Hz, C_{ortho} of P—C_6H_5), 138.10 138.63, 149.38 (carbons of isoquinoline), 167.26 (C=O), 169.46 (d, $^2J_{PC} = 13.1$ Hz, C=O), 172.05 (C=O) ppm; ^{31}P NMR ($CDCl_3$): $\delta = 24.21$ (s, C—P) ppm.

Selected Data for 4b

Yellow solid, yield 74%; M.P. 194–197°C (dec); IR (KBr): $\bar{\nu}$ 1741, 1718, 1595 cm^{-1}; MS, *m/z* (%): 519 (M^+-isoquinoline and Br, 5), 489 (20), 445 (5), 417 (20), 371 (10), 345 (20), 287 (15), 278 (25), 262 (85), 201 (25), 183 (100), 129 (65), 108 (55), 51 (30); ^{1}H NMR ($CDCl_3$): $\delta = 0.33$ (3H, t, $^3J_{HH} = 7.1$ Hz, O—CH_2—CH_3), 1.17 (3H, t, $^3J_{HH} = 7.1$ Hz, O—CH_2—CH_3), 1.19 (3H, t, $^3J_{HH} = 7.1$ Hz, O—CH_2—CH_3), 3.37–3.69 (3H, m, P—C—CH, OCH_2), 4.01–4.33 (4H, m, 2O—CH_2), 6.77 (H, d, $^3J_{HH} = 11.5$ Hz, CH—N), 7.16–9.75 (22H, m, H—Ar) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 13.73$ (O—CH_2—CH_3), 13.90 (O—CH_2—CH_3), 13.97 (O—CH_2—CH_3), 37.59 (d, $^1J_{PC} = 123.8$ Hz, C—P), 47.17 (d, $^2J_{PC} = 14.2$ Hz, P—C—CH), 58.13, 61.71, 63.67 (3O—CH_2), 70.83 (d, $^3J_{PC} = 5.8$ Hz, CH—N), 117.45 (d, $^1J_{PC} = 87.4$ Hz, C_{ipso} of P—C_6H_5), 127.47 (carbon of isoquinoline), 128.93 (d, $^3J_{PC} = 11.93$ Hz, C_{meta} of P—C_6H_5), 129.77, 129.92, 130.10, 132.54 (carbons of isoquinoline), 132.83 (C_{para} of P—C_6H_5), 133.27 (d, $^2J_{PC} = 8.8$ Hz, C_{ortho} of P—C_6H_5), 138.13 138.67, 149.06 (carbons of isoquinoline), 166.78 (C=O), 169.36 (d, $^2J_{PC} = 11.$ 8 Hz, C=O), 171.64 (C=O) ppm; ^{31}P NMR ($CDCl_3$): $\delta = 24.34$ (s, C—P) ppm.

Selected Data for 4c

Yellow solid, yield 71%; mp 173–175°C (dec); IR (KBr): $\bar{\nu}$1740, 1720, 1600 cm^{-1}; MS, *m/z* (%): 575 (M^+-isoquinoline and Br, 5), 417 (5), 371 (8), 345 (5), 278 (35), 262 (90), 201 (25), 183 (100), 129 (75),

108 (45), 41 (85); ^{1}H NMR ($CDCl_3$): δ =0.33 (3H, t, $^3J_{HH}$ = 7.0 Hz, O─CH_2─CH_3), 1.36, 1.4 (18H, 2s, 2O─C$(CH_3)_3$), 3.15 (H, dd, $^3J_{PH}$ = 15.7 Hz,$^3J_{HH}$ = 10.9 Hz P─C─CH), 3.58–3.650 (2H, m, O─CH_2), 4.01–4.33 (4H, m, 2O─CH_2), 6.77 (H, d, $^3J_{HH}$ = 11.5 Hz, CH─N), 7.12–9.82 (22H, m, H─Ar) ppm; ^{13}C NMR ($CDCl_3$): δ = 13.73 (O─CH_2─CH_3), 27.53, 27.94 (2O─C$(CH_3)_3$), 37.35 (d, $^1J_{PC}$ = 123.9 Hz, C─P), 47.57 (d, $^2J_{PC}$ = 13.8 Hz, P─C─CH), 58.02 (O─CH_2), 71.89 (d, $^3J_{PC}$ = 5.6 Hz, CH─N), 82.28, 85.20 (2O─C$(CH_3)_3$), 117.64 (d, $^1J_{PC}$ = 87.3 Hz, C_{ipso} of P─C_6H_5), 127.43, 129.52 (carbons of isoquinoline), 130.06 (d, $^3J_{PC}$ = 12.98 Hz, C_{meta} of P─C_6H_5), 131.30, 131.64, 132.57 (carbons of isoquinoline), 134.56 (C_{para} of P─C_6H_5), 134.72 (d, $^2J_{PC}$ = 9.5 Hz, C_{ortho} of P─C_6H_5), 137.88, 138.04, 138.64, 151.80 (carbons of isoquinoline), 165.43 (C═O), 169.36 (d, $^2J_{PC}$ = 12.2 Hz, C═O), 170.49 (C═O) ppm; ^{31}P NMR ($CDCl_3$): δ = 24.81 (s, C─P) ppm.

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Selected Data for 4d

Yellow solid, yield 72%; mp 115–118°C (dec); IR (KBr): $\bar{\nu}$1729, 1600 cm^{-1}; MS, *m/z* (%): 575 (M^+- pyridine and Br, 3), 419 (8), 403 (10), 345 (25), 287 (15), 278 (35), 262 (87), 201 (20), 183 (100), 152 (25), 108 (40), 77 (20), 51 (30); ^{1}H NMR ($CDCl_3$): δ =0.36 (3H, t, $^3J_{HH}$ = 7.1 Hz, O─CH_2─CH_3), 3.34–3.41 (H, m, P─C─CH), 3.69, 3.73 (6H, 2s, 2O─CH_3), 4.08–4.18 (2H, m, O─CH_2), 7.27 (H, d, $^3J_{HH}$ = 11.5 Hz, CH─N), 7.55–10.06 (20H, m, H─Ar) ppm; ^{13}C NMR ($CDCl_3$): δ = 13.62 (O─CH_2─CH_3), 36.97 (d, $^1J_{PC}$ = 123.3 Hz, C─P), 47.29 (d, $^2J_{PC}$ = 13.87 Hz, P─C─CH), 52.53, 54.35 (2O─CH_3), 58.38 (O─CH_2), 70.75 (d, $^3J_{PC}$ = 5.8 Hz CH─N), 117.45 (d, $^1J_{PC}$ = 87.5 Hz, C_{ipso} of P─C_6H_5), 127.31 (carbon of pyridine), 129.22 (d, $^3J_{PC}$ = 12.2 Hz, C_{meta} of P─C_6H_5), 133.02 (d, $^4J_{PC}$ = 2.4 Hz, C_{para} of P─C_6H_5), 133.35 (d, $^2J_{PC}$ = 9.8 Hz, C_{ortho} of P─C_6H_5), 145.79, 148.99 (carbons of pyridine), 167.41 (C═O), 169.47 (d, $^2J_{PC}$ = 9.9 Hz, C═O), 172.02 (C═O) ppm; ^{31}P NMR ($CDCl_3$): δ = 27.45 (s, C─P) ppm.

Selected Data for 4e

Yellow solid, yield 68%; mp 175–178°C (dec); IR (KBr): $\bar{\nu}$1723, 1607 cm^{-1}; MS, *m/z* (%): 519 (M^+- pyridine and Br, 5), 489 (30), 418 (10), 397 (15), 345 (25), 287 (14), 278 (20), 262 (90), 201 (25), 183 (100), 152 (20), 108 (45), 77 (20), 51 (25); ^{1}H NMR ($CDCl_3$): δ =0.38 (3H, t, $^3J_{HH}$ = 7.1 Hz, O─CH_2─CH_3), 1.14 (3H, t, $^3J_{HH}$ = 7.1 Hz, O─CH_2─CH_3), 1.19 (3H, t, $^3J_{HH}$ = 7.1 Hz, O─CH_2─CH_3), 3.59–3.65 (H, m, P─C─CH), 4.08–4.44 (6H, m, 3O─CH_2), 6.60 (H, d, $^3J_{HH}$ = 12.2 Hz, CH─N), 7.55–10.20 (20H, m, H─Ar) ppm; ^{13}C NMR ($CDCl_3$): δ = 13.63, 13.72, 14.03

(3O—CH_2—CH_3), 36.92 (d, $^1J_{PC} = 123.8$ Hz, C—P), 47.41 (d, $^2J_{PC} = 13.92$ Hz, P—C—CH), 58.33, 61.79, 63.73 (3O—CH_2), 71.00 (d, $^3J_{PC} = 5.8$ Hz CH—N), 117.55 (d, $^1J_{PC} = 87.4$ Hz, C_{ipso} of P—C_6H_5), 127.50 (carbon of pyridine), 129.16 (d, $^3J_{PC} = 12.3$ Hz, C_{meta} of P—C_6H_5), 133.98 (C_{para} of P—C_6H_5), 133.35 (d, $^2J_{PC} = 9.8$ Hz, C_{ortho} of P—C_6H_5), 143.81, 148.98 (carbons of pyridine), 166.88 (C=O), 169.38 (d, $^2J_{PC} = 11.70$ Hz, C=O), 172.00 (C=O) ppm; ^{31}P NMR ($CDCl_3$): $\delta = 26.94$ (s, C—P) ppm.

Selected Data for 4f

Yellow solid, yield 62%; mp 174–176°C (dec); IR (KBr): $\bar{\nu}$1722, 1612 cm^{-1}; MS, *m/z* (%): 575 (M^+- pyridine and Br, 3), 418 (3), 397 (4), 301 (5), 278 (20), 262 (90), 201 (25), 183 (100), 152 (25), 108 (40), 79 (30), 57 (20), 41 (60); ^{1}H NMR ($CDCl_3$): $\delta = 0.38$ (3H, t, $^3J_{HH} = 7.0$ Hz, O—CH_2—CH_3), 1.34, 1.37 (18H, 2s, 2O—$C(CH_3)_3$), 3.08 (H, dd, $^3J_{PH} = 15.5$ Hz,$^3J_{HH} = 10.9$ Hz P—C—CH), 3.56, 3.70 (2H, 2m, O—CH_2), 6.61 (H, d, $^3J_{HH} = 10.9$ Hz, CH—N), 7.55–9.43 (20H, m, H—Ar) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 13.64$ (O—CH_2—CH_3), 27.49, 27.89 (2O—$C(CH_3)_3$), 36.53 (d, $^1J_{PC} = 123.3$ Hz, C—P), 47.86 (d, $^2J_{PC} = 14.1$ Hz, P—C—CH), 58.15 (O—CH_2), 72.13 (d, $^3J_{PC} = 5.7$ Hz, CH—N), 82.38, 85.33 (2O—$C(CH_3)_3$), 117.87 (d, $^1J_{PC} = 87.6$ Hz, C_{ipso} of P—C_6H_5), 127.46 (carbon of pyridine), 129.08 (d, $^3J_{PC} = 12.98$ Hz, C_{meta} of P—C_6H_5), 132.90 (C_{para} of P—C_6H_5), 133.39 (d, $^2J_{PC} = 9.4$ Hz, C_{ortho} of P—C_6H_5), 143.23, 149.15 (carbons of pyridine), 165.58 (C=O), 168.92 (d, $^2J_{PC} = 12.4$ Hz, C=O), 170.33 (C=O) ppm; ^{31}P NMR ($CDCl_3$): $\delta = 24.64$ (s, C—P) ppm.

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