

An Efficient One-Pot Synthesis of Pyrrolo[4,3,2-*ij*]isoquinoline Derivatives by a Consecutive Aza-Wittig/Electrocyclic Ring-Closure/Intramolecular Acylation Process

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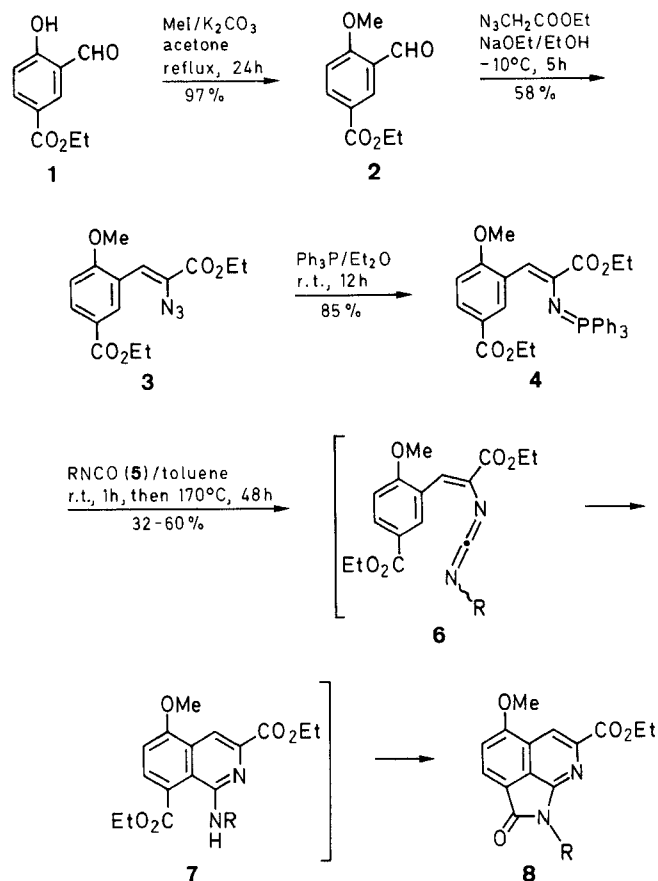
Iminophosphorane, ethyl 5-ethoxycarbonyl-2-methoxy- α -[(triphenylphosphoranylidene)amino]cinnamate (**4**) reacts with aliphatic and aromatic isocyanates in toluene at 170°C to give directly the corresponding pyrrolo[4,3,2-*ij*]isoquinolines **8** in moderate yields.

We have been interested recently in exploiting the unique reactivities afforded by the iminophosphorane function in developing efficient strategies for the preparation of polyheterocycles¹. In this context, we have found that the tandem aza-Wittig/electrocyclization strategy has shown to be a useful protocol for the preparation of fused indoles,² pyridines³ and isoquinoline⁴ derivatives. In essence our synthetic approach consists of treating iminophosphoranes bearing an unsaturated group with easily accessible heterocumulenes such as isocyanates in an aza-Wittig-type reaction to give a 1,3,5-hexatriene moiety containing cumulated double bonds at one end, which subsequently undergo electrocyclic ring closure.

As a further extension of the above methodology we report herein a new general synthesis of the previously unreported pyrrolo[4,3,2-*ij*]isoquinoline ring system, under neutral conditions, based on the aza-Wittig-type reaction of iminophosphorane **4**. One-pot double-annulation occurs via an unsaturated carbodiimide, available from the iminophosphorane **4** and isocyanates, which then undergoes electrocyclic ring-closure followed by 1,3-hydrogen shift and finally intramolecular acylation of the exocyclic amino group.

The salicylaldehyde **1** is prepared in 35% yield by Duff reaction of ethyl *p*-hydroxybenzoate with hexamethylenetetramine (HMT) in trifluoroacetic acid.⁵ Treatment of **1** with methyl iodide in the presence of potassium carbonate leads to **2** in 97% yield. The azide **3** is easily prepared in 58% by condensation of **2** with ethyl azidoacetate in the presence of sodium ethoxide at -10°C. Staudinger reaction⁶ of azide **3** with triphenylphosphine at room temperature affords the iminophosphorane **4** in 85% yield. The reaction of iminophosphorane **4** with several isocyanates in dry toluene at room temperature for 1 hour and then at 170°C in a sealed tube for 48 hours leads directly to the corresponding pyrrolo[4,3,2-*ij*]isoquinoline derivatives **8** in moderate yields (Table).

The IR spectra of pyrrolo[4,3,2-*ij*]isoquinolines **8** show absorption bands due to the two carbonyl stretch at $\nu = 1742\text{--}1721\text{ cm}^{-1}$ and at $\nu = 1719\text{--}1706\text{ cm}^{-1}$ respectively, except for compound **8a** which only show an absorption band at $\nu = 1705\text{ cm}^{-1}$. The ¹H NMR spectra reveal the presence of only one ethoxy group, while the ¹³C NMR spectra show two signals at $\delta = 164.59\text{--}165.77$ and $\delta = 166.42\text{--}167.31$ due to the carbon atoms of the two carbonyl groups. Salient features of the ¹H and ¹³C NMR spectra are given in the Table.



8	R	8	R
a	<i>i</i> -Pr	e	3-MeC ₆ H ₄
b	Bn	f	4-MeOC ₆ H ₄
c	Ph	g	4-FC ₆ H ₄
d	4-MeC ₆ H ₄	h	4-ClC ₆ H ₄

The EI-mass spectra show the expected molecular ion peaks.

We believe that the conversion **4** to **8** involves an initial aza-Wittig-type reaction between the iminophosphorane and the isocyanate to give a carbodiimide **6** (as evidenced by IR) as intermediate, which undergoes electrocyclic ring-closure and further 1,3-hydrogen shift to give the isoquinoline derivative **7** which, under the reaction conditions, cyclizes to give **8**.

The above method demonstrated that the tandem aza-Wittig/electrophilic ring-closure annulation strategy affords a general entry to a variety of pyrrolo[4,3,2-*ij*]isoquinolines with variable substituents at the pyrrole ring.

Table. Pyrrolo[4,3,2-*ij*]isoquinoline Derivatives **8** Prepared

Prod- uct	Yield (%) ^a	mp (°C) ^b	Molecular Formula ^c	IR (Nujol) ^d ν (cm ⁻¹)	¹ H NMR (200 MHz) ^e (CDCl ₃ /TFA/TMS) δ, <i>J</i> (Hz)	¹³ C NMR (50 MHz) ^e (CDCl ₃ /TFA/TMS) δ, <i>J</i> (Hz)	MS (70 eV) ^f <i>m/z</i> (%)
8a	32	225–226	C ₁₇ H ₁₈ N ₂ O ₄ (314.3)	1705, 1585, 1437, 1297	1.47 (t, 3H, <i>J</i> = 7.1, CH ₃), 1.64 [d, 6H, <i>J</i> = 6.9, CH(CH ₃) ₂], 4.09 (s, 3H, CH ₃ O), 4.47 (q, 2H, <i>J</i> = 7.1, CH ₂), 4.86 [sept, 1H, <i>J</i> = 6.9, CH(CH ₃) ₂], 7.08 (d, 1H, <i>J</i> = 7.8, H-4), 8.00 (d, 1H, <i>J</i> = 7.8, H-3), 8.33 (s, 1H, H-6)	14.39 (CH ₃), 20.37 [CH(CH ₃) ₂], 44.12 [CH(CH ₃) ₂], 56.4 (CH ₃ O), 61.63 (CH ₂), 110.91 (C ₄), 113.26 (C ₆), 119.05 (q), 122.57 (q), 122.59 (q), 128.90 (C ₃), 143.11 (q), 156.15 (C _{8a}), 160.62 (C ₅), 165.77 (CO ₂ Et), 167.31 (C ₂)	314 (M ⁺ , 50), 299 (20), 272 (100), 225 (43), 114 (11), 113 (9), 101 (9)
8b	44	230–231	C ₂₁ H ₁₈ N ₂ O ₄ (362.4)	1742, 1719, 1495, 1295	1.47 (t, 3H, <i>J</i> = 7.1, CH ₃), 4.05 (s, 3H, CH ₃ O), 4.52 (q, 2H, <i>J</i> = 7.1, CH ₂), 5.17 (s, 2H, NCH ₂), 7.03 (d, 1H, <i>J</i> = 7.9, H-4), 7.22–7.35 (m, 3H), 7.55–7.59 (m, 2H), 8.00 (d, 1H, <i>J</i> = 7.9, H-3), 8.35 (s, 1H, H-6)	14.43 (CH ₃), 42.81 (NCH ₂), 56.56 (CH ₃ O), 61.72 (CH ₂), 111.09 (C ₄), 113.85 (C ₆), 118.68 (q), 122.75 (q), 122.77 (q), 127.61 (C ₄), 128.56 (C ₂ , C ₃), 128.92 (C ₂ , C ₃), 129.41 (C ₃), 137.11 (C ₁), 143.26 (q), 155.75 (C _{8a}), 160.81 (C ₅), 165.71 (CO ₂ Et), 167.17 (C ₂)	362 (M ⁺ , 100), 361 (6), 290 (41), 288 (44), 260 (28), 185 (39), 91 (93)
8c	55	264	C ₂₀ H ₁₆ N ₂ O ₄ (348.3)	1731, 1709, 1436, 1292	1.40 (t, 3H, <i>J</i> = 7.1, CH ₃), 4.09 (s, 3H, CH ₃ O), 4.42 (q, 2H, <i>J</i> = 7.1, CH ₂), 7.15 (d, 1H, <i>J</i> = 7.9, H-4), 7.37–7.44 (m, 1H), 7.50–7.55 (m, 2H), 7.60–7.66 (m, 2H), 8.13 (d, 1H, <i>J</i> = 7.9, H-3), 8.41 (s, 1H, H-6)	14.14 (CH ₃), 56.78 (CH ₃ O), 62.26 (CH ₂), 111.96 (C ₄), 114.78 (C ₆), 117.45 (q), 122.90 (q), 122.93 (q), 126.54 (C ₂), 128.10 (C ₄), 129.26 (C ₃), 130.63 (C ₃), 133.01 (C ₁), 141.81 (q), 155.64 (C _{8a}), 161.24 (C ₅), 165.36 (CO ₂ Et), 166.56 (C ₂)	348 (M ⁺ , 56), 277 (19), 276 (100), 77 (18), 55 (5)
8d	34	276–277	C ₂₁ H ₁₈ N ₂ O ₄ (362.4)	1722, 1708, 1493, 1297	1.41 (t, 3H, <i>J</i> = 7.1, CH ₃), 2.41 (s, 3H, CH ₃ –Ar), 4.09 (s, 3H, CH ₃ O), 4.43 (q, 2H, <i>J</i> = 7.1, CH ₂), 7.15 (d, 1H, <i>J</i> = 7.8, H-4), 7.32 (d, 2H, <i>J</i> = 8.1), 7.50 (d, 2H, <i>J</i> = 8.1), 8.12 (d, 1H, <i>J</i> = 7.8, H-3), 8.41 (s, 1H, H-6)	14.19 (CH ₃), 21.15 (CH ₃ –Ar), 56.74 (CH ₃ O), 62.14 (CH ₂), 111.82 (C ₄), 114.60 (C ₆), 117.70 (q), 122.93 (q), 126.45 (C ₂), 129.90 (C ₃), 130.40 (C ₃), 130.48 (C ₁), 138.15 (C ₄), 142.17 (q), 155.89 (C _{8a}), 161.15 (C ₅), 165.38 (CO ₂ Et), 166.65 (C ₂) ^g	362 (M ⁺ , 50), 333 (5), 291 (21), 290 (100), 247 (6), 144 (5), 91 (11)
8e	39	274–275	C ₂₁ H ₁₈ N ₂ O ₄ (362.4)	1721, 1709, 1438, 1295	1.39 (t, 3H, <i>J</i> = 7.1, CH ₃), 2.39 (s, 3H, CH ₃ –Ar), 4.14 (s, 3H, CH ₃ O), 4.42 (q, 2H, <i>J</i> = 7.1, CH ₂), 7.21–7.42 (m, 5H), 8.21 (d, 1H, <i>J</i> = 7.9, H-3), 8.47 (s, 1H, H-6)	14.03 (CH ₃), 21.16 (CH ₃ –Ar), 56.95 (CH ₃ O), 62.63 (CH ₂), 112.63 (C ₄), 115.24 (C ₆), 117.15 (q), 123.04 (q), 123.33 (q), 124.26, 127.83, 129.28, 129.78, 131.52 (C ₃), 132.19 (C ₃), 139.71 (C ₁), 140.55 (q), 155.60 (C _{8a}), 161.56 (C ₅), 164.83 (CO ₂ Et), 166.81 (C ₂)	362 (M ⁺ , 63), 333 (5), 291 (22), 290 (100), 144 (6), 91 (31), 65 (18)
8f	45	280–281	C ₂₁ H ₁₈ N ₂ O ₅ (378.4)	1724, 1706, 1252, 1063	1.39 (t, 3H, <i>J</i> = 7.1, CH ₃), 3.81 (s, 3H, CH ₃ O), 4.16 (s, 3H, CH ₃ O), 4.42 (q, 2H, <i>J</i> = 7.1, CH ₂), 6.96 (d, 2H, <i>J</i> = 8.8), 7.27 (d, 1H, <i>J</i> = 7.9, H-4), 7.38 (d, 2H, <i>J</i> = 8.8), 8.22 (d, 1H, <i>J</i> = 7.9, H-3), 8.47 (s, 1H, H-6)	14.03 (CH ₃), 55.41 (CH ₃ O), 57.01 (CH ₃ O), 62.74 (CH ₂), 112.83 (C ₄), 114.78 (C ₃), 115.31 (C ₆), 117.03 (q), 122.99 (q), 123.43 (q), 124.67 (C ₁), 128.79 (C ₂), 131.75 (C ₃), 140.03 (q), 155.64 (C _{8a}), 159.98 (C ₄), 161.63 (C ₅), 164.59 (CO ₂ Et), 167.00 (C ₂)	378 (M ⁺ , 68), 306 (100), 28 (6), 263 (7), 137 (9), 91 (12), 85 (11)
8g	60	303–304	C ₂₀ H ₁₅ FN ₂ O ₄ (366.3)	1727, 1710, 1294, 1233	1.40 (t, 3H, <i>J</i> = 7.0, CH ₃), 4.18 (s, 3H, CH ₃ O), 4.44 (q, 2H, <i>J</i> = 7.1, CH ₂), 7.19 (t, 2H, <i>J</i> = 8.5), 7.31 (d, 1H, <i>J</i> = 8.1, H-4), 7.49 (dd, 2H, <i>J</i> = 4.7, 8.9), 8.27 (d, 1H, <i>J</i> = 8.1, H-3), 8.53 (s, 1H, H-6)	13.98 (CH ₃), 57.14 (CH ₃ O), 63.15 (CH ₂), 113.25 (C ₄), 115.80 (C ₆), 116.66 (d, ² <i>J</i> _{F-C} = 23.2, C ₃), 116.67 (q), 123.19 (q), 123.58 (q), 127.91 (d, ⁴ <i>J</i> _{F-C} = 3.2, C ₁), 129.41 (d, ³ <i>J</i> _{F-C} = 6.9, C ₂), 132.39 (C ₃), 139.33 (q), 155.15 (C _{8a}), 161.97 (C ₅), 162.82 (d, ¹ <i>J</i> _{F-C} = 250.1, C ₄), 164.59 (CO ₂ Et), 166.76 (C ₂)	366 (M ⁺ , 43), 337 (5), 295 (19), 294 (100), 293 (10), 266 (5), 251 (6), 95 (12)

Table. (continued)

Prod- uct	Yield (%) ^a	mp (°C) ^b	Molecular Formula ^c	IR (Nujol) ^d ν (cm ⁻¹)	¹ H NMR (200 MHz) ^e (CDCl ₃ /TFA/TMS) δ , J (Hz)	¹³ C NMR (50 MHz) ^e (CDCl ₃ /TFA/TMS) δ , J (Hz)	MS (70 eV) ^f m/z (%)
8h	47	306–307	C ₂₀ H ₁₅ ClN ₂ O ₄ (382.8)	1723, 1708, 1502, 1265	1.42 (t, 3H, J = 7.1, CH ₃), 4.17 (s, 3H, CH ₃ O), 4.45 (q, 2H, J = 7.1, CH ₂), 7.30 (d, 1H, J = 8.0, H-4), 7.40–7.51 (m, 4H), 8.25 (d, 1H, J = 8.0, H-3), 8.51 (s, 1H, H-6)	13.97 (CH ₃), 57.11 (CH ₃ O), 63.11 (CH ₂), 113.15 (C ₄), 115.76 (C ₆), 116.54 (q), 123.13 (q), 123.46 (q), 128.34 (C ₃), 129.64 (C ₂), 130.68 (C ₄), 132.30 (C ₃), 134.92 (C ₁), 139.50 (q), 154.79 (C _{8a}), 161.95 (C ₅), 164.77 (CO ₂ Et), 166.42 (C ₂)	384 (M ⁺ + 2, 15), 382 (M ⁺ , 44), 312 (35), 311 (24), 310 (100), 111 (12)

^a Yield of isolated pure product.^b Uncorrected.^c Satisfactory microanalyses obtained: C \pm 0.25, H \pm 0.29, N \pm 0.28.^d Recorded on a Nicolet FT 5DX spectrophotometer.^e Recorded on a Bruker AC-200 spectrometer.^f Recorded on a Hewlett-Packard 5993C instrument.^g One quaternary carbon not observed.**Ethyl 3-Formyl-4-hydroxybenzoate (1):**

To a well-stirred solution of ethyl *p*-hydroxybenzoate (4.98 g, 30 mmol) in TFA (24 mL) was added HMT (8.41 g, 60 mmol). The mixture was heated at 80°C for 3 h. After cooling at 0°C, 50% H₂SO₄ (15 mL) and H₂O (90 mL) were added and the resultant mixture was stirred at r.t. for 1 h, then extracted with Et₂O (2 \times 50 mL). The combined organic layers were washed with H₂O (2 \times 25 mL), and dried (MgSO₄). The solvent was evaporated and the crude product **1** was chromatographed on a silica gel column (40 cm \times 3.5 cm; 70–230 mesh) using CH₂Cl₂ as eluent; yield: 1.86 g (32%); mp 69–70°C.

C₁₀H₁₀O₄ calc. C 61.85 H 5.19
(194.2) found 61.63 4.95

IR (Nujol): ν = 3347, 1707, 1662 cm⁻¹.

¹H NMR (CDCl₃/TMS): δ = 1.41 (t, 3H, J = 7.0 Hz, CH₃CH₂O), 4.38 (q, 2H, J = 7.0 Hz, CH₃CH₂O), 7.02 (d, 1H, J = 8.8 Hz, H-5), 8.18 (dd, 1H, J = 2.1, 8.8 Hz, H-6), 8.31 (d, 1H, J = 2.1 Hz, H-2), 9.95 (s, 1H, CHO), 11.38 (s, 1H, OH).

¹³C NMR (CDCl₃/TMS): δ = 14.31 (CH₃), 61.16 (CH₂), 117.82 (C₅), 120.02 (C₁), 122.58 (C₃), 135.97 (C₂), 137.72 (C₆), 164.91 (C₄), 165.02 (CO₂Et), 196.34 (CHO).

Ethyl 3-Formyl-4-methoxybenzoate (2):

To a solution of ethyl 3-formyl-4-hydroxybenzoate (**1**; 3.88 g, 20 mmol) in acetone (40 mL) were added MeI (5.68 g, 40 mmol) and K₂CO₃ (2.76 g, 20 mmol). The resultant mixture was stirred at reflux temperature for 24 h. After cooling, the solution was poured into H₂O (50 mL) and extracted with EtOAc (3 \times 50 mL). The combined organic layers were washed with H₂O (2 \times 50 mL) and dried (MgSO₄). The solvent was evaporated and the crude product **2** was chromatographed on a silica gel column (40 cm \times 3.5 cm; 70–230 mesh) using hexane/Et₂O (2:3) as eluent; yield 4.03 g (97%); mp 76–77°C (Et₂O/hexane).

C₁₁H₁₂O₄ calc. C 63.45 H 5.81
(208.2) found 63.59 5.63

IR (Nujol): ν = 1708, 1682 cm⁻¹.

¹H NMR (CDCl₃/TMS): δ = 1.39 (t, 3H, J = 7.2 Hz, CH₃CH₂O), 4.01 (s, 3H, CH₃O), 4.36 (q, 2H, J = 7.2 Hz, CH₃CH₂O), 7.04 (d, 1H, J = 8.8 Hz, H-5), 8.22 (dd, 1H, J = 2.2, 8.8 Hz, H-6), 8.46 (d, 1H, J = 2.2 Hz, H-2), 10.44 (s, 1H, CHO).

¹³C NMR (CDCl₃/TMS): δ = 14.25 (CH₃), 56.00 (CH₃O), 60.97 (CH₂), 111.49 (C₅), 123.13 (C₃), 124.28 (C₁), 130.31 (C₂), 137.02 (C₆), 164.62 (C₄), 165.38 (CO₂Et), 188.78 (CHO).

Ethyl α -Azido-5-ethoxycarbonyl-2-methoxycinnamate (3):

A mixture of ethyl azidoacetate (10.32 g, 80 mmol) and ethyl 3-formyl-4-methoxybenzoate (**2**; 4.16 g, 20 mmol) was added dropwise under N₂ at –10°C to a well-stirred solution containing Na (1.84 g) in anhyd. EtOH (100 mL). The mixture was stirred for 5 h,

poured into aq. NH₄Cl (150 mL), and then extracted with Et₂O (3 \times 100 mL). The combined organic layers were washed with H₂O (2 \times 100 mL) and dried (MgSO₄). Concentration to dryness yielded a crude material which was recrystallized from EtOH to give **3**; yield: 3.70 g (58%); mp 92–93°C.

C₁₅H₁₇N₃O₅ calc. C 56.42 H 5.37 N 13.16
(319.3) found 56.28 5.49 13.27

MS (EI, 70 eV): m/z (%) = 319 (M⁺, 19), 218 (100).IR (Nujol): ν = 2112, 1706 cm⁻¹.

¹H NMR (CDCl₃/TMS): δ = 1.40 (t, 6H, J = 7.2 Hz, 2CH₃CH₂O), 3.91 (s, 3H, CH₃O), 4.37 (q, 4H, J = 7.2 Hz, 2CH₃CH₂O), 6.88 (d, 1H, J = 8.8 Hz, H-3), 7.29 (s, 1H, H- β), 7.98 (dd, 1H, J = 2.0, 8.8 Hz, H-4), 8.82 (d, 1H, J = 2.0 Hz, H-6).

¹³C NMR (CDCl₃/TMS): δ = 14.15 (CH₃), 14.32 (CH₃), 55.85 (CH₃O), 60.79 (CH₂), 62.22 (CH₂), 109.90 (C₃), 118.09 (C₆), 121.94 (C₁*), 122.61 (C₅*), 126.33 (C₄), 132.09 (C₆), 132.27 (C₄), 160.71 (C₂), 163.41 (CO₂Et), 166.08 (CO₂Et).

* Interchangeable.

Ethyl 5-Ethoxycarbonyl-2-methoxy- α [(triphenylphosphoranylidene)amino]cinnamate (4):

A solution of Ph₃P (2.62 g, 10 mmol) in dry Et₂O (30 mL) was added dropwise under N₂ at r.t. to a solution of **3** (3.19 g, 10 mmol) in the same solvent (15 mL). The mixture was stirred at r.t. for 12 h. The separated solid was collected by filtration, air-dried and recrystallized from benzene/hexane (1:1) to give **4**; yield: 4.70 g (85%); mp 135–136°C.

C₃₃H₃₂NO₅P calc. C 71.60 H 5.83 N 2.53
(553.6) found 71.48 5.96 2.39

MS (EI, 70 eV): m/z (%) = 553 (M⁺, 6), 183 (100).IR (Nujol): ν = 1718, 1693 cm⁻¹.

¹H NMR (CDCl₃/TMS): δ = 0.99 (t, 3H, J = 7.1 Hz), 1.12 (t, 3H, J = 7.1 Hz), 3.85 (q, 2H, J = 7.1 Hz), 3.87 (s, 3H, CH₃O), 4.22 (q, 2H, J = 7.1 Hz), 6.85 (d, 1H, J = 8.6 Hz, H-3), 6.94 (d, 1H, J = 7.2 Hz, H- β), 7.33–7.40 (m, 9H), 7.73–7.88 (m, 7H), 9.73 (d, 1H, J = 2.0 Hz, H-6).

¹³C NMR (CDCl₃/TMS): δ = 14.06 (CH₃), 14.17 (CH₃), 55.59 (CH₃O), 60.21 (CH₂), 60.74 (CH₂), 107.48 (³ J_{P-C} = 19.9 Hz, C₆), 109.13 (C₃), 122.25 (C₁*), 127.10 (C₅*), 128.10 (³ J_{P-C} = 12.2 Hz, C_m), 128.52 (C₄), 130.80 (⁴ J_{P-C} = 2.6 Hz, C_p), 131.77 (C₆), 132.52 (² J_{P-C} = 9.8 Hz, C_o), 133.11 (¹ J_{P-C} = 103.1 Hz, C_i), 137.65 (² J_{P-C} = 6.7 Hz, C₂), 159.74 (C₂), 167.02 (5-CO₂Et), 167.93 (³ J_{P-C} = 7.7 Hz, α -CO₂Et).

Ethyl 1-Alkyl(aryl)-1,2-dihydro-5-methoxy-2-oxo-1H-pyrrolo[4,3,2-*ij*]isoquinoline-7-carboxylates **8; General Procedure:**

To a solution of the iminophosphorane **4** (1.11 g, 2 mmol) in dry toluene (15 mL), the appropriate isocyanate (2 mmol) was added

dropwise. The resultant solution was stirred at r.t. for 1 h and then heated in a sealed tube at 170°C for 48 h. After cooling, the precipitated solid was collected by filtration and recrystallized from toluene to give **8**. Compound **8b** is recrystallized from EtOH.

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