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A novel synthesis of functionalized spirooxaphospholes

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Abstract: The reaction of ethyl propiolate with triphenylphosphine in the presence of *N*-alkylisatins led to ethyl oxaphosphole in good yields. The reaction of dialkyl acetylenedicarboxylates with Ph_3P in the presence of N-alkylisatins led to oxaphosphole-3,4-dicarboxylate in good yields.

Keywords: Ethyl propiolate; Triphenylphosphine; Alkylisatins; Oxaphosphole.

Introduction

Organophosphorus compounds are widely used in organic synthesis [1].In recent years there has been increasing interest in the synthesis of organophosphorus compounds, that is, those bearing a carbon atom bound directly to a phosphorus atom. This interest has resulted from the recognition of the value of such compounds in a variety of biological, industrial and chemical synthetic uses. A large number of methods have appeared describing novel syntheses of organophosphorus compounds [2-4]. Thesuccessful attack by nucleophilic trivalent phosphines on a carbon atom is facilitated when the latter is conjugated with a carbonyl group, or when it is part of an unsaturated bond otherwise activated [5-10].

Result and discussion

The reaction of Ph_3P with ethyl propiolate in the presence of *N*-alkylisatins led to 1,2-λ^5-oxaphosphole-4-carboxylate **4** in 85-94% yields (Scheme **1**). Structures of compounds**4a**–**c** were apparent from their mass spectra, which displayed, in each case, the molecular ion peak at appropriate *m/z* values.

The ^{1}H- and ^{13}C-NMR spectroscopic data, as well as IR spectra, are in agreement with the proposed structures. The ^{1}H-NMR spectrum of **4a** exhibited a singlet at (δ = 3.25 ppm) arising from the *N*Me proton. The carbonyl groups resonances in the ^{13}C-NMR spectra of **4a** appear at δ = 168.4 ($^3J_{CP}$ = 21.2) and 169.7 ppm. The ^{31}P-NMR signal of **4a** was found at (δ = -50.35 ppm). The mass spectrum of **4a** displayed the molecular ion peak at *m/z* = 521, which is consistent with the 1:1:1 adduct of Ph_3P, ethyl propiolate and *N*-methylisatin.
Mechanistically, it is conceivable that the reaction involves the initial formation of a 1,3-dipolar intermediate **5** between triphenylphosphine**1** and ethyl propiolate**2a**, which reacts with the carbonyl group of *N*-alkylisatin **3**to produce **4**. Cyclization of this zwitterionic intermediate leads to the spiro compound **4** (Scheme **2**).

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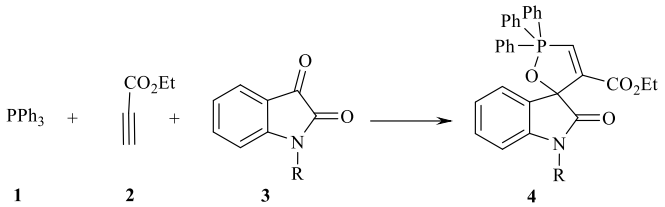
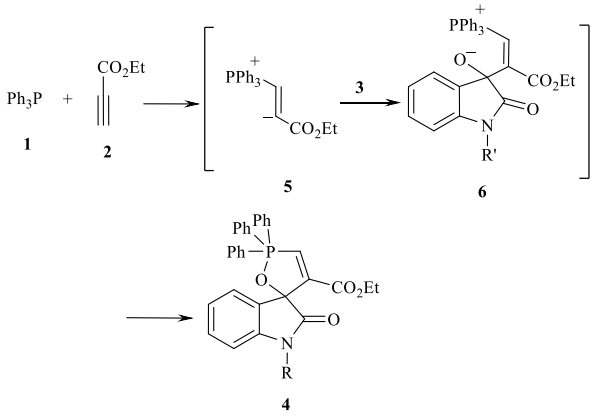


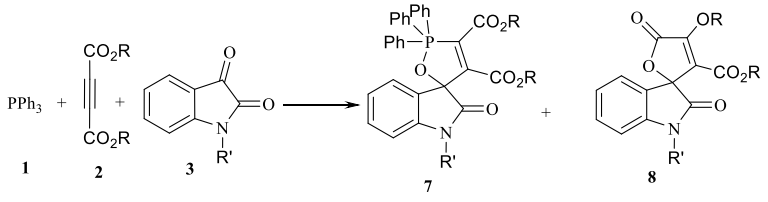
4	R	%Yield of 4
a	Me	94
b	Et	90
c	Bn	85

Scheme 1: Synthesis of compounds **4**



Scheme 2: Proposed mechanism for the synthesis of compounds **4**

The reaction of Ph_3P and dialkyl acetylenedicarboxylates **2** in the presence of *N*-alkylisatins **3** proceeds smoothly in CH_2Cl_2 at ambient temperature to produce **7** and **8** in about 3:1 ratios (Scheme **3**).



7, 8	R	R'	% Yield	7:8 ratio
a	Me	Me	75	3:1
b	Et	Me	75	3:1
c	Me	Bn	70	7:3
d	Et	Bn	75	3:1
e	Me	Et	70	7:3
f	Et	Et	70	7:3

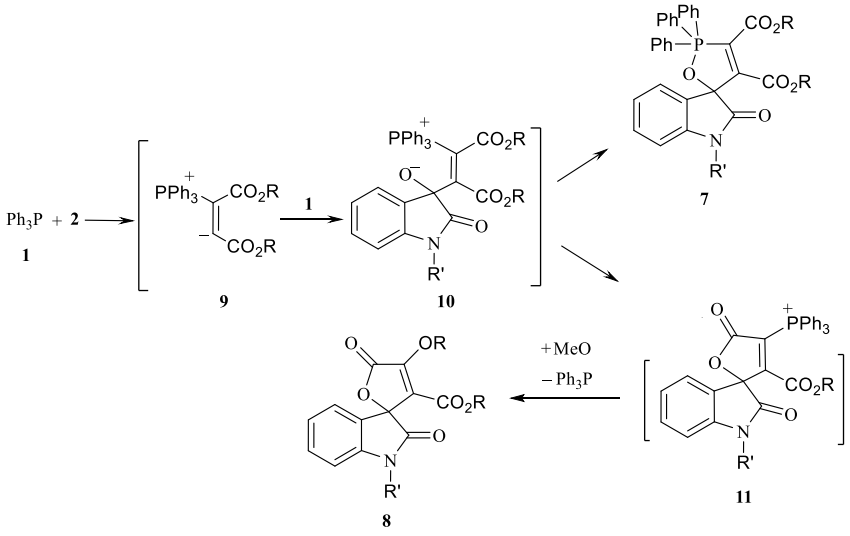
Scheme 3. Synthesis of spiro compounds **7** and **8**

The reactions were carried out by mixing the acetylenic ester **5** with **3** and then Ph_3P was added slowly. The reactions were complete within 24 hr. The structures of compounds**7** and **8**were apparent from their mass spectra, which displayed, in each case, the molecular ion peak at appropriate *m/z* values. The ^{1}H- and ^{13}C-NMR spectroscopic data, as well as IR spectra, are in agreement with the proposed structures.

The ^{1}H-NMR spectrum of **7a** exhibited three singlets readily recognized at as arising from the NMe (δ = 3.27 ppm) and two methoxy (δ = 3.69 and 3.98 ppm) protones. The carbonyl resonances in the ^{13}C-NMR spectrum of **7a** appear at 163.0 (d, $^2J_{CP}$ = 24.2), 168.4 (d, $^3J_{CP}$ = 21.2) and 169.7 ppm. The ^{31}P-NMR signal of **7a** was found at δ = -79.45 ppm. The mass spectrum of **7a** displayed the molecular ion peak at *m/z* = 565, which is consistent with the

1:1:1 adduct of Ph_3P, DMAD, and *N*-methylisatin.The ^{1}H-NMR spectrum of **8a** exhibited three singlet for *N*Me (δ = 3.26 ppm) and the methoxy (δ = 3.58 and 4.35 ppm) protons. The carbonyl groups resonances in the ^{13}C-NMR spectrum of **8a** appear at 160.5, 165.4 and 169.9 ppm. The mass spectrum of **8a** displayed the molecular ion peak at *m*/*z* = 303.

A tentative mechanism for this transformation is proposed in Scheme 4. It is conceivable that, the reaction involves the initial formation of a 1,3-dipolar intermediate **8** between Ph_3P and the acetylenic compound,[7] which reacts with the carbonyl group of *N*-alkylisatin to produce **9**. Cyclization of this zwitterionic intermediate leads to the spiro compound **6.** Another way, intermediate **10** is formed then leads to the spiro compound **7**.



Scheme 3. Proposed mechanism for theSynthesis of spiro compounds **7** and **8**

Conclsion

In summary, the reaction of ethyl propiolate with *N*-alkylisatins in the presence of Ph_3P led to ethyl oxaphospholes of potential synthetic interest. Under similar conditions, dialkyl acetylenedicarboxylates react with *N*-alkylisatins to produce oxaphosphole-3,4-dicarboxylate and furancarboxylate in nearly 3:1 ratio. The present procedure has the advantage that the reaction is performed under neutral conditions, and the starting material can be used without any activation or modification.

Experimental

M.p.: *Electrothermal-9100* apparatus; uncorrected. IR Spectra: *Shimadzu IR-460* spectrometer. ^{1}H-, ^{13}C-, and ^{31}P-NMR spectra: Bruker DRX-500 AVANCE instrument; in $CDCl_3$ at 500.1, 125.7, and 202.4 MHz, resp.; δ in ppm, *J* in Hz. EI-MS (70 eV): Finnigan-MAT-8430 mass spectrometer, in *m/z*. Elemental analyses (C, H, N) were performed with a Heraeus CHN-O-Rapid analyzer.

Ethyl propiolate, Ph_3P, and **5** were obtained from *Fluka* and were used without further purification. Alkylisatins were prepared according to the literature procedure.[11]

3.1. General procedure for preparation of compounds 2a-c:

To a stirred solution of ethyl propiolate (2 mmol) and *N*-alkylisatin (2 mmol) in 15 mL CH_2Cl_2 was added Ph_3P (2 mmol) at room temperature. The reaction mixture was then stirred for 24 h. The solvent was removed under reduced pressure, and the residue was separated by silica gel (Merck 230–400 mesh) column chromatography using hexane–ethyl acetate mixture as eluent.

Ethyl 1,2-dihydro-2-oxo-1-methyl-spiro-[3H-indol-2,2,2-triphenyl-2,5-dihydro-1,2-λ^5-oxaphosphole]-4-carboxylate **(4a):**

Yellow crystals, mp 210-212°C, 0.98 g, yield 94%. IR (KBr) (ν_{max}/cm^{-1}): 1726, 1682, 1459, 1110, 1031 and 1009.MS, *m/z* (%):521(M^+, 5), 476 (66), 278 (85), 243(64), 201 (62), 111 (34), 169 (100), 45 (100). Anal. Calcd for

$C_{32}H_{28}NO_4P$ (521.5): C, 73.69; H, 5.41; N, 2.69; found: C, 73.70; H, 5.40; N, 2.70%. ^{1}H-NMR: δ1.25 (3 H, t, $^3J_{HH}$ = 7.2 Hz, Me), 3.25 (3 H, s, NMe), 4.17 (2 H, q, $^3J_{HH}$ = 7.2 Hz, OCH_2), 6.89 (1 H, d, $^2J_{HP}$ = 22.7 Hz, CH), 7.09 (1 H, d, $^3J_{HH}$ = 7.2 Hz, CH), 7.32 (1 H, t, $^3J_{HH}$ = 7.3 Hz, CH), 7.42 (1 H, d, $^3J_{HH}$ = 7.3 Hz, CH), 7.48 (1 H, d, $^3J_{HH}$ = 7.2 Hz, CH), 7.52-7.78 (15 H, m, 15 CH). ^{13}C-NMR: δ 14.3 (Me), 28.1 (NMe), 61.7 (OCH_2), 91.2 (d, $^2J_{CP}$ = 49.1 Hz, C_{ipso}), 116.7 (CH), 120.3 (CH), 123.6 (CH), 128.1 (CH), 128.6 (d, $^3J_{CP}$ = 10.2 Hz, C), 129.2 (d, $^3J_{CP}$ = 21.1 Hz, 6 CH), 129.4 (3 CH), 131.9 (d, $^2J_{CP}$ = 31.9 Hz, CH), 135.1 (d, $^1J_{CP}$ = 230.1 Hz, 3 C), 149.3 (d, $^1J_{CP}$ = 192.3 Hz, CH), 150.4 (C), 157.3 (d, $^2J_{CP}$ = 19.3 Hz, C), 168.4 (d, $^3J_{CP}$ = 21.2 Hz, C=O), 169.7 (d, $^3J_{CP}$ = 17.4 Hz, C=O). ^{31}P-NMR: δ -50.35.

Ethyl 1,2-dihydro-2-oxo-1-ethyl-spiro-[3H-indol-2,2,2-triphenyl-2,5-dihydro-1,2-λ^5-oxaphosphole]-4-carboxylate (4b):

Yellow powder, mp 196-198°C, 0.96 g, yield 90%. IR (KBr) (ν_{max}/cm^{-1}):1727, 1680, 1450, 1100, 1029 and 1010. MS, *m/z* (%): 535(M^+, 15), 490 (74), 461(54), 278 (68), 257 (62), 175 (34), 74 (46), 45 (94).Anal. Calcd for $C_{33}H_{30}NO_4P$ (535.6): C, 74.01; H, 5.65; N, 2.62; found: C, 74.00; H, 5.60; N, 2.60%. ^{1}H-NMR: δ 1.24 (3 H, t, $^3J_{HH}$ = 7.2 Hz, Me), 1.37 (3 H, t, $^3J_{HH}$ = 7.2 Hz, Me), 4.13 (2 H, q, $^3J_{HH}$ = 7.2 Hz, OCH_2), 4.35 (2 H, m, CH_2), 6.75 (1 H, d, $^2J_{PH}$ = 25.4 Hz, CH), 7.34 (1 H, d, $^3J_{HH}$ = 7.2 Hz, CH), 7.42 (1 H, t, $^3J_{HH}$ = 7.2 Hz, CH), 7.50 (1 H, d, $^3J_{HH}$ = 7.3 Hz, CH), 7.73 (1 H, d, $^3J_{HH}$ = 7.2 Hz, CH), 7.45-7.84 (15H, m, 15 CH).^{13}C-NMR: δ 13.3 (Me), 14.0 (Me), 38.4 (CH_2), 62.1 (OCH_2), 93.2 (d, $^2J_{CP}$ = 35.4 Hz, C_{ipso}), 118.3 (CH), 120.4 (CH), 124.2 (CH), 127.4 (CH), 127.9 (d, $^3J_{CP}$ = 8.0 Hz, C), 128.4 (d, $^3J_{CP}$ = 21.1 Hz, 6 CH), 129.1 (3 CH), 132.0 (d, $^2J_{CP}$ = 31.9 Hz, 6 CH), 135.4 (d,$^1J_{CP}$ = 226.5 Hz, 3 C), 144.1 (d, $^1J_{CP}$ = 194.1 Hz, CH), 149.2 (C), 154.2 (d, $^2J_{CP}$ = 15.4 Hz, C), 166.5 (d, $^3J_{CP}$ = 21.2 Hz, C=O), 168.7 (d, $^3J_{CP}$ = 19.8 Hz, C=O). ^{31}P-NMR: δ -52.42.

Ethyl 1,2-dihydro-2-oxo-1-benzyl-spiro-[3H-indol-2,2,2-triphenyl-2,5-dihydro-1,2-λ^5-oxaphosphole]-4-carboxylate (4c):

Pale yellow crystals, mp 223-225°C, 1.01 g, yield 85%. IR (KBr) (ν_{max}/cm^{-1}):1730, 1685, 1462, 1210, 1054 and 1022. MS, *m/z*(%): 597(M^+, 10), 506 (70), 319 (64), 278 (64), 217 (62), 91 (96), 45 (100). Anal. Calcd for $C_{38}H_{32}NO_4P$ (597.65): C, 76.37; H, 5.40; N, 2.34; found: C, 76.40; H, 5.40; N, 2.35%. ^{1}H-NMR: δ 1.23 (3 H, t, $^3J_{HH}$ = 7.2 Hz, Me), 4.24 (2 H, q, $^3J_{HH}$ = 7.2 Hz, OCH_2), 4.82 (2 H, m, CH_2), 6.94 (1 H, d, $^2J_{PH}$ = 20.8 Hz, CH), 7.15 (1 H, d, $^3J_{HH}$ = 7.2 Hz, CH); 7.26-7.29 (3 H, m, 3 CH), 7.34 (1 H, d, $^3J_{HH}$ = 7.2 Hz, 2 CH), 7.37 (1 H, t, $^3J_{HH}$ = 7.2 Hz, CH), 7.44 (1 H, d, $^3J_{HH}$ = 7.3 Hz, CH), 7.45-7.80 (16 H, m, 16 CH). ^{13}C-NMR: δ 14.1 (Me), 49.2 (CH_2), 61.4 (OCH_2), 91.7 (d, $^2J_{CP}$ = 30.2 Hz, C_{ipso}), 117.4 (CH), 120.0 (CH), 122.4 (2 CH), 123.9 (CH), 125.8 (CH), 127.9 (2 CH), 128.2 (CH), 128.6 (d, $^3J_{CP}$ = 9.4 Hz, C), 129.1 (*d*, $^3J_{CP}$ = 18.5 Hz, 6 CH), 129.9 (3 CH), 132.4 (d, $^2J_{CP}$ = 28.4 Hz, 6 CH), 135.6 (C), 137.4 (d,$^1J_{CP}$ = 230.2 Hz, 3 C), 145.4 (d, $^1J_{CP}$ = 201.3 Hz, CH), 150.4 (C), 157.1 (d, $^2J_{CP}$ = 16.2 Hz, C), 169.5 (d, $^3J_{CP}$ = 23.5 Hz, C=O), 170.1 (d, $^3J_{CP}$ = 20.1 Hz, C=O). ^{31}P-NMR: δ -59.58.

General procedure for preparation of compounds 7 and 8:

To a stirred solution of dialkyl acetylenedicarboxylate (2 mmol) and *N*-alkylisatin (2 mmol) in 15 mL CH_2Cl_2 was added Ph_3P (2 mmol) at room temperature. The reaction mixture was then stirred for 24 h. The solvent was removed under reduced pressure, and the residue was separated by silica gel (Merck 230–400 mesh) column chromatography using hexane–ethyl acetate mixture as eluent.

Dimethyl 1,2-dihydro-2-oxo-1-methyl-spiro-[3H-indol-2,2,2-triphenyl-2,5-dihydro-1,2-λ^5-oxaphosphole]-3,4-dicarboxylate (7a):

Pale yellow crystals, mp 195-197°C, 0.85 g, yield 75%. IR (KBr) (ν_{max}/cm^{-1}):1752, 1732, 1672, 1478, 1135, 1097 and 1019. MS, *m/z* (%): 565 (M^+, 15), 533 (85), 502 (72), 403 (54), 278 (96), 161 (38), 146 (88), 31 (100). Anal. Calcd for $C_{33}H_{28}NO_6P$ (565.56): C, 70.08; H, 4.99; N, 2.48; found: C, 70.10; H, 5.00; N, 2.45%. ^{1}H-NMR: δ3.27 (3 H, s, NMe), 3.69 (3 H, s, OMe), 3.98 (3 H, s, OMe), 6.91 (1 H, d, $^3J_{HH}$ = 7.2 Hz, CH), 7.08 (1 H, t, $^3J_{HH}$ = 7.3 Hz, CH), 7.11 (1 H, d, $^3J_{HH}$ = 7.3 Hz, CH), 7.43 (1 H, d, $^3J_{HH}$ = 7.2 Hz, CH), 7.47-7.84 (15 H, m, 15 CH). ^{13}C-NMR: δ 26.9 (NMe), 51.7 (OMe), 52.3 (OMe), 90.1 (d, $^2J_{CP}$ = 51.2 Hz, C_{ipso}), 116.7 (CH), 120.3 (CH), 123.6 (CH), 128.1 (CH), 128.6 (d, $^3J_{CP}$ = 22.4 Hz, C), 129.2 (d, $^3J_{CP}$ = 21.1 Hz, 6 CH), 129.4 (3 CH), 131.9 (d, $^2J_{CP}$ = 31.9 Hz, 6 CH), 135.1 (d, $^1J_{CP}$ = 230.1 Hz, 3 C), 149.3 (C),

150.4 (d, $^1J_{CP}$ = 192.3 Hz, C), 163.0 (d, $^2J_{CP}$ = 24.2 Hz, C=O), 165.1 (C), 168.4 (d, $^3J_{CP}$ = 21.2 Hz, C=O), 169.7 (C=O). ^{31}P-NMR: δ -79.45.

Diethyl 1,2-dihydro-2-oxo-1-methyl-spiro-[3H-indol-2,2,2-triphenyl-2,5-dihydro-1,2-λ^5-oxaphosphole]-3,4-dicarboxylate (6b):

Yellow powder, mp 190-192°C, 0.89 g, yield 75%. IR (KBr) (ν_{max}/cm^{-1}):1727, 1720, 1643, 1478, 1166, 1086 and 1004. MS, *m/z* (%): 593 (M^+, 10), 548 (82), 503 (76), 315 (54), 278 (96), 161 (46), 146 (88), 45 (100). Anal. Calcd for $C_{35}H_{32}NO_6P$ (593.6): C, 70.82; H, 5.43; N, 2.36; found: C, 70.80; H, 5.40; N, 2.35%. ^{1}H-NMR: δ 1.23 (3 H, t, $^3J_{HH}$ = 7.2 Hz, Me), 1.48 (3 H, t, $^3J_{HH}$ = 7.2 Hz, Me), 3.25 (3 H, s, NMe), 3.84 (2 H, q, $^3J_{HH}$ = 7.2 Hz, OCH_2), 4.08 (2 H, q, $^3J_{HH}$ = 7.2 Hz, OCH_2), 6.95 (1 H, t, $^3J_{HH}$ = 7.2 Hz, CH), 7.08 (1 H, d, $^3J_{HH}$ = 7.2 Hz, CH), 7.33 (1 H, d, $^3J_{HH}$ = 7.2 Hz, CH), 7.35-7.72 (16 H, m, 16 CH). ^{13}C-NMR: δ 13.0 (Me), 13.2 (Me), 26.4 (NMe),61.4 (OCH_2), 62.4 (OCH_2), 92.0 (d, $^2J_{CP}$ = 49.5 Hz, C_{ipso}), 116.2 (CH), 119.5 (CH), 122.9 (CH), 127.9 (CH), 128.4 (d, $^3J_{CP}$ = 23.9 Hz, C), 130.1 (d, $^3J_{CP}$ = 20.1 Hz, 6 CH), 130.5 (3 CH), 132.0 (d, $^2J_{CP}$ = 32.9 Hz, 6 CH), 134.9 (d, $^1J_{CP}$ = 230.1 Hz, 3 C), 149.2 (C), 150.4 (d, $^1J_{CP}$ = 195.3 Hz, C), 162.9 (d, $^2J_{CP}$ = 23.6 Hz, C=O), 166.1 (C), 168.2 (d, $^3J_{CP}$ = 23.2 Hz, C=O), 169.2 (C=O). ^{31}P-NMR: δ -75.45.

Dimethyl 1,2-dihydro-2-oxo-1-benzyl-spiro-[3H-indol-2,2,2-triphenyl-2,5-dihydro-1,2-λ^5-oxaphosphole]-3,4-dicarboxylate (7c):

Pale yellow crystals, mp 178-180°C, 0.89 g, yield 70%. IR (KBr) (ν_{max}/cm^{-1}): 1725, 1720, 1642, 1472, 1165, 1090 and 1012. MS, *m/z* (%): 641 (M^+, 10), 610 (84), 579 (74), 368 (54), 278 (96), 237 (46), 146 (88), 91 (96), 31 (100). Anal. Calcd for $C_{39}H_{32}NO_6P$ (641.66): C, 73.00; H, 5.03; N, 2.18; found: C, 73.00; H, 5.05; N, 2.20%. ^{1}H-NMR: δ 3.75 (3 H, s, OMe), 4.11 (3 H, s, OMe), 4.80 (1 H, d, $^2J_{HH}$ = 15.6 Hz, CH), 5.01 (1 H, d, $^2J_{HH}$ = 15.6 Hz, CH), 7.15 (1 H, d, $^3J_{HH}$ = 7.4 Hz, CH), 7.30 (1 H, t, $^3J_{HH}$ = 7.5 Hz, CH), 7.36 (1 H, d, $^3J_{HH}$ = 7.5 Hz, CH), 7.38 (2 H, t, $^3J_{HH}$ = 7.5 Hz, 2 CH), 7.45 (2 H, t, $^3J_{HH}$ = 7.7 Hz, 2 CH), 7.54 (2 H, d, $^3J_{HH}$ = 7.5 Hz, 2 CH),7.62-7.84 (15 H, m, 15 CH). ^{13}C-NMR: δ 46.2 (NCH_2), 51.4 (OMe), 52.2 (OMe), 89.3 (d, $^2J_{CP}$ = 47.8 Hz, C_{ipso}), 116.5 (CH), 119.1 (CH), 123.4 (2 CH), 123.6 (CH), 125.9 (CH); 127.7 (2 CH), 128.3 (CH), 128.5 (d, $^3J_{CP}$ = 24.2 Hz, C), 128.9 (d, $^3J_{CP}$ = 20.1 Hz, 6 CH), 130.2 (3 CH), 132.4 (d, $^2J_{CP}$ = 34.2 Hz, 6 CH), 135.9 (C), 136.2 (d, $^1J_{CP}$ = 234.5 Hz, 3 C), 148.4 (C), 151.2 (d, $^1J_{CP}$ = 190.1 Hz, C), 162.4 (d, $^2J_{CP}$ = 26.5 Hz, C=O), 164.8 (C), 167.5 (d, $^3J_{CP}$ = 20.3 Hz, C=O), 169.5 (C=O). ^{31}P-NMR: δ -44.2.

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