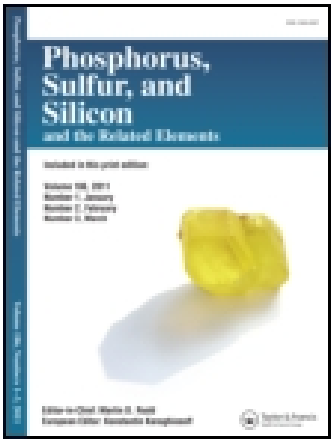

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Efficient, Simple Synthesis of Stable Phosphorus Ylides Derived from 4-Aryl Urazoles

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EFFICIENT, SIMPLE SYNTHESIS OF STABLE PHOSPHORUS YLIDES DERIVED FROM 4-ARYL URAZOLES

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Crystalline phosphorus ylides are obtained in nearly quantitative yields from the 1:1:1 addition reaction between triphenylphosphine, dialkyl acetylenedicarboxylates, and 4-aryl urazoles.

Keywords Acetylenic esters; 4-aryl urazoles; phosphoranes; triphenylphosphine

INTRODUCTION

Among a large variety of nitrogen-containing heterocyclic compounds, those containing a urazole moiety are of interest because they constitute an important class of natural and non-natural products, many of which exhibit useful biological activities.[1] Some urazole derivatives have been shown to possess anticonvulsant,[2] fungicidal,[3] antineoplastic,[4] hypolipidemic,[5] antiinflammatory,[6] and antidepressant[7] activities as well as catalytic activity in radical polymerization.[8]

Phosphorus ylides are reactive intermediates that take part in many valuable reactions in organic synthesis.[9–22] Several methods have been developed for the preparation of phosphorus ylides. These ylides are usually prepared by treatment of a phosphonium salt with a base, and phosphonium salts are usually prepared from the phosphine and an alkyl halide.[9–11] Phosphonium salts are also prepared by Michael addition of phosphorus nucleophiles to activated olefins among other methods.[9]

Recently, there are many reports in literature on the synthesis of phosphorus ylides by three-component reaction of electron-deficient acetylenic compounds and triphenylphosphine in the presence of various organic N-H, O-H, C-H, and S-H acids.[23]

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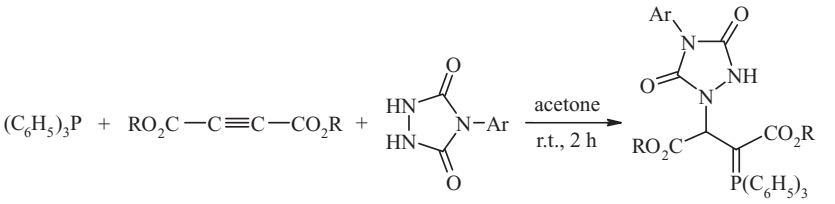
RESULTS AND DISCUSSION

As part of our current studies on the development of efficient and facile methods for the preparation of organic compounds,[24] we report in this article an efficient synthetic route to phosphorus ylides derived from 4-aryl urazoles. Thus, a mixture of triphenylphosphine, a dialkyl acetylenedicarboxylate **1**, and a 4-aryl urazole **2** underwent a one-pot, three-component reaction to afford the corresponding phosphorus ylides **3** in nearly quantitative yields (Scheme 1).



1 **2** **3**

1	R
a	CH_3
b	C_2H_5

2	Ar
a	C_6H_5
b	3,4-$Cl_2C_6H_3$
c	3-Cl-4-$CH_3C_6H_3$

3	R	Ar	% Yield
a	CH_3	C_6H_5	99
b	CH_2CH_3	C_6H_5	98
c	CH_3	3,4-$Cl_2C_6H_3$	99
d	CH_2CH_3	3,4-$Cl_2C_6H_3$	96
e	CH_3	3-Cl-4-$CH_3C_6H_3$	98
f	CH_2CH_3	3-Cl-4-$CH_3C_6H_3$	94

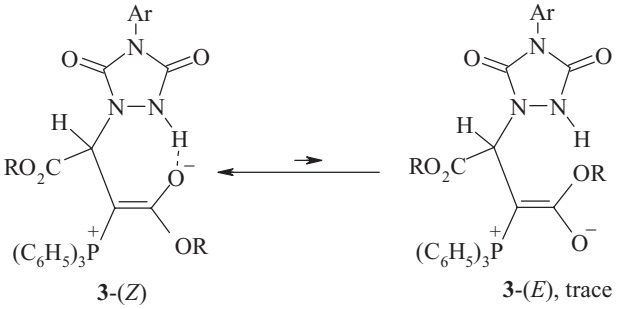
Scheme 1

The reaction of triphenylphosphine with dialkyl acetylenedicarboxylates in the presence of 4-aryl urazoles proceeded spontaneously at room temperature in acetone, and was complete within 2 h. ^1H and ^{13}C NMR spectra of the crude product clearly indicated the formation of the phosphoranes **3**. Any product other than **3** could not be detected by NMR spectroscopy.

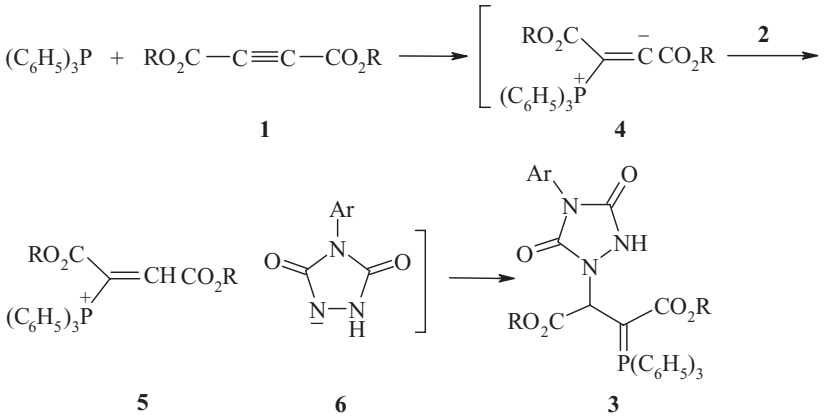
The structures of the isolated products **3a–f** were deduced on the basis of IR, ^1H, ^{13}C, and ^{31}P NMR spectroscopy, mass spectrometry, and elemental analysis. The nature of these compounds as 1:1:1 adducts was apparent from their mass spectra, which displayed molecular ion [M^+] peaks at $m/z = 581$, 609, 650, 678, 630, and 658, respectively. Initial fragmentations involve loss from or complete loss of the side chains and scission of the heterocyclic ring system.

We have reported[25,26] the synthesis of several functionalized phosphoranes via one-pot, three-component reactions between triphenylphosphine, dialkyl acetylenedicarboxylates, and some NH-acids. Dynamic NMR effects were observed in the ^1H NMR spectra of the reported ylides and were attributed to restricted rotation around the carbon–carbon partial double bond resulting from conjugation of the ylide moiety with the adjacent carbonyl

group. However, the ^{1}H, ^{13}C NMR spectra of the ylides synthesized from 4-aryl urazoles **3a–f** are consistent with the presence of only one isomer, and the minor isomer is present in less than 3% (on the basis of ^{31}P NMR). In the *Z* geometrical isomer of these ylides, there is strong hydrogen bonding between O^- of the ylide moiety and the adjacent NH group, which could stabilize the *Z* isomer.



On the basis of the well established chemistry of trivalent phosphorus nucleophiles,[9–22] it is reasonable to assume that phosphorus ylide **3** results from the initial addition of triphenylphosphine to the acetylenic ester and subsequent protonation of the zwitterionic intermediate **4** by the NH-acid **2**. Then the positively charged ion **5** is attacked by the conjugate base of the NH-acid **6** to form phosphorane **3** (Scheme 2).



Scheme 2

Functionalized phosphorus ylides **3a–f** may be considered as potentially useful synthetic intermediates.[9,10] Excellent yields of the products and relatively short reaction times are the main advantages of this method. The reactions were performed under neutral and mild conditions, and the starting materials and reagents can be mixed without any activation or modification. The procedure described here may be an acceptable method for the preparation of phosphoranes with variable functionalities. The one-pot nature of the present procedure makes it an interesting alternative to multistep approaches.[9–18] In view of widespread biological activities of urazole derivatives, the urazole-containing phosphorus

ylides prepared in the present study may find useful applications in synthetic organic and bioorganic chemistry.

EXPERIMENTAL

Dialkyl acetylenedicarboxylates and triphenylphosphine were obtained from Merck (Germany) and were used without further purification. 4-Aryl urazoles were prepared according to the procedure in the literature.[27] Melting points were measured on an Electrothermal 9100 apparatus. Elemental analyses for C, H, and N were performed using a Heraeus CHN-O-Rapid analyzer. Mass spectra were recorded on a Finnigan-MATT 8430 mass spectrometer operating at an ionization potential of 20 eV. ^{1}H, ^{13}C, and ^{31}P NMR spectra were measured ($CDCl_3$ solution) with a Bruker DRX-500 AVANCE spectrometer at 500.1, 125.8, and 202.5 MHz, respectively. IR spectra were recorded on a Shimadzu IR-460 spectrometer.

Preparation of Dimethyl 2-(4-Phenyl-3,5-dioxo-1,2,4-triazolin-1-yl)-3-(triphenylphosphoranylidene)butanedioate (3a)

To a magnetically stirred solution of triphenylphosphine (0.262 g, 1 mmol) and 4-phenyl urazole **2a** (0.177 g, 1 mmol) in acetone (6 mL), a solution of dimethyl acetylenedicarboxylate **1a** (0.148 g, 1 mmol) in acetone (2 mL) at −5°C was added dropwise over 10 min. The reaction mixture was then allowed to warm to room temperature and stirred for 2 h. The white precipitate was filtered and washed with cold acetone. The product **3a** was recrystallized from EtOAc/*n*-hexane (2:1) as colorless crystals, mp 149–151°C, yield 0.58 g, 99%. IR (KBr) (ν_{max}/cm^{-1}): 3210 (NH), 1760 (shoulder), 1732 (shoulder), 1691 (C=O), 1647, 1610, 1489, 1423, 1301, 1230, 1130, 1100, 761, 712, 689, 536. MS, *m/z* (%): 581 (M^+, 25), 566 (20), 447 (10), 419 (75), 262 (100), 183 (46), 177 (45), 144 (29), 94 (20), 77 (10). Anal. Calcd. for $C_{32}H_{28}N_3O_6P$ (581.56): C, 66.09; H, 4.85; N, 7.23. Found: C, 66.4; H, 4.8; N, 7.0%. ^{1}H NMR: δ 3.15 and 3.75 (6 H, 2 s, 2 OCH_3), 4.74 (1 H, d, $^{3}J_{PH}$ 16.6 Hz, P-C-CH), 7.34 (1 H, t, *J* 7.1 Hz, CH), 7.42 (2 H, t, *J* 8.0 Hz, 2 CH), 7.51 (2 H, d, *J* 7.8 Hz, 2 CH), 7.57–7.75 (9 H, m, 6 CH_{meta} and 3 CH_{para}), 7.81 (6 H, dd, *J* 8.1 Hz and *J* 12.3 Hz, 6 CH_{ortho}), 9.93 (1 H, br. s, NH). ^{13}C NMR: δ 40.16 (d, $^{1}J_{PC}$ 128.2 Hz, P=C), 48.08 and 51.26 (2 OCH_3), 57.39 (d, $^{2}J_{PC}$ 16.8 Hz, P-C-*C*H), 124.53 (CH), 124.95 (d, $^{1}J_{PC}$ 92.4 Hz, C_{ipso}), 126.21 and 127.53 (2 CH), 128.15 (d, $^{3}J_{PC}$ 12.4 Hz, CH_{meta}), 131.66 (d, $^{4}J_{PC}$ 2.9 Hz, CH_{para}), 131.78 (C), 132.62 (d, $^{2}J_{PC}$ 9.9 Hz, CH_{ortho}), 150.77 and 151.03 (2 C=O, urea), 169.73 and 170.65 (2 d, $^{2}J_{PC}$ 10.9 Hz and $^{3}J_{PC}$ 12.5 Hz, 2 C=O ester). ^{31}P NMR: δ 23.81 (Ph_3P^+-C).

Diethyl 2-(4-Phenyl-3,5-dioxo-1,2,4-triazolin-1-yl)-3-(triphenylphosphoranylidene)butanedioate (3b)

Colorless crystals, mp 132–134°C, yield 0.59 g, 98%. IR (KBr) (ν_{max}/cm^{-1}): 3195 (NH), 1761, 1732, 1696 (C=O), 1631, 1595, 1489, 1423, 1367, 1298, 1227, 1130, 1101, 1022, 771, 748, 710, 688, 548, 511. MS, *m/z* (%): 609 (M^+, 17), 490 (25), 462 (31), 262 (89), 183 (100), 177 (10), 77 (28). Anal. Calcd. for $C_{34}H_{32}N_3O_6P$ (609.62): C, 66.99; H, 5.29; N, 6.89. Found: C, 66.6; H, 5.2; N, 6.7%. ^{1}H NMR: δ 0.50 and 1.29 (6 H, 2 t, *J* 7.1 Hz, 2 OCH_2CH_3), 3.75 and 4.23 (4 H, 2 m, 2 OCH_2CH_3), 4.80 (1 H, d, $^{3}J_{PH}$ 16.5 Hz, P-C-CH), 7.28 (1 H, t, *J* 7.4 Hz, CH), 7.39 (2 H, t, *J* 7.7 Hz, 2 CH), 7.48 (2 H, d, *J* 7.9 Hz,

2 CH), 7.53 (6 H, dt, *J* 2.5 Hz and *J* 7.5 Hz, 6 CH_{meta}), 7.62 (3 H, t, *J* 7.1 Hz, 3 CH_{para}), 7.72 (6 H, dd, *J* 7.7 Hz and *J* 12.5 Hz, 6 CH_{ortho}), 10.03 (1 H, s, NH). ^{13}C NMR: δ 13.86 and 14.21 (2 OCH_2CH_3), 42.39 (d, $^1J_{PC}$ 128.4 Hz, P=C), 57.89 (d, $^2J_{PC}$ 16.5 Hz, P-C-*C*H), 58.74 and 61.99 (2 OCH_2CH_3), 125.62 (d, $^1J_{PC}$ 92.7 Hz, C_{ipso}), 125.71, 127.53, and 128.90 (3 CH), 129.06 (d, $^3J_{PC}$ 12.7 Hz, CH_{meta}), 132.14 (C), 132.65 (CH_{para}), 133.65 (d, $^2J_{PC}$ 9.9 Hz, CH_{ortho}), 151.28 and 151.42 (2 C=O, urea), 170.37 and 172.11 (2 d, $^2J_{PC}$ 10.2 Hz and $^3J_{PC}$ 12.3 Hz, 2 C=O ester). ^{31}P NMR: δ 23.00 (Ph_3P^+-C).

Dimethyl 2-[4-(3,4-Dichlorophenyl)-3,5-dioxo-1,2,4-triazolin-1-yl]-3-(triphenylphosphoranylidene)butanedioate (3c)

Colorless crystals, mp 145–147°C, yield 0.64 g, 99%. IR (KBr) (ν_{max}/cm^{-1}): 3150 (NH), 1762, 1736, 1697 (C=O), 1607, 1468, 1421, 1320, 1294, 1225, 1144, 1100, 747, 711, 688, 512. MS, *m/z* (%): 650 (M^+, 8), 635 (7), 404 (14), 376 (61), 262 (97), 183 (100), 174 (24), 161 (16), 78 (25). Anal. Calcd. for $C_{32}H_{26}Cl_2N_3O_6P$ (650.45): C, 59.09; H, 4.03; N, 6.46. Found: C, 59.3; H, 3.8; N, 6.4%. ^{1}H NMR: δ 3.20 and 3.78 (6 H, 2 s, 2 OCH_3), 4.79 (1 H, d, $^3J_{PH}$ 16.2 Hz, P-C-CH), 7.46 (2 H, br., 2 CH), 7.55 (6 H, dt, *J* 2.3 Hz and *J* 7.4 Hz, 6 CH_{meta}), 7.64 (3 H, t, *J* 7.1 Hz, 3 CH_{para}), 7.70 (6 H, dd, *J* 7.8 Hz and *J* 12.5 Hz, 6 CH_{ortho}), 7.72 (1 H, br., CH), 10.08 (1 H, s, NH). ^{13}C NMR: δ 42.59 (d, $^1J_{PC}$ 128.3 Hz, P=C), 50.10 and 53.20 (2 OCH_3), 57.78 (d, $^2J_{PC}$ 17.0 Hz, P-C-*C*H), 124.43 (CH), 125.35 (d, $^1J_{PC}$ 92.6 Hz, C_{ipso}), 127.04 (CH), 129.19 (d, $^3J_{PC}$ 11.7 Hz, CH_{meta}), 130.41 (CH), 131.28, 131.71, and 132.67 (3 C), 132.79 (CH_{para}), 133.57 (d, $^2J_{PC}$ 9.8 Hz, CH_{ortho}), 150.09 and 150.43 (2 C=O, urea), 170.78 and 172.58 (2 d, $^2J_{PC}$ 10.3 Hz and $^3J_{PC}$ 12.3 Hz, 2 C=O ester). ^{31}P NMR: δ 23.38 (Ph_3P^+-C).

Diethyl 2-[4-(3,4-Dichlorophenyl)-3,5-dioxo-1,2,4-triazolin-1-yl]-3-(triphenylphosphoranylidene)butanedioate (3d)

Colorless crystals, mp 127–129°C, yield 0.65 g, 96%. IR (KBr) (ν_{max}/cm^{-1}): 3190 (NH), 1761, 1732 (shoulder), 1700 (C=O), 1628, 1594, 1469, 1426, 1368, 1305, 1232, 1129, 1100, 1021, 711, 689, 549, 511. MS, *m/z* (%): 678 (M^+, 5), 649 (11), 432 (15), 404 (43), 245 (15), 262 (100), 183 (84), 174 (14), 161 (18), 77 (31). Anal. Calcd. for $C_{34}H_{30}Cl_2N_3O_6P$ (678.51): C, 60.19; H, 4.46; N, 6.19. Found: C, 59.9; H, 4.5; N, 6.4%. ^{1}H NMR: δ 0.48 and 1.26 (6 H, 2 t, *J* 7.1 Hz, 2 OCH_2CH_3), 3.72 and 4.20 (4 H, 2 m, 2 OCH_2CH_3), 4.73 (1 H, d, $^3J_{PH}$ 16.5 Hz, P-C-CH), 7.43 (2 H, br., 2 CH), 7.51 (6 H, dt, *J* 2.4 Hz and *J* 7.4 Hz, 6 CH_{meta}), 7.61 (3 H, t, *J* 7.0 Hz, 3 CH_{para}), 7.68 (6 H, dd, *J* 7.9 Hz and *J* 12.6 Hz, 6 CH_{ortho}), 7.69 (1 H, br., CH), 10.19 (1 H, s, NH).^{13}C NMR: δ 13.80 and 14.15 (2 OCH_2CH_3), 41.43 (d, $^1J_{PC}$ 128.4 Hz, P=C), 57.94 (d, $^2J_{PC}$ 16.9 Hz, P-C-*C*H), 58.84 and 62.08 (2 OCH_2CH_3), 124.40 (CH), 125.48 (d, $^1J_{PC}$ 92.6 Hz, C_{ipso}), 126.99 (CH), 129.05 (d, $^3J_{PC}$ 11.7 Hz, CH_{meta}), 130.34 (CH), 131.21, 131.70, and 132.62 (3 C), 132.68 (CH_{para}), 133.57 (d, $^2J_{PC}$ 10.1 Hz, CH_{ortho}), 150.46 and 150.51 (2 C=O, urea), 170.08 and 172.20 (2 d, $^2J_{PC}$ 10.3 Hz and $^3J_{PC}$ 12.4 Hz, 2 C=O ester). ^{31}P NMR: δ 23.02 (Ph_3P^+-C).

Dimethyl 2-[4-(3-Chloro-4-methylphenyl)-3,5-dioxo-1,2,4-triazolin-1-yl]-3-(triphenylphosphoranylidene)butanedioate (3e)

Colorless crystals, mp 162–164°C, yield 0.62 g, 98%. IR (KBr) (ν_{max}/cm^{-1}): 3140 (NH), 1760, 1738, 1694 (C=O), 1609, 1489, 1421, 1322, 1293, 1224, 1145, 1100, 711,

689, 527. MS, *m/z* (%): 630 (M^+, 21), 404 (16), 376 (29), 262 (100), 225 (54), 183 (79), 144 (19), 78 (10). Anal. Calcd. for $C_{33}H_{29}ClN_3O_6P$ (630.04): C, 62.91; H, 4.64; N, 6.67. Found: C, 62.6; H, 4.3; N, 6.6%. ^{1}H NMR: δ 2.33 (3 H, s, CH_3), 3.17 and 3.76 (6 H, 2 s, 2 OCH_3), 4.76 (1 H, d, $^3J_{PH}$ 16.2 Hz, P-C-CH), 7.23 (1 H, d, *J* 8.2 Hz, CH), 7.28 (1 H, dd, *J* 8.2 Hz and *J* 1.2 Hz, CH), 7.48 (1 H, d, *J* 1.2 Hz, CH), 7.52 (6 H, dt, *J* 1.7 Hz and *J* 7.3 Hz, 6 CH_{meta}), 7.61 (3 H, t, *J* 7.4 Hz, 3 CH_{para}), 7.67 (6 H, dd, *J* 7.6 Hz and *J* 12.4 Hz, 6 CH_{ortho}), 9.84 (1 H, s, NH). ^{13}C NMR: δ 19.67 (CH_3), 42.55 (d, $^1J_{PC}$ 128.4 Hz, P=C), 49.96 and 53.09 (2 OCH_3), 57.63 (d, $^2J_{PC}$ 17.0 Hz, P-C-*C*H), 123.84 (CH), 125.42 (d, $^1J_{PC}$ 92.4 Hz, C_{ipso}), 126.17 (CH), 129.10 (d, $^3J_{PC}$ 12.5 Hz, CH_{meta}), 130.63 (C), 130.90 (CH), 132.67 (CH_{para}), 133.54 (d, $^2J_{PC}$ 9.9 Hz, CH_{ortho}), 134.32 and 135.57 (2 C), 150.46 and 150.91 (2 C=O, urea), 170.89 and 172.41 (2 d, $^2J_{PC}$ 10.3 Hz and $^3J_{PC}$ 12.6 Hz, 2 C=O ester). ^{31}P NMR: δ 22.96 (Ph_3P^+-C).

Diethyl 2-[4-(3-Chloro-4-methylphenyl)-3,5-dioxo-1,2,4-triazolin-1-yl]-3-(triphenylphosphoranylidene)butanedioate (3f)

Colorless crystals, mp 125–127°C, yield 0.62 g, 94%. IR (KBr) (υ_{max}/cm^{-1}): 3210 (NH), 1759, 1729, 1697 (C=O), 1632, 1489, 1423, 1366, 1299, 1228, 1134, 1101, 690. MS, *m/z* (%): 658 (M^+, 11), 629 (8), 432 (20), 404 (52), 262 (100), 183 (84), 174 (12), 141 (36), 78 (15). Anal. Calcd. for $C_{35}H_{33}ClN_3O_6P$ (658.09): C, 63.88; H, 5.05; N, 6.39. Found: C, 63.4; H, 4.9; N, 6.5%. ^{1}H NMR: δ 0.41 and 1.21 (6 H, 2 t, *J* 7.0 Hz, 2 OCH_2CH_3), 2.23 (3 H, s, CH_3), 3.67 and 4.18 (4 H, 2 m, 2 OCH_2CH_3), 4.73 (1 H, d, $^3J_{PH}$ 16.5 Hz, P-C-CH), 7.15 (1 H, d, *J* 8.2 Hz, CH), 7.25 (1 H, dd, *J* 8.2 Hz and *J* 1.2 Hz, CH), 7.40–7.48 (7 H, m, CH and 6 CH_{meta}), 7.53 (3 H, t, *J* 7.3 Hz, 3 CH_{para}), 7.66 (6 H, dd, *J* 7.6 Hz and *J* 12.4 Hz, 6 CH_{ortho}), 10.15 (1 H, s, NH).^{13}C NMR: δ 13.77 and 14.19 (2 OCH_2CH_3), 19.60 (CH_3), 41.89 (d, $^1J_{PC}$ 127.8 Hz, P=C), 58.12 (d, $^2J_{PC}$ 17.0 Hz, P-C-*C*H), 58.72 and 61.94 (2 OCH_2CH_3), 123.80 (CH), 125.50 (d, $^1J_{PC}$ 92.1 Hz, C_{ipso}), 126.05 (CH), 129.00 (d, $^3J_{PC}$ 12.6 Hz, CH_{meta}), 130.77 (C), 130.84 (CH), 132.64 (CH_{para}), 133.60 (d, $^2J_{PC}$ 9.8 Hz, CH_{ortho}), 134.16 and 135.36 (2 C), 150.10 and 151.18 (2 C=O, urea), 170.24 and 171.95 (2 d, $^2J_{PC}$ 10.2 Hz and $^3J_{PC}$ 12.4 Hz, 2 C=O ester). ^{31}P NMR: δ 23.08 (Ph_3P^+-C).

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