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Green synthesis of functionalized oxaphosphole-1,2,4-triazoles: Investigation of biological Activity

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Abstract: The reaction of activated acetylenic compounds with triphenylphosphine (Ph_3P) in the presence of isatins or its derivatives and hydrazonoyl chlorides led to oxaphosphole-1,2,4-triazole derivatives in good yields. Also, antioxidant property of some prepared compounds was investigated using diphenyl-picrylhydrazine (DPPH) radical trapping experiment.

Keywords: Acid chlorides, Ammonium thiocyanate, *N*-formylmorpholine, 3-Hydroxy-2-butanone, Esterification.

Introduction

Among the heterocyclic compounds, pyridine and their analogues are the most interesting structural units due to their broad spectrum of applications [1] in natural products [2] pharmaceutical [3], agrochemical [4] and material science [5, 6]. The successful 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 [1-10]. Organophosphorus compounds are synthetic targets of interest, not least because of their value for a variety of industrial, biological, and chemical synthetic uses [11-16]. The physical properties and chemical reactivity of phosphate esters interlinks many areas in chemistry and biology. Introduction of a phosphate monoester into a molecule such as a drug candidate enhances the water solubility, hence altering its bioavailability [17-19]. As a result, a large number of methods have appeared describing novel syntheses of organophosphorus compounds.

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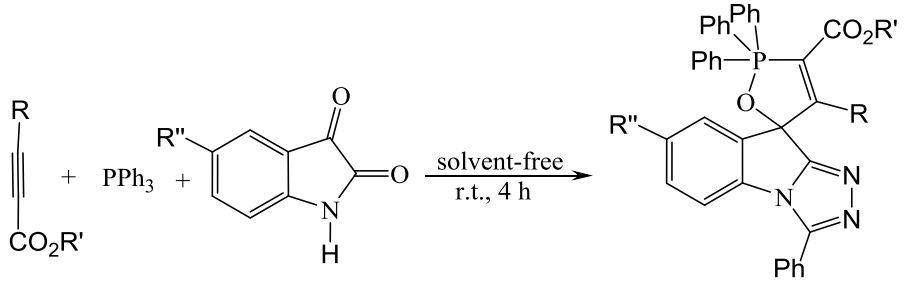
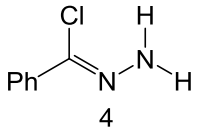
Spiro compounds having cyclic structures fused at a central carbon are of interest due to their interesting conformational features and their structural implications on biological systems. The asymmetric characteristic of the molecule due to the chiral spiro carbon is one of the important criteria of the biological activities. The presence of the sterically constrained spiro structure in various natural products also adds to the interest in the investigations of spiro compounds. Another topic in this research work is evaluation of antioxidant activity in some of synthesized spiro derivatives. Usually the compounds that because of reductive property and chemical structure of them could be reduced or deleted negative effect of free radicals have antioxidant property. Much disease such as cardiovascular, inflammatory bowel syndrome, cancer, ageing, and Alzheimer could be prevent or decrease with compounds with antioxidant properties. In recent times, biologists, medicinal and food chemist analysis for protecting of persons against these diseases discovered new and economical synthetic antioxidant compounds. The reaction of Ph_3P **2** with activated acetylenic compounds **1** in the presence of isatins **3** and

hydrazonoyl chloride **4** led to oxaphosphole-4-carboxylate **5** in excellent yields (Scheme **1**).



1 **2** **3** **5**



4

5	R	R'	R"	%Yield of **5**
a	CO_2Me	Me	Me	95
b	CO_2Me	Me	Et	87
c	CO_2Me	Me	OMe	86
d	H	Me	Me	89
e	H	Et	Et	90
f	H	Et	OMe	93

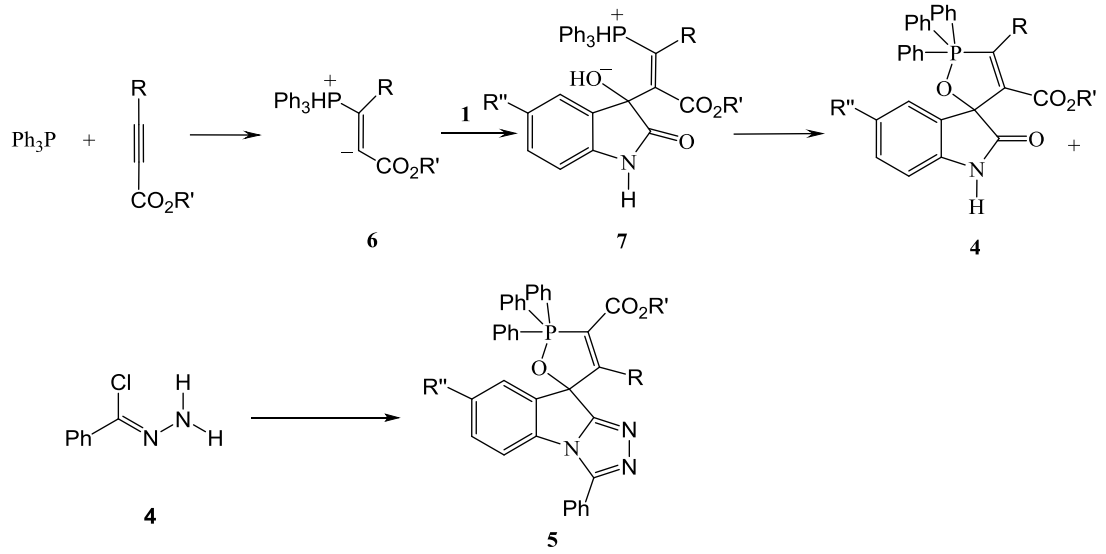
Scheme 1: Synthesis of isatin 1,2,4-triazole derivatives

Result and Discussion

Structures of compounds **5a**–**5f** 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 **5a** 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 **5a** appear at δ = 168.4 ($^3J_{CP}$ = 21.2) and 169.7 ppm. The mass spectrum of **5a** displayed the molecular ion peak at *m*/*z* = 521, which is consistent with the 1:1:1:1 adduct of Ph_3P, ethyl propiolate and methylisatin and phenyl hydrazoyle chloride. Mechanistically, it is conceivable that the reaction involves the initial formation of a 1,3-dipolar intermediate **6** between triphenylphosphine **2** and activated acetylenic compounds **1**, which reacts with the carbonyl group of isatins to produce **7**. Cyclization of this zwitterionic intermediate leads to the spiro compound **8**. Intermediate **8** react with hydrazonoyle chloride **4** and by intermolecular cyclization produced products **5** (Scheme **2**).

Diphenyl-2-picrylhydrazyl (DPPH) utilizing for evaluation of antioxidant ability of spirotriazoles

DPPH radical trapping examin is broadly employed for confirmation the power of synthezied compounds to take free radicals. In this valuation, antioxidant ability of produced spiropyridines was proved by taking the hydrogen atom or one electron by DPPH radical. The synthesized compounds have one NH groups and because of having acidic hydrogen have antioxidant activity. The antioxidant activity of investigated compounds **5a-5d** has not much different from each other. In general antioxidant activity of these compounds are due to having one NH groups. The percentage of DPPH free radical trapping indicates the antioxidant degree of the synthesized spirotriazoles **5a-5d**. In other words, the order of antioxidant ability of spiropyridines **5a-5d** is determined basis of the electron or hydrogen donating power of spirotriazoles **5a-5d** to the DPPH radical. When DPPH give one electron or hydrogen from antioxidant or a radical typs, its absorbtion was decreased from 517 nm. In this research, the ability of getting free radicals by spirotriazoles **5a-5d** was compared with BHT and TBHQ as standard syntheiszed antioxidant at different concentrations.



Scheme 2: Proposed mechanism for the synthesis of **5**

In general, the order of antioxidant activity of some synthesized spirotriazoles **5a-5d** is **TBHQ> BHT>5b>5a>5c>5d** (Figure **1**). Good difference relative to BHT and TBHQ existed in all concentrations of the new prepared spiropyridine derivatives that are showed in Figure 6. In among investigated spirotriazole, **5b** exhibited good activity for trapping of radical relative to BHT and TBHQ as standard antioxidant.

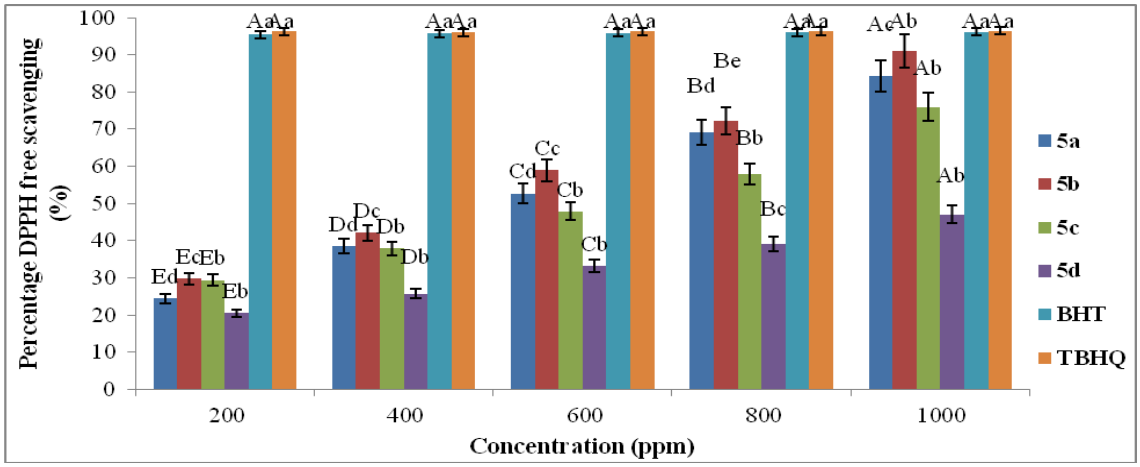


Figure 1. Radical scavenging activity (RSA) of spirotriazoles **5a-5d**

Conclusion

In summary, the reaction of activatedacetylenic compounds with isatins in the presence of Ph_3P and hydrazonoyl chloride led to oxaphosphole-4-carboxylate-1,2,4-triazole derivatives with potential synthetic interest. 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.; □ 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.

General procedure for preparation of compounds 5a-f:

To a stirred solution of activated acetylenic compounds **1** (2 mmol) and isatin **3** (2 mmol) undersolvent-free conditions was added Ph_3P **2** (2 mmol) and hydrazonoyl chloride **4** (2 mmol) at room temperature. The reaction mixture was then stirred for 4 h. After completion of reactions (monitored by TLC (5:1) n-hexane/ethyl acetate, 15 mL water poured into the mixture of reaction. The solid residue was filtered and washed with Et_2O to afforded pure title compounds.

Dimethyl 7'-methyl-2,2,2,3'-tetraphenyl-2H-2l5-spiro[[1,2]oxaphosphole-5,9'-[1,2,4]triazolo[4,3-a]indole]-3,4-dicarboxylate (5a):

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: δ 1.25 (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.

Dimethyl 7'-ethyl-2,2,2,3'-tetraphenyl-2H-2l5-spiro[[1,2]oxaphosphole-5,9'-[1,2,4]triazolo[4,3-a]indole]-3,4-dicarboxylate (5b):

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.

Methyl 1,2-dihydro-2-oxo-1-benzyl-spiro-[3H-indol-2,2,2-triphenyl-2,5-dihydro-1,2-□^{5}-oxaphosphole]-4-carboxylate (5c):

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.

Dimethyl 1,2-dihydro-2-oxo-1-methyl-spiro-[3H-indol-2,2,2-triphenyl-2,5-dihydro-1,2-□^{5}-oxaphosphole]-3,4-dicarboxylate (5d):

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: δ 3.27 (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, $^{2}J_{CP}$ = 51.2 Hz, C_{ipso}), 116.7 (CH), 120.3 (CH), 123.6 (CH), 128.1 (CH), 128.6 (d, $^{3}J_{CP}$ = 22.4 Hz, C), 129.2 (d, $^{3}J_{CP}$ = 21.1 Hz, 6 CH), 129.4 (3 CH), 131.9 (d, $^{2}J_{CP}$ = 31.9 Hz, 6 CH), 135.1 (d, $^{1}J_{CP}$ = 230.1 Hz, 3 C), 149.3 (C), 150.4 (d, $^{1}J_{CP}$ = 192.3 Hz, C), 163.0 (d, $^{2}J_{CP}$ = 24.2 Hz, C=O), 165.1 (C), 168.4 (d, $^{3}J_{CP}$ = 21.2 Hz, C=O), 169.7 (C=O). ^{31}P-NMR: δ79.45.

Diethyl 1,2-dihydro-2-oxo-1-methyl-spiro-[3H-indol-2,2,2-triphenyl-2,5-dihydro-1,2-□^{5}-oxaphosphole]-3,4-dicarboxylate (5e):

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, $^{3}J_{HH}$ = 7.2 Hz, Me), 1.48 (3 H, t, $^{3}J_{HH}$ = 7.2 Hz, Me), 3.25 (3 H, s, NMe), 3.84 (2 H, q, $^{3}J_{HH}$ = 7.2 Hz, OCH_2), 4.08 (2 H, q, $^{3}J_{HH}$ = 7.2 Hz, OCH_2), 6.95 (1 H, t, $^{3}J_{HH}$ = 7.2 Hz, CH), 7.08 (1 H, d, $^{3}J_{HH}$ = 7.2 Hz, CH), 7.33 (1 H, d, $^{3}J_{HH}$ = 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, $^{2}J_{CP}$ = 49.5 Hz, C_{ipso}), 116.2 (CH), 119.5 (CH), 122.9 (CH), 127.9 (CH), 128.4 (d, $^{3}J_{CP}$ = 23.9 Hz, C), 130.1 (d, $^{3}J_{CP}$ = 20.1 Hz, 6 CH), 130.5 (3 CH), 132.0 (d, $^{2}J_{CP}$ = 32.9 Hz, 6 CH), 134.9 (d, $^{1}J_{CP}$ = 230.1 Hz, 3 C), 149.2 (C), 150.4 (d, $^{1}J_{CP}$ = 195.3 Hz, C), 162.9 (d, $^{2}J_{CP}$ = 23.6 Hz, C=O), 166.1 (C), 168.2 (d, $^{3}J_{CP}$ = 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-□^{5}-oxaphosphole]-3,4-dicarboxylate (5f):

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, $^{2}J_{HH}$ = 15.6 Hz, CH), 5.01 (1 H, d, $^{2}J_{HH}$ = 15.6 Hz, CH), 7.15 (1 H, d, $^{3}J_{HH}$ = 7.4 Hz, CH), 7.30 (1 H, t, $^{3}J_{HH}$ = 7.5 Hz, CH), 7.36 (1 H, d, $^{3}J_{HH}$ = 7.5 Hz, CH), 7.38 (2 H, t, $^{3}J_{HH}$ = 7.5 Hz, 2 CH), 7.45 (2 H, t, $^{3}J_{HH}$ = 7.7 Hz, 2 CH), 7.54 (2 H, d, $^{3}J_{HH}$ = 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, $^{2}J_{CP}$ = 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, $^{3}J_{CP}$ = 24.2 Hz, C), 128.9 (d, $^{3}J_{CP}$ = 20.1 Hz, 6 CH), 130.2 (3 CH), 132.4 (d, $^{2}J_{CP}$ = 34.2 Hz, 6 CH), 135.9 (C), 136.2 (d, $^{1}J_{CP}$ = 234.5 Hz, 3 C), 148.4 (C), 151.2 (d, $^{1}J_{CP}$ = 190.1 Hz, C), 162.4 (d, $^{2}J_{CP}$ = 26.5 Hz, C=O), 164.8 (C), 167.5 (d, $^{3}J_{CP}$ = 20.3 Hz, C=O), 169.5 (C=O). ^{31}P-NMR: δ 44.2.

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