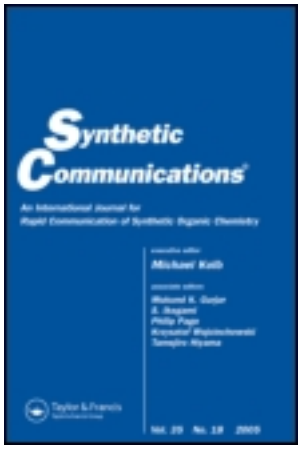

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Three-Component Reaction of Triphenylphosphine, Dialkyl Acetylenedicarboxylate, and 2-Aminothiazole or 2-Aminobenzothiazole in the Presence of Arylglyoxals: An Efficient One-Pot Synthesis of Highly Functionalized Pyrroles

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THREE-COMPONENT REACTION OF TRIPHENYLPHOSPHINE, DIALKYL ACETYLENEDICARBOXYLATE, AND 2-AMINOTHIAZOLE OR 2-AMINOBENZOTHIAZOLE IN THE PRESENCE OF ARYLGLYOXALS: AN EFFICIENT ONE-POT SYNTHESIS OF HIGHLY FUNCTIONALIZED PYRROLES

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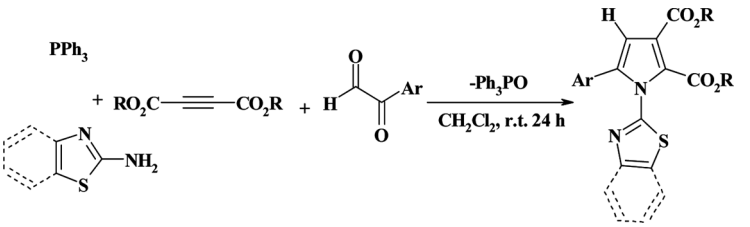
GRAPHICAL ABSTRACT



Abstract *A new and efficient one-pot synthesis of polysubstituted pyrrole derivatives by three-component reaction of dialkyl acetylenedicarboxylates, triphenylphosphine, 2-aminothiazole or 2-aminobenzothiazole in the presence of arylglyoxals is described. The reactions were performed in dichloromethane at room temperature and neutral conditions and afforded good yields of products.*

Keywords 2-Aminobenzothiazole; 2-aminothiazole; arylglyoxals; dialkyl acetylenedicarboxylates; intramolecular Wittig reaction; multicomponent reaction; pyrrole derivatives; triphenylphosphine

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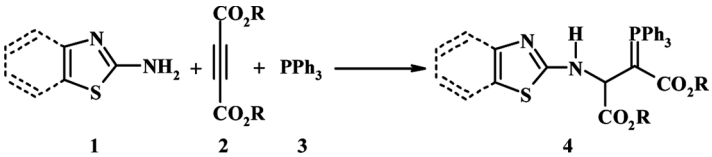
INTRODUCTION

Pyrroles are heterocycles of great importance because of their presence in numerous natural products such as heme, vitamin B_{12}, and various cytochrome enzymes.[1] Some of the recently isolated pyrrole-containing marine natural products have been found to exhibit considerable cytotoxicity and function as multidrug-resistant (MDR) reversal agents.[2] Many of these biologically active compounds have emerged as chemotherapeutic agents. In addition, polysubstituted pyrroles are molecular frameworks having immense importance in material science. They have been employed as antioxidants; antibacterial, ionotropic, antitumor, anti-inflammatory, and antifungal agents,[3] P38 kinase, prolyl-4-hydroxylase, and poly(ADP-ribose)polymerase inhibitors; estrogen receptor β selective ligands; AT_1-selective angiotensin II receptor antagonists, and minor groove-recognition elements.[4] Moreover, they are a highly versatile class of intermediates in the synthesis of natural products as well as in heterocyclic chemistry.[5] Consequently, a large number of methods have been developed for their synthesis. which include 1,3-dipolar cycloaddition reactions,[6] reductive coupling,[7] and aza-Wittig reactions.[8] These methodologies typically require the preparation of the precursors prior to cyclization, which can complicate both the synthesis and the structural modification of the substituted pyrroles. This has stimulated significant interest in the design of new synthetic routes to pyrroles, including several efficient multicomponent reactions[9–12] and metal-catalyzed routes.[13] Addition reaction between phosphines and activated carbon–carbon triple bonds is well known to produce a reactive zwitterionic intermediate, which may be trapped by various electrophiles.[14–18] Reaction of triphenylphosphine with dimethyl acetylenedicarboxylate (DMAD) has been studied in the presence of a variety of organic acidic compounds to trap the zwitterionic intermediate. Trapping of the PPh_3-DMAD zwitterion by an organic acidic compound containing a carbonyl group has been used as an efficient, one-pot route for the synthesis of a variety of heterocyclic and carbocyclic compounds.[17–22] Treatment of triphenylphosphine with DMAD in the presence of primary amines has been reported to produce β-amino phosphoranes **4** (Scheme 1).[18]

Recently we reported the one-pot reaction of these ylides with aryl glyoxals for the synthesis of substituted *N*-Aryl pyrroles.[23]

Here we report an extention of this route for the synthesis of polysubstituted pyrrole derivatives by the three-component reaction of dialkyl acetylenedicarboxylates, triphenylphosphine, and 2-aminothiazole or 2-aminobenzothiazole in the presence of arylglyoxals. We first prepared ylide **4** by the reaction of triphenylphosphine, DMAD, and 2-aminothiazole by the previously reported procedure.[15]

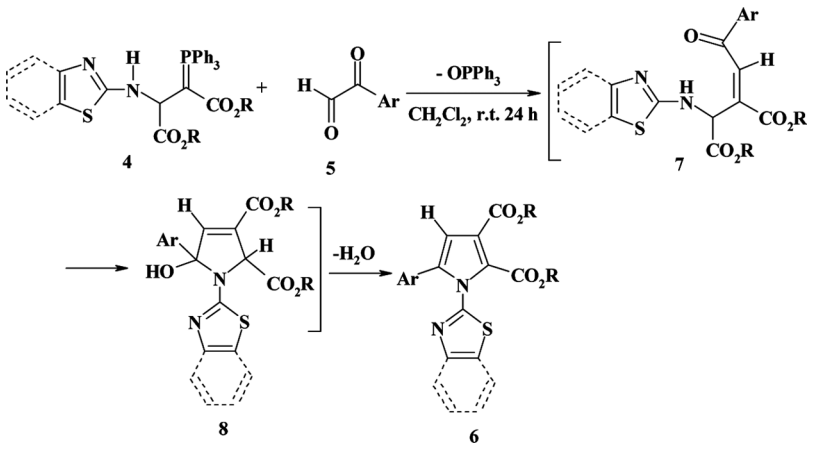


Scheme 1. Synthesis of 2-aminophosphoranes by reaction of PPh3-DAAD zwitterion with 2-aminothiazole or 2-aminobenzothiazole.

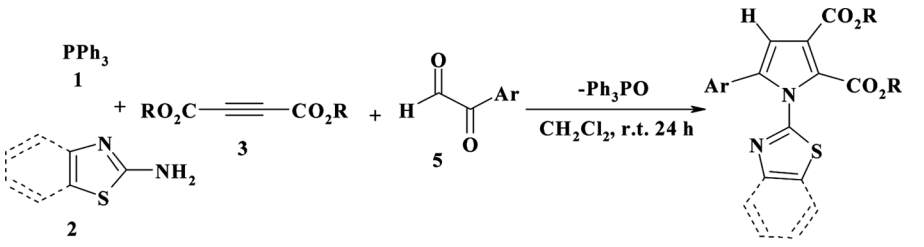
When phosphorane **4** was stirred with an equimolar amount of phenylglyoxal in dichloromethane, a smooth reaction took place. After completion of the reaction (monitored by thin-layer chromatography, TLC) dimethyl 4-phenyl-1-(thiazol-2-yl)-1*H*-pyrrole-2,3-dicarboxylate **6a** was obtained in 85% yield. As shown in Scheme 2, it is reasonable to assume that the Wittig reaction between phosphorane **4** and phenylglyoxal **5** affords intermediate **6**, which then cyclizes to dihydropyrrole intermediate **7** and subsequently loses water to produce pyrrole derivative **6a**.

Being successful in this reaction, we decided to investigate the one-pot synthesis of dimethyl 4-phenyl-1-(thiazol-2-yl)-1*H*-pyrrole-2,3-dicarboxylate **6a** (Scheme 3). Thus, equimolar amounts of triphenylphosphine, 2-aminothiazole, and DMAD were mixed in dichloromethane as solvent. After stirring for 1 min at room temperature, phenylglyoxal was added, and the progress of the reaction was monitored by TLC. After 24 h, the TLC of the mixture of the reaction showed only the presence of pyrrole derivative **6a** and triphenylphosphine oxide. Silica-gel chromatography afforded the product 2-aminothiazole **6a** in 85% yield. To investigate the scope of the reaction, 2-aminothiazole or 2-aminobenzothiazole was reacted with arylglyoxals, triphenylphosphine, and dialkylacetylenedicarboxylates, and the corresponding pyrroles were obtained in good yields (Scheme 3).

The structures of compounds **6a–h** were deduced from their elemental analyses and their infrared (IR), ^{1}H NMR, and ^{13}CNMR spectral data. The mass spectrum of compound **6a** displayed the molecular ion peak at $m/z = 342$ as the base peak. The 500-MHZ ^{1}H NMR spectrum of **6a** exhibited three sharp signals at δ 3.74, 3.76, and 7.14 ppm for two methoxy groups protons and the proton of pyrrole ring, respectively. Aromatic protons resonated between 7.21 and 7.58 ppm as multiplets. The ^{13}C NMR spectrum of compound **6a** showed 15 distinct resonances in agreement with the proposed structure. The structural assignments made on the basis of the NMR spectra of compound **6a** were supported by its IR spectrum. The ester carbonyl groups exhibited strong absorption bands at about 1708 cm^{-1}. In conclusion, here we report a three-component reaction of dialkyl acetylenedicarboxylate,



Scheme 2. Reaction of 2-aminophosphoranes **4** with arylglyoxals **5** for the synthesis of pyrrole derivatives **6**.



6	R	Ar	%Yield*
a	Me	C_6H_5	85
b	Et	4-ClC_6H_4	85
c	Me	4-ClC_6H_4	80
d	Et	4-$NO_2C_6H_4$	80
e	Me	4-$NO_2C_6H_4$	85
f	Me	C_6H_5	89
g	Me	4-ClC_6H_4	86
h	Et	4-ClC_6H_4	85

*Isolated yields

6a-e :thiazole and **6f-h** :benzothiazole

Scheme 3. Three-component reaction of 2-aminothiazole or 2-aminobenzothiazole, DAADs, and arylglyoxals promoted by PPh_3.

2-aminothiazole or 2-aminobenzothiazole, and arylglioxals promoted by triphenylphosphine to produce functionalized pyrrole derivatives in good yields. Not only is the reaction performed under neutral conditions, but also the substances can be mixed without any activation or modification.

EXPERIMENTAL

Melting points were determined with an Electrothermal 9100 apparatus. Elemental analyses were performed using a Costech ECS 4010 CHNS-O analyzer at the analytical laboratory of the Islamic Azad University Yazd branch. Mass spectra were recorded on a Finnigan-MAT 8430 mass spectrometer operating at an ionization potential of 70 eV. IR spectra were recorded on a Shimadzu IR-470 spectrometer. ^1H and ^{13}C NMR spectra were recorded on a Bruker DRX-500 Avance spectrometer at 500.1 and 125.8 MHz, respectively. ^1H and ^{13}C NMR spectra were obtained in solution in $CDCl_3$ using tetramethylsilane (TMS) as internal standard. Column chromatography was performed with Merck silica gel 60, 230–400 mesh. The chemicals used in this work were purchased from Fluka (Buchs, Switzerland) and were used without further purification.

General Procedure for Preparation of Compounds 6a–h

A mixture of dialkyl acetylenedicarboxylate (1 mmol) in dichloromethane (3 mL) was added dropwise to a magnetically stirred solution of triphenylphosphine

(1 mmol) and 2-aminothiazole or 2-aminobenzothiazole (1 mmol) in dichloromethane (10 mL). At room temperature over 2 min. The reaction mixture was then stirred for 1 min. Arylglyoxal (1 mmol) was added, and the reaction mixture was stirred for 24 hs. The solvent was evaporated, and the residue was purified by column chromatography on silicagel using ethyl acetate–hexane mixture as eluent.

Dimethyl 4-Phenyl-1-(thiazol-2-yl)-1H-pyrrole-2,3-dicarboxylate (6a)

Yellow powder, mp 125–127 °C; IR (KBr) (ν_{max}/cm^{-1}): 1708 (C=O). Anal. Calcd. for $C_{17}H_{14}N_2O_4S$ (342): C, 59.64; H, 4.12; N, 8.18%. Found: C, 59.5; H, 4.2; N, 8.3%. MS, m/z (%): 342 ($M^{+\cdot}$, 100). ^{1}H NMR (500 MHZ, $CDCl_3$): $\delta = 3.74$ and 3.76 (6H, 2 s, 2 OCH_3), 7.14 (1H, s, CH of pyrrole), 7.21–7.37 (6H, m, C_6H_5 and CH of C_3H_2NS), 7.58 (1H, d, $^3J_{HH} = 3.4$ HZ, CH of C_3H_2NS) ppm. ^{13}C NMR (125.8 MHZ, $CDCl_3$): $\delta = 52.73$ and 52.94 (2 OCH_3), 119.81, 122.30, 124.84, 126.90, 127.99, 128.53, 128.99, 132.85, 140.70, 158.83, 161.98 (aromatic), 165.73 and 168.46 (2 C=O, ester).

Diethyl 4-(4-Chlorophenyl)-1-(thiazol-2-yl)-1H-pyrrole-2,3-dicarboxylate (6b)

Yellow powder, mp 112–114 °C; IR (KBr) (ν_{max}/cm^{-1}): 1726 (C=O). Anal. calcd. for $C_{19}H_{17}ClN_2O_4S$ (404): C, 56.36; H, 4.23; N, 6.92%. Found: C, 56.3; H, 4.3; N, 6.8%. MS, m/z (%): 404 ($M^{+\cdot}$, 100). ^{1}H NMR (500 MHZ, $CDCl_3$): $\delta = 1.99$ (6H, m, 2 OCH_2CH_3), 4.22 (4H, m, 2 OCH_2CH_3), 7.14 (1H, s, CH of pyrrole), 7.25 (1 H, d, $^3J_{HH} = 3.5$ HZ, CH of C_3H_2NS), 7.27–7.31 (4 H, m, 2 CH of 4Cl-C_6H_4), 7.58 (1 H, d, $^3J_{HH} = 3.5$ HZ, CH of C_3H_2NS) ppm. ^{13}C NMR (125.8 MHZ, $CDCl_3$): $\delta = 14.32$, 14.47 ($2OCH_2CH_3$), 61.77 and 62.07 ($2OCH_2CH_3$), 119.83, 122.15, 124.53, 125.71, 128.99, 129.99, 131.50, 133.93, 140.67, 158.75, 160.44 (aromatic), 165.01 and 168.26 (2 C=O, ester).

Dimethyl 4-(4-Chlorophenyl)-1-(thiazol-2-yl)-1H-pyrrole-2,3-dicarboxylate (6c)

Yellow powder, mp 137–139 °C; IR (KBr) (ν_{max}/cm^{-1}): 1723 (C=O). Anal. calcd. for $C_{17}H_{13}ClN_2O_4S$ (376): C, 54.19; H, 3.48; N, 7.43%. Found: C, 54.3; H, 3.5; N, 7.6%. MS, m/z (%): 376 ($M^{+\cdot}$, 100). ^{1}H NMR (500 MHZ, $CDCl_3$): $\delta = 3.73$ and 3.76 (6 H, 2 s, 2 OCH_3), 7.15 (1H, s, CH of pyrrole), 7.24 (1 H, d, $^3J_{HH} = 3.5$ HZ, CH of C_3H_2NS), 7.27–7.30 (4 H, m, 2 CH of 4Cl-C_6H_4), 7.58 (1 H, d, $^3J_{HH} = 3.5$ HZ, CH of C_3H_2NS) ppm. ^{13}C NMR (125.8 MHZ, $CDCl_3$): $\delta = 52.73$ and 53.04 (2 OCH_3), 119.73, 121.65, 124.53, 125.90, 129.05, 129.98, 131.30, 134.02, 140.76, 158.62, 161.03 (aromatic), 165.36 and 168.16 (2 C=O, ester).

Diethyl 4-(4-Nitrophenyl)-1-(thiazol-2-yl)-1H-pyrrole-2,3-dicarboxylate (6d)

Yellow powder, mp 131–133 °C; IR (KBr) (ν_{max}/cm^{-1}): 1726 (C=O). Anal. Calcd. for $C_{19}H_{17}N_3O_6S$ (415): C, 54.93; H, 4.12; N, 10.12%. Found: C, 54.8; H,

4.3; N, 10.0%. MS, *m/z* (%): 415 ($M^{+\cdot}$, 100). ^{1}H NMR (500 MHZ, $CDCl_3$): δ = 1.21 (6H, m, 2 OCH_2CH_3), 4.29 (4H, m, 2 OCH_2CH_3), 7.32 (1H, s, CH of pyrrole), 7.35 (1 H, d, $^3J_{HH}$ = 3.5 HZ, CH of C_3H_2NS), 7.59 (2 H, d, $^3J_{HH}$ = 8.7 HZ, 2CH of 4-$NO_2C_6H_4$), 7.65 (1 H, d, $^3J_{HH}$ = 3.5 HZ, CH of C_3H_2NS), 8.21 (2 H, d, $^3J_{HH}$ = 8.7 HZ, 2CH of 4-$NO_2C_6H_4$) ppm. ^{13}C NMR (125.8 MHZ, $CDCl_3$): δ = 14.29, 14.41 ($2OCH_2CH_3$), 61.65 and 62.12 ($2OCH_2CH_3$), 119.99, 121.45, 124.23, 124.76, 126.91, 129.21, 139.90, 140.78, 147.46, 158.34, 160.36 (aromatic), 164.57 and 168.13 (2 C=O, ester).

Dimethyl 4-(4-Nitrophenyl)-1-(thiazol-2-yl)-1H-pyrrole-2,3-dicarboxylate (6e)

Yellow powder, mp 117–119 °C; IR (KBr) (ν_{max}/cm^{-1}): 1721 (C=O). Anal. calcd. for $C_{17}H_{13}N_3O_6S$ (387): C, 52.71; H, 3.38; N, 10.85%. Found: C, 52.8; H, 3.2; N, 10.7%. MS, *m/z* (%): 387 ($M^{+\cdot}$, 100). ^{1}H NMR (500 MHZ, $CDCl_3$): δ = 3.79 and 3.84 (6 H, 2 s, 2 OCH_3), 7.32 (1H, s, CH of pyrrole), 7.33 (1 H, d, $^3J_{HH}$ = 3.5 HZ, CH of C_3H_2NS), 7.58 (2 H, d, $^3J_{HH}$ = 8.7 HZ, 2CH of 4-$NO_2C_6H_4$), 7.64 (1 H, d, $^3J_{HH}$ = 3.5 HZ, CH of C_3H_2NS), 8.19 (2 H, d, $^3J_{HH}$ = 8.7 HZ, 2CH of 4-$NO_2C_6H_4$) ppm. ^{13}C NMR (125.8 MHZ, $CDCl_3$): δ = 52.85 and 53.23 (2 OCH_3), 119.94, 120.93, 124.12, 124.92, 126.89, 129.38, 139.75, 140.86, 147.47, 158.19, 160.95 (aromatic), 164.91 and 168.37 (2 C=O, ester).

Dimethyl 1-(Benzo[d]thiazol-2-yl)-4-phenyl-1H-pyrrole-2,3-dicarboxylate (6f)

Yellow powder, mp 112–113 °C, IR (KBr) (ν_{max}/cm^{-1}): 1722 (C=O). Anal. calcd. for $C_{21}H_{16}N_2O_4S$ (392): C, 64.27; H, 4.11; N, 7.14%. Found: C, 64.3; H, 4.3; N, 7.3%. MS, *m/z* (%): 392 ($M^{+\cdot}$, 100). ^{1}H NMR (500 MHZ, $CDCl_3$): δ = 3.80 and 3.90 (6 H, 2 s, 2 OCH_3), 7.31 (1H, s, CH of pyrrole), 7.32–7.58 (7 H, m, aromatic), 7.83 (1 H, d, $^3J_{HH}$ = 8 HZ, CH of aromatic), 7.97 (1 H, d, $^3J_{HH}$ = 8 HZ, CH of aromatic) ppm. ^{13}C NMR (125.8 MHZ, $CDCl_3$): δ = 52.65 and 52.23 (2 OCH_3), 121.55, 121.99, 123.73, 12386, 126.35, 126.60, 127.37, 127.77, 128.10, 128.82, 128.85, 132.80, 134.88, 150.37, 157.68 (aromatic), 161.52 and 165.18 (2 C=O, ester).

Dimethyl 1-(Benzo[d]thiazol-2-yl)-4-(4-chlorophenyl)-1H-pyrrole-2,3-dicarboxylate (6g)

Yellow powder, mp 147–149 °C, IR (KBr) (ν_{max}/cm^{-1}): 1732 (C=O). Anal. calcd. for $C_{21}H_{15}ClN_2O_4S$ (426): C, 59.09; H, 3.54; N, 6.56%. Found: C, 59.2; H, 3.4; N, 6.6%. MS, *m/z* (%): 426 ($M^{+\cdot}$, 100). ^{1}H NMR (500 MHZ, $CDCl_3$): δ = 3.80 and 3.90 (6 H, 2 s, 2 OCH_3), 7.23 (1H, s, CH of pyrrole), 7.25–7.53 (6 H, m, aromatic), 7.96 (1 H, d, $^3J_{HH}$ = 8.3 HZ, CH of aromatic), 8.05 (1 H, d, $^3J_{HH}$ = 8.3 8.3 HZ, CH of aromatic) ppm. ^{13}C NMR (125.8 MHZ, $CDCl_3$): δ = 52.66 and 53.32 (2 OCH_3), 121.99, 123.43, 123.88, 126.41, 126.76, 127.43, 128.96, 129.23, 129.67, 129.84, 130.29, 131.30, 132.88, 150.47, 156.26 (aromatic), 162.31 and 168.19 (2 C=O, ester).

Diethyl 1-(Benzo[d]thiazol-2-yl)-4-(4-chlorophenyl)-1H-pyrrole-2,3-dicarboxylate (6h)

Yellow powder, mp 130–132 °C, IR (KBr) (ν_{max}/cm^{-1}): 1716 (C=O). Anal. calcd. for $C_{23}H_{19}ClN_2O_4S$ (454): C, 60.72; H, 4.21; N, 6.16%. Found: C, 60.8; H, 4.1; N, 6.4%. MS, m/z (%): 454 ($M^{+\cdot}$, 100). ^1H NMR (500 MHZ, $CDCl_3$): $\delta = 1.29$ (6H, m, 2 OCH_2CH_3), 4.29 (2H, d, $^3J_{HH} = 7.2$ HZ, OCH_2CH_3), 4.37 (2H, d, $^3J_{HH} = 7.2$ HZ, OCH_2CH_3), 7.29 (1H, s, CH of pyrrole), 7.34–7.87 (6 H, m, aromatic), 7.96 (1 H, d, $^3J_{HH} = 8.3$ HZ, CH of aromatic), 8.45 (1 H, d, $^3J_{HH} = 8.3$ HZ, CH of aromatic) ppm.. ^{13}C NMR (125.8 MHZ, $CDCl_3$): $\delta = 14.35$, 14.47 ($2OCH_2CH_3$), 61.67 and 62.44 ($2OCH_2CH_3$), 121.97, 123.29, 123.82, 126.35, 126.76, 127.39, 128.88, 129.22, 129.69, 130.35, 131.30, 131.44, 132.88, 150.47, 156.26 (aromatic), 160.36 and 168.18 (2 C=O, ester).

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