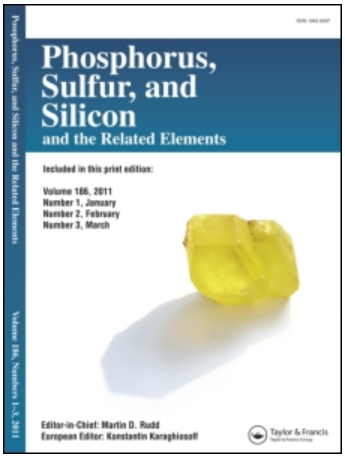

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An Efficient, One-Pot Synthesis of Dialkyl 5-Hydroxy-4-aryl-2,5-dihydrofuran-2,3-dicarboxylate Derivatives

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AN EFFICIENT, ONE-POT SYNTHESIS OF DIALKYL 5-HYDROXY-4-ARYL-2,5-DIHYDROFURAN-2,3-DICARBOXYLATE DERIVATIVES

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The reaction between arylglyoxalmonohydrates, dialkyl acetylenedicarboxylates, and triphenylphosphine is described as a simple and efficient method for the synthesis of 5-hydroxy-4-aryl-2,5-dihydrofuran-2,3-dicarboxylate derivatives.

Keywords Arylglyoxals; dialkyl acetylenedicarboxylates; 2,5-dihydrofuran derivatives; intramolecular Wittig reaction; triphenylphosphine

INTRODUCTION

Dihydrofurans are among the most important heterocycles commonly found in a large variety of naturally occurring substances.[1,2] The development of new and efficient methods for their synthesis remains an area of current interest, and a whole series of new synthetic methods has appeared in literature.[3–16] Among the synthetic methodologies toward dihydrofurans, non-ionic as well as ionic procedures have been exploited. Radical[2] or carbenoid[7–9] additions to olefins have been utilized as non-ionic procedures. Among ionic reaction conditions, dihydrofuran syntheses via tandem nucleophilic reaction of 1,3-dicarbonyl compounds[10–12] or ylides[13–16] with enones have been reported.

The nucleophilic addition of triphenylphosphine to activated acetylenes is well known to produce a reactive zwitterionic intermediate,[17–19] which may be trapped by acidic organic compounds such as alcohols. The reaction between dimethyl acetylenedicarboxylate (DMAD) and triphenylphosphine in the presence of alcohols has been reported to produce phosphorus ylides as an intermediate or the final product. When hydroxy carbonyl compounds were used, the intramolecular Wittig reaction of the ylide intermediate afforded oxygen heterocycles.[20–24] Using α-hydroxy ketones in order to trap the PPh_3-DMAD zwitterion, 2,5-dihydrofuran derivatives were obtained.[21–24] Similar reactions have been developed for the synthesis of a variety of carbocycles and heterocycles, using N-H,[23,25,26] S-H,[27] and C-H[28] acidic compounds in order to trap the zwitterionic DMAD-PPh_3 intermediate. In continuation of our previous studies on the reaction of PPh_3-DMAD zwitterion with organic acidic compounds,[25,29–31] in this article we report that the reaction between

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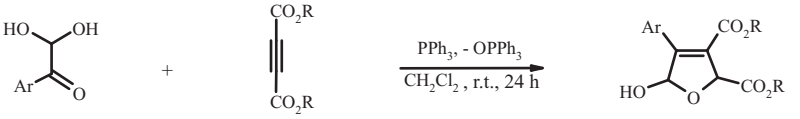
dialkyl acetylenedicarboxylates, triphenylphosphine, and arylglyoxalmonohydrates affords 2,5-dihydrofuran derivatives in good yields.

RESULTS AND DISCUSSION

Treatment of DMAD with triphenylphosphine and phenylglyoxalmonohydrate in dichloromethane at room temperature after 24 h afforded dimethyl 5-hydroxy-4-phenyl-2,5-dihydrofuran-2,3-dicarboxylate (**3a**) in 85% yield (Scheme 1). Compound **3a** may



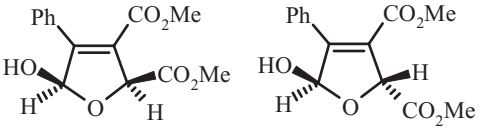
1 **2** **3**

3	Ar	R	%Yield*	*cis*/*trans***
a	C_6H_5	CH_3	85	33/67
b	C_6H_5	t-Bu	79	40/60
c	4-Br C_6H_5	CH_3	80	36/64
d	4-Br C_6H_5	C_2H_5	71	62/38
e	4-Br C_6H_5	*t*-Bu	80	22/78
f	4- NO_2 C_6H_5	CH_3	81	10/90
g	4- NO_2 C_6H_5	C_2H_5	77	20/80

*Isolated yields
**Obtained by ^{1}H NMR spectra

Scheme 1 Synthesis of 2,5-dihydrofuran derivatives by condensation of aryglyoxalmonohydrates with acetylene diesters promoted by PPh_3.

exist as two diastereomers, *cis* and *trans* (Figure 1). The homoallylic coupling constant of C-2 and C-5 hydrogens of dihydrofuran ring can be used for determining the stereochemistry of compound **3a**; the $^5J_{HH}$ is considerably greater when the two hydrogens have *cis* arrangement than when they are *trans* to each other.[32] The ^{1}H NMR spectrum of compound **3a** exhibited the presence of both *cis* and *trans* isomers. Two doublets ($^5J_{HH} = 5$ H_Z) were observed at 5.67 and 6.49 ppm for two methine protons of the *cis* isomer, and two doublets ($^5J_{HH} = 1$ H_Z) were observed at 5.45 and 6.11 ppm for two methine protons of the *trans*



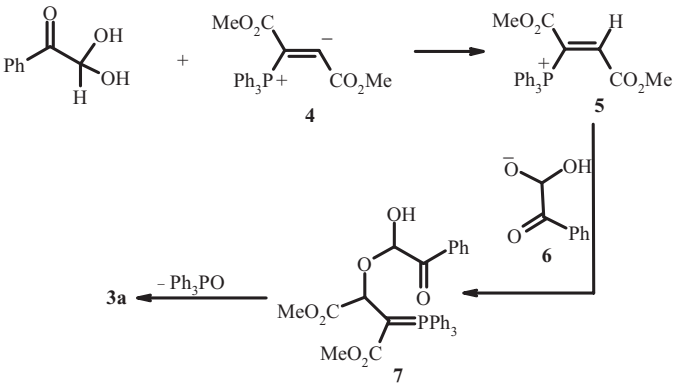
Cis-**3a** *Trans*-**3a**

Figure 1 Two stereoisomers of compound **3a**.

isomer. The methoxy protons were observed as two singlets at 3.66 and 3.77 ppm for the *cis* isomer and two singlets at 3.70 and 3.83 for the *trans* isomer. The aromatic protons resonated between 7.36 and 8.12 ppm. A broad signal was observed at 4.73 ppm for the OH proton, which disappeared by addition of D_2O to $CDCl_3$ solution of **3a**. ^{13}C NMR spectrum of compound **3a** showed the signals due to sp^3 carbons of furan ring at 83.9 and 106.1 ppm for the *cis* isomer and at 83.7 and 106.4 ppm for the *trans* isomer. These chemical shifts are consistent with those expected for similar ether and acetal carbons, respectively.[33] Sixteen distinct signals were observed for each isomer in the ^{13}C NMR spectrum of compound **3a**, which is in agreement with the proposed structure. The above structural assignments based on NMR spectroscopy were supported by the IR spectrum of compound **3a**, which exhibited an absorption band at 3430 cm^{-1} for the OH group and a strong broad absorption band at 1732 cm^{-1} for carbonyl groups. The *cis*:*trans* ratio can be calculated to be 33:67 using the ^1H NMR spectrum of compound **3a**.

In order to investigate the generality of the method, the reactions of different arylglyoxalmonohydrates and dialkyl acetylenedicarboxylates were carried out with triphenylphosphine, and the corresponding functionalized 2,5-dihydrofuran derivatives **3a–g** were obtained in good yields.

A reasonable mechanism for the formation of compound **3a** is presented in Scheme 2. Protonation of the DMAD-PPh_3 zwitterion **4** by phenylglyoxalmonohydrate was followed by the conjugate addition of the alkoxide anion **6** to phosphonium cation **5** to form ylide **7**, which was converted to dihydrofuran **3a** by intramolecular Wittig reaction.



Scheme 2 Suggested mechanism for formation of compound **3a**.

In summary, the reaction of the DMAD-PPh_3 zwitterion with arylglyoxalmonohydrates provides an acceptable, one-pot method for the preparation of dialkyl 5-hydroxy-4-aryl-2,5-dihydrofuran-2,3-dicarboxylates. The present procedure carries the advantage that, not only is the reaction performed under neutral conditions, but the starting materials and reagents also 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 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. ^{1}H and ^{13}C NMR spectra were recorded on Bruker DRX-500 AVANCE, at 250 and 62.9 MH_Z, respectively, at solutions in $CDCl_3$ using TMS as internal standard. The chemicals used in this work were purchased from Fluka (Buchs, Switzerland) and were used without further purification.

General Procedure for Preparation of Compounds 3a–g by Reaction Between Dialkyl Acetylenedicarboxylates, Triphenylphosphine, and Arylglyoxalmonohydrates

To a magnetically stirred solution of arylglyoxalmonohydrate **1** (2 mmol) and dialkyl acetylenedicarboxylate **2** (2 mol) in dichloromethane (20 mL), a mixture of triphenylphosphine (2 mmol) in dichloromethane (2 mL) was added at room temperature. The reaction mixture was then allowed to stir for 24 h. The solvent was evaporated at reduced pressure, and the residue was purified by silica gel column chromatography using hexane-ethyl acetate (4:1) as eluent. The solvent was removed under reduced pressure to afford the product. For all compounds **3a–g**, a mixture of isomers was obtained (the isomeric ratios were obtained by ^{1}H NMR data).

Dimethyl 5-Hydroxy-4-phenyl-2,5-dihydrofuran-2,3-dicarboxylate (3a)

Yield: 85%; viscous oil, IR (KBr) (ν_{max}, cm^{-1}): 3430 (OH), 1732 (C=O). MS (m/z,%): 278 ($M^{\cdot+}$, 7). NMR data for *cis* isomer: ^{1}H NMR (250 MH_Z, $CDCl_3$):δ 3.66 and 3.77 (6 H, 2 s, 2 OCH_3), 4.73 (1 H, broad s, OH), 5.67 (1 H, d, $^5J_{HH} = 5$ H_Z, C*H*), 6.49 (1 H, d, $^5J_{HH} = 5$ H_Z, C*H*), 7.36–8.12 (5 H, aromatic). ^{13}C NMR (62.9 MH_Z, $CDCl_3$):δ 52.1, 52.8 (2 OCH_3), 83.9, 106.1 (2 CH), 129.6, 149.4, 126.2, 128.1, 128.8 and 133.2 (aromatic and olefinic carbons), 169.1 and 169.7 (2 C=O). NMR data for *trans* isomer: ^{1}H NMR (250 MH_Z, $CDCl_3$): δ 3.70 and 3.83 (6 H, 2 s, 2 OCH_3), 4.52 (1 H, broad s, OH), 5.45 (1 H, d, $^5J_{HH} = 1$ H_Z, C*H*), 6.18 (1 H, d, $^5J_{HH} = 1$ H_Z, C*H*), 7.36–8.12 (5 H, aromatic). ^{13}C NMR (62.9 MH_Z, $CDCl_3$):δ 52.2, 53.4 (2 OCH_3), 83.7, 106.4 (2 CH), 130.5, 150.1, 125.9, 128.2, 129.9 and 133.6 (aromatic and olefinic carbons), 171.6 and 172.7 (2 C=O). Analysis: Calcd. for $C_{14}H_{14}O_6$: C, 60.43; H, 5.07%. Found: C, 60.3; H, 5.2%.

Di-*t*-butyl 5-Hydroxy-4-phenyl-2,5-dihydrofuran-2,3-dicarboxylate (3b)

Yield: 79%; viscous oil, IR(KBr) (ν_{max}, cm^{-1}): 3465 (OH), 1719 (C=O). MS (m/z,%): 362 ($M^{\cdot+}$, 9). NMR data for *cis* isomer: ^{1}H NMR (250 MH_Z, $CDCl_3$): δ 1.38 (9 H, s, *t*-Bu), 1.50 (9 H, s, *t*-Bu), 4.23 (1 H, broad s, OH), 5.49 (1 H, d, $^5J_{HH} = 5$ H_Z, C*H*), 6.47 (1 H, d, $^5J_{HH} = 5$ H_Z, C*H*), 7.26–7.59 (5 H, aromatic). ^{13}C NMR (62.9 MH_Z, $CDCl_3$):δ 28.7 and 28. 8 (6 CH_3 of 2 *t*-Bu), 81.2 and 82.4 (2 C of 2 *t*-Bu), 84.0, 105.1 (2 CH), 129.7, 147.6, 127.3, 127.7, 128.3 and 133.0 (aromatic and olefinic carbons), 160.5 and 170.1 (2 C=O). NMR data for *trans* isomer: ^{1}H NMR (250 MH_Z, $CDCl_3$): δ 1.39 (9 H, s, *t*-Bu), 1.53 (9 H, s, *t*-Bu), 4.31 (1 H, broad s, OH), 5.41 (1 H, d, $^5J_{HH} = 1$ H_Z, C*H*), 6.07 (1 H, d, $^5J_{HH} = 1$ H_Z, C*H*), 7.26–7.59 (5 H, aromatic). ^{13}C NMR (62.9 MH_Z, $CDCl_3$):δ 26.7 and 26. 8 (6 CH_3 of 2 *t*-Bu), 81.3 and 82.5 (2 C of 2 *t*-Bu), 84.2, 105.6 (2 CH), 129.8, 147.9, 127.0, 128.0, 129.4 and 132.9 (aromatic and olefinic carbons), 160.7 and 170.4 (2 C=O). Analysis: Calcd. for $C_{20}H_{26}O_6$: C, 66.28; H, 7.23%. Found: C, 66.4; H, 7.1%.

Dimethyl 4-(4-Bromophenyl)-5-hydroxy-2,5-dihydrofuran-2,3-dicarboxylate (3c)

Yield: 80%; viscous oil, IR (KBr) (ν_{max}, cm^{-1}): 3380 (OH), 1746 (C=O). MS (m/z,%): 356 ($M^{\cdot+}$, 4). NMR data for *cis* isomer: ^1H NMR (250 MH_Z, $CDCl_3$):δ 3.72 and 3.78 (6 H, 2 s, 2 OCH_3), 4.68 (1 H, broad s, OH), 5.61 (1 H, d, $^5J_{HH} = 4$ H_Z, C*H*), 6.14 (1 H, d, $^5J_{HH} = 4$ H_Z, C*H*), 7.26–7.52 (4 H, aromatic). ^{13}C NMR (62.9 MH_Z, $CDCl_3$):δ 52.5, 53.1 (2 OCH_3), 83.9, 110.7 (2 CH), 129.3, 146.5, 130.5, 130.7, 131.7 and 131.8 (aromatic and olefinic carbons), 168.2 and 167.8 (2 C=O). NMR data for *trans* isomer: ^1H NMR (250 MH_Z, $CDCl_3$): δ 3.69 and 3.80 (6 H, 2 s, 2 OCH_3), 4.70 (1 H, broad s, OH), 5.45 (1 H, d, $^5J_{HH} = 1$ H_Z, C*H*), 5.99 (1 H, d, $^5J_{HH} = 1$ H_Z, C*H*), 7.26–7.52 (4 H, aromatic). ^{13}C NMR (62.9 MH_Z, $CDCl_3$):δ 52.6, 53.2 (2 OCH_3), 84.7, 110.7 (2 CH), 129.9, 146.3, 130.8, 130.9, 131.6 and 131.7 (aromatic and olefinic carbons), 169.8 and 170.2 (2 C=O). Analysis: Calcd. for $C_{14}H_{13}BrO_6$: C, 47.08; H, 3.67%. Found: C, 47.2; H, 3.5%.

Diethyl 4-(4-Bromophenyl)-5-hydroxy-2,5-dihydrofuran-2,3-dicarboxylate (3d)

Yield: 71%; White powder, mp 121–123°C, IR(KBr) (ν_{max}, cm^{-1}): 3415 (OH), 1728 (C=O). MS (m/z,%): 384 ($M^{\cdot+}$, 7). NMR data for *cis* isomer: ^1H NMR (250 MH_Z, $CDCl_3$):δ 1.26 and 1.30 (6 H, 2 t, $^3J_{HH} = 7$ H_Z, 2 CH_3), 4.20 and 4.24 (4 H, 2 q, $^3J_{HH} = 7$ H_Z, 2 OCH_2), 4.52 (1 H, broad s, OH), 5.57 (1 H, d, $^5J_{HH} = 5$ H_Z, C*H*), 6.14 (1 H, d, $^5J_{HH} = 5$ H_Z, C*H*), 7.40—7.50 (4 H, aromatic). ^{13}C NMR (62.9 MH_Z, $CDCl_3$):δ 14.4 and 15.6 (2 CH_3), 61.6 and 62.1 (2 O*C*H_2), 84.9, 111.3 (2 CH), 129.2, 146.6, 130.2, 130.9, 131.6 and 131.7 (aromatic and olefinic carbons), 169.3 and 169.7 (2 C=O). NMR data for *trans* isomer: ^1H NMR (250 MH_Z, $CDCl_3$): δ 1.15 and 1.20 (6 H, 2 t, $^3J_{HH} = 7$ H_Z, 2 CH_3), 3.61 and 3.79 (4 H, 2 q, $^3J_{HH} = 7$ H_Z, 2 OCH_2), 4.46 (1 H, broad s, OH), 5.41 (1 H, d, $^5J_{HH} = 1$ H_Z, C*H*), 5.98 (1 H, d, $^5J_{HH} = 1$ H_Z, C*H*), 7.40–7.50 (4 H, aromatic). ^{13}C NMR (62.9 MH_Z, $CDCl_3$):δ 14.3 and 14.5 (2 CH_3), 61.7 and 62.2 (2 O*C*H_2), 84.1, 110.6 (2 CH), 128.9, 145.6, 130.1, 130.8, 131.4 and 131.5 (aromatic and olefinic carbons), 165.3 and 165.6 (2 C=O). Analysis: Calcd. for $C_{16}H_{17}BrO_6$: C, 49.89; H, 4.45%. Found: C, 49.7; H, 4.6%.

Di-*t*-butyl 4-(4-Bromophenyl)-5-hydroxy-2,5-dihydrofuran-2,3-dicarboxylate (3e)

Yield: 80%; viscous oil, IR (KBr) (ν_{max}, cm^{-1}): 3395 (OH), 1726 (C=O). MS (m/z,%): 440 ($M^{\cdot+}$, 6). NMR data for *cis* isomer: ^1H NMR (250 MH_Z, $CDCl_3$): δ 1.39 (9 H, s, *t*-Bu), 1.51 (9 H, s, *t*-Bu), 4.43 (1 H, broad s, OH), 5.43 (1 H, d, $^5J_{HH} = 5$ H_Z, C*H*), 6.13 (1 H, d, $^5J_{HH} = 5$ H_Z, C*H*), 7.27–7.68 (4 H, aromatic). ^{13}C NMR (62.9 MH_Z, $CDCl_3$):δ 26.9 and 27.0 (6 CH_3 of 2 *t*-Bu), 81.3 and 81.4 (2 C of 2 *t*-Bu), 84.5, 110.0 (2 CH), 129.5, 144.0, 122.4.3, 129.1, 130.1 and 133.6 (aromatic and olefinic carbons), 160.4 and 167.6 (2 C=O). NMR data for *trans* isomer: ^1H NMR (250 MH_Z, $CDCl_3$): δ 1.40 (9 H, s, *t*-Bu), 1.47 (9 H, s, *t*-Bu), 4.46 (1 H, broad s, OH), 5.95 (1 H, d, $^5J_{HH} = 1$ H_Z, C*H*), 6.67 (1 H, d, $^5J_{HH} = 1$ H_Z, C*H*), 7.27–7.68 (4 H, aromatic). ^{13}C NMR (62.9 MH_Z, $CDCl_3$):δ 28.6 and 28.7 (6 CH_3 of 2 *t*-Bu), 81.1 and 81.2 (2 C of 2 *t*-Bu), 83.7, 109.2 (2 CH), 129.4, 143.8, 122.3, 129.1, 130.3 and 133.4 (aromatic and olefinic carbons), 160.6 and 167.1 (2 C=O). Analysis: Calcd. for $C_{20}H_{25}BrO_6$: C, 54.43; H, 5.71%. Found: C, 54.6; H, 5.8%.

Dimethyl 4-(4-Nitrophenyl)-5-hydroxy-2,5-dihydrofuran-2,3-dicarboxylate (3f)

Yield: 81%; viscous oil, IR (KBr) (ν_{max}, cm^{-1}): 3485 (OH), 1728 (C=O). MS (m/z,%): 323 ($M^{\cdot+}$, 3). NMR data for *cis* isomer: ^1H NMR (250 MH_Z, $CDCl_3$): δ 3.70 and 3.85 (6 H, 2 s, 2 OCH_3), 4.23 (1 H, broad s, OH), 5.69 (1 H, d, $^5J_{HH} = 4$ H_Z, C*H*), 6.16 (1 H, d, $^5J_{HH} = 4$ H_Z, C*H*), 7.26–7.25 (4 H, aromatic). ^{13}C NMR (62.9 MH_Z, $CDCl_3$):δ 52.6, 53.5 (2 OCH_3), 84.1, 106.5 (2 CH), 129.3, 149.3, 123.6, 130.2, 137.1 and 148.6 (aromatic and olefinic carbons), 162.1 and 172.5 (2 C=O). NMR data for *trans* isomer: ^1H NMR (250 MH_Z, $CDCl_3$): δ 3.73 and 3.87 (6 H, 2 s, 2 OCH_3), 4.38 (1 H, broad s, OH), 5.48 (1 H, d, $^5J_{HH} = 1$ H_Z, C*H*), 6.11 (1 H, d, $^5J_{HH} = 1$ H_Z, C*H*), 7.26–7.25 (4 H, aromatic). ^{13}C NMR (62.9 MH_Z, $CDCl_3$):δ 52.8, 53.9 (2 OCH_3), 84.3, 106.7 (2 CH), 129.2, 149.5, 123.7, 130.4, 137.2 and 148.8 (aromatic and olefinic carbons), 162.5 and 172.8 (2 C=O). Analysis: Calcd. for $C_{14}H_{13}NO_8$: C, 52.02; H, 4.05; N, 4.33%. Found: C, 52.2; H, 4.2; N, 4.5%.

Diethyl 4-(4-Nitrophenyl)-5-hydroxy-2,5-dihydrofuran-2,3-dicarboxylate (3g)

Yield: 77%; viscous oil, IR (KBr) (ν_{max}, cm^{-1}): 3440 (OH), 1742 (C=O). MS (m/z,%): 351 ($M^{\cdot+}$, 5). NMR data for *cis* isomer: ^1H NMR (250 MH_Z, $CDCl_3$):δ 1.16 and 1.29 (6 H, 2 t, $^3J_{HH} = 7$ H_Z, 2 CH_3), 4.16 and 4.25 (4 H, 2 q, $^3J_{HH} = 7$ H_Z, 2 OCH_2), 4.56 (1 H, broad s, OH), 5.57 (1 H, d, $^5J_{HH} = 4$ H_Z, C*H*), 6.52 (1 H, d, $^5J_{HH} = 4$ H_Z, C*H*), 7.72–8.28 (4 H, aromatic). ^{13}C NMR (62.9 MH_Z, $CDCl_3$):δ 13.0 and 13.1 (2 CH_3), 60.6 and 61.6 (2 O*C*H_2), 83.1, 104.7 (2 CH), 129.2, 147.2, 122.1, 128.0, 129.4 and 136.2 (aromatic and olefinic carbons), 161.2 and 168.7 (2 C=O). NMR data for *trans* isomer: ^1H NMR (250 MH_Z, $CDCl_3$): δ 1.20 and 1.35 (6 H, 2 t, $^3J_{HH} = 7$ H_Z, 2 CH_3), 4.20 and 4.31 (4 H, 2 q, $^3J_{HH} = 7$ H_Z, 2 OCH_2), 4.59 (1 H, broad s, OH), 5.47 (1 H, d, $^5J_{HH} = 1$ H_Z, C*H*), 6.12 (1 H, d, $^5J_{HH} = 1$ H_Z, C*H*), 7.72–8.28 (4 H, aromatic). ^{13}C NMR (62.9 MH_Z, $CDCl_3$):δ 12.8 and 12.9 (2 CH_3), 60.5 and 61.8 (2 O*C*H_2), 82.9, 105.2 (2 CH), 128.8, 147.5, 122.2, 128.2, 129.0 and 136.0 (aromatic and olefinic carbons), 160.6 and 170.9 (2 C=O). Analysis: Calcd. for $C_{16}H_{17}NO_8$: C, 54.70; H, 4.88; N, 3.99%. Found: C, 54.9; H, 4.7; N, 4.1%.

REFERENCES

1. F. M. Dean and M. V. Sargent, In *Comprehensive Heterocyclic Chemistry*, C. W. Bird and G. W. H. Cheeseman, Eds. (Pergamon, New York, 1984), vol. 4, 4, p. 531.
2. B. H. Lipshutz, *Chem. Rev.*, **86,** 795 (1986).
3. V. Nair, J. Mathew, and L. G. Nair, *Synth. Commun.*, **26,** 4531 (1996).
4. S. C. Roy and P. K. Mandal, *Tetrahedron*, **52,** 2193 (1996).
5. S. C. Roy and P. K. Mandal, *Tetrahedron*, **52,** 12495 (1996).
6. Y. R. Lee, N. S. Kim, and B. S. Kim, *Tetrahedron Lett.*, **38,** 5671 (1997).
7. M. C. Pirrung, J. Zhang, and A. T. McPhail, *J. Org. Chem.*, **56,** 6269 (1991).
8. H. M. L. Davies, G. Ahmed, R. L. Calvo, M. R. Churchill, and D. G. Churchill, *J. Org. Chem.*, **63,** 2641 (1998).
9. M. C. Pirrung and F. Blume, *J. Org. Chem.*, **64,** 3642 (1999).
10. A. A. Jaxa-Chamiec and P. G. Sammes, *J. Chem. Soc., Perkin Trans. 1*, 170 (1980).
11. H. Hagiwara, K. Sato, T. Suzuki, and M. Ando, *Tetrahedron Lett.*, **38,** 2103 (1997).

12. S. Arai, K. Nakayama, Y. Suzuki, K. Hatano, and T. Shioiri, *Tetrahedron Lett.*, **39,** 9739 (1998).
13. Y. Jiang and D. Ma, *Tetrahedron: Asymmetry*, **13,** 1033 (2002).
14. W. Cao, W. Ding, J. Chen, Y. Chen, Q. Zang, and G. Chen, *Synth. Commun.*, **34,** 1599 (2004).
15. Z. Yang, M. Fan, R. Mu, W. Liu, and Y. Liang, *Tetrahedron*, **61,** 9140 (2005).
16. C. P. Chuang and A. I. Tsai, *Synthesis*, 675 (2006).
17. F. Palacios, A. M. O. Retana, and J. Pagalday, *Tetrahedron*, **55,** 14451 (1999).
18. J. Caesar, D. V. Griffiths, P. A. Griffiths, and T. J. Tebby, *J. Chem. Soc., Perkin Trans. 1*, 2425 (1989).
19. A. Ramazani, A. R. Kazemizadeh, E. Ahmadi, N. Noshiranzadeh, and A. Souldozi, *Cur. Org. Chem.*, **12,** 59 (2008).
20. I. Yavari and A. Ramazani, *J. Chem. Res. (S)*, 382 (1996).
21. I. Yavari, M. Adib, and M. H. Sayahi, *Tetrahedron Lett.*, **43,** 2927 (2002).
22. I. Yavari and M. H. Mosslemin, *Tetrahedron*, **54**, **31,** 9169 (1998).
23. L. A. Evans, K. E. Griffiths, H. Guthmann, and P. J. Murphy, *Tetrahedron Lett.*, **43,** 299 (2002).
24. M. Anary-Abbasinejad, M. Mazraeh-Seffid, and A. Hassanabadi, *Arkivoc*, **12,** 265 (2008).
25. M. Anary-Abbasinejad, M. H. Mosslemin, A. Hassanabadi, and M. Tabatabaee, *Synth. Commun.*, **38**, **21,** 3700 (2008).
26. M. Kalantari, M. R. Islami, Z. Hassani, and K. Saidi, *Arkivoc*, **10,** 55 (2006).
27. R. Hekmatshoar, S. Javanshir, and M. M. Heravi, *J. Chem. Res.*, 60 (2007).
28. M. H. Mosslemin, I. Yavari, M. Anary-Abbasinejad, and M. R. Nateghi, *Synthesis*, **7,** 1029 (2004).
29. M. Anary-Abbasinejad and S. Tahan, *Phosphorus, Sulfur, and Silicon*, **2,** 315 (2007).
30. M. Anary-Abbasinejad, H. Anaraki-Ardakani, A. Dehghan, A. Hassanabadi, and M. R. Seyedmir, *J. Chem. Res.*, 574 (2007).
31. M. Anary-Abbasinejad, A. Hassanabadi, and M. Mazraeh-Seffid, *J. Chem. Res.*, 708 (2007).
32. H. Friebolin, *Basic One and Two-Dimensional NMR Spectroscopy* (Wiley-VCH, Weinheim, 1998), p. 96.
33. E. Breitmaier and W. Voelter, *Carbon-13 NMR Spectroscopy*, 3rd ed. (VCH, New York, 1990), pp. 213–220.