Selective Substitution Reactions of Methoxycarbonylamino-1-(1-benzotriazolyl)alkanes with Active Methylene Compounds

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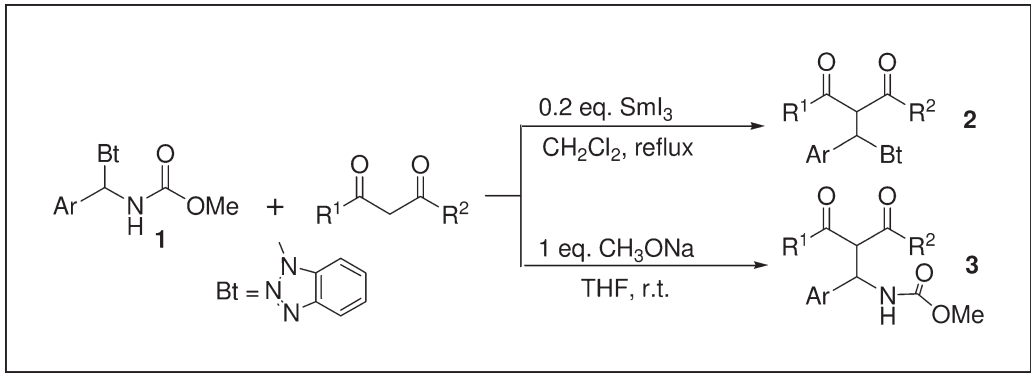

Benzotriazole adducts methoxycarbonylamino-1-(1-benzotriazolyl)alkanes **1** were derived from the condensation of an aldehyde, benzotriazole, and methylcarbamate. The leaving tendency of methoxycarbonylamino group (MeOCONH) and benzotriazole group (Bt) was investigated by treatment of the adducts with active methylene compounds under either Lewis acid-catalyzed or basic conditions. In the presence of SmI_3, MeOCONH take priority over Bt in the leaving process, whereas in the presence of MeONa, the Bt was substituted in preference. Thus, the tunable substitution of the two leaving groups could be used for different synthetic purposes.

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INTRODUCTION

Benzotriazole is a useful synthetic auxiliary due to the readily introduction and good leaving ability of the benzotriazolyl (Bt). A large number of benzotriazole derivatives have been reported as key intermediates for the synthesis of various compounds [1].

Benzotriazole derivatives of type Bt—C—OR was reported to be ionized [2] either by Bt—C bond scission to form alkyleneonium cations C^+—OR and Bt^- or by C—O bond scission to give RO^- anion and a benzotriazole stabilized carbocation Bt=C^+, depending on the substituents in the molecules. Recently, the selective substitution of Bt or amido group in *N*-(α-benzotriazolylalkyl)amides (Bt—C—NHCOR type) were achieved by using different Lewis acid catalysts. Promoted by $AlCl_3$ [3a] or SmI_3 [3b], the Bt could be substituted selectively by active methylene compounds. However, in the presence of $Dy(OTf)_3$, selective substitution of the amido group (—NHCOR) in the same substrates was found [3c]. The Bt in 1-(benzotriazole-1-yl)alkyl ester [4a] (Bt—C—OCOR type) could be selectively substituted by C-nucleophiles such as cyanide anion [4b] and the carbonanion from organozinc reagents [4c]. Interestingly, the acyloxy (—OCOR) in the same substrates could be selectively removed via SmI_2-mediated reduction [4d]. Nevertheless, examples concerning such conditions-dependent selectivity are limited and more examples for the selective scission of either Bt or the other concurrent leaving group are required so as to make it more applicable in organic synthesis.

RESULTS AND DISCUSSION

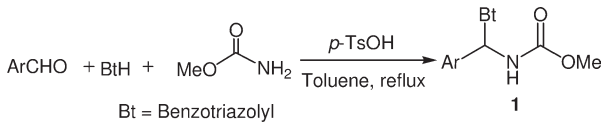
As a part of our continuing work in benzotriazole chemistry, we wish to report the selective substitution reactions of methoxycarbonylamino-1-(1-benzotriazolyl) alkanes **1** with active methylene compounds.

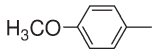
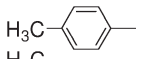
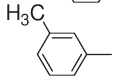
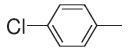
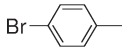
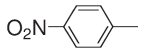
The substrates methoxycarbonylamino-1-(1-benzotriazoly)alkanes **1** could be facilely prepared by the condensation of an aldehyde, benzotriazole, and methylcarbamate (Table 1). They were obtained in moderate to good yields (Table 1, entries 1–8).

With compounds **1** in hand, we then tried to investigate the substitution reaction with active methylene compounds.

Table 1

Synthesis of Methoxycarbonylamino-1-(1-benzotriazoly)alkanes **1**.[a]



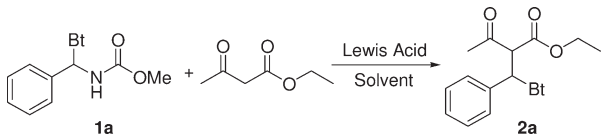
Entry	Ar	Compounds 1	Time (h)	Yields (%)[b]
1		**1a**	24	83
2		**1b**	24	56
3		**1c**	24	61
4		**1d**	24	60
5		**1e**	24	58
6		**1f**	24	72
7		**1g**	24	68
8		**1h**	24	70

[a] Reaction conditions: aldehyde (10 mmol), benzotriazole (10 mmol), methyl methyl-carbamate (10 mmol), toluene (30 mL), *p*-TsOH (0.1 mmol), reflux.
[b] Isolated yield.

Methyl *N*-(1-benzotriazol-1-phenylmethyl)carbamate **1a** and ethyl acetoacetate were initially used as the model substrates. The first attempt was performed in THF with SmI_3 (20 mol %) as the catalyst under reflux (Table 2, entry 1). Interestingly, in contrast with the previous results [3b], the major product now obtained was β,β-dicarbonyl derivatives **2a** (35% yield), where the methoxycarbonylamino (—NHCOOMe) was replaced and the Bt was reserved. We then tested other Lewis acid catalysts such as $AlCl_3$, and metal triflates (Table 2, entries 2–4).

Table 2

Substitution of **1a** with ethyl acetoacetate under different conditions.[a]

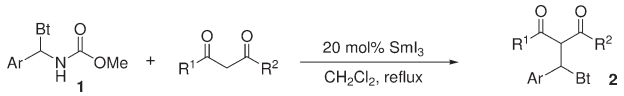


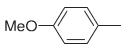
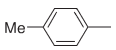
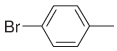
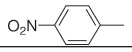
Entry	Catalyst (equiv.)	Solvent	Temperature (°C)	Time (h)	Yield (%)[b]
1	SmI_3 (0.2)	THF	Reflux	1	35
2	$AlCl_3$ (1.0)	THF	Reflux	12	19
3	$Zn(OTf)_2$ (0.2)	THF	Reflux	12	Trace
4	$Cu(OTf)_2$ (0.2)	THF	Reflux	12	Trace
5	SmI_3 (0.2)	CH_3CN	Reflux	1	37
6	SmI_3 (0.2)	Toluene	70	4	40
7	SmI_3 (0.2)	Dioxane	Reflux	2	48
8	SmI_3 (0.2)	CH_2Cl_2	Reflux	1	67
9	SmI_3 (0.2)	CH_2Cl_2	Room temperature	5	36

[a] Reaction conditions: acetoacetate (1.1 mmol), **1** (1 mmol), solvent (10 mL).
[b] Isolated yield.

Table 3

SmI_3-catalyzed nucleophilic substitution reaction of methoxycarbonyl-amino-1-(1-benzotriazoly)alkanes **1** with 1,3-dicarboyl compounds.[a]



Entry	Ar in 1	R^1	R^2	Time (h)	Products	Yields (%)[b]
1		Me-	EtO-	1	**2a**	67
2		Me-	MeO-	10	**2b**	70
3		Me-	Me-	24	**2c**	40
4		EtO-	EtO-	10	**2d**	58
5		Me-	MeO-	8	**2e**	63
6		Me-	EtO-	4	**2f**	54
7		Me-	EtO-	10	**2g**	59
8		Me-	MeO-	10	**2h**	60
9		Me-	MeO-	10	**2i**	65
10		EtO-	EtO-	10	**2j**	53
11		Me-	EtO-	10	–	–[c]
12		Me-	Me-	10	–	–[c]

[a] Reaction conditions: **1** (1 mmol), active methylene compounds (1 mmol), SmI_3 (0.2 mmol), solvent (10 mL), reflux.
[b] Isolated yield.
[c] Complete decomposition of compound **1**.

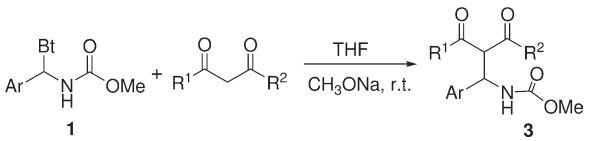
However, these acid catalysts did not give encouraging results and serious decomposition of substrate **1** to the original aldehyde, benzotriazole, and methyl carbamate was observed. Fortunately, screening of the solvents (Table 2, entries 5–8) and examination of the temperature (Table 2, entries 8–9) showed that refluxing in CH_2Cl_2 with 20 mol % of SmI_3 could afford **2a** in improved yield (67% yield) (Table 2, entry 8).

After the reaction conditions were optimized, various methoxycarbonylamino-1-(1-benzotri-azoly)alkanes **1** and active methylene compounds were used as the substrates to examine the scope and limitations. The results were summarized in Table 3. We were pleased to find a variety of active methylene compounds could smoothly react with **1** (Table 3, entries 1–10). Both β-keto esters and malonates were good C-nucleophiles for the reaction, whereas the substitutions with a β-diketone required longer reaction time and gave a lower yield (Table 3, entry 3). Compounds **1** bearing electron-donating groups (such as methyl and methoxy) on the phenyl ring could afford the desired products **2** in moderate yields (Table 3, entries 1–10). However, electron-withdrawing groups (such as bromo and nitro groups) led to the rapid decomposition of compounds **1**, and the desired products **2** could not be obtained in these cases (Table 3, entry 11 and entry 12).

Because Bt is also a good leaving group, it could be anticipated that the Bt might be substituted under suitable conditions. Recent studies showed that the Bt in benzyloxycarbonylamino-1-(1-benzotriazoly)alkanes could be smoothly substituted by *tert*-butyl acetates using lithium diisopropyl amine (LDA) as a base [5]. The above results inspired us to envision a selective substitution of Bt in **1** by the active methylene compounds via forming an enolate under proper basic conditions. We

Table 4

MeONa-promoted nucleophilic substitution of methyl (1H-benzotriazolylaryl)methylcarbamate with 1,3-dicarboyl compounds.[a]



Entry	Ar	R^1	R^2	Time (min)	Product 3	Yields (%)[b]
1		Me-	EtO-	45	**3a**	77
2		Me-	EtO-	60	**3b**	83
3		Me-	EtO-	35	**3c**	86
4		Me-	EtO-	40	**3d**	72
5		Me-	EtO-	120	**3e**	64
6		EtO-	EtO-	210	**3f**	65

[a] Reaction conditions: **1** (1 mmol), active methylene compounds (1 mmol), CH_3ONa (1 mmol), solvent (10 mL), room temperature.
[b] Isolated yields.

found that this substitution could proceed successfully in the presence of MeONa. Generally, moderate to good yield of the expected β-amino acid derivatives **3** could be obtained within 3.5 h despite the electronic effect (Table 4).

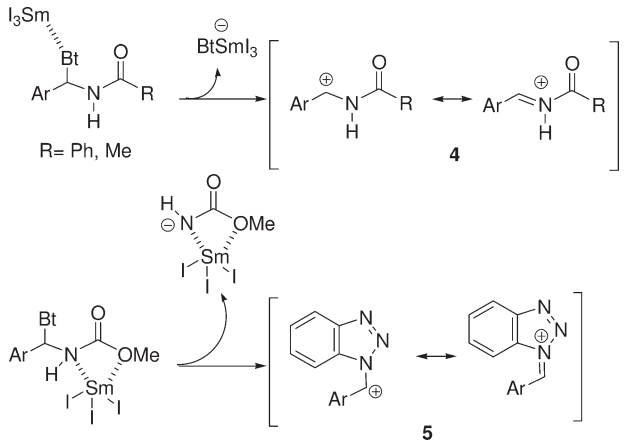
Very recently, the reaction between *N*-(α-amidoalkyl)-benzotriazoles and 1,3-diketones-dervied potassium enolates was used for the preparation of the β-amido β-diketones by the loss of Bt [6]. Combination with Katritzky's research [5a] and ours, it could be concluded that Bt is a better leaving group than either amido or alkoxycarbonylamino because the enolates consistently substitute the Bt in the three types of substrates. In the base conditions, the above reactions proceeded more probably via S_N2 mechanism. However, in the presence of SmI_3, the benzotriazole derivatives should ionize either to the benzotriazole anion and cation **4** [5b] or to the alkoxycarbonylamino anion and cation **5** [2] depending upon the substrate structures (Scheme 1). In contrast with the Bt—C—NHCOR stucutre [3b], which tended to ionize to cation **4** in the presence of SmI_3, good chelation may exist for the Bt—C—NHCOOMe moeity because samarium salt was reported to coordinate well with both O— and N— donors [7]. Hence, the leaving ability of —NHCOOMe was effectively enhanced until an alternative ionization mode to form cation **5** predominated. Attack of cation **5** with active methylene compounds afforded product **2**.

In summary, selective substitution of either NHCOOMe or Bt in *N*-(α-benzotriazol-1-ylalkyl) methylcarbamates was achieved by performing the reaction under different conditions. With SmI_3 as the Lewis acid catalyst, NHCOOMe could be selectively substituted by the active methylenyl group, and the benzotriazole derivatives could be obtained in moderate to good yields; whereas using MeONa as the base, the Bt was substituted preferentially and the β-amino acid derivatives were prepared under mild conditions. The tunable leaving tendency of the two leaving groups may be useful for other synthetic purposes.

EXPERIMENTAL

Methylene chloride was distilled from calcium hydride immediately before use. Melting points are uncorrected. ^{1}H-NMR

Scheme 1. Proposed mechanism for the selective substitution of —NHCOOMe with SmI_3 as a catalyst.



(400 MHz) spectra were recorded on a Bruker AV400 NMR instrument as $CDCl_3$ solutions using tetramethyl silane (TMS) as internal standard. Chemical shifts (σ) are reported in parts per million (ppm) and coupling constants J are given in hertz. IR spectra were recorded in film or using KBr disks with a Nicolet Nexus 670 FTIR spectrometer. Mass spectra were recorded on a HP 5989B MS spectrometer (70 eV). Elemental analyses were performed on a Vario-ELIII instrument. Compounds **1** were prepared using the method analogous to that for the preparation of benzyloxycarbonylamino-1-(1-benzotriazolyl)alkanes [8].

General procedure for the preparation of β,β-dicarbonyl benzotriazole derivatives 2. An oven-dried 50 mL flask was charged with samarium powder (0.03 g, 0.2 mmol), anhydrous THF (10 mL), and I_2 (0.076 g, 0.3 mmol). The mixture was stirred at room temperature for 1 h and concentrated *in vacuo*. Then anhydrous CH_2Cl_2 (10 mL), 1,3-dicarbonyl compounds (1 mmol) and *N*-(1-benzotriazol-1-ylalky1)carbamate derivatives **1** (1 mmol) were added. The mixture was stirred under reflux until the disappearance of **1** was observed [reaction monitored by thin-layer chromatography (TLC)]. The reaction was quenched with aq. HCl (0.1*M*, 3 mL) and extracted with diethyl ether (3× 10 mL). The combined organic extracts were washed with saturated $Na_2S_2O_3$ (5 mL), then with brine, and were dried over anhydrous Na_2SO_4. Removal of the solvent under reduced pressure afforded the crude product, which was purified by preparative TLC on silica gel using ethyl acetate/petroleum ether (1/4, v/v) as eluent to give the corresponding benzotriazole derivatives **2**.

General procedure for the preparation of 3. A mixture of **1** (1 mmol), active methylene compounds (1 mmol), and CH_3ONa (1 mmol) in THF (5 mL) was stirred until the disappearance of **1** (monitored by TLC). Then the reaction was quenched with H_2O and extracted with ethyl acetate (3× 10 mL). The combined organic extracts were washed with saturated Na_2CO_3, then with brine, and were dried over anhydrous Na_2SO_4, then filtrated and concentrated under reduced pressure. The residue was purified by preparative TLC on silica gel to give product **3**.

Methyl (1H-benzo[d][1,2,3]triazol-1-yl)(phenyl) methylcarbamate (1a). White solid, mp 120–122°C (123–124°C) [8]. See Table 1, entry 1.

Methyl (1H-benzo[d][1,2,3]triazol-1-yl)(4-methoxy-phenyl)-methylcarbamate (1b). White solid, mp 156–158°C. IR (KBr): 3414, 3193, 1715, 1546, 1400, 1252 cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.07 (d, J = 8.4 Hz, 1H), 7.63–7.57 (m, 2H), 7.45–7.37 (m, 2H), 7.19 (d, J = 8.4 Hz, 2H), 6.84 (d, J = 6.8 Hz, 2H), 6.50 (s, 1H), 3.77(s, 3H), 3.68 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 160.2, 156.0, 145.9, 132.5, 128.0, 127.8, 127.7, 124.3, 120.1, 114.4, 109.8, 67.1, 55.4., 52.9. Anal. Calcd. for $C_{16}H_{16}N_4O_3$: C, 61.53; H, 5.16; N, 17.94. Found: C, 61.62; H, 5.15; N, 17.90. See Table 1, entry 2.

Methyl (1H-benzo[d][1,2,3]triazol-1-yl)(p-tolyl)-methylcarbamate (1c). White solid, mp 154–157°C. IR (KBr): 3413, 3212, 1732, 1532, 1400, 1241 cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.08 (d, J = 8.0 Hz, 1H), 7.61 (d, J = 9.6 Hz, 1H), 7.58–7.36 (m, 4H), 7.15 (m, 4H), 6.35 (s, 1H), 3.71 (s, 3H), 2.32 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 155.1, 146.0, 139.4, 129.8, 127.8, 126.7, 126.2, 124.3, 120.1, 118.5, 109.8, 67.3, 52.5, 21.1. Anal. Calcd. for $C_{16}H_{16}N_4O_2$: C, 64.85; H, 5.44; N, 18.91. Found: C, 64.62; H, 5.45; N, 18.94. See Table 1, entry 3.

Methyl (1H-benzo[d][1,2,3]triazol-1-yl)(m-tolyl)-methylcarbamate (1d). White solid, mp 168–170°C. IR (KBr): 3337, 3131, 1702, 1530, 1400, 1234 cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.09 (d, J = 8.0 Hz, 1H), 7.63–7.60 (m, 2H), 7.47–7.36 (m, 2H), 7.26–7.06 (m, 4H), 6.42 (s, 1H), 3.70 (s, 3H), 2.30 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 155.8, 145.9, 139.1, 130.1, 129.0, 127.9, 126.9, 126.7, 124.3, 123.3, 120.1, 118.5, 109.8, 66.9, 52.9, 21.4. Anal. Calcd. for $C_{16}H_{16}N_4O_2$: C, 64.85; H, 5.44; N, 18.91. Found: C, 65.05; H, 5.42; N, 18.87. See Table 1, entry 4.

Methyl (1H-benzo[d][1,2,3]triazol-1-yl)(o-tolyl-methylcarbamate (1e). White solid, mp 153–156°C. IR (KBr): 3290, 3133, 1718, 1531, 1400, 1246 cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.07 (d, J = 8.0 Hz, 1H), 7.74 (d, J = 9.6 Hz, 1H), 7.50–7.27 (m, 3H), 7.26–7.14 (m, 4H), 6.39 (s, 1H), 3.70 (s, 3H), 2.37 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 155.9, 146.0, 135.8, 134.1, 132.4, 131.2, 129.5, 127.8, 126.7, 125.8, 124.3, 120.1, 109.8, 65.2, 53.0, 19.2. Anal. Calcd. for $C_{16}H_{16}N_4O_2$: C, 64.85; H, 5.44; N, 18.91. Found: C, 64.98; H, 5.43; N, 18.96. See Table 1, entry 5.

Methyl (1H-benzo[d][1,2,3]triazol-1-yl)(4-chloro-phenyl)-methylcarbamate (1f). White solid, mp 154–156°C. IR (KBr): 3295, 3023, 1718, 1549, 1400, 1250 cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.90–7.87 (m, 2H), 7.65 (s, 1H), 7.43–7.41 (m, 2H), 7.31 (d, J = 8.4 Hz, 2H), 7.18 (d, J = 8.4 Hz, 2H), 6.40 (s, 1H), 3.74 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 156.3, 145.5, 134.8, 132.4, 128.3, 127.8, 127.1, 124.5, 123.7, 118.6, 109.2, 73.0, 53.2. Anal. Calcd. for $C_{15}H_{13}ClN_4O_2$: C, 56.88; H, 4.14; N, 17.69. Found: C, 57.06; H, 4.13; N, 17.73. See Table 1, entry 6.

Methyl (1H-benzo[d][1,2,3]triazol-1-yl)(4-bromo-phenyl)-methylcarbamate (1g). White solid, mp 158–160°C. IR (KBr): 3272, 3019, 1720, 1535, 1400, 1241 cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.89–7.87 (m, 2H), 7.65 (s, 1H), 7.47 (d, J = 8.4Hz, 2H), 7.42–7.40 (m, 2H), 7.12 (d, J = 8.4 Hz, 2H), 6,45 (s,1H), 3.74 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 156.2, 144.3, 135.4, 132.2, 128.1, 127.8, 127.0, 124.5, 123.7, 118.5, 109.5, 73.6, 53.1. Anal. Calcd. for $C_{15}H_{13}BrN_4O_2$: C, 49.88; H, 3.63; N, 15.51. Found: C, 49.73; H, 3.64; N, 15.55. See Table 1, entry 7.

Methyl (1H-benzo[d][1,2,3]triazol-1-yl)(4-nitro-phenyl)methylcarbamate (1h). White solid, mp 147–150°C. IR (KBr): 3278, 3043, 1717, 1545, 1400, 1239 cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.21 (d, J = 8.8 Hz, 2H), 8.11 (d, J = 8.4 Hz, 1H), 7.77 (d, J = 9.6 Hz, 1H), 7.66–7.42 (m, 5H), 6.61 (d, J = 9.6 Hz, 1H), 3.73 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 156.2, 147.6, 137.5, 134.1, 130.9, 128.5, 127.7, 124.8, 124.2, 120.3, 109.4, 66.1, 53.3. Anal. Calcd. for $C_{15}H_{13}N_5O_4$: C, 55.05; H, 4.00; N, 21.40. Found: C, 55.21; H, 4.01; N, 21.34. See Table 1, entry 8.

Ethyl 2-((1H-benzo[d][1,2,3]triazol-1-yl)(phenyl)-methyl)-3-oxobutanoate (2a). White solid, mp 137–139°C. IR (KBr): 3414, 1743, 1717, 1618, 1400 cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.98 (d, J = 8.4 Hz, 1H), 7.48–7.45 (m, 3H), 7.42–7.40 (m, 1H), 7.30–7.28 (m, 4H), 6.43 (d, J = 11.2 Hz, 1H), 5.45 (d, J = 11.2 Hz, 1H), 4.06–4.03 (m, 2H), 2.40 (s, 3H), 1.07 (t, J = 7.2 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 199.2, 165.5, 146.1, 135.9, 132.9, 129.1, 129.0, 128.9, 127.9, 127.6, 124.3, 119.8, 109.9, 64.1, 62.1, 61.0, 30.3, 13.8. Anal. Calcd. for $C_{19}H_{19}N_3O_3$: C, 67.64; H, 5.68; N, 12.46. Found: C, 67.78; H, 5.66; N, 12.43. MS (ES): *m/z* 337.9, ([M + Na]$^+$) (ES): ([M + Na]$^+$): 359.8. See Table 3, entry 1.

Methyl 2-((1H-benzo[d][1,2,3]triazol-1-yl)(phenyl)-methyl)-3-oxobutanoate (2b). White solid, mp 103–106°C. IR (KBr): 3129, 1745, 1720, 1617, 1401 cm^{-1}. Major: ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.98 (d, *J* = 8.4 Hz, 1H), 7.49–7.27 (m, 8H), 6.44 (d, *J* = 11.2 Hz, 1H), 5.46 (d, *J* = 11.2 Hz, 1H), 3.60 (s, 3H), 2.41 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 199.2, 165.9, 146.5, 136.0, 129.1, 129.0, 127.8, 127.7, 124.3, 119.9, 109.9, 63.9, 61.0, 53.0, 30.4.

Minor: ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.98 (d, *J* = 8.4 Hz, 1H), 7.49–7.27 (m, 8H), 6.41 (d, *J* = 11.2 Hz, 1H), 5.46 (d, *J* = 11.2 Hz, 1H), 3.60 (s, 3H), 2.12 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 199.2, 166.4, 146.5, 135.7, 129.1, 129.0, 127.7, 127.6, 124.2, 119.8, 109.9, 109.7, 63.4, 61.2, 53.0, 31.9. Anal. Calcd. for $C_{18}H_{17}N_3O_3$: C, 66.86; H, 5.30; N, 13.00. Found: C, 66.65; H, 5.31; N, 13.03. MS (ES): *m/z* 323.7, ($[M + Na]^+$) (ES): ($[M + Na]^+$): 345.7. See Table 3, entry 2.

3-((1H-Benzo[d][1,2,3]triazol-1-yl)(phenyl)methyl)-pentane-2,4-dione (2c). White solid, mp 146–148°C. IR (KBr): 3131, 1743, 1721, 1696, 1406 cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.83–7.81 (m, 2 H), 7.50–7.47 (m, 2H), 7.36–7.30 (m, 5H), 6.72 (d, *J* = 11.6 Hz, 1H), 5.53 (d, *J* = 11.6 Hz, 1H), 2.23 (s, 3H), 2.09 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 199.6, 199.4, 144.3, 135.8, 129.3, 129.1, 127.7, 126.6, 118.2, 109.6, 72.9, 68.6, 30.8, 29.4. Anal. Calcd. for $C_{18}H_{17}N_3O_2$: C, 70.34; H, 5.58; N, 13.67. Found: C, 70.46; H, 5.56; N, 13.65. MS (ES): *m/z* 307, ($[M + Na]^+$). (ES): ($[M + Na]^+$): 329.7. See Table 3, entry 3.

Diethyl 2-((1H-benzo[d][1,2,3]triazol-1-yl)(phenyl)-methyl)-malonate (2d). White solid, mp 105–108°C. IR (KBr): 3132, 1745, 1721, 1400 cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.01 (d, *J* = 8.4 Hz, 1H), 7.56–7.50 (m, 3H), 7.45–7.43 (m, 1H), 7.34–7.30 (m, 4H), 6.39 (d, *J* = 11.6 Hz, 1H), 5.17 (d, *J* = 11.6 Hz, 1H), 4.09–4.00 (m, 4H), 1.07 (t, *J* = 7.2 Hz, 3H), 1.01 (t, *J* = 7.2 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 166.3, 166.2, 146.1, 135.4, 133.0, 129.2, 128.9, 128.0, 127.6, 124.2, 120.0, 109.7, 62.2, 62.1, 61.6, 57.2, 13.8, 13.7. Anal. Calcd. for $C_{20}H_{21}N_3O_4$: C, 65.38; H, 5.76; N, 11.44. Found: C, 65.61; H, 5.66; N, 11.45. MS (ESI): *m/z* 367.8, ($[M + Na]^+$) (ESI): 389.9. See Table 3, entry 4.

Methyl 2-((1H-benzo[d][1,2,3]triazol-1-yl)(4-meth-oxyphenyl)methyl)-3-oxobutanoate (2e). White solid, mp 102–104°C. IR (KBr): 3135, 1744, 1719, 1401 cm^{-1}. Major: ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.99 (d, *J* = 8.4 Hz, 1H), 7.46–7.26 (m, 5H), 6.80 (d, *J* = 8.4 Hz, 1H), 6.39 (d, *J* = 11.2 Hz, 1H), 5.42 (d, *J* = 11.2 Hz, 1H), 3.73 (s, 3H), 3.62 (s, 3H), 2.40 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 199.4, 166.0, 159.9, 146.1, 132.8, 129.0, 127.9, 127.6, 124.3, 119.8, 114.2, 110.0, 64.0, 62.1, 55.2, 53.1, 30.5.

Minor: ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.99 (d, *J* = 8.4 Hz, 1H), 7.46–7.26 (m, 5H), 6.80 (d, *J* = 8.4 Hz, 1H), 6.34 (d, *J* = 11.2 Hz, 1H), 5.42 (d, *J* = 11.2 Hz, 1H), 3.73 (s, 3H), 3.59 (s, 3H), 2.13 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 200.4, 166.5, 159.9, 145.9, 132.8, 129.1, 127.9, 127.6, 124.2, 119.8, 114.4, 109.8, 63.4, 60.7, 55.3, 53.0, 30.5. Anal. Calcd. for $C_{19}H_{19}N_3O_4$: C, 64.58; H, 5.42; N, 11.89. Found: C, 64.88; H, 5.44; N, 11.87. MS (ES): *m/z* 352.9, ($[M + Na]^+$) (ES): 386.9. See Table 3, entry 5.

Ethyl 2-((1H-benzo[d][1,2,3]triazol-1-yl)(p-tolyl)-methyl)-3-oxobutanoate (2f). White solid, mp 124–127°C. IR (KBr): 3130, 1743, 1717, 1400 cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.98 (d, *J* = 8.0 Hz, 1H), 7.50 (d, *J* = 8.0 Hz, 1H), 7.42–7.27 (m, 4H), 7.09 (d, *J* = 7.6 Hz, 2H), 6.40 (d, *J* = 11.2 Hz, 1H), 5.43 (d, *J* = 11.2 Hz, 1H), 4.08–4.04 (m, 2 H), 2.40 (s, 3H), 2.27 (s, 3H), 1.10 (d, *J* = 11.2 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 199.4, 165.5, 138.9, 133.0, 129.5, 127.7, 127.6, 124.2, 119.8, 110.0, 64.1, 62.1, 60.8, 30.4, 21.1, 13.8. Anal. Calcd. for $C_{20}H_{21}N_3O_3$: C, 68.36; H, 6.02; N, 11.96. Found: C, 68.54; H, 6.04; N, 12.01. MS (ES): *m/z* 353, ($[M + Na]^+$) (ESI): 389.9. See Table 3, entry 6.

Ethyl 2-((1H-benzo[d][1,2,3]triazol-1-yl)(m-tolyl)-methyl)-3-oxobutanoate (2g). Colorless oil. IR (KBr): 3435, 1745, 1721, 1608 cm^{-1}. Major: ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.98 (d, *J* = 8.4 Hz, 1H), 7.55–7.41 (m, 2H), 7.31–7.06 (m, 5H), 6.37 (d, *J* = 11.2 Hz, 1H), 5.44 (d, *J* = 11.2 Hz, 1H), 4.07–4.00 (m, 2H), 2.40 (s, 3H), 2.28 (s, 3H), 1.08 (t, *J* = 7.2 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 199.3, 165.5, 146.1, 138.7, 135.8, 132.9, 129.9, 128.7, 128.3, 127.6, 125.0, 124.2, 119.8, 109.9, 64.1, 62.1, 61.1, 30.3, 21.4, 13.8.

Minor: ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.98 (d, *J* = 8.4 Hz, 1H), 7.55–7.41 (m, 2H), 7.31–7.06 (m, 5H), 6.35 (d, *J* = 11.2 Hz, 1H), 5.44 (d, *J* = 11.2 Hz, 1H), 4.07–4.00 (m, 2H), 2.28 (s, 3H), 2.15 (s, 3H), 1.03 (t, *J* = 6.8 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 200.2, 165.8, 145.9, 138.9, 135.7, 129.8, 128.9, 128.3, 127.6, 125.0, 124.2, 119.8, 109.7, 63.8, 62.1, 60.9, 31.8, 21.3, 13.7. Anal. Calcd. for $C_{20}H_{21}N_3O_3$: C, 68.36; H, 6.02; N, 11.96. Found: C, 68.45; H, 6.01; N, 11.98. MS (ES): *m/z* 351.9, ($[M + Na]^+$) (ESI): 373.8. See Table 3, entry 7.

Methyl 2-((1H-benzo[d][1,2,3]triazol-1-yl)(m-tolyl)-methyl)-3-oxobutanoate (2h). White solid, mp 122–125°C. IR (KBr): 3130, 1748, 1717, 1400 cm^{-1}. Major: ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.02–8.00 (m, 1H), 7.55–7.39 (m, 2H), 7.32–7.16 (m, 5H), 7.07 (d, *J* = 8.0 Hz, 1H), 6.43–6.36 (m, 1H), 5.45 (d, *J* = 11.2 Hz, 1H), 3.62 (s, 3H), 2.41 (s, 3H), 2.28 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 200.2, 166.0, 146.1, 138.8, 135.9, 129.9, 128.7, 128.1, 127.6, 124.9, 124.3, 119.8, 110.0, 63.9, 61.0, 53.0, 30.5, 21.4.

Minor: ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.00–8.02 (m, 1H), 7.55–7.39 (m, 2H), 7.32–7.16 (m, 5H), 7.07 (d, *J* = 8.0 Hz, 1H), 6.43–6.36 (m, 1H), 5.45 (d, *J* = 11.2 Hz, 1H), 3.59 (s, 3H), 2.28 (s, 3H), 2.12 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 199.3, 166.5, 146.1, 139.0, 135.6, 130.0, 129.0, 128.3, 128.1, 127.6, 124.9, 124.2, 119.8, 109.8, 63.4, 61.2, 52.9, 31.9, 21.4. Anal. Calcd. for $C_{19}H_{19}N_3O_3$: C, 67.64; H, 5.68; N, 12.46. Found: C, 67.80; H, 5.67; N, 12.44. MS (ES): *m/z* 337.9, ($[M + Na]^+$) (ESI): 359.7. See Table 3, entry 8.

Methyl 2-((1H-benzo[d][1,2,3]triazol-1-yl)(o-tolyl)-methyl)-3-oxobutanoate (2i). White solid, mp 98–101°C. IR (KBr): 3434, 1745, 1720, 1450 cm^{-1}. Major: ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.97 (d, *J* = 8.8 Hz, 1H), 7.53–7.30 (m, 4H), 7.18–7.15 (m, 3H), 6.75 (d, *J* = 11.2 Hz, 1H), 5.51 (d, *J* = 11.2 Hz, 1H), 3.56 (s, 3H), 2.64 (s, 3H), 2.38 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 199.3, 166.0, 146.0, 136.2, 133.9, 132.8, 131.0, 129.0, 127.9, 127.6, 126.9, 124.2, 119.9, 109.8, 63.9, 56.5, 52.9, 30.4, 19.7.

Minor: ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.98 (d, *J* = 8.8 Hz, 1H), 7.53–7.30 (m, 4H), 7.18–7.15 (m, 3H), 6.70 (d, *J* = 11.2 Hz, 1H), 5.50 (d, *J* = 11.2 Hz, 1H), 3.58 (s, 3H), 2.63 (s, 3H), 2.10 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 200.1, 166.7, 146.0, 136.2, 134.0, 132.7, 131.2, 129.0, 127.9, 127.6, 127.0, 124.2, 119.9, 109.6, 63.2, 56.5, 53.0, 32.0, 19.8. Anal. Calcd. for $C_{19}H_{19}N_3O_3$: C, 67.64; H, 5.68; N, 12.46. Found: C, 67.53; H, 5.69; N, 12.45. MS (ES): *m/z* 337.8, ($[M + Na]^+$) (ES): 359.8. See Table 3, entry 9.

Diethyl 2-((1H-benzo[d][1,2,3]triazol-1-yl)(o-tolyl)-methyl)-malonate (2j). Colorless oil. IR (KBr): 3440, 1748, 1716, 1400 cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.01 (d, J = 8.4 Hz, 1H), 7.69 (d, J = 8.0 Hz, 1H), 7.53–7.31 (m, 3H), 7.17–7.02 (m, 3H), 6.73 (d, J = 10.0 Hz, 1H), 5.19 (d, J = 10.0 Hz, 1H), 4.14–3.69 (m, 4H), 2.49 (s, 3H), 1.04–0.97 (m, 6H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 166.6, 166.1, 145.8, 136.2, 133.6, 133.0, 130.9, 129.0, 128.3, 127.6, 126.8, 124.1, 119.9, 109.7, 62.2, 62.0, 57.2, 57.1, 19.8, 13.9, 13.6. Anal. Calcd. for $C_{21}H_{23}N_3O_4$: C, 66.13; H, 6.08; N, 11.02; Found: C, 66.40; H, 6.06; N, 11.00. MS (ES): *m/z* 381.8, ($[M + Na]^+$) (ESI): 404.0. See Table 3, entry 10.

Ethyl 2-((4-bromophenyl)(methoxycarbonyl)methyl)-3-oxobutanoate (3a). IR (KBr): 3306, 1749, 1716, 1694 cm^{-1}; Major: ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.45–7.42 (m, 2H), 7.19–7.16 (m, 2H), 6.15 (m, 1H), 5.40 (m, 1H), 4.17–4.04 (m, 2H), 4.00 (m, 1H), 3.63 (s, 3H), 2.16 (s, 3H), 1.20–1.13 (m, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 200.9, 167.8, 156.5, 138.7, 131.8, 128.0, 121.8, 63.9, 62.0, 53.9, 52.5, 29.0, 13.9.

Minor: ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.45–7.42 (m, 2H), 7.19–7.16 (m, 2H), 6.40 (m, 1H), 5.52 (m, 1H), 4.17–4.04 (m, 2H), 3.93 (m, 1H), 3.62 (s, 3H), 2.30 (s, 3H), 1.20–1.13 (m, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 200.2, 166.5, 156.5, 138.7, 131.8, 128.4, 121.8, 62.8, 61.8, 53.9, 52.4, 29.0, 13.9. Anal. Calcd. for $C_{15}H_{17}BrO_5$: C, 50.44; H, 4.80; Found: C, 50.61; H, 4.79. See Table 4, entry 1.

Ethyl 2-((methoxycarbonyl)(4-methoxyphenyl)-methyl)-3-oxobutanoate (3b). IR (KBr): 3316, 1744, 1713, 1697 cm^{-1}. Major: ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.26–7.20 (m, 2H), 6.85–6.82 (m, 2H), 6.10 (d, J = 0.8 Hz, 1H), 5.38 (m, 1H), 4.13–4.06 (m, 2H), 4.00 (d, J = 0.8 Hz, 1H), 3.77 (s, 3 H), 3.65 (s, 3H), 2.30 (s, 3H), 1.19–1.14 (m, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 200.9, 167.5, 159.0, 156.5, 131.6, 127.4, 114.0, 63.4, 61.8, 55.2, 53.9, 52.4, 28.8, 13.9.

Minor: ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.26–7.20 (m, 2 H), 6.85–6.82 (m, 2H), 6.30 (m, 1H), 5.50 (s, 1H), 4.13–4.06 (m, 2H), 3.90 (m, 1H), 3.78 (s, 3H), 3.63 (s, 3H), 2.17 (s, 3H), 1.19–1.14 (m, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 200.9, 169.0, 159.0, 156.5, 131.5, 127.4, 114.0, 64.3, 61.6, 55.2, 53.9, 52.4, 28.8, 13.9. Anal. Calcd. for $C_{16}H_{20}O_6$: C, 62.33; H, 6.54; Found: C, 62.25; H, 6.55. See Table 4, entry 2.

Ethyl 2-((methoxycarbonyl)(o-tolyl)methyl)-3-oxobutanoate (3c). IR (KBr): 3334, 1740, 1717, 1692 cm^{-1}. Major: ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.23–7.12 (m, 4H), 6.06 (d, J = 9.2, 1H), 5.68 (m, 1H), 4.12–4.04 (m, 2H), 3.97 (d, J = 7.6 Hz, 1H), 3.57 (s, 3H), 2.47 (s, 3H), 2.18 (s, 3H), 1.13 (t, J = 7.2 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 200.8, 167.1, 156.2, 137.8, 134.8, 130.8, 127.8, 126.4, 125.6, 62.8, 61.7, 52.3, 50.7, 29.0, 19.3, 13.8.

Minor: ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.24–7.13 (m, 4H), 6.49 (m, 1H), 5.68 (m, 1H), 4.12–4.04 (m, 2H), 3.84 (d, J = 5.2 Hz, 1H), 3.60 (s, 3H), 2.49 (s, 3H), 2.26 (s, 3H), 1.08 (t, J = 7.2 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 168.5, 167.1, 156.2, 137.7, 134.8, 130.8, 127.8, 126.4, 126.0, 62.9, 61.6, 52.3, 50.1, 30.2, 19.2, 13.9. Anal. Calcd. for $C_{16}H_{20}O_5$: C, 65.74; H, 6.90; Found: C, 65.90; H, 6.88. See Table 4, entry 3.

Ethyl 2-((methoxycarbonyl)(m-tolyl)methyl)-3-oxobutanoate (3d). IR (KBr): 3339, 1745, 1716, 1701 cm^{-1}. Major: ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.17–7.04 (m, 4H), 6.18 (s, 1H), 5.40 (s, 1H), 4.09–4.01 (m, 3H), 3.62 (s, 3H), 2.30 (s, 3H), 2.15 (s, 3H), 1.15–1.09 (m, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 203.1, 167.2, 156.3, 139.3, 138.3, 128.6, 127.0, 123.5, 123.2, 63.4, 61.8, 54.4, 52.3, 29.0, 21.4, 13.9.

Minor: ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.17–7.04 (m, 4H), 6.40 (s, 1H), 5.54 (s, 1H), 4.09–4.01 (m, 3H), 3.60 (s, 3H), 2.30 (s, 3H), 2.29 (s, 3H), 1.15–1.09 (m, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 201.2, 168.3, 156.3, 139.4, 138.3, 128.5, 127.3, 123.5, 123.2, 64.2, 61.5, 53.3, 52.2, 30.4, 21.4, 13.9. Anal. Calcd. for $C_{16}H_{20}O_5$: C, 65.74; H, 6.90; Found: C, 65.85; H, 6.91. See Table 4, entry 4.

Ethyl 2-((methoxycarbonyl)(4-nitrophenyl)methyl)-3-oxobutanoate (3e). IR (KBr): 3349, 1732, 1715, 1698 cm^{-1}. Major: ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.17–8.13 (m, 2H), 7.51–7.48 (m, 2H), 6.33 (d, J = 0.8 Hz, 1H), 5.50 (m, 1H), 4.14–4.01 (m, 3H), 3.63 (s, 3H), 2.32 (s, 3H), 1.17 (t, J = 7.2 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 200.3, 167.8, 166.5, 156.4, 147.4, 147.0, 127.7, 123.9, 63.4, 62.4, 53.8, 52.9, 30.7, 13.9.

Minor: ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.17–8.13 (m, 2H), 7.51–7.48 (m, 2H), 6.50 (m, 1H), 5.60 (m, 1H), 4.14–4.01 (m, 3H), 3.61 (s, 3H), 2.17 (s, 3H), 1.10 (t, J = 7.2 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 202.5, 167.8, 166.5, 156.4, 147.3, 146.8, 127.4, 123.8, 63.4, 62.3, 53.8, 52.6, 29.0, 13.9. Anal. Calcd. for $C_{15}H_{17}NO_7$: C, 55.73; H, 5.30; N, 4.33; Found: C, 55.82; H, 5.29; N, 4.34. See Table 4, entry 5.

Diethyl 2-((methoxycarbonyl)(4-nitrophenyl)-methyl)-malonate (3f). IR (KBr): 3358, 1715, 1710, 1700 cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.16 (d, J = 8.8 Hz, 2H), 7.50 (d, J = 8.4 Hz, 2H), 6.5 (m, 1H), 5.6 (m, 1H), 4.21–4.03 (m, 4H), 3.90 (m, 1H), 3.64 (s, 3H), 1.22 (t, J = 7.2 Hz, 3H), 1.13 (t, J = 7.2 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 167.5, 166.4, 156.6, 147.5, 146.7, 127.5, 123.8, 62.3, 62.0, 56.2, 53.6, 52.5, 13.9, 13.8. Anal. Calcd. for $C_{16}H_{19}NO_8$: C, 54.39; H, 5.42; N, 3.96; Found: C, 54.29; H, 5.43; N, 3.95. See Table 4, entry 6.

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