Investigation of the Reaction between 3-Benzisothiazolones, an Isoindole Isoster and Activated Acetylenes: Synthesis of Heterocyclic Backbones for Building Bioactive Molecules

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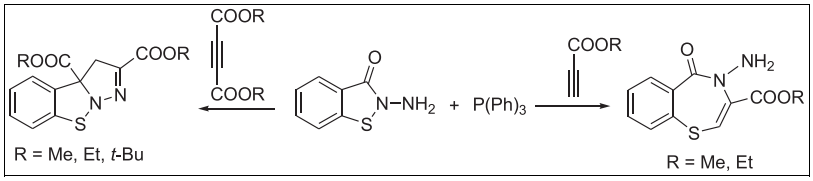

Investigation of the reaction between benzo[*d*]isothiazol-3-one, 2-aminobenzo[*d*]isothiazol-3-one, its isoster 2-aminoisoindolin-1-one, and activated acetylenes, in the presence of triphenylphosphine, led us to synthesize novel heterocyclic compounds that could be attractive for the building of biologically active molecules. A one-pot PPh_3-promoted tandem reaction, with acetylene dicarboxylates and dibenzoylacetylene, afforded new tricyclic pyrazolo-fused benzisothiazoles. The PPh_3-promoted reaction between benzisothiazolones and methyl propiolate afforded 1,4-benzothiazepine-5-one derivatives, via an isothiazole ring expansion. These studies are providing additional insights in benzisothiazolone chemistry and describe simple and original synthetic accesses to novel derivatives.

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INTRODUCTION

In the search for chemical modification of heterocyclic compounds that give access to molecular diversity, recently we have focused our attention on PPh_3-promoted tandem reaction between 2-aminobenzo[*d*]isothiazol-3-one (**1**) and activated acetylenic reagents such as dimethyl acetylenedicarboxylate (DMAD) and alkyl propiolate [1,2]. Following our work on exploiting PPh_3-promoted reactions to obtain novel benzisothiazole derivatives of interest in drug discovery, which have been the object of many years of our studies [3–12], herein we wish to report the reaction of **1** with further dialkyl acetylenedicarboxylates, with dibenzoylacetylene (DBA), and with phenylmethylpropiolate. In order to explore the applicability of the reactions over different benzisothiazolones and to have deeper understanding of the structural requirements and of the processes involved, benzo[*d*]isothiazol-3-one (**8**) and 2-aminoisoindolin-1-one (**10**), an isoindole isoster of **1**, were also subjected to identical reaction conditions. The results are summarized in Table 1. The structures of the obtained compounds were elucidated mainly by means of ^{1}H-NMR and ^{13}C-NMR techniques.

RESULTS AND DISCUSSION

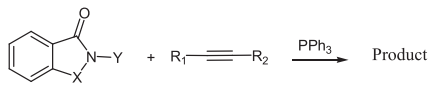
In our initial investigations, we examined the reaction between 2-aminobenzo[*d*]isothiazol-3-one (**1**) and DMAD in the presence of an equimolar amount of triphenylphosphine. The one-pot reaction proceeded smoothly as a tandem sequence of a phospha-Michael, an aza-Michael addition, and a final intramolecular Wittig-type reaction followed by a 1,2-methyl ester migration to give 3-*H*-benzo[*d*]pyrazolo [1,5-*b*]isothiazole-2,3*a*-dicarboxylic acid dimethyl ester (**2**), a novel functionalized pyrazolo-fused benzisothiazole, in satisfactory (65%) yield [1]. The reaction, here described, of **1** with diethyl and di-*tert*-butyl acetylenedicarboxylate in refluxing toluene for 24 h resulted in complete consumption of the starting materials and formation of **3** (40% yield) and **4** (54% yield), respectively, along with the recovery of triphenylphosphine oxide (Fig. 1). The mechanism for the whole reaction process is outlined in Figure 2.

A first Michael addition of triphenylphosphine to the acetylenic esters gives a 1:1 adduct that undergoes protonation by the NH_2-group of 2-aminobenzo[*d*]isothiazol-3-one. Then, a second addition of the NH anion to the positively charged Michael acceptor provides phosphorane, which spontaneously undergoes an intramolecular Wittig-type reaction to produce, through the intermediacy of betaine, a tricyclic heterocycle and triphenylphosphine oxide. At this point, 1,2-hydride migration provides iminium compound, upon which a 1,2-methyl ester migration occurs via cyclopropane intermediate, furnishing the reaction products **2–4**.

Afterward, 2-aminobenzo[*d*]isothiazol-3-one (**1**) was reacted with DBA, as activated alkyne. A new tricyclic pyrazolo-fused benzisothiazole, namely 2,3-dibenzoyl-2-*H*-benzo[*d*]pyrazolo[1,5-*b*]isothiazole (**5**), was obtained (Fig. 1). Contrary to the use of dialkyl acetylenedicarboxylates (refluxed in toluene), the reaction with DBA turned out to take place in 5 h, at room temperature, in CH_2Cl_2 solution. Moreover, remarkably, the ^{1}H-NMR analysis of **5** pointed out that no benzoyl group migration occurred, as deduced

Table 1

Reactions of benzisothiazolones **1** and **8** and isoindolone **10** with various acetylenic reagents in the presence of triphenylphosphine.



Substrate	R_1	R_2	Product
1 (X = S; Y = NH_2)	–COOMe	–COOMe	**2**[1]
1	–COOEt	–COOEt	**3**
1	–COO*t*-Bu	–COO*t*-Bu	**4**
1	–COPh	–COPh	**5**
1	–H	–COOMe	**6**[2]
1	–H	–COOEt	**7**[2]
1	–Ph	–COOMe	—
8 (X = S; Y = H)	–COOMe	–COOMe	—
8	–COOEt	–COOEt	—
8	–COO*t*-Bu	–COO*t*-Bu	—
8	–COPh	–COPh	—
8	–H	–COOMe	**9**
8	–Ph	–COOMe	—
10 (X = CH_2; Y = NH_2)	–COOMe	–COOMe	—
10	–COOEt	–COOEt	—
10	–COO*t*-Bu	–COO*t*-Bu	—
10	–COPh	–COPh	—
10	–H	–COOMe	—
10	–Ph	–COOMe	—

by no signal of diastereotopic H-atoms as was, on the contrary, previously observed in the ^{1}H-NMR spectra of the tricyclic products **2–4**. The presence of signals at δ = 6.78 ppm, of a CH proton, and, downfield, at δ = 13.44 ppm, of a typical H-atom bound to a heteroatom (D_2O exchangeable) further supported the formation of compound **5**. It is thus the nature of the ethyne substituents that does govern the last step of compounds **2**, **3**, **4**, and **5** formation. Mechanistically, it is conceivable that the reaction process involved in the formation of compound **5** proceeded as described earlier for **2**, **3**, and **4** (Fig. 2); however, the reaction ended with the intramolecular Wittig-type cyclization.

A successful nucleophilic attack of the dipolar intermediate, formed by a Michael addition of triphenylphosphine to alkyl propiolate, on the nitrogen atom of the isothiazole ring of **1**, was recently reported by us to afford, through an isothiazole ring expansion, in 45% yield, the noteworthy functionalized 1,4-benzothiazepine-5-ones **6** and **7** (Fig. 1) [2].

Given the interest of the unprecedented reactions discovered between 2-aminobenzo[*d*]isothiazol-3-one (**1**) and activated acetylenic reagents, we tried under the same conditions (equimolar amounts of the reagents, in the presence of triphenylphosphine) the reaction between **1** and methylphenylpropiolate. As shown in Figure 1, despite few trials with different solvents as toluene, CH_2Cl_2, and THF, the reaction did not succeed. At room temperature, the reagents failed to react and were recovered unchanged, while, by heating in different solvents, an unsolvable mixture was formed. This led us to suppose that the steric bulk and the electronic feature of the phenyl ring directly linked to the alkyne prevents the first Michael addition of triphenylphosphine, at room temperature, to the methylphenylpropiolate; refluxed in different solvents, the reactants underwent decomposition.

In order to expand the scope of the approach described in Figure 1, benzo[*d*]isothiazol-3-one (**8**) was subjected to the same reaction conditions applied for **1**. The results in Table 1 show that the only successful reaction was the one with the activated acetylene methyl propiolate (representative of the alkyl propiolates) that afforded, in 46% yield, through an isothiazole ring expansion, methyl 5-oxo-4,5-dihydrobenzo[*f*][1,4]thiazepine-3-carboxylate (**9**). A plausible reaction mechanism for the formation of compound **9**, in full agreement with the sequence proposed for the tandem reaction that led to methyl 4-amino-5-oxo-4,5-tetrahydrobenzo[*f*][1,4]thiazepine-3-carboxylate **6** [2], is depicted in Figure 3.

The nitrogen atom of the isothiazole ring of **8** is susceptible to nucleophilic attack as activated for the adjacent sulfur and carbonyl group. The nucleophilic attack of the dipolar intermediate, formed *in situ* by a Michael addition of triphenylphosphine to methyl propiolate, is accompanied

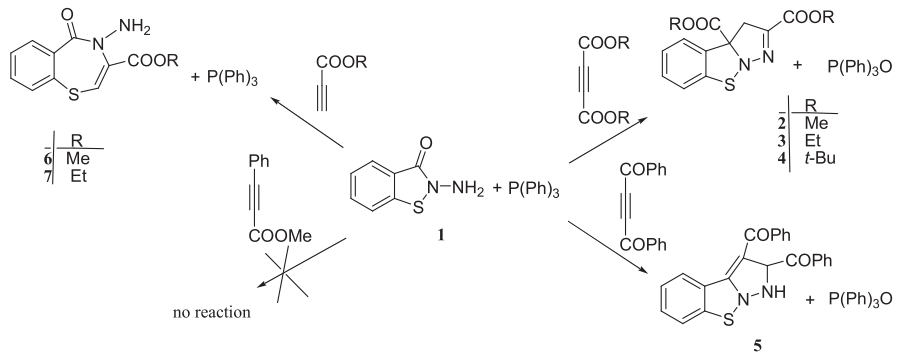


Figure 1. PPh_3-promoted reaction between 2-aminobenzo[*d*]isothiazol-3-one (**1**) and acetylenic reagents.

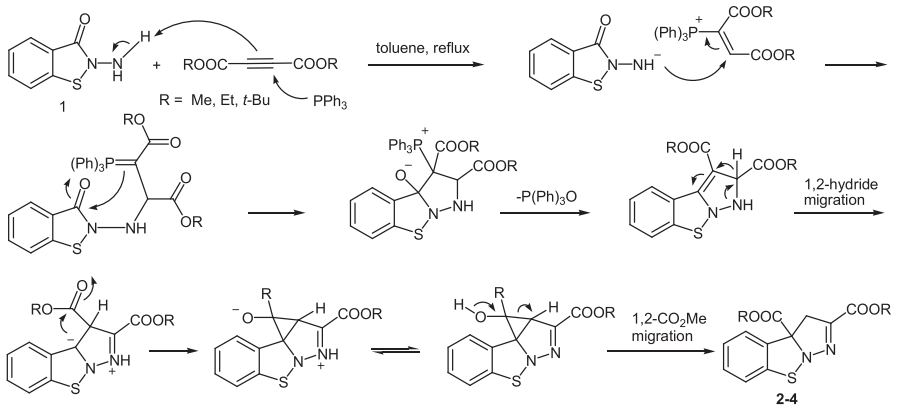


Figure 2. Mechanistic pathway for the formation of compounds **2–4**.

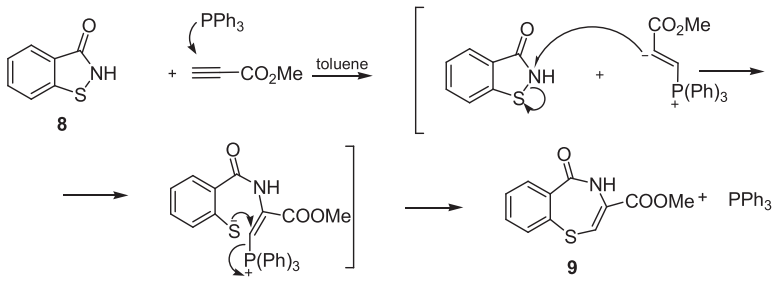


Figure 3. Mechanistic pathway for the formation of compound **9**.

by a spontaneous cleavage of the N–S bond, reasonably supported by the chemistry of isothiazolones that well explains the liability of N–S bond [13–15]. The open-chain undergoes a further nucleophilic attack of the sulfur to the electrophilic centre C(2) of the betainic intermediate. Thus, benzothiazepine **9** is formed by elimination of PPh_3 and intramolecular cyclocondensation with ring expansion to the stable seven-membered benzofused ring. To explain the complete lack of reactivity between benzo[*d*]isothiazol-3-one (**8**) and dialkyl acetylenedicarboxylates or DBA, which gave, on the contrary, products **2–5** in good yields starting from **1**, we want to point out the absence in compound **8** of the NH_2 nitrogen as the fifth term of a stable transition intermediate in the Wittig reaction leading to cyclization, as what occurred for compound **1**. Despite the presence in **8** of the isothiazole NH as proton source, whose importance has been widely demonstrated in Yavari's studies [16–19] the aza Michael addition to the protonated 1:1 adduct did not succeed. In fact the instable four terms cycle prevented the cyclization step that led to tryciclic derivatives **2**, **3**, **4**, and **5**. The ^{1}H-NMR monitoring of the reactions between **8** and disubstituted acetylenes confirmed lack of reaction at room temperature and, by heating, consumption of the reactants along with formation of a complex mixture of unidentified products.

To further extend the study of the previously reported processes, a desulfurated isoster of **1**, namely 2-aminoisoindolin-1-one (**10**, Table 1), was subjected, under similar conditions, to the reactions described between benzisothiazolones **1** and **8** and activated acetylenes. As expected, no significant reaction was observed, and the lack of reaction led us to confirm the pivotal role, in benzisothiazolones, of the S-atom, which is crucial for the Michael addition of the NH group to the phosphorated adduct that starts these tandem processes.

Synthetic methods, characterization data for compounds **3–5** and **9**, including mp, ^{1}H-NMR and ^{13}C-NMR, MS data, and the results of elemental analysis are reported in the experimental section.

In conclusion, this work provides a direct route to the synthesis of some novel biologically relevant heterocyclic compounds. In any case, all these studies are providing few additional insights in benzisothiazolone chemistry as well as simple and original synthetic accesses. When applicable, the selectivity, good to high yields, simple work up procedures, and mild conditions are advantages of these processes. We hope in the future to further exploit, with the present approach, the benzisothiazolone scaffold as a source of highly functionalized heterocycles for the building of biologically active molecules.

EXPERIMENTAL

2-Aminobenzo[*d*]isothiazol-3-one (**1**) and benzo[*d*]isothiazol-3-one one (**8**) were synthesized as reported earlier by us [12]. DBA was prepared according to [20], and 2-aminoisoindolin-1-one (**10**) was synthesized following a modification of a described procedure [21,22]. Unless otherwise noted, reagents were obtained from commercial suppliers and were used without purification. Anhydrous toluene was obtained by distillation from Na, and anhydrous CH_2Cl_2 was obtained by distillation from calcium hydride. All reactions were carried out using flame-dried glassware under a nitrogen atmosphere. Melting points were measured on a Buechi 512 apparatus (Buechi Italia, Milano, Italia) and are uncorrected. The progress of the reactions was monitored by TLC with F_{254} silica-gel precoated sheets (Merck, Darmstadt, Germany). UV light was used for detection. Flash chromatography was performed using Merck silica gel 60 (Si 60, 40–63 μm, 230–400 mesh ASTM). Elemental analyses for C, H, and N were performed using a ThermoQuest Flash 1112 elemental analyzer (Termoquest Italia, Milano, Italy). IR spectra were recorded on a Jasco FT-IR 300E spectrometer (Jasco Europe, Carpi (MO), Italia). Mass spectra were recorded on an Applied Biosystem API 150 EX LC-MS system spectrometer (AB/SCIEX, Toronto, Canada). ^{1}H-NMR and ^{13}C-NMR spectra were recorded on a Bruker Avance 300 spectrometer (Brucker Italia, Milano, Italia). Spectra were acquired from samples in DMSO-d_6 (25 mg mL^{-1}). ^{1}H and ^{13}C spectra (at 300 and 75 MHz, respectively) were measured at 25°C in 5 mm o.d. tubes. Chemical shifts are reported as δ (ppm); coupling constants (*J*) are expressed in Hz.

General procedure for the preparation of 3-*H*-benzo[*d*]pyrazolo[1,5-*b*]isothiazole-2,3*a*-dicarboxylic acid dialkyl esters. To a magnetically stirred mixture of 2-aminobenzo[*d*]isothiazol-3-one (**1**) (0.5 g, 3.0 mmol) and triphenylphosphine (0.8 g, 3.0 mmol) in anhydrous toluene (7 mL), a solution of DMAD (0.43 g, 3.0 mmol) in anhydrous toluene (7 mL) was added dropwise over 5 min keeping the temperature at −5°C. The reaction mixture was allowed to warm to room temperature and then refluxed for 24 h. The solvent was removed under reduced pressure, and the oily residue was purified by silica gel chromatography using CH_2Cl_2/ethyl acetate 90:10 (**3**) or 95/5 (**4**) v/v mixture as eluent. The solid obtained was then recrystallized from ethyl acetate.

3-*H*-Benzo[*d*]pyrazolo[1,5-*b*]isothiazole-2,3*a*-dicarboxylic acid diethyl ester (3). Yield 40%; mp 146–147°C; ^{1}H-NMR (300 MHz DMSO-d_6): δ = 0.94 (t, 3H, *J* = 7.2 CH_3), 1.33 (t, 3H, *J* = 7.1 CH_3), 3.64 (d, 1H, *J* = 17.70 CH), 3.82 (d, 1H, *J* = 17.70 CH), 4.02 (q, 2H, *J* = 7.2 CH_2), 4.29 (q, 2H, *J* = 7.2 CH_2) 7.37–7.52 (m,3H, Ar), 8.01 (dd,1H, *J* = 1.5 *J* = 8.1 Ar); ^{13}C-NMR (75 MHz DMSO-d_6): δ = 27.27 (CH_3), 123.89 (C), 27.71 (CH_3), 44.19 (CH_2), 67.84 (CH_2), 84.31 (C), 85.03 (C), 126.56 (CH), 126.94 (CH), 129.86 (C), 130.08 (CH), 130.92 (C), 131.43 (CH), 157.32 (C), 162.61 (C═O), 167.67 (C═O); IR (KBr) (ν_{max}/cm^{-1}): 3039 (CH Ar), 2960 and 2836 (CH_3), 1727 and 1690 (C═O ester). MS (APCI) *m/z*: 321 (M + 1). *Anal*. Calcd. for $C_{15}H_{16}N_2O_4S$ (320.36): C, 56.24; H, 5.03; N, 8.74. Found C, 55.97; H, 5.10; N, 8.54%.

3-*H*-Benzo[*d*]pyrazolo[1,5-*b*]isothiazole-2,3*a*-dicarboxylic acid di-*tert*-butyl ester (4). Yield 54%; mp 147–148°C; ^{1}H-NMR (300 MHz DMSO-d_6): δ = 0.94 (s, 9H, $(CH_3)_3$), 1.54 (s, 9H, $(CH_3)_3$), 3.45 (d, 1H, *J* = 17.70 CH), 3.69 (d, 1H, *J* = 17.40 CH), 7.38–7.55 (m, 3H, Ar), 8.00 (dd, 1H, *J* = 1.2 *J* = 8.1 Ar); ^{13}C-NMR (75 MHz DMSO-d_6): δ = 13.52 (CH_3), 14.27 (CH_3), 42.91 (C), 126.22 (CH), 63.25 (C), 67.21 (C), 68.43 (C), 126.70 (CH), 127.19 (CH), 129.30 (C), 130.27 (CH), 130.39 (C), 131.71 (CH), 157.48 (C), 164.76 (C═O), 168.92 (C═O); IR (KBr) (ν_{max}/cm^{-1}): 3041 (CH Ar), 2977 and 2911 (CH_3), 1720 and 1681 (C═O ester). MS (APCI) *m/z*: 376 (M + 1). *Anal*. Calcd. for $C_{19}H_{24}N_2O_4S$ (376.47): C, 60.62; H, 6.43; N, 7.44. Found C, 60.32; H, 6.40; N, 7.28%.

2,3-Dibenzoyl-2-*H*-benzo[*d*]pyrazolo[1,5-*b*]isothiazole (5). To a magnetically stirred solution of triphenylphosphine (0.22 g, 0.84 mmol) and compound **1** (0.14 g, 0.84 mmol) in anhydrous dichloromethane (10 mL), kept under nitrogen atmosphere was added dropwise a solution of DBA (0.20 g, 0.84 mmol) in anhydrous dichloromethane (1 mL). After stirring for 5 h at room temperature, the solvent was removed under reduced pressure. The reddish solid residue was purified by silica gel flash chromatography (dichloromethane/ethyl acetate 95:5 v/v) to afford compound **5**, which was recrystallized from ethanol/water. Yield 45%; mp 176–177°C; ^{1}H-NMR (300 MHz DMSO-d_6): δ = 6.78 (d, 1H, *J* = 1.5 CH), 7.26–7.56 (m, 9H, Ar), 7.63–7.66 (d, 2H, *J* = 7.5 Ar), 7.67–7.70 (d, 2H, *J* = 7.5 Ar,), 7.95 (d, 1H, *J* = 7.8 Ar), 13.44 (d, 1H, *J* = 1.5 NH); ^{13}C-NMR (75 MHz DMSO-d_6): δ = 102.51 (CH), 123.89 (C), 126.11 (CH), 126.22 (CH), 126.75 (CH), 128.06 (CH), 128.23 (CH), 128.48 (CH), 128.72 (CH), 129.08 (C), 131.74 (CH), 133.60 (CH), 136.54 (C), 139.14 (C), 145.89 (C), 150.59 (C═O), 163.31 (C═O); (IR (KBr) (ν_{max}/cm^{-1}): 3242 (NH), 3061 (CH Ar), 2919 (CH), 1725 and 1703 (C═O). MS (APCI) *m/z*: 385 (M + 1). *Anal*. Calcd. $C_{23}H_{16}N_2O_2S$ (384.45): C, 71.85; H, 4.19; N, 7.29. Found. C, 71.90; H, 3.98; N, 7.08, %.

Methyl 5-oxo-4,5-dihydrobenzo[*f*][1,4]thiazepine-3-carboxylate (9). To a magnetically stirred mixture of 2-aminobenzo[*d*]isothiazol-3-one **1** (0.5 g, 3.0 mmol) and triphenylphosphine (0.8 g, 3.0 mmol) in anhydrous toluene (7 mL), a solution of alkyl propiolate (3.0 mmol) in anhydrous toluene (2 mL) was added. The reaction mixture was stirred at room temperature for 5 h. The solvent was removed under reduced pressure, and the oily residue was purified by silica gel flash chromatography (CH_3Cl/ethanol 98:2 v/v). The solid obtained was recrystallized from toluene furnishing the product as a white solid. Yield 40%; mp 115°C; ^{1}H-NMR (300 MHz DMSO-d_6): δ = 3.72 (s, 3H, CH_3), 7.24 (s, 1H, CH), 7.41 (td, 1H, *J* = 1.2 *J* = 7.8 Ar), 7.45–7.53 (m, 2H, Ar,), 7.66 (dd, 1H, *J* = 1.5 *J* = 7.2 Ar), 9.80 (s, 1H, NH); ^{13}C-NMR (75 MHz DMSO-d_6): δ = 52.74 (CH), 120.85 (CH), 128.38 (CH), 131.22 (CH), 132.15 (CH), 132.42 (CH), 133.36 (C), 135.77 (C), 136.46 (C), 161.79 (C═O), 167.95 (C═O); IR (KBr) (ν_{max}/cm^{-1}): 3190 (NH), 3086 (CH Ar), 2957 (CH_3), 1724 (C═O ester) and 1653 (C═O). MS (APCI) *m/z*: 236 (M + 1). *Anal*. Calcd. for $C_{11}H_9NO_3S$ (235.26): C, 56.16; H, 3.86; N, 5.94. Found C, 56.35; H, 3.87; N, 5.94%.

2-Aminoisoindolin-1-one (10). A modification of a described procedure [21] was used: to a magnetically stirred solution of *N*-(*t*-butyloxycarbonyl)-2-aminoisoindolin-1-one [22] (0.10 g, 0.40 mmol) in ethyl acetate (10 mL) cooled at 0°C was added a solution of 3*M* HCl in ethyl acetate (5 mL). The reaction mixture was then allowed to warm to room temperature and stirred overnight. Evaporation of the solvent under reduce pressure afforded a white solid. Yield 94%; mp 94–96. ^{1}H-NMR (300 MHz DMSO-d_6): δ = 4.28 (s, 2H, NH_2), 4.80 (s, 2H, CH_2), 7.37–7.44 (m, 2H, Ar), 7.50 (t, 1H, *J* = 6.9 Ar), 7.78 (d, 1H, *J* = 7.8 Ar); ^{13}C-NMR (75 MHz DMSO-d_6): δ = 53.30 (CH_2), 122.82(CH), 123.67 (CH), 128.25 (CH), 131.48 (CH), 132.34 (C), 140.31 (C), 166.09 (C═O); IR (KBr) (ν_{max}/cm^{-1}): 3290 and 3185 (NH_2), 3045 (CH Ar), 2823 (CH_2), 1690 (C═O), MS (APCI) *m/z*: 185 (M + 1).

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