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New Synthesis of 1*H*-[1,2,4]Triazolo[3,4-*a*]isoindoles by Intramolecular Condensation of 1-Substituted-1,2,4-triazol-4-ium Methylides

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Abstract—In this paper we report the first approach of synthesis of 1*H*-[1,2,4]triazolo[3,4-*a*]isoindoles in moderate to good yields by intramolecular annulation of 1-substituted-1,2,4-triazol-4-ium methylides.

Introduction

Fused heterocycles including a triazole ring have become of interest to synthetic chemists and biologists because of their unusual structures and wide range of biological and medicinal properties.[1] Among this class of compounds, the triazoloindole derivatives have received increasing attention in past years as witnessed by the recent articles and patents dealing with their synthesis and emphasising their pharmaceutical and medicinal activities.[2]

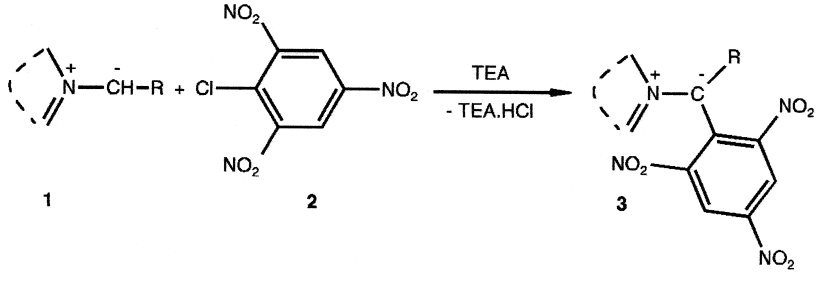
In previous work, monosubstituted cycloimmonium ylides **1** have been identified by condensation with picryl chloride **2** giving the corresponding disubstituted cycloimmonium ylides **3** (Scheme 1).[3–6]

In this paper, we report, for the first time, the synthesis of 1*H*-[1,2,4]triazolo[3,4-*a*]isoindoles by intramolecular cyclisation of 1-substituted-1*H*-[1,2,4]triazol-4-iumbenzoyl-2,4,6-trinitrophenylmethylides (Scheme 2).

Results and Discussion

Initially, the required 1-benzyl-1*H*-[1,2,4]triazole **4a** was obtained from the commercially available 1*H*-[1,2,4]-triazole via the corresponding anion and benzyl bromide.[7] The 1-(4-methoxyphenyl)-1*H*-[1,2,4] triazole **4b** was swiftly synthesised by deprotonation of the 4-[1,2,4]-triazol-1-yl phenol with KOH pellets followed by addition of iodomethane.

The "salt method"[7] has been applied in order to obtain the triazol-4-ium ylides **8**. Thus, the 1-substituted-1H-[1,2,4]-triazoles **4a**–**b** treated with 4-substituted ω-bromo-acetophenones **5a**–**b** in dry boiling acetone furnish, after recrystallisation (ethanol), the corresponding salts **6a**–**d**, in good yields (Table 1). Next these salts in the presence of triethylamine (TEA) form 'in situ' the monosubstituted carbanions ylides **7** which, with picryl chloride in excess of TEA offer directly the [1,2,4]-triazol-4-ium trinitrophenyl-methylides **8a**–**d**. After purification these disubstituted



1 2 3

Scheme 1.

Keywords: ylids; annulation; isoindoles.

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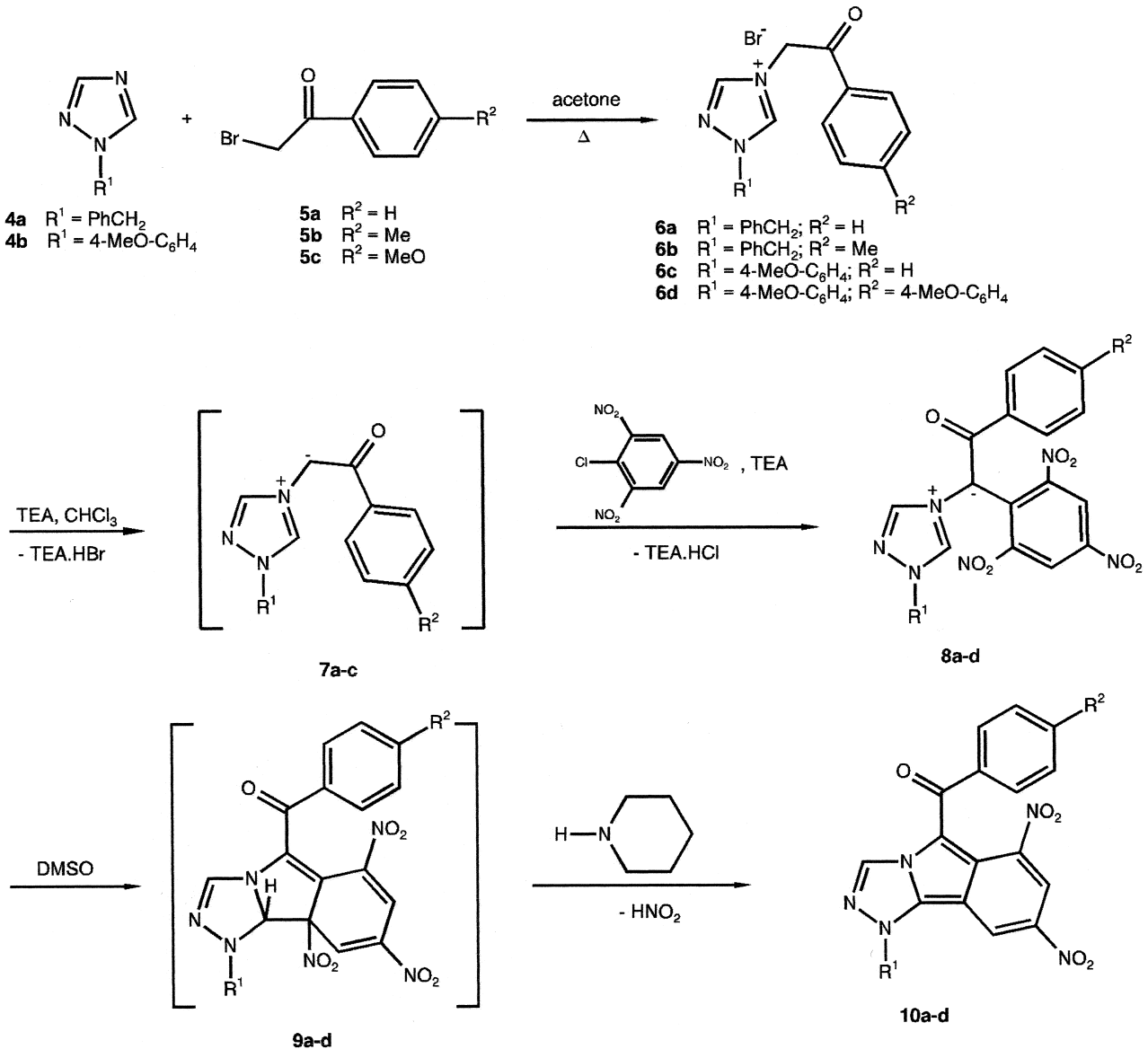

Scheme 2.

Table 1. [1,2,4]-Triazol-4-ium salts **6a**–**d**, [1,2,4]-triazol-4-ium disubstituted methylides **8a**–**d** and isoindoles **10a**–**d** prepared

Compound	R^1	R^2	Yield[a] (%)
6a	$PhCH_2$	H	60
6b	$PhCH_2$	CH_3	75
6c	4-MeO-C_6H_4	H	59
6d	4-MeO-C_6H_4	4-MeO-C_6H_4	65
8a	$PhCH_2$	H	68
8b	$PhCH_2$	CH_3	62
8c	4-MeO-C_6H_4	H	77
8d	4-MeO-C_6H_4	4-MeO-C_6H_4	75
10a	$PhCH_2$	H	76
10b	$PhCH_2$	CH_3	75
10c	4-MeO-C_6H_4	H	67
10d	4-MeO-C_6H_4	4-MeO-C_6H_4	72

[a] Yields are given for purified products.

carbanion ylides have been isolated in fairly good yields (Table 1). However, this reaction must be carried out under 10°C and without light[8] in order to prevent cleavage of the C^-–N^+ bond. The [1,2,4]-triazol-4-ium disubstituted methylides **8**, dissolved in dimethylsulfoxide (DMSO) in the presence of piperidine at room temperature are converted to 1*H*-[1,2,4]triazolo[3,4-*a*]isoindoles **10a**–**d**, indicated by the disappearance of the initial deep purple colour corresponding to ylides **8** during the experiments and the disappearance of the signals near 8 ppm in ^1H NMR data assigned to the proton in the five position in the triazole ring. Nevertheless, in order to explain the formation of the final structures **10**, an intermediate adduct of the type **9** may be envisaged and the relatively good yields (Table 1) of these transformations could be explained only by the existence of a high degree of conjugation in the final compounds **10**.

Conclusion

The ylides **7** and **8** represent a new class of intermediates which could be used in the synthesis of isoindoles derivatives. The extension of the established strategy to the synthesis of other fused heterocycles is under active investigation.

Experimental

^1H and ^{13}C NMR spectra were recorded on a Brüker AM 250 spectrometer with tetramethylsilane as internal standard. Chemical shifts are expressed in ppm. The abbreviations are: s (singlet), d (doublet), t (triplet) and m (multiplet). Mass spectra were taken on a Platform II Micromass apparatus. For column chromatography SDS silica gel 60 (70–200 μm) was used. Chloroform and acetone were freshly distilled over CaH_2 and K_2CO_3, respectively. Dry glassware for moisture-sensitive reactions was obtained by oven-drying and assembly under Ar. An inert atmosphere was obtained with a stream of Ar and glassware used in experiments was equipped with rubber septa. The reagent transfer was performed by syringe techniques.

Compound **4a** was prepared following the reported experimental procedure.[7]

1-(4-Methoxy-phenyl)-1*H*-[1,2,4]triazole 4b. A suspension of 4-[1,2,4]triazo-1-yl phenol (4.4 g, 0.3 mmol), KOH (2.2 g, 0.4 mmol) and CH_3I (2.8 mL, 0.4 mmol) in EtOH (200 mL) was refluxed under Ar for 12 h. The mixture was filtered and evaporated to give a crude solid which was treated with water and extracted twice with CH_2Cl_2 (2×100 mL). The organic layer was made alkaline by the addition of 10% aqueous KOH, dried on $MgSO_4$ and evaporated in vacuum to afford colourless crystals. (Rdt=86%). ^1H- and ^{13}C NMR spectra of this compound were consistent with literature data.[9]

General procedure for the synthesis of 1-substituted-1*H*-[1,2,4]triazol-4-ium bromide 6a–d. The ω-bromoacetophenone compounds **5a**–**c** are commercially available. A solution of ω-bromoacetophenone derived **5a**–**c** (1 mmol) in anhydrous acetone (30 mL) was added under Ar at room temperature to a solution of 1-substituted-1*H*-[1,2,4] triazol **4a** and **b** (1 mmol) in anhydrous acetone (80 mL). The solution was warmed to reflux for 8 h. The crude precipitated product was filtered and finally recrystallised in EtOH.

1-Benzyl-4-phenacyl-1*H*-[1,2,4]triazol-4-ium bromide 6a. Mp 175–176°C; ^1H NMR (DMSO-d_6, δ, *J* Hz): 5.77 (2H, s, NCH_2Ar), 6.14 (2H, s, CH_2CO), 7.42–7.47 (5H, m, H_{arom}), 7.61–7.66 (2H, m, H_{arom}), 7.77 (1H, dd, *J*=7.3 Hz, H_{arom}), 8.05 (2H, d, *J*=7.3 Hz, H_{arom}), 9.17 (1H, s, H_{arom}), 10.22 (1H, s, H_{arom}); ^{13}C NMR (DMSO, δ): C, 190.4, 133.4, 133.3; CH, 146.1, 143.7, 134.8, 129.2, 129.0, 128.8, 128.3; CH_2, 54.8, 54.0; *m*/*z* (%): 279 (M−Br, 100), 279 (M+1−Br, 25); IR (KBr, cm^{-1}):3049, 1694, 1572, 1443, 1396, 1338, 1243, 1149, 747, 882; Anal. Calcd for $C_{17}H_{16}N_3OBr$: C, 57.00; H, 4.50; N, 11.73; found: C, 59.12; H, 4.48; N, 11.46.

1-Benzyl-4-(4-methyl)phenacyl-1*H*-[1,2,4]triazol-4-ium bromide 6b. Mp 155–156°C; ^1H NMR (DMSO-d_6, δ, *J* Hz): 2.43 (3H, s, CH_3), 5.79 (2H, s, NCH_2Ar), 6.14 (2H, s, CH_2CO), 7.44–7.50 (7H, m, H_{arom}), 7.97 (2H, d, *J*=8.1 Hz, H_{arom}), 9.20 (1H, s, H_{arom}), 10.27 (1H, s, H_{arom}); ^{13}C NMR (DMSO, δ): C, 189.9, 145.4, 133.3, 130.9; CH, 146.1, 143.6, 129.7, 128.9, 128.8, 128.3; CH_2, 54.8, 53.9; CH_3, 21.3; *m*/*z* (%): 292 (M−Br, 100) 293 (M+1−Br, 21); IR (KBr, cm^{-1}): 2928, 1694, 1603, 1573, 1446, 1341, 1237, 1154, 999, 731.; Anal. Calcd for $C_{18}H_{18}N_3OBr$: C, 58.21; H, 4.89; N, 11.32; found: C, 58.16; H, 4.67; N, 11.30.

1-(4-Methoxy-phenyl)-4-phenacyl-1*H*-[1,2,4]triazole 6c. Mp 197–198°C; ^1H NMR (DMSO-d_6, δ, *J* Hz): 3.88 (3H, s, OCH_3), 6.27 (2H, s, CH_2CO), 7.26 (2H, d, *J*=8.8 Hz, H_{arom}), 7.65–7.85 (3H, m, H_{arom}), 7.91 (2H, d, *J*=9.1 Hz, H_{arom}), 8.13 (2H, d, *J*=7.3 Hz, H_{arom}), 9.43 (1H, s, H_{arom}), 10.89 (1H, s, H_{arom}); ^{13}C NMR (DMSO, δ): C, 190.1, 160.7, 133.3, 128.0; CH, 146.0, 141.8, 134.9, 129.2, 128.4, 122.7, 115.3; CH_2, 55.8; CH_3, 54.0; *m*/*z* (%): 294 (M−Br, 100), 295 (M+1−Br, 14); IR (KBr, cm^{-1}): 2916, 1692, 1596, 1573, 1524, 1498, 1343, 1237, 835, 763; Anal. Calcd for $C_{17}H_{16}N_3OBr$: C, 53.56; H, 4.31; N, 11.23; found: C, 53.32; H, 4.47; N, 10.95.

1-(4-Methoxy-phenyl)-4-(4-methoxy)phenacyl-1*H*-[1,2,4]-triazole 6d. Mp 205–206; ^1H NMR (DMSO-d_6, δ, *J* Hz): 3.87 (3H, s, OCH_3), 3.91 (3H, s, OCH_3), 6.20 (2H, s, CH_2CO), 7.19 (2H, d, *J*=8.9 Hz, H_{arom}), 7.26 (2H, d, *J*=9.1 Hz, H_{arom}), 7.90 (2H, d, *J*=8.9 Hz, H_{arom}), 8.10 (2H, d, *J*=8.9 Hz, H_{arom}), 9.41 (1H, s, H_{arom}), 10.87 (1H, s, H_{arom}); ^{13}C NMR (DMSO, δ): C, 188.2, 164.4, 160.7, 128.0, 126.1; CH, 146.0, 141.8, 130.8, 122.7, 115.3, 114.5; CH_2, 53.6; CH_3, 55.8 (×2); *m*/*z* (%): 324 (M−Br, 100); IR (KBr, cm^{-1}): 2925, 1688, 1600, 1571, 1501, 1274, 1239, 1174, 990, 836.; Anal. Calcd for $C_{17}H_{16}N_3OBr$: C, 54.56; H, 4.31; N, 11.23; found: C, 54.29; H, 3.96; N, 11.01.

General procedure for the synthesis of 1-substituted-1*H*-[1,2,4]triazol-4-iumbenzoyl-2,4,6-trinitrophenylmethylides 8a–d. A solution of freshly distilled Et_3N (5.6 mmol) in dry $CHCl_3$ (5 mL) was added at 0°C under Ar to a stirred suspension of **6** (2.8 mmol) and picryl chloride in dry $CHCl_3$ (30 mL). The mixture was warmed to room temperature, without light for 3 h. The solvent was evaporated and the deep purple crude product was chromotographed on SiO_2 using acetone–hexane (40/60) as eluent.

1-Benzyl-1*H*-[1,2,4]triazol-4-iumbenzoyl-2,4,6-trinitrophenylmethylide 8a. Mp 97–98°C; ^1H NMR ($CDCl_3$, δ, *J* Hz): 5.43 (2H, s, CH_2Ph), 7.17–7.33 (7H, m, H_{arom}), 7.46–7.48 (3H, m, H_{arom}), 8.16 (1H, s, H_{arom}), 8.49 (2H, s, H_{arom}), 9.45 (1H, s, H_{arom}); *m*/*z* (%): 489 (M+1, 10); 443 (M+1−NO_2, 100) IR (KBr, cm^{-1}): 3055, 1698, 1608, 1528, 1299, 1159, 1081, 721, 873.

1-Benzyl-1*H*-[1,2,4]triazol-4-ium-4-methylbenzoyl-2,4,6-trinitrophenylmethylide 8b. Mp 101–102°C; ^1H NMR ($CDCl_3$, δ, *J* Hz): 2.29 (3H, s, CH_3), 5.40 (2H, s, CH_2Ph), 6.98 (2H, d, *J*=7.9 Hz, H_{arom}), 7.13 (2H, d, *J*=7.9 Hz,

H_{arom}), 7.27–7.31 (2H, m, H_{arom}), 7.43–7.46 (3H, m, H_{arom}), 8.13 (1H, s, H_{arom}), 8.46 (2H, s, H_{arom}), 9.35 (1H, s, H_{arom}); *m*/*z* (%): 503 (M+1, 8); 457 (M+1−NO_2, 100), 365 (10); IR (KBr, cm^{-1}): 2923, 1700, 1606, 1522, 1458, 1297, 1162, 1083, 711.

1-(4-Methoxy-phenyl)-1*H*-[1,2,4]triazol-4-iumbenzoyl-2,4,6-trinitrophenylmethylide 8c. Mp 109–110; ^{1}H NMR ($CDCl_3$, δ, *J* Hz): 3.90 (3H, s, OCH_3), 7.06 (2H, d, *J*=9.1 Hz, H_{arom}), 7.22–7.37 (5H, m, H_{arom}), 7.64 (2H, d, *J*=9.1 Hz, H_{arom}), 8.29 (1H, s, H_{arom}), 8.53 (2H, s, H_{arom}), 9.91 (1H, s, H_{arom}); *m*/*z* (%): 527 (M+Na, 80), 505 (M+1, 100), 459 (M+1−NO_2, 20), 175 (25); IR (KBr, cm^{-1}): 2945, 1606, 1520, 1519, 1259, 1257, 1165, 914, 831, 716.

1-(4-Methoxy-phenyl)-1*H*-[1,2,4]triazol-4-ium-4-methoxybenzoyl-2,4,6-trinitrophenylmethylide 8d. Mp 99–100°C; ^{1}H NMR ($CDCl_3$, δ, *J* Hz): 3.80 (3H, s, OCH_3), 3.89 (3H, s, OCH_3), 6.72 (2H, d, *J*=8.6 Hz, H_{arom}), 7.04 (2H, d, *J*=9.0 Hz, H_{arom}), 7.24 (2H, d, *J*=8.6 Hz, H_{arom}), 7.65 (2H, d, *J*=9.0 Hz, H_{arom}), 8.30 (1H, s, H_{arom}), 8.49 (2H, s, H_{arom}), 9.95 (1H, s, H_{arom}); *m*/*z* (%): 557 (M+Na, 50), 535 (M+1, 100), 489 (M+1−NO_2, 25), 295 (25), 175 (25); IR (KBr, cm^{-1}): 2932, 1603, 1517, 1257, 1255, 1165, 1022, 833.

General procedure for the synthesis of 1*H*-[1,2,4]triazolo[3,4-*a*]isoindoles. A solution of **8** (1.5 mmol) in DMSO (20 mL) was stirred under Ar at room temperature in the presence of piperidine (3 mmol) for 5 h without light. Initially the solution had a deep purple colour and progressively became red. The solution was acidified with 3N acetic acid solution and washed with water to afford a red precipitate which was filtered and washed in boiling EtOH.

1-Benzyl-5-benzoyl-6,8-dinitro-1*H*-[1,2,4]triazolo[3,4-*a*]isoindoles 10a. Mp 225–226°C; ^{1}H NMR (DMSO-d_6, δ, *J* Hz): 5.87 (2H, s, CH_2Ar), 7.38–7.56 (10H, m, H_{arom}), 8.45 (1H, s, H_{arom}), 8.79 (1H, s, H_{arom}), 9.41 (1H, s, H_{arom}); ^{13}C NMR (DMSO, δ): C, 183.3, 140.7, 140.3, 138.1, 136.4, 134.4, 124.1, 107.3, 107.0; CH, 132.9, 131.8, 128.9, 128.5, 128.0, 127.1, 125.1, 121.0; CH_2, 54.1; *m*/*z* (%): 481 (M+CH_3CN, 30), 464 (M+Na, 80), 442 (M+1, 20), 295 (40), 91 (100), 63 (15), 36 (35); IR (KBr, cm^{-1}): 2933, 1706, 1558, 1498, 1450, 1385, 1304, 1266, 1142, 1053, 732, 700; Anal. Calcd for $C_{23}H_{15}N_5O_5$: C, 62.57; H, 3.43; N, 15.87; found: C, 62.06; H, 3.38; N, 15.76.

1-Benzyl-5-(4-methyl)benzoyl-6,8-dinitro-1*H*-[1,2,4]-triazolo[3,4-*a*]isoindoles 10b. Mp 235–236°C; ^{1}H NMR (DMSO-d_6, δ, *J* Hz): 2.51 (3H, s, CH_3), 6.14 (2H, s, CH_2Ar), 7.26 (2H, d, *J*=7.8 Hz, H_{arom}), 7.37–7.50 (7H, m, H_{arom}), 8.69 (1H, s, H_{arom}), 9.18 (1H, s, H_{arom}), 9.53 (1H, s, H_{arom}); ^{13}C NMR (DMSO, δ): C, 183.1, 141.9, 138.1, 138.0, 136.2, 134.4, 132.8, 123.9, 107.4, 106.8; CH, 129.4, 128.9, 128.5, 128.0, 127.3, 125.1, 121.0; CH_2, 54.1; CH_3, 21.1; *m*/*z* (%): 494 (M+K, 10), 478 (M+Na, 100), 456 (M+1, 20), 295 (30), 36 (20); IR (KBr, cm^{-1}): 2362, 1621, 1558, 1528, 1302, 1255, 1136, 1105; Anal. Calcd for $C_{24}H_{17}N_5O_5$: C, 63.28; H, 3.75; N, 15.38; found: C, 63.05; H, 3.56; N, 14.99.

1-(4-Methoxyphenyl)-5-benzoyl-6,8-dinitro-1*H*-[1,2,4]-triazolo[3,4-*a*]isoindoles 10c. Mp 276–277°C; ^{1}H NMR (DMSO-d_6, δ, *J* Hz): 3.93 (3H, s, OCH_3), 7.35 (2H, d, *J*=78.9 Hz, H_{arom}), 7.45–7.60 (5H, m, H_{arom}), 7.94 (2H, d, *J*=8.9 Hz, H_{arom}), 8.72 (1H, s, H_{arom}), 8.77 (1H, s, H_{arom}), 9.72 (1H, s, H_{arom}); ^{13}C NMR (DMSO, δ): C, 183.3, 160.4, 140.6, 139.2, 138.3, 136.0, 133.6, 124.1, 107.3, 107.2; CH, 131.9, 129.0, 127.2, 125.3, 123.1, 121.0, 115.5; CH_3, 55.8; *m*/*z* (%): 496 (M+K, 15), 480 (M+Na, 100), 458 (M+1, 5), 295 (35), 36 (30); IR (KBr, cm^{-1}): 3123, 1602, 1511, 1455, 1287, 1167, 1078, 1050, 840, 732; Anal. Calcd for $C_{23}H_{15}N_5O_6$: C, 60.38; H, 3.31; N, 15.32; found: C, 60.09; H, 3.27; N, 15.06.

1-(4-Methoxyphenyl)-5-(4-methoxy)benzoyl-6,8-dinitro-1*H*-[1,2,4]triazolo[3,4-*a*]isoindoles 10d. Mp 273–274°C; ^{1}H NMR (DMSO-d_6, δ, *J* Hz): 3.86 (3H, s, OCH_3), 3.96 (3H, s, OCH_3), 7.04 (2H, d, *J*=8.4 Hz, H_{arom}), 7.37 (2H, d, *J*=8.6 Hz, H_{arom}), 7.62 (2H, d, *J*=8.4 Hz, H_{arom}), 7.95 (2H, d, *J*=8.6 Hz, H_{arom}), 8.76 (1H, s, H_{arom}), 8.80 (1H, s, H_{arom}), 9.68 (1H, s, H_{arom}); ^{13}C NMR (DMSO, δ): C, 182.4, 162.4, 160.4, 138.9, 138.0, 135.6, 133.5, 133.2, 123.4, 107.1, 106.7; CH, 129.5, 129.0, 125.2, 123.2, 120.9, 115.5, 114.3; CH_3, 55.8, 55.5; *m*/*z* (%): 525 (M+K, 30), 510 (M+Na, 100), 488 (M+1, 5), 295 (6+8), 36 (61); IR (KBr, cm^{-1}): 3097, 1589, 1560, 1510, 1303, 1258, 1164, 1078, 1021, 911, 833; Anal. Calcd for $C_{24}H_{17}N_5O_7$: C, 59.12; H, 3.52; N, 14.37; found: C, 58.86; H, 3.34; N, 14.28.

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