Facile One-pot Synthesis of 1,2,3-triazoles Featuring Oxygen, Nitrogen, and Sulfur Functionalized Pendant Arms

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Supporting information

Experimental details for compounds **1a-f**, **2a-f**, **3a-f** and **4a-f**

¹H and ¹³C NMR spectra of compounds **1a-f**, **2a-f**, **3a-f** and **4a-f**

Experimental details for compounds **1a-f**, **2a-f**, **3a-f** and **4a-f**.

4.1 General methods

Commercially available reagents and solvents were used as received. Alkynes **I**,¹² **II**,¹³ **III**²³ and **IV**²² were synthesized as reported in the literature. Compound **3a** has been reported elsewhere⁵ and its spectroscopic characterization is consistent with the one described in this article. Flash column chromatography was performed on Kieselgel silica gel 60 (230-400 mesh). Melting points were determined on a Fisher-Johns apparatus and are uncorrected. IR spectra were recorded on a Bruker Alpha FT-IR/ATR spectrometer. NMR spectra were obtained with a JEOL ECA-500 (500 MHz) spectrometer. Chemical shifts (δ) are given in ppm downfield from Me₄Si as an internal reference; coupling constants are given in Hertz. High-resolution mass spectra (HRMS) were recorded on a JEOL JMS-SX 102a and Agilent-MSD-TOF-1069A spectrometers 41.

1.1. Typical procedure for the synthesis of 1,2,3-triazoles derived from methylaniline

(1-Benzyl-1H-1,2,3-triazole-4-ylmethyl)-methyl-phenyl-amine (1a). To a 20 mL round-bottomed flask equipped with a magnetic stirrer, were charged 11 mg (0.053 mmol, 5 mol%) of Cu(OAc)₂•H₂O, 10 mg (0.053 mmol, 5 mol%) of 1,10-phenanthroline monohydrate, and 204 mg (1.03 mmol) of sodium L-ascorbate. After addition of 7 mL of a mixture EtOH-H₂O (4:1 v/v), the resulting suspension was stirred for five minutes at room temperature. Subsequently, 150 mg (1.03 mmol) of (**I**), 81 mg (1.24 mmol) of sodium azide, and 0.14 mL (1.24 mmol) of benzyl chloride were added to the reaction mixture, which was stirred during 18 h at room temperature. 5 mL of H₂O were added to the reaction mixture and the precipitate was filtered off, washed thoroughly with H₂O, petroleum ether, and dried under vacuum. The crude product was purified by column chromatography

(CH₂Cl₂) to afford 200 mg (70% Yield) of **3a** as a white solid. mp = 82-84 °C. ¹H-NMR (CDCl₃, 500 MHz): δ = 2.96 (s, 3H, NCH₃), 4.61 (s, 2H, ArCH₂N), 5.45 (s, 2H, NCH₂C=C), 6.72 (t, 7.9 Hz, 1H, CH_{ar}), 6.76 (d, 7.8 Hz, 2H, CH_{ar}), 7.18-7.22 (m, 4H, CH_{ar}), 7.21 (s, 1H, CH_{triazole}), 7.33-7.35 (m, 3H, CH_{ar}). ¹³C-NMR (CDCl₃, 125.76 MHz): δ = 38.5 (NCH₃), 48.8 (ArCH₂N), 54.1 (NCH₂C=C), 113.0 (CH_{ar}), 117.2 (CH_{ar}), 121.4 (CH_{triazole}), 127.8 (CH_{ar}), 128.6 (CH_{ar}), 129.0 (CH_{ar}), 129.2 (CH_{ar}), 134.7 (C_{ar}), 145.9 (C_{triazole}), 149.1 (C_{ar}). FT-IR/ATR ν_{max} cm⁻¹: 3124, 3059, 3032, 2944, 2900, 1820, 1726, 1598, 1570. HRMS (ESI-TOF) calculated for C₁₇H₁₈N₄ + H⁺: 279.1610; Found: 279.1608.

[1-(4-Fluorobenzyl)-1H-1,2,3-triazole-4-ylmethyl]-methyl-phenyl-amine (1b). The procedure described for (**1a**) was followed using (1.24 mmol) of 4-fluorobenzyl chloride. The crude product was purified by column chromatography (CH₂Cl₂-MeOH 98:2 v/v) to afford 220 mg (72% Yield) of the title product as a white solid. mp = 62-64 °C. ¹H-NMR (CDCl₃, 500 MHz): δ = 2.97 (s, 3H, NCH₃), 4.61 (s, 2H, ArCH₂N), 5.41 (s, 2H, NCH₂C=C), 6.73 (t, 8.0 Hz, 1H, CH_{ar}), 6.75 (d, 7.8 Hz, 2H, CH_{ar}), 7.01-7.04 (m, 2H, CH_{ar}), 7.17-7.21 (m, 4H, CH_{ar}), 7.20 (s, 1H, CH_{triazole}). ¹³C-NMR (CDCl₃, 125.76 MHz): δ = 38.6 (NCH₃), 48.8 (ArCH₂N), 53.3 (NCH₂C=C), 113.0 (CH_{ar}), 116.1 (d, 22.6 Hz, CH_{ar-F}), 117.2 (CH_{ar}), 121.3 (CH_{triazole}), 129.2 (CH_{ar}), 129.7 (d, 8.8 Hz, CH_{ar-F}), 130.6 (d, 2.5 Hz, C_{ar-F}), 146.1 (C_{triazole}), 149.1 (C_{ar}), 162.8 (d, 247.8 Hz, C_{ar-F}). FT-IR/ATR ν_{max} cm⁻¹: 3124, 3040, 2947, 2883, 2823, 2110, 1912, 1595, 1571. HRMS (ESI-TOF) calculated for C₁₇H₁₇FN₄ + H⁺: 297.1515; Found: 297.1517

[1-(4-Chlorobenzyl)-1H-1,2,3-triazole-4-ylmethyl]-methyl-phenyl-amine (1c). The procedure described for (**1a**) was followed using (1.24 mmol) of 4-chlorobenzyl chloride. The crude product was purified by column chromatography (CH₂Cl₂-MeOH 99:1 v/v) to

afford 229 mg (71% Yield) of the title product as a white solid. mp = 64-66 °C. ¹H-NMR (CDCl₃, 500 MHz): δ = 2.97 (s, 3H, NCH₃), 4.61 (s, 2H, ArCH₂N), 5.40 (s, 2H, NCH₂C=C), 6.73 (t, 7.9 Hz, 1H, CH_{ar}), 6.75 (d, 7.8 Hz, 2H, CH_{ar}), 7.12 (d, 8.0 Hz, 2H, CH_{ar}), 7.19-7.22 (m, 2H, CH_{ar}), 7.21 (s, 1H, CH_{triazole}), 7.30 (d, 8.0 Hz, 2H, CH_{ar}). ¹³C-NMR (CDCl₃, 125.76 MHz): δ = 38.5 (NCH₃), 48.7 (ArCH₂N), 53.2 (NCH₂C=C), 113.0 (CH_{ar}), 117.2 (CH_{ar}), 121.3 (CH_{triazole}), 129.1 (CH_{ar}), 129.18 (CH_{ar}), 129.2 (CH_{ar}), 133.2 (C_{ar}), 134.6 (C_{ar}), 146.1 (C_{triazole}), 148.9 (C_{ar}). FT-IR/ATR ν_{max} cm⁻¹: 3113, 3064, 2949, 2909, 2832, 1915, 1595, 1504, 1490. HRMS (ESI-TOF) calculated for C₁₇H₁₇ClN₄ + H⁺: 313.1220; Found: 313.1215.

[1-(4-Bromobenzyl)-1H-1,2,3-triazole-4-ylmethyl]-methyl-phenyl-amine (1d). The procedure described for (1a) was followed using (1.24 mmol) of 4-bromobenzyl chloride. The crude product was purified by column chromatography (CH₂Cl₂-MeOH 99:1 v/v) to afford 283 mg (77% Yield) of the title product as a white solid. mp = 80-82 °C. ¹H-NMR (CDCl₃, 500 MHz): δ = 2.97 (s, 3H, NCH₃), 4.61 (s, 2H, ArCH₂N), 5.39 (s, 2H, NCH₂C=C), 6.71-6.76 (m, 3H, CH_{ar}), 7.06 (d, 7.8 Hz, 2H, CH_{ar}), 7.19-7.22 (m, 2H, CH_{ar}), 7.21 (s, 1H, CH_{triazole}), 7.46 (d, 7.8 Hz, 2H, CH_{ar}). ¹³C-NMR (CDCl₃, 125.76 MHz): δ = 38.1 (NCH₃), 48.2 (ArCH₂N), 52.8 (NCH₂C=C), 112.5 (CH_{ar}), 116.7 (CH_{ar}), 120.9 (CH_{triazole}), 122.2 (C_{ar}), 128.7 (CH_{ar}), 128.9 (CH_{ar}), 131.7 (CH_{ar}), 133.2 (C_{ar}), 145.6 (C_{triazole}), 148.5 (C_{ar}). FT-IR/ATR ν_{max} cm⁻¹: 3123, 3075, 3043, 2954, 2928, 2928, 2895, 2099, 1912, 1720, 1686, 1598, 1506. HRMS (ESI-TOF) calculated for C₁₇H₁₇BrN₄ + H⁺: 357.0715; Found: 357.0710.

[1-(4-Iodobenzyl)-1H-1,2,3-triazole-4-ylmethyl]-methyl-phenyl-amine (1e). The procedure described for (1a) was followed using (1.24 mmol) of 4-iodobenzyl bromide.

The crude product was purified by column chromatography (CH₂Cl₂-MeOH 99:1 v/v) to afford 345 mg (83% Yield) of the title product as a white solid. mp = 91-93 °C. ¹H-NMR (CDCl₃, 500 MHz): δ = 2.97 (s, 3H, NCH₃), 4.62 (s, 2H, ArCH₂N), 5.38 (s, 2H, NCH₂C=C), 6.71-6.76 (m, 3H, CH_{ar}), 6.93 (d, 7.9 Hz, 2H, CH_{ar}), 7.19-7.23 (m, 2H, CH_{ar}), 7.20 (s, 1H, CH_{triazole}), 7.65 (d, 7.9 Hz, 2H, CH_{ar}). ¹³C-NMR (CDCl₃, 125.76 MHz): δ = 38.6 (NCH₃), 48.8 (ArCH₂N), 53.5 (NCH₂C=C), 94.4 (C_{ar}), 113.0 (CH_{ar}), 117.2 (CH_{ar}), 121.4 (CH_{triazole}), 129.2 (CH_{ar}), 129.6 (CH_{ar}), 134.3 (C_{ar}), 138.2 (CH_{ar}), 146.2 (C_{triazole}), 149.0 (C_{ar}). FT-IR/ATR ν_{max} cm⁻¹: 3124, 3038, 2947, 2883, 2823, 2110, 1912, 1595, 1504. HRMS (ESI-TOF) calculated for C₁₇H₁₇N₄ + H⁺: 405.0576; Found: 405.0574.

[1-(4-Methoxybenzyl)-1*H*-1,2,3-triazole-4-ylmethyl]-methyl-phenyl-amine (1f). The procedure described for (1a) was followed using (1.24 mmol) of 4-methoxybenzyl chloride. The crude product was purified by column chromatography (CH₂Cl₂-MeOH 97:3 v/v) to afford 200 mg (63% Yield) of the title product as a white solid. mp = 79-81 °C. ¹H-NMR (CDCl₃, 500 MHz): δ = 2.96 (s, 3H, NCH₃), 3.79 (s, 3H, OCH₃), 4.59 (s, 2H, ArCH₂N), 5.37 (s, 2H, NCH₂C=C), 6.72 (t, 7.9 Hz, 1H, CH_{ar}), 6.74 (d, 7.9 Hz, 2H, CH_{ar}), 6.86 (d, 8.0 Hz, 2H, CH_{ar}), 7.15 (d, 8.0 Hz, 2H, CH_{ar}), 7.18 (s, 1H, CH_{triazole}), 7.19-7.22 (m, 2H, CH_{ar}). ¹³C-NMR (CDCl₃, 125.76 MHz): δ = 38.5 (NCH₃), 48.8 (ArCH₂N), 53.6 (NCH₂C=C), 55.3 (OCH₃), 113.0 (CH_{ar}), 114.4 (CH_{ar}), 117.1 (CH_{ar}), 121.2 (CH_{triazole}), 126.7 (C_{ar}), 129.2 (CH_{ar}), 129.4 (CH_{ar}), 145.6 (C_{triazole}), 149.1 (C_{ar}), 159.8 (C_{ar}). FT-IR/ATR ν_{max} cm⁻¹: 3111, 3061, 3037, 2991, 2934, 2905, 2836, 1598, 1573, 1506. HRMS (ESI-TOF) calculated for C₁₈H₂₀N₄O + H⁺: 309.1715 Found: 309.1716.

4.2 Typical procedure for the synthesis of 1,2,3-triazoles derived from thiophenol

1-Benzyl-4-phenylthiomethyl-1H-1,2,3-triazole (2a). To a 20 mL round-bottomed flask equipped with a magnetic stirrer, were charged 6 mg (0.031 mmol, 5 mol%) of Cu(OAc)₂•H₂O, 6 mg (0.031 mmol, 5 mol%) of 1,10-phenanthroline monohydrate, and 150 mg (0.76 mmol) of sodium L-ascorbate. After addition of 5 mL of a mixture EtOH-H₂O (4:1 v/v), the resulting suspension was stirred for five minutes at room temperature. Subsequently, 90 mg (0.61 mmol) of (**II**), 54 mg (0.83 mmol) of sodium azide, and 0.09 mL (0.83 mmol) of benzyl chloride were added to the reaction mixture, which was stirred during 16 h at room temperature. 5 mL of H₂O were added to the reaction mixture and the precipitate was filtered off, washed thoroughly with H₂O, petroleum ether, and dried under vacuum. The crude product was purified by column chromatography (CH₂Cl₂-MeOH 99:1 v/v) to afford 136 mg (79% Yield) of **2a** as a white solid. mp = 71-73 °C. ¹H-NMR (CDCl₃, 500 MHz): δ = 4.23 (s, 2H, ArCH₂N), 5.47 (s, 2H, ArSCH₂), 7.17-7.20 (m, 3H, CH_{ar}), 7.23-7.26 (m, 3H, CH_{ar}), 7.30-7.32 (m, 2H, CH_{ar}), 7.36-7.37 (m, 2H, CH_{ar}), 7.36 (s, 1H, CH_{triazole}). ¹³C-NMR (CDCl₃, 125.76 MHz): δ = 29.1 (ArCH₂N), 54.1 (ArSCH₂), 122.0 (CH_{triazole}), 126.5 (CH_{ar}), 127.9 (CH_{ar}), 128.7 (CH_{ar}), 128.9 (CH_{ar}), 129.1 (CH_{ar}), 129.8 (CH_{ar}), 134.6 (C_{ar}), 135.4 (C_{ar}), 145.5 (C_{triazole}). FT-IR/ATR ν_{max} cm⁻¹: 3140, 3073, 3046, 3013, 2986, 2951, 2923, 1956, 1726, 1586, 1553, 1476, 1455. HRMS (ESI-TOF) calculated for C₁₆H₁₅N₃S + H⁺: 281.0987; Found: 282.1058.

1-(4-Fluorobenzyl)-4-phenylthiomethyl-1H-1,2,3-triazole (2b). The procedure described for (**2a**) was followed using (0.83 mmol) of 4-fluorobenzyl chloride. The crude product was purified by column chromatography (CH₂Cl₂-MeOH 99:1 v/v) to afford 137 mg (75% Yield) of the title product as a white solid. mp = 81-83 °C. ¹H-NMR (CDCl₃, 500 MHz): δ = 4.20 (s, 2H, ArCH₂N), 5.41 (s, 2H, ArSCH₂), 7.01-7.05 (m, 2H, CH_{ar}), 7.14-7.18 (m, 3 H,

CH_{ar}), 7.21 (s, 1H, CH_{triazole}), 7.21-7.24 (m, 2H, CH_{ar}), 7.28 (d, 7.9 Hz, 2H, CH_{ar}). ¹³C-NMR (CDCl₃, 125.76 MHz): δ = 29.0 (ArCH₂N), 53.4 (ArSCH₂), 116.1 (d, 21.4 Hz, CH_{ar-F}), 121.9 (CH_{triazole}), 126.5 (CH_{ar}), 128.9 (CH_{ar}), 129.7 (CH_{ar}), 129.8 (d, 8.8 Hz, CH_{ar-F}), 130.5 (d, 3.8 Hz, C_{ar-F}), 135.3 (C_{ar}), 145.5 (C_{triazole}), 162.8 (d, 247.8 Hz, C_{ar-F}). FT-IR/ATR ν_{max} cm⁻¹: 3121, 3079, 3064, 2986, 2964, 2934, 1874, 1726, 1605, 1580, 1522, 1506, 1481. HRMS (ESI-TOF) calculated for C₁₆H₁₄N₃SF + H⁺: 300.0971; Found: 300.0959.

1-(4-Chlorobenzyl)-4-phenylthiomethyl-1H-1,2,3-triazole (2c). The procedure described for (2a) was followed using (0.83 mmol) of 4-chlorobenzyl chloride. The crude product was purified by column chromatography (CH₂Cl₂-MeOH 99:1 v/v) to afford 156 mg (81% Yield) of the title product as a white solid. mp = 95-97 °C. ¹H-NMR (CDCl₃, 500 MHz): δ = 4.20 (s, 2H, ArCH₂N), 5.41 (s, 2H, ArSCH₂), 7.09 (d, 7.8 Hz, 2H, CH_{ar}), 7.17 (t, 7.9 Hz, 1H, CH_{ar}), 7.21-7.24 (m, 2H, CH_{ar}), 7.22 (s, 1H, CH_{triazole}), 7.27-7.29 (m, 2H, CH_{ar}), 7.31 (d, 7.8 Hz, 2H, CH_{ar}). ¹³C-NMR (CDCl₃, 125.76 MHz): δ = 28.9 (ArCH₂N), 53.3 (ArSCH₂), 121.9 (CH_{triazole}), 126.5 (CH_{ar}), 128.9 (CH_{ar}), 129.1 (CH_{ar}), 129.2 (CH_{ar}), 129.7 (CH_{ar}), 133.1 (C_{ar}), 134.7 (C_{ar}), 135.2 (C_{ar}), 145.5 (C_{triazole}). FT-IR/ATR ν_{max} cm⁻¹: 3121, 3078, 3055, 2960, 2924, 1892, 1723, 1579, 1551, 1490, 1461. HRMS (ESI-TOF) calculated for C₁₆H₁₄N₃SCl + H⁺: 316.0631; Found: 316.0672.

1-(4-Bromobenzyl)-4-phenylthiomethyl-1H-1,2,3-triazole (2d). The procedure described for (2a) was followed using (0.83 mmol) of 4-bromobenzyl bromide. The crude product was purified by column chromatography (CH₂Cl₂-MeOH 99:1 v/v) to afford 176 mg (80% Yield) of the title product as a white solid. mp = 91-93 °C. ¹H-NMR (CDCl₃, 500 MHz): δ = 4.23 (s, 2H, ArCH₂N), 5.42 (s, 2H, ArSCH₂), 7.05 (d, 7.8 Hz, 2H, CH_{ar}), 7.20 (t, 7.9 Hz, 1H, CH_{ar}), 7.24 (s, 1H, CH_{triazole}), 7.25-7.27 (m, 2H, CH_{ar}), 7.30-7.32 (m, 2H, CH_{ar}), 7.49

(d. 7.8 Hz, 2H, CH_{ar}). ¹³C-NMR (CDCl₃, 125.76 MHz): δ = 29.0 (ArCH₂N), 53.4 (ArSCH₂), 122.0 (CH_{triazole}), 122.9 (C_{ar}), 126.6 (CH_{ar}), 128.9 (CH_{ar}), 129.5 (CH_{ar}), 129.8 (CH_{ar}), 132.2 (CH_{ar}), 133.7 (C_{ar}), 135.2 (C_{ar}), 145.6 (C_{triazole}). FT-IR/ATR ν_{max} cm⁻¹: 3121, 3078, 3053, 2960, 2923, 1892, 1658, 1573, 1551. HRMS (ESI-TOF) calculated for C₁₆H₁₄N₃SBr + H⁺:360.0170; Found: 360.0165.

1-(4-Iodobenzyl)-4-phenylthiomethyl-1H-1,2,3-triazole (2e). The procedure described for (2a) was followed using (0.83 mmol) of 4-iodobenzyl bromide. The crude product was purified by column chromatography (CH₂Cl₂-MeOH 99:1 v/v) to afford 216 mg (87% Yield) of the title product as a white solid. mp = 93-95 °C. ¹H-NMR (CDCl₃, 500 MHz): δ = 4.23 (s, 2H, ArCH₂N), 5.40 (s, 2H, ArSCH₂), 6.92 (d, 7.7 Hz, 2H, CH_{ar}), 7.19 (t, 7.9 Hz, 1H, CH_{ar}), 7.23 (s, 1H, CH_{triazole}), 7.25-7.27 (m, 2H, CH_{ar}), 7.29-7.32 (m, 2H, CH_{ar}), 7.69 (d. 7.7 Hz, 2H, CH_{ar}). ¹³C-NMR (CDCl₃, 125.76 MHz): δ = 29.0 (ArCH₂N), 53.5 (ArSCH₂), 94.4 (C_{ar}), 122.0 (CH_{triazole}), 126.6 (CH_{ar}), 128.9 (CH_{ar}), 129.6 (CH_{ar}), 129.8 (CH_{ar}), 134.3 (C_{ar}), 135.2 (C_{ar}), 138.2 (CH_{ar}), 145.6 (C_{triazole}). FT-IR/ATR ν_{max} cm⁻¹: 3120, 3077, 3050, 2960, 2922, 1981, 1908, 1724, 1659, 1570, 1550. HRMS (ESI-TOF) calculated for C₁₆H₁₄N₃SI + H⁺:408.0031; Found: 408.0029.

1-(4-Methoxybenzyl)-4-phenylthiomethyl-1H-1,2,3-triazole (2f). The procedure described for (2a) was followed using (0.83 mmol) of 4-Methoxybenzyl chloride. The crude product was purified by column chromatography (CH₂Cl₂-MeOH 99:1 v/v) to afford 133 mg (70% Yield) of the title product as a white solid. mp = 76-78 °C. ¹H-NMR (CDCl₃, 500 MHz): δ = 3.80 (s, 3H, OCH₃), 4.19 (s, 2H, ArCH₂N), 5.38 (s, 2H, ArSCH₂), 6.86 (d, 7.8 Hz, 2H, CH_{ar}), 7.18 (t, 7.9 Hz, 1H, CH_{ar}), 7.19 (s, 1H, CH_{triazole}), 7.21-7.24 (m, 2H, CH_{ar}), 7.29 (d. 7.8 Hz, 2H, CH_{ar}). ¹³C-NMR (CDCl₃, 125.76 MHz): δ = 29.0 (ArCH₂N),

53.6 (ArSCH₂), 55.3 (OCH₃), 114.4 (CH_{ar}), 121.7 (CH_{triazole}), 126.4 (CH_{ar}), 126.6 (C_{ar}), 128.9 (CH_{ar}), 129.4 (CH_{ar}), 129.7 (CH_{ar}), 135.4 (C_{ar}), 145.2 (C_{triazole}), 159.8 (C_{ar}). FT-IR/ATR $\nu_{\max}$ cm⁻¹: 3106, 3061, 3008, 2949, 2920, 2834, 1911, 1723, 1610, 1582, 1544. HRMS (ESI-TOF) calculated for C₁₇H₁₇N₃SO + H⁺: 312.1171; Found: 312.1159.

4.3 Typical procedure for the synthesis of Triazoles derived from benzyl alcohol

4-[phenylmethoxy)methyl]-1-(benzyl)-1H-1,2,3-Triazole (3a). To a 20 mL round-bottomed flask equipped with a magnetic stirrer, were charged 7 mg (0.035 mmol, 5 mol%) of Cu(OAc)₂•H₂O, 7 mg (0.035 mmol, 5 mol%) of 1,10-phenanthroline monohydrate, and 139 mg (0.70 mmol) of sodium L-ascorbate. After addition of 5 mL of a mixture EtOH-H₂O (4:1 v/v), the resulting suspension was stirred for five minutes at room temperature. Subsequently, 103 mg (0.70 mmol) of (**1**), 50 mg (0.77 mmol) of sodium azide, and 0.09 mL (0.83 mmol) of benzyl chloride were added to the reaction mixture, which was stirred during 36 h at room temperature. 5 mL of H₂O were added to the reaction mixture and the precipitate was filtered off, washed thoroughly with H₂O, petroleum ether, and dried under vacuum. The crude product was purified by column chromatography (CH₂Cl₂-MeOH 98:2 v/v) to afford 150 mg (77% Yield) of **1a** as a white solid. mp = 53-55 °C. ¹H-NMR (CDCl₃, 500 MHz): δ = 4.58 (s, 2H, ArCH₂N), 4.65 (s, 2H, C-CH₂O), 5.50 (s, 2H, ArCH₂O), 7.25-7.27 (m, 3H, CH_{ar}), 7.32-7.34 (m, 4H, CH_{ar}), 7.35-7.37 (m, 3H, CH_{ar}), 7.45 (s, 1H, CH_{triazole}). ¹³C-NMR (CDCl₃, 125.76 MHz): δ = 54.2 (ArCH₂N), 63.8 (ArOCH₂), 72.6 (C-CH₂O), 122.5 (CH_{triazole}), 127.8 (CH_{ar}), 127.9 (CH_{ar}), 128.1 (CH_{ar}), 128.4 (CH_{ar}), 128.7 (CH_{ar}), 129.1 (CH_{ar}), 134.6 (C_{ar}), 137.8 (C_{ar}), 145.7 (C_{triazole}). FT-IR/ATR $\nu_{\max}$ cm⁻¹: 3128, 3063, 3031, 2954, 2862, 1495, 1455. HRMS (ESI-TOF) calculated for C₁₇H₁₇N₃O + H⁺: 280.1450; Found: 280.1443.

4-[phenylmethoxy)methyl]-1-(4-(fluoro)benzyl)]-1*H*-1,2,3-Triazole (3b). The procedure described for (3a) was followed using (0.83 mmol) of 4-fluorobenzyl chloride. The crude product was purified by column chromatography (CH₂Cl₂-MeOH 98:2 v/v) to afford 152 mg (73% Yield) of the title product as a white solid. mp = 70-72 °C. ¹H-NMR (CDCl₃, 500 MHz): δ = 4.58 (s, 2H, ArCH₂N), 4.66 (s, 2H, C-CH₂O), 5.48 (s, 2H, ArCH₂O), 7.04-7.07 (m, 2H, CH_{ar}), 7.25-7.29 (m, 3H, CH_{ar}), 7.31-7.34 (m, 4H, CH_{ar}), 7.44 (s, 1H, CH_{triazole}). ¹³C-NMR (CDCl₃, 125.76 MHz): δ = 53.4 (ArCH₂N), 63.7 (ArOCH₂), 72.6 (C-CH₂O), 116.1 (d, 22.6 Hz, CH_{ar-F}), 122.2 (CH_{triazole}), 127.8 (CH_{ar}), 127.9 (CH_{ar}), 128.4 (CH_{ar}), 129.9 (d, 8.8 Hz, CH_{ar-F}), 130.4 (d, 3.8 Hz, C_{ar-F}), 137.7 (C_{ar}), 145.8 (C_{triazole}), 162.8 (d, 247.8 Hz, C_{ar-F}). FT-IR/ATR ν_{max} cm⁻¹: 3130, 3072, 3041, 3022, 2926, 2850, 2809, 1901, 1725, 1603, 1507. HRMS (ESI-TOF) calculated for C₁₇H₁₆N₃OF + H⁺: 298.1356; Found: 298.1357.

4-[phenylmethoxy)methyl]-1-(4-(chloro)benzyl)]-1*H*-1,2,3-Triazole (3c). The procedure described for (3a) was followed using (0.83 mmol) of 4-chlorobenzyl chloride. The crude product was purified by column chromatography (CH₂Cl₂-MeOH 98:2 v/v) to afford 160 mg (73% Yield) of the title product as a white solid. mp = 65-67 °C. ¹H-NMR (CDCl₃, 500 MHz): δ = 4.61 (s, 2H, ArCH₂N), 4.69 (s, 2H, C-CH₂O), 5.50 (s, 2H, ArCH₂O), 7.23 (d, 8.0 Hz, 2H, CH_{ar}), 7.29 (t, 7.9 Hz, 1H, CH_{ar}), 7.31-7.32 (m, 4H, CH_{ar}), 7.37 (d, 7.2 Hz, 2H, CH_{ar}), 7.48 (s, 1H, CH_{triazole}). ¹³C-NMR (CDCl₃, 125.76 MHz): δ = 53.4 (ArCH₂N), 63.7 (ArOCH₂), 72.7 (C-CH₂O), 122.3 (CH_{triazole}), 127.8 (CH_{ar}), 127.9 (CH_{ar}), 128.4 (CH_{ar}), 129.3 (CH_{ar}), 129.4 (CH_{ar}), 133.1 (C_{ar}), 134.8 (C_{ar}), 137.7 (C_{ar}), 145.9 (C_{triazole}). FT-IR/ATR ν_{max} cm⁻¹: 3131, 3072, 3042, 3024, 2864, 2809, 1963, 1604, 1508. HRMS (ESI-TOF) calculated for C₁₇H₁₆N₃OCl + H⁺: 314.1060; Found: 314.1055.

4-[phenylmethoxy)methyl]-1-(4-(bromo)benzyl)]-1*H*-1,2,3-Triazole (3d). The procedure described for (3a) was followed using (0.83 mmol) of 4-bromobenzyl bromide. The crude product was purified by column chromatography (CH₂Cl₂-MeOH 98:2 v/v) to afford 213 mg (85% Yield) of the title product as a white solid. mp = 84-86 °C. ¹H-NMR (CDCl₃, 500 MHz): δ = 4.61 (s, 2H, ArCH₂N), 4.69 (s, 2H, C-CH₂O), 5.48 (s, 2H, ArCH₂O), 7.16 (d, 7.9 Hz, 2H, CH_{ar}), 7.29 (t, 7.8 Hz, 1H, CH_{ar}), 7.32-7.36 (m, 4H, CH_{ar}), 7.51 (s, 1H, CH_{triazole}), 7.53 (d, 7.2 Hz, 2H, CH_{ar}). ¹³C-NMR (CDCl₃, 125.76 MHz): δ = 53.5 (ArCH₂N), 63.7 (ArOCH₂), 72.7 (C-CH₂O), 122.3 (CH_{triazole}), 122.9 (C_{ar}), 127.8 (CH_{ar}), 127.9 (CH_{ar}), 128.4 (CH_{ar}), 129.7 (CH_{ar}), 132.3 (CH_{ar}), 133.6 (C_{ar}), 137.7 (C_{ar}), 145.9 (C_{triazole}). FT-IR/ATR ν_{max} cm⁻¹: 3131, 3092, 3033, 2947, 2877, 2855, 2800, 1915, 1720, 1587, 1559. HRMS (ESI-TOF) calculated for C₁₇H₁₆N₃OBr + H⁺: 358.0555; Found: 358.0552.

4-[phenylmethoxy)methyl]-1-(4-(iodo)benzyl)]-1*H*-1,2,3-Triazole (3e). The procedure described for (3a) was followed using (0.83 mmol) of 4-iodobenzyl bromide. The crude product was purified by column chromatography (CH₂Cl₂-MeOH 98:2 v/v) to afford 224 mg (79% Yield) of the title product as a white solid. mp = 101-103 °C. ¹H-NMR (CDCl₃, 500 MHz): δ = 4.58 (s, 2H, ArCH₂N), 4.66 (s, 2H, C-CH₂O), 5.44 (s, 2H, ArCH₂O), 7.00 (d, 7.9 Hz, 2H, CH_{ar}), 7.29 (t, 7.7 Hz, 1H, CH_{ar}), 7.30-7.34 (m, 4H, CH_{ar}), 7.44 (s, 1H, CH_{triazole}), 7.70 (d, 7.9 Hz, 2H, CH_{ar}). ¹³C-NMR (CDCl₃, 125.76 MHz): δ = 53.5 (ArCH₂N), 63.7 (ArOCH₂), 72.6 (C-CH₂O), 94.5 (C_{ar}), 122.3 (CH_{triazole}), 127.8 (CH_{ar}), 127.9 (CH_{ar}), 128.4 (CH_{ar}), 129.9 (CH_{ar}), 134.2 (C_{ar}), 137.7 (C_{ar}), 138.2 (CH_{ar}), 145.9 (C_{triazole}). FT-IR/ATR ν_{max} cm⁻¹: 3134, 3091, 2940, 2869, 2857, 2807, 1720, 1567. HRMS (ESI-TOF) calculated for C₁₇H₁₆N₃OI + H⁺: 406.0416; Found: 406.0411.

4-[(phenylmethoxy)methyl]-1-(4-(methoxy)benzyl)-1H-1,2,3-Triazole (3f). The procedure described for (3a) was followed using (0.83 mmol) of 4-methoxybenzyl chloride. The crude product was purified by column chromatography (CH₂Cl₂-MeOH 98:2 v/v) to afford 145 mg (67% Yield) of the title product as a white solid. mp = 54-56 °C. ¹H-NMR (CDCl₃, 500 MHz): δ = 3.80 (s, 3H, OCH₃), 4.57 (s, 2H, ArCH₂N), 4.64 (s, 2H, C-CH₂O), 5.44 (s, 2H, ArCH₂O), 6.90 (d, 7.9 Hz, 2H, CH_{ar}), 7.22 (d, 7.9 Hz, 2H, CH_{ar}), 7.28 (t, 7.8 Hz, 1H, CH_{ar}), 7.31-7.33 (m, 4H, CH_{ar}), 7.41 (s, 1H, CH_{triazole}). ¹³C-NMR (CDCl₃, 125.76 MHz): δ = 53.7 (ArCH₂N), 55.3 (OCH₃), 63.7 (ArOCH₂), 72.5 (C-CH₂O), 114.4 (CH_{ar}), 122.1 (CH_{triazole}), 126.5 (C_{ar}), 127.7 (CH_{ar}), 127.9 (CH_{ar}), 128.4 (CH_{ar}), 129.7 (CH_{ar}), 137.7 (C_{ar}), 145.5 (C_{triazole}), 159.9 (C_{ar}). FT-IR/ATR ν_{max} cm⁻¹: 3120, 3076, 3059, 3011, 2957, 2857, 2836, 1890, 1723, 1611, 1586, 1514. HRMS (ESI-TOF) calculated for C₁₈H₁₉N₃O₂ + H⁺: 310.1556; Found: 310.1553.

4.4 Typical procedure for the synthesis of bis-1,2,3-triazoles derived from di-propargyl aniline

Bis-(1-benzyl-1H-1,2,3-triazole-4-ylmethyl)-phenyl-amine (4a). To a 20 mL round-bottomed flask equipped with a magnetic stirrer, were charged 6 mg (0.030 mmol, 5 mol%) of Cu(OAc)₂•H₂O, 6 mg (0.030 mmol, 5 mol%) of 1,10-phenanthroline monohydrate, and 117 mg (0.59 mmol) of sodium L-ascorbate. After addition of 5 mL of a mixture EtOH-H₂O (4:1 v/v), the resulting suspension was stirred for five minutes at room temperature. Subsequently, 100 mg (0.59 mmol) of (IV), 92 mg (1.42 mmol) of sodium azide, and 0.16 mL (1.42 mmol) of benzyl chloride were added to the reaction mixture, which was stirred during 24 h at room temperature. 5 mL of H₂O were added to the reaction mixture and the precipitate was filtered off, washed thoroughly with H₂O, petroleum ether, and dried under

vacuum. The crude product was purified by column chromatography (CH₂Cl₂-MeOH 98:2 v/v) to afford 185 mg (72% Yield) of **4a** as a white solid. mp = 146-148 °C. ¹H-NMR (CDCl₃, 500 MHz): δ = 4.65 (s, 4H, ArCH₂N), 5.45 (s, 4H, NCH₂C=C), 6.75 (t, 7.9 Hz, 1H, CH_{ar}), 6.85 (d, 7.8 Hz, 2H, CH_{ar}), 7.18-7.20 (m, 3H, CH_{ar}), 7.21-7.22 (m, 3H, CH_{ar}), 7.31 (s, 2H, CH_{triazole}), 7.34-7.36 (m, 4H, CH_{ar}), 7.37-7.38 (m, 2H, CH_{ar}). ¹³C-NMR (CDCl₃, 125.76 MHz): δ = 46.9 (ArCH₂N), 54.1 (NCH₂C=C), 113.7 (CH_{ar}), 117.8 (CH_{ar}), 121.9 (CH_{triazole}), 127.8 (CH_{ar}), 128.7 (CH_{ar}), 129.0 (CH_{ar}), 129.3 (CH_{ar}), 134.7 (C_{ar}), 145.8 (C_{triazole}), 147.9 (C_{ar}). FT-IR/ATR ν_{max} cm⁻¹: 3116, 3066, 3027, 3003, 3027, 3003, 2960, 2894, 1719, 1597, 1503. HRMS (ESI-TOF) calculated for C₂₆H₂₅N₇ + H⁺: 436.2250; Found: 436.2247.

Bis-[1-(4-fluorobenzyl)-1*H*-1,2,3-triazole-4-ylmethyl]-phenyl-amine (4b). The procedure described for (**1a**) was followed using (1.42 mmol) of 4-fluorobenzyl chloride. The crude product was purified by column chromatography (CH₂Cl₂-MeOH 98:2 v/v) to afford 203 mg (73% Yield) of the title product as a white solid. mp = 122-124 °C. ¹H-NMR (CDCl₃, 500 MHz): δ = 4.61 (s, 4H, ArCH₂N), 5.40 (s, 4H, NCH₂C=C), 6.73 (t, 7.9 Hz, 1H, CH_{ar}), 6.82 (d, 7.8 Hz, 2H, CH_{ar}), 7.00-7.04 (m, 4H, CH_{ar}), 7.15-7.19 (m, 6H, CH_{ar}), 7.31 (s, 2H, CH_{triazole}). ¹³C-NMR (CDCl₃, 125.76 MHz): δ = 46.8 (ArCH₂N), 53.3 (NCH₂C=C), 113.7 (CH_{ar}), 116.1 (d, 22.6 Hz, CH_{ar-F}), 117.9 (CH_{ar}), 121.9 (CH_{triazole}), 129.3 (CH_{ar}), 129.7 (d, 7.5 Hz, CH_{ar-F}), 130.5 (d, 3.8 Hz, C_{ar-F}), 145.9 (C_{triazole}), 147.9 (C_{ar}), 162.7 (d, 247.7 Hz, C_{ar-F}). FT-IR/ATR ν_{max} cm⁻¹: 3120, 3071, 3006, 2956, 2937, 2874, 1719, 1690, 1596, 1549, 1504. HRMS (ESI-TOF) calculated for C₂₆H₂₃F₂N₇ + H⁺: 472.2061; Found: 472.2059.

Bis-[1-(4-chlorobenzyl)-1*H*-1,2,3-triazole-4-ylmethyl]-phenyl-amine (4c). The procedure described for (**1a**) was followed using (1.42 mmol) of 4-chlorobenzyl chloride. The crude product was purified by column chromatography (CH₂Cl₂-MeOH 99:1 v/v) to afford 232 mg (78% Yield) of the title product as a white solid. mp = 112-114 °C. ¹H-NMR (CDCl₃, 500 MHz): δ = 4.62 (s, 4H, ArCH₂N), 5.39 (s, 4H, NCH₂C=C), 6.74 (t, 7.9 Hz, 1H, CH_{ar}), 6.82 (d, 7.8 Hz, 2H, CH_{ar}), 7.11 (d, 7.8 Hz, 4H, CH_{ar}), 7.16-7.19 (m, 2H, CH_{ar}), 7.30 (s, 2H, CH_{triazole}), 7.31 (d, 4H, 7.8 Hz, CH_{ar}). ¹³C-NMR (CDCl₃, 125.76 MHz): δ = 46.8 (ArCH₂N), 53.2 (NCH₂C=C), 113.7 (CH_{ar}), 117.9 (CH_{ar}), 121.9 (CH_{triazole}), 129.1 (CH_{ar}), 129.2 (two signals overlapping, CH_{ar}), 133.1 (C_{ar}), 134.7 (CH_{ar}), 145.9 (C_{triazole}), 147.8 (C_{ar}). FT-IR/ATR ν_{max} cm⁻¹: 3118, 3068, 2930, 2875, 2244, 1996, 1896, 1720, 1597, 1503. HRMS (ESI-TOF) calculated for C₂₆H₂₃Cl₂N₇ + H⁺: 504.1470; Found: 504.1468.

Bis-[1-(4-bromobenzyl)-1*H*-1,2,3-triazole-4-ylmethyl]-phenyl-amine (4d). The procedure described for (**1a**) was followed using (1.42 mmol) of 4-bromobenzyl chloride. The crude product was purified by column chromatography (CH₂Cl₂-MeOH 99:1 v/v) to afford 262 mg (75% Yield) of the title product as a white solid. mp = 128-130 °C. ¹H-NMR (CDCl₃, 500 MHz): δ = 4.62 (s, 4H, ArCH₂N), 5.37 (s, 4H, NCH₂C=C), 6.74 (t, 8.0 Hz, 1H, CH_{ar}), 6.82 (d, 7.8 Hz, 2H, CH_{ar}), 7.05 (d, 7.8 Hz, 4H, CH_{ar}), 7.18-7.19 (m, 2H, CH_{ar}), 7.30 (s, 2H, CH_{triazole}), 7.46 (d, 4H, 7.8 Hz, CH_{ar}). ¹³C-NMR (CDCl₃, 125.76 MHz): δ = 46.8 (ArCH₂N), 53.3 (NCH₂C=C), 113.7 (CH_{ar}), 117.9 (CH_{ar}), 121.9 (CH_{triazole}), 122.8 (C_{ar}), 129.2 (CH_{ar}), 129.4 (CH_{ar}), 132.2 (CH_{ar}), 133.7 (C_{ar}), 145.9 (C_{triazole}), 147.8 (C_{ar}). FT-IR/ATR ν_{max} cm⁻¹: 3122, 3108, 3065, 3041, 2955, 2937, 2887, 1915, 1719, 1681, 1597, 1577, 1503. HRMS (ESI-TOF) calculated for C₂₆H₂₃Br₂N₇ + H⁺: 594.0439; Found: 594.0483.

Bis-[1-(4-iodobenzyl)-1*H*-1,2,3-triazole-4-ylmethyl]-phenyl-amine (4e). The procedure described for (**1a**) was followed using (1.42 mmol) of 4-iodobenzyl bromide. The crude product was purified by column chromatography (CH₂Cl₂-MeOH 99:1 v/v) to afford 340 mg (84% Yield) of the title product as a white solid. mp = 155-157 °C. ¹H-NMR (CDCl₃, 500 MHz): δ = 4.62 (s, 4H, ArCH₂N), 5.36 (s, 4H, NCH₂C=C), 6.74 (t, 7.9 Hz, 1H, CH_{ar}), 6.81 (d, 7.8 Hz, 2H, CH_{ar}), 6.91 (d, 7.8 Hz, 4H, CH_{ar}), 7.16-7.19 (m, 2H, CH_{ar}), 7.29 (s, 2H, CH_{triazole}), 7.67 (d, 4H, 7.8 Hz, CH_{ar}). ¹³C-NMR (CDCl₃, 125.76 MHz): δ = 46.9 (ArCH₂N), 53.5 (NCH₂C=C), 94.4 (C_{ar}), 113.7 (CH_{ar}), 118.0 (CH_{ar}), 122.0 (CH_{triazole}), 129.3 (CH_{ar}), 129.6 (CH_{ar}), 134.3 (C_{ar}), 138.2 (CH_{ar}), 146.0 (C_{triazole}), 147.8 (C_{ar}). FT-IR/ATR ν_{max} cm⁻¹: 3115, 3062, 2919, 2872, 2110, 1918, 1718, 1671, 1596, 1503. HRMS (ESI-TOF) calculated for C₂₆H₂₃I₂N₇ + H⁺: 688.0183; Found: 688.0159.

Bis-[1-(4-methoxybenzyl)-1*H*-1,2,3-triazole-4-ylmethyl]-phenyl-amine (4f). The procedure described for (**1a**) was followed using (1.42 mmol) of 4-methoxybenzyl chloride. The crude product was purified by column chromatography (CH₂Cl₂-MeOH 99:1 v/v) to afford 190 mg (65% Yield) of the title product as a white solid. mp = 118-120 °C. ¹H-NMR (CDCl₃, 500 MHz): δ = 3.78 (s, 6H, OCH₃), 4.60 (s, 4H, ArCH₂N), 5.35 (s, 4H, NCH₂C=C), 6.72 (t, 8.0 Hz, 1H, CH_{ar}), 6.82 (d, 7.9 Hz, 2H, CH_{ar}), 6.85 (d, 7.8 Hz, 4H, CH_{ar}), 7.13 (d, 4H, 7.8 Hz, CH_{ar}), 7.16-7.18 (m, 2H, CH_{ar}), 7.24 (s, 2H, CH_{triazole}). ¹³C-NMR (CDCl₃, 125.76 MHz): δ = 46.8 (ArCH₂N), 53.6 (NCH₂C=C), 55.3 (OCH₃), 113.6 (CH_{ar}), 114.4 (CH_{ar}), 117.7 (CH_{ar}), 121.6 (CH_{triazole}), 126.6 (C_{ar}), 129.2 (CH_{ar}), 129.4 (CH_{ar}), 145.9 (C_{triazole}), 147.9 (C_{ar}), 159.9 (C_{ar}). FT-IR/ATR ν_{max} cm⁻¹: 3117, 3069, 2924, 2890, 2134, 1919, 1728, 1684, 1590, 1509. HRMS (ESI-TOF) calculated for C₂₈H₂₉N₇O₂ + H⁺: 496.2461; Found: 496.2454.

^1H and ^{13}C NMR spectra of compounds **1a-f**, **2a-f**, **3a-f** and **4a-f**

(1-Benzyl-1*H*-1,2,3-triazole-4-ylmethyl)-methyl-phenyl-amine (**1a**)

[1-(4-Fluorobenzyl)-1H-1,2,3-triazole-4-ylmethyl]-methyl-phenyl-amine (1b)

[1-(4-Chlorobenzyl)-1H-1,2,3-triazole-4-ylmethyl]-methyl-phenyl-amine (1c)

[1-(4-Bromobenzyl)-1H-1,2,3-triazole-4-ylmethyl]-methyl-phenyl-amine (1d)

[1-(4-Iodobenzyl)-1H-1,2,3-triazole-4-ylmethyl]-methyl-phenyl-amine (1e)

[1-(4-Methoxybenzyl)-1H-1,2,3-triazole-4-ylmethyl]-methyl-phenyl-amine (1f)

1-Benzyl-4-phenylthiomethyl-1H-1,2,3-triazole (2a)

1-(4-Fluorobenzyl)-4-phenylthiomethyl-1*H*-1,2,3-triazole (2b)

1-(4-Chlorobenzyl)-4-phenylthiomethyl-1H-1,2,3-triazole (2c)

1-(4-Bromobenzyl)-4-phenylthiomethyl-1H-1,2,3-triazole (2d)

1-(4-Iodobenzyl)-4-phenylthiomethyl-1H-1,2,3-triazole (2e)

1-(4-Methoxybenzyl)-4-phenylthiomethyl-1*H*-1,2,3-triazole (2f)

4-[phenylmethoxy)methyl]-1-(benzyl)-1*H*-1,2,3-Triazole (3a)

4-[phenylmethoxy)methyl]-1-(4-(fluoro)benzyl)-1*H*-1,2,3-Triazole (3b)

4-[phenylmethoxy)methyl]-1-(4-(chloro)benzyl)-1*H*-1,2,3-Triazole (3c)

4-[phenylmethoxy)methyl]-1-(4-(bromo)benzyl)-1*H*-1,2,3-Triazole (3d)

4-[phenylmethoxy)methyl]-1-(4-(iodo)benzyl)-1*H*-1,2,3-Triazole (3e)

4-[(phenylmethoxy)methyl]-1-(4-(methoxy)benzyl)-1*H*-1,2,3-Triazole (3f)

Bis-(1-benzyl-1*H*-1,2,3-triazole-4-ylmethyl)-phenyl-amine (4a)

Bis-[1-(4-fluorobenzyl)-1*H*-1,2,3-triazole-4-ylmethyl]-phenyl-amine (4b)

Bis-[1-(4-chlorobenzyl)-1*H*-1,2,3-triazole-4-ylmethyl]-phenyl-amine (4c)

Bis-[1-(4-bromobenzyl)-1*H*-1,2,3-triazole-4-ylmethyl]-phenyl-amine (4d)

Bis-[1-(4-iodobenzyl)-1*H*-1,2,3-triazole-4-ylmethyl]-phenyl-amine (4e)

Bis-[1-(4-methoxybenzyl)-1*H*-1,2,3-triazole-4-ylmethyl]-phenyl-amine (4f)

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