Supporting Information

Iron-Catalyzed C–H/N–H Activation by Triazole Guidance: Versatile Alkyne Annulation

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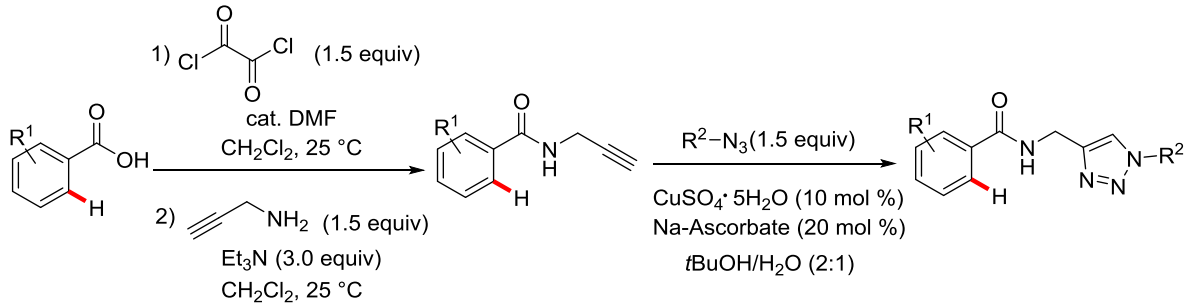
General Remarks

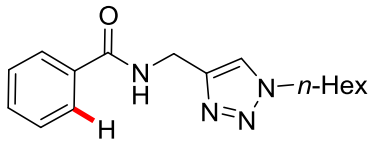
All reactions were carried out in Schlenk tubes under a N_2 atmosphere using pre-dried glassware. THF was dried using a solvent purification system (SPS) from MBRAUN. All starting materials were synthesized according to previously described methods.[1-2] Alkynes **4** were synthesized according to known procedures.[3] *i*PrMgBr (3.0 M in 2Me-THF) was purchased from Sigma-Aldrich. Other chemicals were obtained from commercial sources and were used without further purification. Yields refer to isolated compounds, estimated to be > 95% pure as determined by ^{1}H-NMR and GC analysis. Chromatography: Merck silica gel 60 (40-63 μm). NMR: Spectra were recorded on Varian Unity 300, Mercury 300 or Inova 500 in the solvent indicated; chemical shifts (δ) are given in ppm. All IR spectra were recorded on a Bruker FT-IR Alpha device. MS: EI-MS-spectra were recorded with Finnigan MAT 95, 70 eV; High resolution mass spectrometry (HRMS) with APEX IV 7T FTICR, Bruker Daltonic. Preparative HPLC was performed on a system from JAI (LC-92XX II Series, Injection- and Control-Valve, UV and RI Detector) connected to JAIGEL HH series columns. $CHCl_3$ of HPLC-grade was employed. M. p.: Stuart melting point apparatus SMP3, Barlworld Scientific, values are uncorrected.

Representative procedure for the synthesis of amides 1



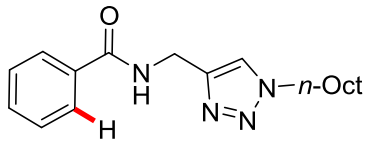
Oxalyl chloride (1.1 equiv) was added dropwise to a mixture of carboxylic acid (1.0 equiv), DMF (20 µl) in CH_2Cl_2 (10 ml) under N_2 atmosphere at 0 °C. The mixture was stirred at the same temperature for 5 h upon which it was allowed to warm up to ambient temperature. The crude acid chloride was cooled to 0 °C and it was added dropwise to a solution of propargylamine (1.5 equiv), NEt_3 (3.0 equiv) in dry CH_2Cl_2 (10 ml) at 0 °C. The mixture was initially stirred at the same temperature and then at ambient temperature for 12 h. To the reaction was added sat. aqueous $NaHCO_3$ (20 ml). The aqueous layers were extracted with CH_2Cl_2 (3x20 ml). The combined organic extracts were washed with HCl (1 M, 20 ml), brine and dried over Na_2SO_4 and the filtrate was concentrated under reduced pressure. The crude product was further submitted to the corresponding alkylazide (1.5 equiv), $CuSO_4 \cdot 5H_2O$ (10 mol %), sodium ascorbate (20 mol %) in a mixture of *t*BuOH/H_2O (2:1, 40:20 ml). After 3 h, to the reaction was added with sat. aqueous NH_4Cl (40 ml). The aqueous layers were extracted with EtOAc (3x40 ml). The combined organic extracts were dried over Na_2SO_4 and the filtrate was concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel.

***N*-[(1-*n*-Hexyl-1*H*-1,2,3-triazol-4-yl)methyl]benzamide (1b)**



The representive procedure was followed using benzoic acid (366 mg, 3.00 mmol) and 1-azido-*n*-hexane (571 mg, 4.50 mmol). Purification by column chromatography (*n*-hexane/EtOAc 7:3→ 1:1) yielded **1b** (705 mg, 82%) as a white solid. M. p. = 92-94 °C. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 7.85-7.77 (m, 2H), 7.64-7.60 (s, 1H), 7.48-7.42 (m, 2H), 7.41-7.35 (m, 2H), 4.66 (d, *J* = 5.3 Hz, 2H), 4.33-4.25 (m, 2H), 1.89-1.82 (m, 2H), 1.33-1.24 (m, 6H), 0.88-0.81 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 167.4 (C_q), 134.0 (C_q), 131.5 (C_q), 128.4 (CH), 127.3 (CH), 127.0 (CH), 122.4 (CH), 50.4 (CH_2), 35.3 (CH_2), 31.1 (CH_2), 30.1 (CH_2), 26.1 (CH_2), 22.3 (CH_2), 13.9 (CH_3). IR (ATR): 2954, 1638, 1486, 1373, 1059, 748, 589 cm^{-1}. MS (ESI) *m/z* (relative intensity): 595 (21) $[2M+Na]^+$, 309 (100) $[M+Na]^+$, 287 (43) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{16}H_{23}N_4O$ $[M+H]^+$ 287.1872 found 287.1870.

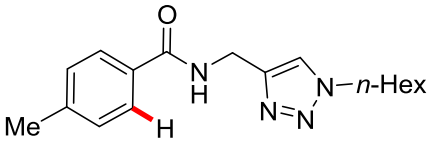
***N*-[(1-*n*-Octyl-1*H*-1,2,3-triazol-4-yl)methyl]benzamide (1c)**



The representive procedure was followed using benzoic acid (366 mg, 3.00 mmol) and 1-azido-*n*-octane (698 mg, 4.50 mmol). Purification by column chromatography (*n*-hexane/EtOAc 7:3→ 1:1) yielded **1c** (791 mg, 84%) as a white solid. M. p. = 105-107 °C. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 7.80 (d, *J* = 7.6 Hz, 2H), 7.62 (s, 1H), 7.49-7.41 (m, 1H), 7.38-7.34 (m, 2H), 7.32 (s, 1H), 4.66 (d, *J* = 5.3 Hz, 2H), 4.28 (t, *J* = 7.2 Hz, 2H), 1.89-1.81 (m, 2H), 1.30-1.19 (m, 10H), 0.85-0.80 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 167.4 (C_q), 134.0 (C_q), 131.5 (C_q), 128.4 (CH), 128.4 (CH), 127.0 (CH), 122.3 (CH), 50.4 (CH_2), 35.3 (CH_2), 31.6 (CH_2), 30.2 (CH_2), 29.0 (CH_2), 28.9 (CH_2), 26.4 (CH_2), 22.5 (CH_2), 14.0 (CH_3). IR (ATR): 2955, 1637, 1523, 1290, 1059, 785, 692, cm^{-1}. MS (ESI) *m/z* (relative

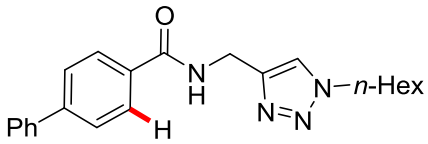
intensity): 651 (5) $[2M+Na]^+$, 337 (18) $[M+Na]^+$, 315 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{18}H_{27}N_4O$ $[M+H]^+$ 315.2185 found 315.2181.

_N_-[(1-_n_-Hexyl-1_H_-1,2,3-triazol-4-yl)methyl]-4-methylbenzamide (1f)



The representive procedure was followed using 4-methylbenzoic acid (409 mg, 3.00 mmol) and 1-azido-*n*-hexane (571 mg, 4.50 mmol). Purification by column chromatography (*n*-hexane/EtOAc 7:3→ 1:1) yielded **1f** (721 mg, 80%) as a white solid. M. p. = 126-128 °C. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 7.68 (d, J = 8.3 Hz, 2H), 7.60 (s, 1H), 7.29-7.23 (m, 1H), 7.17 (d, J = 8.3 Hz, 2H), 4.65 (d, J = 5.6 Hz, 2H), 4.28 (t, J = 7.2 Hz, 2H), 2.34 (s, 3H), 1.89-1.81 (m, 2H), 1.30-1.24 (m, 6H), 0.86-0.82 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 167.4 (C_q), 144.7 (C_q), 142.0 (C_q), 131.1 (CH), 129.1 (CH), 127.0 (CH), 122.3 (C_q), 50.4 (CH_2), 35.3 (CH_2), 31.1 (CH_2), 30.2 (CH_2), 26.1 (CH_2), 22.4 (CH_2), 21.4 (CH_3), 13.9 (CH_3). IR (ATR): 2857, 1636, 1547, 1261, 1052, 798, 752, cm^{-1}. MS (ESI) *m/z* (relative intensity): 623 (5) $[2M+Na]^+$, 323 (22) $[M+Na]^+$, 301 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{17}H_{25}N_4O$ $[M+H]^+$ 301.2028 found 301.2027.

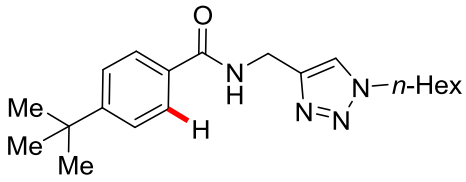
_N_-[(1-_n_-Hexyl-1_H_-1,2,3-triazol-4-yl)methyl]-[1,1'-biphenyl]-4-carboxamide (1g)



The representive procedure was followed using 4-phenylbenzoic acid (597 mg, 3.00 mmol) and 1-azido-*n*-hexane (571 mg, 4.50 mmol). Purification by column chromatography (*n*-hexane/EtOAc 7:3→ 1:1) yielded **1g** (946 mg, 87%) as a white solid. M. p. = 178-180 °C. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 7.89-7.86 (m, 2H), 7.63-7.61 (m, 2H), 7.62-7.60 (m, 2H), 7.58-7.56 (m, 3H), 7.38-7.32 (m, 2H), 4.70 (d, J = 5.7 Hz, 2H), 4.32-4.28 (m, 2H), 1.92-1.84 (m, 2H), 1.32-1.24 (m, 6H), 0.87-0.82 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 167.1 (C_q),

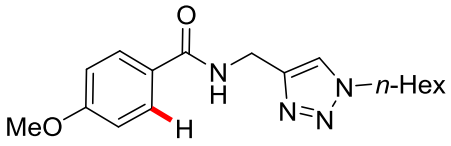
144.3 (C_q), 139.9 (C_q), 132.6 (CH), 128.9 (CH), 127.9 (C_q), 127.9 (CH), 127.6 (CH), 127.2 (CH), 127.2 (CH), 122.4 (C_q), 50.4 (CH_2), 35.4 (CH_2), 31.1 (CH_2), 30.2 (CH_2), 26.1 (CH_2), 22.4 (CH_2), 13.9 (CH_3). IR (ATR): 2953, 1636, 1532, 1058, 851, 741, 685 cm^{-1}. MS (ESI) *m/z* (relative intensity): 747 (39) $[2M+Na]^+$, 385 (47) $[M+Na]^+$, 363 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{22}H_{27}N_4O$ $[M+H]^+$ 363.2185 found 363.2181.

4-(*tert*-Butyl)-*N*-[(1-*n*-hexyl-1*H*-1,2,3-triazol-4-yl)methyl]benzamide (1h)



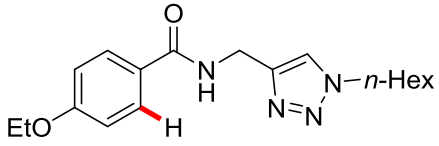
The representive procedure was followed using 4-(*tert*-butyl)benzoic acid (356 mg, 2.00 mmol) and 1-azido-*n*-hexane (381 mg, 3.00 mmol). Purification by column chromatography (*n*-hexane/EtOAc 7:3→ 1:1) yielded **1h** (581 mg, 85%) as a white solid. M. p. = 126-129 °C. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 7.73-7.69 (m, 2H), 7.59 (s, 1H), 7.42-7.39 (m, 2H), 6.96 (bs, 1H), 4.68 (d, *J* = 5.7 Hz, 2H), 4.29 (t, *J* = 7.3 Hz, 2H), 1.89-1.81 (m, 2H), 1.30 (s, 9H), 1.30-1.23 (m, 6H), 0.88-0.83 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 167.3 (C_q), 155.1 (C_q), 131.1 (C_q), 126.9 (CH), 125.5 (CH), 125.2 (C_q), 122.3 (CH), 50.4 (CH_2), 35.3 (CH_2), 34.9 (C_q), 31.2 (CH_3), 31.1 (CH_2), 30.2 (CH_2), 26.1 (CH_2), 22.4 (CH_2), 13.9 (CH_3). IR (ATR): 2951, 1638, 1549, 1313, 854, 667 cm^{-1}. MS (ESI) *m/z* (relative intensity): 707 (37) $[2M+Na]^+$, 365 (59) $[M+Na]^+$, 343 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{20}H_{31}N_4O$ $[M+H]^+$ 343.2498 found 343.2492.

***N*-[(1-*n*-Hexyl-1*H*-1,2,3-triazol-4-yl)methyl]-4-methoxybenzamide (1i)**



The representive procedure was followed using 4-methoxybenzoic acid (457 mg, 3.00 mmol) and 1-azido-*n*-hexane (571 mg, 4.50 mmol). Purification by column chromatography (*n*-hexane/EtOAc 7:3→ 1:1) yielded **1i** (703 mg, 74%) as a white solid. M. p. = 109-110 °C. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 7.76 (d, *J* = 8.5 Hz, 2H), 7.60 (s, 1H), 7.40-7.35 (m, 1H), 6.84 (d, *J* = 8.5 Hz, 2H), 4.63 (d, *J* = 5.7 Hz, 2H), 4.28 (t, *J* = 7.2 Hz, 2H), 3.78 (s, 3H), 1.88-1.81 (m, 2H), 1.31-1.23 (m, 6H), 0.86-0.81 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 166.9 (C_q), 162.2 (C_q), 128.8 (C_q), 126.3 (CH), 122.3 (C_q), 113.7 (CH), 113.5 (CH), 55.3 (CH_2), 50.4 (CH_3), 35.3 (CH_2), 31.1 (CH_2), 30.2 (CH_2), 26.1 (CH_2), 22.2 (CH_2), 13.8 (CH_3). IR (ATR): 2954, 1634, 1503, 1175, 1059, 839, 611 cm^{-1}. MS (ESI) *m/z* (relative intensity): 339 (21) $[M+Na]^+$, 317 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{17}H_{25}N_4O$ $[M+H]^+$ 317.1978 found 317.1980.

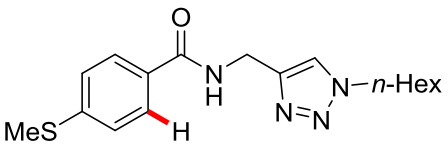
4-Ethoxy-*N*-[(1-*n*-hexyl-1*H*-1,2,3-triazol-4-yl)methyl]benzamide (1j)



The representive procedure was followed using 4-ethoxybenzoic acid (500 mg, 3.00 mmol) and 1-azido-*n*-hexane (571 mg, 4.50 mmol). Purification by column chromatography (*n*-hexane/EtOAc 7:3→ 1:1) yielded **1j** (635 mg, 64%) as a white solid. M. p. = 134-136 °C. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 7.73 (d, *J* = 8.8 Hz, 2H), 7.60 (s, 1H), 6.93 (bs, 1H), 6.86 (d, *J* = 8.8 Hz, 2H), 4.66 (d, *J* = 5.0 Hz, 2H), 4.29 (t, *J* = 7.3 Hz, 2H), 4.04 (q, *J* = 7.0 Hz, 2H), 1.91-1.82 (m, 2H), 1.40 (t, *J* = 7.0 Hz, 3H), 1.33-1.23 (m, 6H), 0.87-0.82 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 167.1 (C_q), 161.7 (C_q), 128.8 (CH), 125.7 (C_q), 122.3 (C_q), 116.2 (CH), 114.1 (CH), 63.6 (CH_2), 50.4 (CH_2), 35.3 (CH_2), 31.1 (CH_2), 30.2 (CH_2), 26.1 (CH_2), 22.4 (CH_2), 14.7 (CH_3), 13.9 (CH_3). IR (ATR): 2932, 1634, 1551, 1251, 1050, 651 cm^{-1}. MS

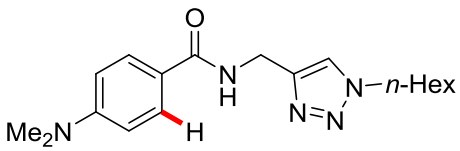
(ESI) *m/z* (relative intensity): 683 (18) $[2M+Na]^+$, 353 (100) $[M+Na]^+$, 331 (47) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{18}H_{27}N_4O_2$ $[M+H]^+$ 331.2134 found 331.2129.

_N_-[(1-_n_-Hexyl-1_H_-1,2,3-triazol-4-yl)methyl]-4-(methylthio)benzamide (1k)



The representive procedure was followed using 4-(methylthio)benzoic acid (336 mg, 2.00 mmol) and 1-azido-*n*-hexane (381 mg, 3.00 mmol). Purification by column chromatography (*n*-hexane/EtOAc 7:3→ 1:1) yielded **1k** (439 mg, 66%) as a white solid. M. p. = 137-139 °C. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 7.73-7.68 (m, 2H), 7.60 (s, 1H), 7.24 (s, 1H), 7.23-7.18 (m, 2H), 4.66 (d, *J* = 5.7 Hz, 2H), 4.29 (t, *J* = 7.6 Hz, 2H), 2.46 (s, 3H), 1.92-1.79 (m, 2H), 1.32-1.21 (m, 6H), 0.87-0.81 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 166.9 (C_q), 144.7 (C_q), 143.5 (C_q), 130.1 (C_q), 127.4 (CH), 125.3 (CH), 122.5 (CH), 50.4 (CH_2), 36.3 (CH_2), 31.1 (CH_2), 30.1 (CH_2), 26.1 (CH_2), 22.3 (CH_2), 15.0 (CH_3), 13.9 (CH_3). IR (ATR): 2954, 1634, 1523, 1059, 837, 789 cm^{-1}. MS (ESI) *m/z* (relative intensity): 687 (44) $[2M+Na]^+$, 355 (92) $[M+Na]^+$, 333 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{17}H_{25}N_4OS$ $[M+H]^+$ 333.1749 found 333.1744.

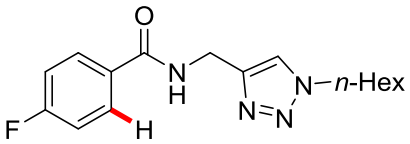
4-(Dimethylamino)-_N_-[(1-_n_-hexyl-1_H_-1,2,3-triazol-4-yl)methyl]benzamide (1l)



The representive procedure was followed using 4-(dimethylamino)benzoic acid (330 mg, 2.00 mmol) and 1-azido-*n*-hexane (381 mg, 3.00 mmol). Purification by column chromatography (*n*-hexane/EtOAc 7:3→ 1:1) yielded **1l** (363 mg, 55%) as a white solid. M. p. = 176-178 °C. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 7.69-7.66 (m, 2H), 7.57 (s, 1H), 6.82 (s, 1H), 6.63-6.60 (m, 2H), 4.66 (d, *J* = 5.7 Hz, 2H), 4.27 (t, *J* = 7.3 Hz, 2H), 2.97 (s, 6H), 1.88-1.76 (m, 2H), 1.33-1.19 (m, 6H), 0.86-0.82 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 167.3 (C_q), 152.5 (C_q),

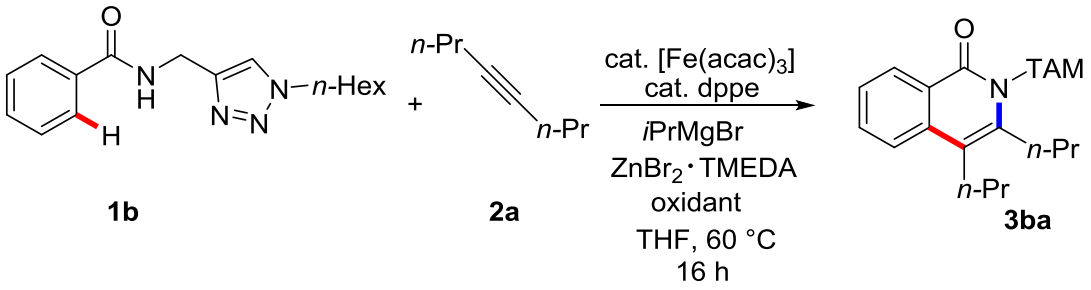
145.2 (C_q), 128.5 (CH), 122.3 (CH), 120.7 (C_q), 111.0 (CH), 50.4 (CH_2), 40.1 (CH_3), 35.2 (CH_2), 31.1 (CH_2), 30.2 (CH_2), 26.1 (CH_2), 22.4 (CH_2), 13.9 (CH_3). IR (ATR): 2922, 1624, 1606, 1517, 1310, 1213, 631 cm^{-1}. MS (ESI) *m/z* (relative intensity): 681 (28) $[2M+Na]^+$, 352 (76) $[M+Na]^+$, 330 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{18}H_{28}N_5O$ $[M+H]^+$ 330.2294 found 330.2299.

4-Fluoro-*N*-[(1-*n*-hexyl-1*H*-1,2,3-triazol-4-yl)methyl]benzamide (1m)



The representive procedure was followed using 4-fluorobenzoic acid (280 mg, 2.00 mmol) and 1-azido-*n*-hexane (381 mg, 3.00 mmol). Purification by column chromatography (*n*-hexane/EtOAc 7:3→ 1:1) yielded **1m** (413 mg, 68%) as a white solid. M. p. = 108-110 °C. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 7.98-7.78 (m, 2H), 7.71-7.59 (m, 1H), 7.11-6.96 (m, 1H), 4.69-4.57 (m, 2H), 4.38-4.23 (m, 2H), 1.92-1.79 (m, 2H), 1.37-1.77 (m, 2H), 1.32-1.18 (m, 6H), 0.90-0.77 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 166.4 (C_q), 165.9 (d, $^1J_{C-F}$ = 251 Hz, C_q,), 144.7 (C_q), 130.1 (d, $^4J_{C-F}$ = 3 Hz, C_q,), 129.5 (d, $^3J_{C-F}$ = 9 Hz, CH), 122.6 (CH), 115.5 (d, $^2J_{C-F}$ = 22 Hz, CH), 50.4 (CH_2), 35.2 (CH_2), 31.0 (CH_2), 30.1 (CH_2), 26.1 (CH_2), 22.3 (CH_2), 13.8 (CH_3). ^{19}F-NMR (283 MHz, $CDCl_3$): δ = -108.1 (m). IR (ATR): 2956, 2856, 1637, 1604, 1461, 1286, 1239, 1059, 849 cm^{-1}. MS (ESI) *m/z* (relative intensity): 327 (17) $[M+Na]^+$, 305 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{16}H_{22}FN_4O$ $[M+H]^+$ 305.1778 found 305.1774.

Variation of key parameters for iron-catalyzed C–H/N–H alkyne annulation

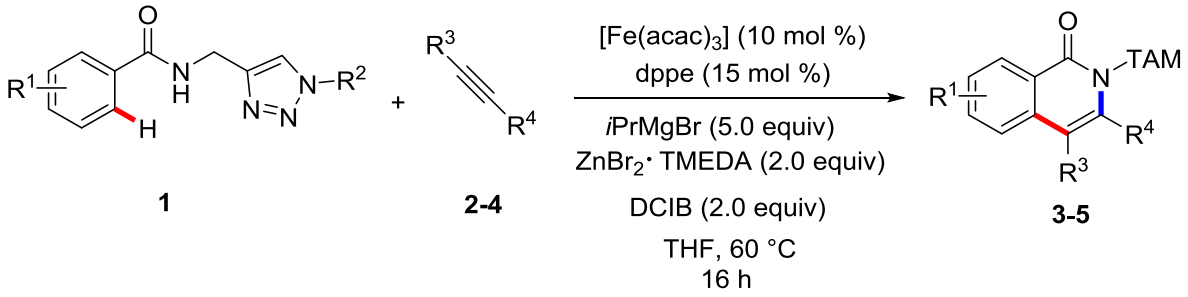


To a stirred solution of **1b** (85.8 mg, 0.30 mmol), $ZnBr_2$•TMEDA (204.6 mg, 0.60 mmol), dppe (17.9 mg, 0.045 mmol) and 4-octyne (**2a**) (66.0 mg, 0.60 mmol) in THF (0.50 ml), *i*PrMgBr (3.0 M in 2-MeTHF) (500 µl, 1.5 mmol) was added dropwise and the reaction mixture was stirred for 5 min at ambient temperature. Then, $Fe(acac)_3$ (10.6 mg, 0.03 mmol) was added in a single portion. After stirring the solution for additional 5 min, an oxidant (0.60 mmol, 2.0 equiv) was added. Then, the mixture was placed in a pre-heated oil bath at 60 °C. After stirring for 16 h, sat. aqueous NH_4Cl (15 ml) was added to the reaction mixture, which was then extracted with CH_2Cl_2 (3 x15 ml). The combined organic extracts were dried over Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography (*n*hexane/EtOAc 9:1→ 3:1).

Entry	**Oxidant**	**Yield (%)[a]**
1	DCIB[b]	81
2	DCP[c]	--
3	2,3-DCB[d]	--
4	1,2-*trans*-dichloro-cyclohexane	--
5	DCE[e]	--

[a] Reaction conditions: **1b** (0.30 mmol), **2a** (0.60 mmol), $Fe(acac)_3$ (0.03 mmol), dppe (0.045 mmol), $ZnBr_2$•TMEDA (0.60 mmol, 2.0 equiv), *i*PrMgBr (1.50 mmol, 5.0 equiv), oxidant (0.60 mmol), THF (0.50 ml), 60 °C, 16 h, isolated yields; [b] 1,2-dichloro-isobutane; [c] 1,2-dichloropropane; [d] 2,3-*trans*-dichlorobutane; [e] dichloroethane.

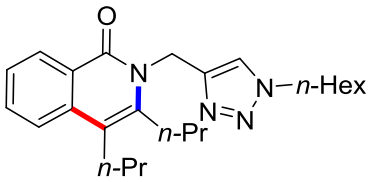
Representative procedure for the iron-catalyzed C–H/N–H alkyne annulation



1 **2-4** **3-5**

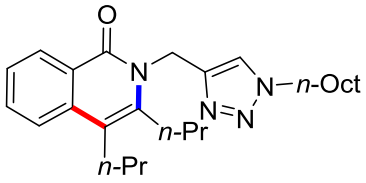
To a stirred solution of **1** (0.30 mmol), $ZnBr_2$•TMEDA (204.6 mg, 0.60 mmol), dppe (17.9 mg, 0.045 mmol) and the corresponding alkyne (0.60 mmol) in THF (0.50 ml), *i*PrMgBr (3.0 M in 2-MeTHF, 500 µl, 1.5 mmol) was added dropwise and the reaction mixture was stirred for 5 min at ambient temperature. Then, $Fe(acac)_3$ (10.6 mg, 0.03 mmol) was added in a single portion. After stirring the solution for additional 5 min, DCIB (70 µl, 0.60 mmol) was added. Then, the mixture was placed in a pre-heated oil bath at 60 °C. After stirring for 16 h, sat. aqueous NH_4Cl (15 ml) was added to the reaction mixture, which was extracted with CH_2Cl_2 (3 x15 ml). The combined organic extracts were dried over Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography (*n*-hexane/EtOAc).

2-[(1-*n*-Hexyl-1*H*-1,2,3-triazol-4-yl)methyl]-3,4-di-*n*-propylisoquinolin-1(2*H*)-one (3ba)



The representive procedure was followed using **1b** (85.8 mg, 0.30 mmol) and 4-octyne (**2a**) (66.0 mg, 0.60 mmol). Purification by column chromatography (*n*-hexane/EtOAc 9:1→ 3:1) yielded **3ba** (95.6 mg, 81%) as a yellowish oil. R_f (*n*-hexane/EtOAc 1:1) = 0.57. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 7.76 (d, *J* = 8.7 Hz, 1H), 7.69 (s, 1H), 7.63-7.59 (m, 2H), 7.43-7.37 (m, 1H), 5.38 (s, 2H), 4.23 (t, *J* = 7.3 Hz, 2H), 3.04-2.95 (m, 2H), 2.71-2.63 (m, 2H), 1.89-1.78 (m, 2H), 1.72-1.63 (m, 2H), 1.60-1.49 (m, 2H), 1.28-1.22 (m, 6H), 1.13 (t, *J* = 7.4 Hz, 3H), 1.03 (t, *J* = 7.4 Hz, 3H), 0.85-0.80 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 162.7 (C_q), 144.4 (C_q), 140.0 (C_q), 136.8 (C_q), 132.2 (CH), 128.1 (CH), 125.6 (CH), 124.8 (C_q), 123.8 (CH), 122.8 (CH), 114.4 (C_q), 50.3 (CH_2), 39.9 (CH_2), 31.4 (CH_2), 31.1 (CH_2), 30.1 (CH_2), 29.8 (CH_2), 26.1 (CH_2), 23.5 (CH_2), 23.3 (CH_2), 22.3 (CH_2), 14.5 (CH_3), 14.3 (CH_3), 13.9 (CH_3). IR (ATR): 2957, 1643, 1591, 1341, 1049, 910, 771 cm^{-1}. MS (ESI) *m/z* (relative intensity): 811 (26) $[2M+Na]^+$, 417 (34) $[M+Na]^+$, 395 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{24}H_{35}N_4O$ $[M+H]^+$ 395.2811 found 395.2806.

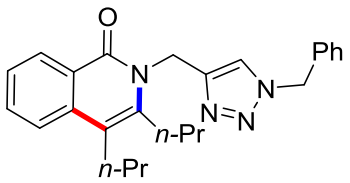
2-[(1-*n*-Octyl-1*H*-1,2,3-triazol-4-yl)methyl]-3,4-di-*n*-propylisoquinolin-1(2*H*)-one (3ca)



The representive procedure was followed using **1c** (94.2 mg, 0.30 mmol) and 4-octyne (**2a**) (66.0 mg, 0.60 mmol). Purification by column chromatography (*n*-hexane/EtOAc 9:1→ 3:1) yielded **3ca** (78.4 mg, 62%) as a yellowish oil. R_f (*n*-hexane/EtOAc 2:1) = 0.54. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 8.42 (d, *J* = 8.4 Hz, 1H), 7.69 (s, 1H), 7.64-7.59 (m, 2H), 7.45-7.36 (m, 1H), 5.38 (s, 2H), 4.23 (t, *J* = 7.4 Hz, 2H), 3.05-2.96 (m, 2H), 2.70-2.6 (m, 2H), 1.87-1.78 (m, 2H), 1.70-1.49 (m, 6H), 1.32-1.19 (m, 8H), 1.13 (t, *J* = 7.4 Hz, 3H), 1.03 (t, *J* = 7.4 Hz, 3H), 0.87-0.78 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ =162.6 (C_q), 144.3 (C_q), 140.0 (C_q),

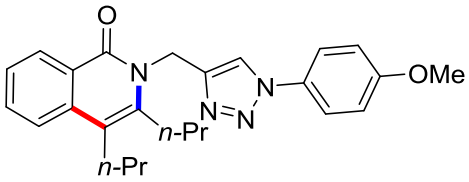
136.8 (C_q), 132.2 (C_q), 128.1 (CH), 125.6 (CH), 124.8 (CH), 123.8 (CH), 122.8 (CH), 114.4 (C_q), 50.3 (CH_2), 39.9 (CH_2), 31.4 (CH_2), 31.1 (CH_2), 31.0 (CH_2), 30.1 (CH_2), 29.8 (CH_2), 26.1 (CH_2), 23.5 (CH_2), 23.3 (CH_2), 22.4 (CH_2), 22.3 (CH_2), 14.5 (CH_3), 14.3 (CH_3), 13.9 (CH_3). IR (ATR): 2957, 2870, 1646, 1590, 1465, 1378, 1047 cm^{-1}. MS (ESI) *m/z* (relative intensity): 867 (35) $[2M+Na]^+$, 445 (100) $[M+Na]^+$, 423 (76) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{26}H_{39}N_4O$ $[M+H]^+$ 423.3124 found 423.3118.

2-[(1-Benzyl-1*H*-1,2,3-triazol-4-yl)methyl]-3,4-di-*n*-propylisoquinolin-1(2*H*)-one (3da)



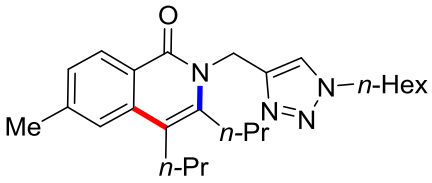
The representive procedure was followed using **1d** (87.6 mg, 0.30 mmol) and 4-octyne (**2a**) (66.0 mg, 0.60 mmol). Purification by column chromatography (*n*-hexane/EtOAc 9:1→ 3:1) yielded **3da** (85.2 mg, 71%) as a white solid. M. p. = 101-103 °C. R_f (*n*-hexane/EtOAc 1:1) = 0.38. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 8.38 (ddd, *J* = 8.2, 1.3, 0.7 Hz, 1H), 7.66 (s, 1H), 7.62-7.59 (m, 2H), 7.38 (ddd, *J* = 8.2, 5.4, 2,7 Hz, 1H), 7.33-7.27 (m, 3H), 7.22-7.19 (m, 2H), 5.41 (s, 2H), 5.37 (s, 2H), 3.02-2.98 (m, 2H), 2.68-2.64 (m, 2H), 1.70-1.61 (m, 2H), 1.58-1.49 (m, 2H), 1.13 (t, *J* = 7.2 Hz, 3H), 1.02 (t, *J* = 7.2 Hz, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 162.6 (C_q), 144.8 (C_q), 140.0 (C_q), 136.8 (C_q), 134.5 (C_q), 132.2 (CH), 129.0 (CH), 128.6 (CH), 128.1 (CH), 128.0 (CH), 125.6 (CH), 124.7 (C_q), 123.9 (CH), 122.8 (CH), 114.4 (C_q), 54.1 (CH_2), 39.8 (CH_2), 31.4 (CH_2), 29.8 (CH_2), 23.5 (CH_2), 23.2 (CH_2), 14.4 (CH_3), 14.2 (CH_3). IR (ATR): 1629, 1581, 1053, 778, 725, 690, 661 cm^{-1}. MS (ESI) *m/z* (relative intensity): 823 (54) $[2M+Na]^+$, 423 (29) $[M+Na]^+$, 401 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{25}H_{29}N_4O$ $[M+H]^+$ 401.2341 found 401.2336.

2-{[1-(4-Methoxyphenyl)-1*H*-1,2,3-triazol-4-yl]methyl}-3,4-di-*n*-propylisoquinolin-1(2*H*)-one (3ea)



The representive procedure was followed using **1e** (92.4 mg, 0.30 mmol) and 4-octyne (**2a**) (66.0 mg, 0.60 mmol). Purification by column chromatography (*n*-hexane/EtOAc 9:1→ 3:1) yielded **3ea** (74.3 mg, 60%) as a viscous yellowish oil. R_f (*n*-hexane/EtOAc 1:1) = 0.53. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 8.43 (dd, *J* = 8.0, 1.0 Hz, 1H), 8.09 (s, 1H), 7.64-7.61 (m, 2H), 7.47-7.54 (m, 2H), 7.41 (ddd, *J* = 8.0, 4.9, 3.2 Hz, 1H), 6.97-6.91 (m, 2H), 5.46 (s, 2H), 3.81 (s, 3H), 3.09-3.00 (m, 2H), 2.72-2.64 (m, 2H), 1.83-1.66 (m, 2H), 1.65-1.48 (m, 2H), 1.16 (t, *J* = 7.2 Hz, 3H), 1.04 (t, *J* = 7.2 Hz, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 162.6 (C_q), 159.6 (C_q), 144.8 (C_q), 139.8 (C_q), 136.8 (C_q), 132.1 (CH), 130.4 (C_q), 128.1 (CH), 125.6 (CH), 124.7 (C_q), 122.8 (CH), 122.3 (CH), 122.0 (CH), 114.6 (CH), 114.5 (C_q), 55.6 (CH_3), 39.9 (CH_2), 31.5 (CH_2), 30.0 (CH_2), 23.6 (CH_2), 23.4 (CH_2), 14.6 (CH_3), 14.4 (CH_3). IR (ATR): 1636, 1516, 1255, 1029, 825, 761 cm^{-1}. MS (ESI) *m/z* (relative intensity): 855 (31) $[2M+Na]^+$, 439 (38) $[M+Na]^+$, 417 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{25}H_{29}N_4O_2$ $[M+H]^+$ 417.2291 found 417.2289.

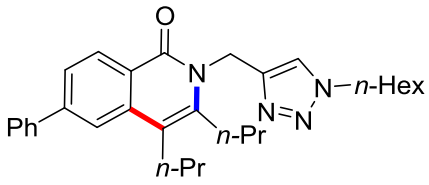
2-[(1-*n*-Hexyl-1*H*-1,2,3-triazol-4-yl)methyl]-6-methyl-3,4-di-*n*-propylisoquinolin-1(2*H*)-one (3fa)



The representive procedure was followed using **1f** (98.4 mg, 0.30 mmol) and 4-octyne (**2a**) (66.0 mg, 0.60 mmol). Purification by column chromatography (*n*-hexane/EtOAc 9:1→ 3:1) yielded **3fa** (103.7 mg, 82%) as a coulorless oil. R_f (*n*-hexane/EtOAc 2:1) = 0.43. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 8.31 (d, *J* = 8.6 Hz, 1H), 7.67 (s, 1H), 7.37 (s, 1H), 7.25-7.21 (d, *J* = 8.6 Hz, 1H), 5.37 (s, 2H), 4.22 (t, *J* = 7.4 Hz, 2H), 3.01-2.93 (m, 2H), 2.68-2.61 (m, 2H), 2.47

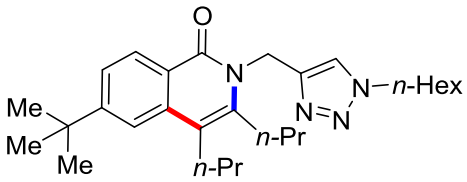
(s, 3H), 1.86-1.77 (m, 2H), 1.70-1.60 (m, 2H), 1.60-1.48 (m, 2H), 1.27-1.20 (m, 6H), 1.12 (t, J = 7.3 Hz, 3H), 1.03 (t, J = 7.4 Hz, 3H), 0.84-0.79 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 162.6 (C_q), 144.5 (C_q), 142.6 (C_q), 140.1 (C_q), 137.0 (C_q), 128.1 (C_q), 127.3 (CH), 123.8 (CH), 122.6 (CH), 122.6 (CH), 114.2 (C_q), 50.3 (CH_2), 39.8 (CH_2), 31.4 (CH_2), 31.1 (CH_2), 30.1 (CH_2), 29.8 (CH_2), 26.1 (CH_2), 23.5 (CH_2), 23.2 (CH_2), 22.4 (CH_2), 22.3 (CH_2), 14.5 (CH_2), 14.2 (CH_2), 13.9 (CH_3). IR (ATR): 2957, 2870, 1643, 1618, 1490, 1458, 1047, 793 cm^{-1}. MS (ESI) *m/z* (relative intensity): 839 (26) $[2M+Na]^+$, 431 (45) $[M+Na]^+$, 409 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{25}H_{37}N_4O$ $[M+H]^+$ 409.2967 found 409.2962.

2-[(1-*n*-Hexyl-1*H*-1,2,3-triazol-4-yl)methyl]-6-phenyl-3,4-di-*n*-propylisoquinolin-1(2*H*)-one (3ga)



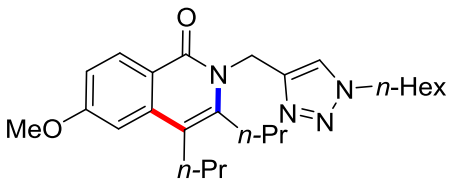
The representive procedure was followed using **1g** (117.0 mg, 0.30 mmol) and 4-octyne (**2a**) (66.0 mg, 0.60 mmol). Purification by column chromatography (*n*-hexane/EtOAc 9:1→ 3:1) yielded **3ga** (72.9 mg, 54%) as a white solid. M. p. = 114-116 °C. R_f (*n*-hexane/EtOAc 1:1) = 0.52. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 8.48 (dd, J = 8.2, 0.7 Hz, 1H), 7.77 (dd, J = 1.4, 0.7 Hz, 1H), 7.70 (s, 1H), 7.66-7.61 (m, 3H), 7.51-7.44 (m, 2H), 7.43-7.37 (m, 1H), 5.34 (s, 2H), 4.24 (t, J = 7.2 Hz, 2H), 3.05-2.99 (m, 2H), 2.77-2.70 (m, 2H), 1.89-1.78 (m, 2H), 1.74-1.54 (m, 4H), 1.32-1.21 (m, 6H), 1.15 (t, J = 7.5 Hz, 3H), 1.05 (t, J = 7.5 Hz, 3H), 0.86-0.80 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 162.4 (C_q), 145.0 (C_q), 144.3 (C_q), 140.8 (C_q), 140.5 (C_q), 137.1 (C_q), 128.9 (CH), 128.7 (CH), 127.9 (CH), 127.5 (CH), 125.0 (CH), 123.7 (CH), 123.6 (C_q), 121.2 (CH), 114.4 (C_q), 50.4 (CH_2), 40.0 (CH_2), 31.5 (CH_2), 31.1 (CH_2), 30.2 (CH_2), 29.9 (CH_2), 26.2 (CH_2), 23.7 (CH_2), 23.4 (CH_2), 22.4 (CH_2), 14.6 (CH_3), 14.4 (CH_3), 14.0 (CH_3). IR (ATR): 1638, 1616, 1588, 1045, 796, 699 cm^{-1}. MS (ESI) *m/z* (relative intensity): 963 (41) $[2M+Na]^+$, 493 (21) $[M+Na]^+$, 471 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{30}H_{39}N_4O$ $[M+H]^+$ 471.3124 found 471.3118.

6-(*tert*-Butyl)-2-[(1-*n*-hexyl-1*H*-1,2,3-triazol-4-yl)methyl]-3,4-di-*n*-propylisoquinolin-1(2*H*)-one (3ha)



The representive procedure was followed using **1h** (102.6 mg, 0.30 mmol) and 4-octyne (**2a**) (66.0 mg, 0.60 mmol). Purification by column chromatography (*n*-hexane/EtOAc 9:1→ 3:1) yielded **3ha** (67.9 mg, 50%) as a viscous coulorless oil. R_f (*n*-hexane/EtOAc 1:1) = 0.56. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 8.36 (d, J = 7.8 Hz, 1H), 7.68 (s, 1H), 7.60 (d, J = 1.7 Hz, 1H), 7.49 (dd, J = 7.8, 1.7 Hz, 1H), 5.41 (s, 2H), 4.23 (t, J = 7.3 Hz, 2H), 3.04-2.96 (m, 2H), 2.70 (dd, J = 10.5, 5.5 Hz, 2H), 1.93-1.76 (m, 2H), 1.73-1.50 (m, 4H), 1.38 (s, 9H), 1.31-1.20 (m, 6H), 1.14 (t, J = 7.3 Hz, 3H), 1.05 (t, J = 7.3 Hz, 3H), 0.86-0.80 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 162.5 (C_q), 155.4 (C_q), 144.5 (C_q), 139.9 (C_q), 136.6 (C_q), 127.9 (CH), 123.8 (CH), 123.7 (CH), 122.5 (C_q), 118.7 (CH), 114.5 (C_q), 50.3 (CH_2), 39.7 (CH_2), 35.3 (C_q), 31.4 (CH_2), 31.2 (CH_3), 31.0 (CH_2), 30.0 (CH_2), 29.8 (CH_2), 26.1 (CH_2), 23.5 (CH_2), 23.3 (CH_2), 22.3 (CH_2), 14.4 (CH_3), 14.2 (CH_3), 13.9 (CH_3). IR (ATR): 1641, 1614, 1587, 1463, 795, 730 cm^{-1}. MS (ESI) *m/z* (relative intensity): 923 (44) $[2M+Na]^+$, 451 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{28}H_{43}N_4O$ $[M+H]^+$ 451.3437 found 451.3431.

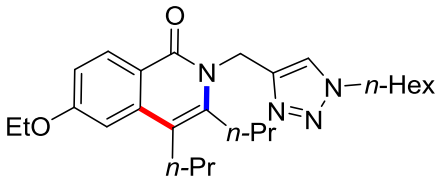
2-[(1-*n*-Hexyl-1*H*-1,2,3-triazol-4-yl)methyl]-6-methoxy-3,4-di-*n*-propylisoquinolin-1(2*H*)-one (3ia)



The representive procedure was followed using **1i** (103.2 mg, 0.30 mmol) and 4-octyne (**2a**) (66.0 mg, 0.60 mmol). Purification by column chromatography (*n*-hexane/EtOAc 9:1→ 3:1) yielded **3ia** (73.7 mg, 58%) as viscous coulorless oil. R_f (*n*-hexane/EtOAc 2:1) = 0.30. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 8.35 (d, J = 8.7 Hz, 1H), 7.68 (s, 1H), 7.02-6.95 (m, 1H), 7.43-7.37 (m, 1H), 5.36 (s, 2H), 4.23 (t, J = 7.2 Hz, 2H), 3.89 (s, 3H), 3.01-2.93 (m, 2H), 2.66-2.60

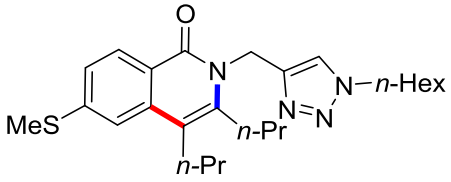
(m, 2H), 1.86-1.77 (m, 2H), 1.71-1.52 (m, 4H), 1.27-1.20 (m, 6H), 1.13 (t, *J* = 7.4 Hz, 3H), 1.03 (t, *J* = 7.5 Hz, 3H), 0.85-0.79 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 162.8 (C_q), 162.3 (C_q), 144.5 (C_q), 140.7 (C_q), 138.8 (C_q), 130.3 (C_q), 123.8 (CH), 118.8 (CH), 114.2 (CH), 113.9 (C_q), 104.9 (CH), 55.3 (CH_3), 50.3 (CH_2), 39.7 (CH_2), 31.5 (CH_2), 31.1 (CH_2), 30.1 (CH_2), 30.0 (CH_2), 26.1 (CH_2), 23.3 (CH_2), 23.3 (CH_2), 22.4 (CH_2), 14.5 (CH_3), 14.3 (CH_3), 13.9 (CH_3). IR (ATR): 2957, 2929, 1639, 1611, 1491, 1238, 1213, 1035, 789, 729 cm^{-1}. MS (ESI) *m/z* (relative intensity): 871 (20) $[2M+Na]^+$, 447 (28) $[M+Na]^+$, 425 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{25}H_{37}N_4O$ $[M+H]^+$ 425.2917 found 425.2912.

6-Ethoxy-2-[(1-*n*-hexyl-1*H*-1,2,3-triazol-4-yl)methyl]-3,4-di-*n*-propylisoquinolin-1(2*H*)-one (3ja)



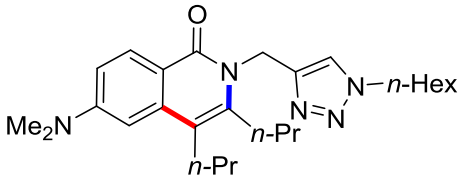
The representive procedure was followed using **1j** (99.2 mg, 0.30 mmol) and 4-octyne (**2a**) (66.0 mg, 0.60 mmol). Purification by column chromatography (*n*-hexane/EtOAc 9:1→ 3:1) yielded **3ja** (97.0 mg, 74%) as a white solid. M. p. = 134-136 °C. R_f (*n*-hexane/EtOAc 1:1) = 0.39. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 8.33 (d, *J* = 8.7 Hz, 1H), 7.65 (s, 1H), 6.99 (d, *J* = 2.1 Hz, 1H), 6.96-6.93 (m, 1H), 5.37 (s, 2H), 4.22 (t, *J* = 7.2 Hz, 2H), 4.11 (q, *J* = 6.8 Hz, 2H), 3.00-2.91 (m, 2H), 2.64-2.57 (m, 2H), 1.87-1.76 (m, 2H), 1.71-1.60 (m, 2H), 1.59-1.48 (m, 2H), 1.44 (t, *J* = 6.8 Hz, 3H), 1.29-1.19 (m, 6H), 1.11 (t, *J* = 7.5 Hz, 3H), 1.02 (t, *J* = 7.5 Hz, 3H), 0.81-0.77 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 162.3 (C_q), 162.1 (C_q), 144.5 (C_q), 140.6 (C_q), 138.8 (C_q), 130.2 (CH), 123.7 (CH), 118.6 (C_q), 114.5 (CH), 113.9 (C_q), 105.5 (CH), 63.6 (CH_2), 50.3 (CH_2), 39.6 (CH_2), 31.4 (CH_2), 31.0 (CH_2), 30.0 (CH_2), 29.9 (CH_2), 26.1 (CH_2), 23.3 (CH_2), 23.2 (CH_2), 22.3 (CH_2), 14.7 (CH_3), 14.5 (CH_3), 14.2 (CH_3), 13.8 (CH_3). IR (ATR): 1639, 1610, 1239, 1216, 1187, 1045 cm^{-1}. MS (ESI) *m/z* (relative intensity): 899 (23) $[2M+Na]^+$, 461 (22) $[M+Na]^+$, 439 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{26}H_{39}N_4O_2$ $[M+H]^+$ 439.3073 found 439.3068.

2-[(1-*n*-Hexyl-1*H*-1,2,3-triazol-4-yl)methyl]-6-(methylthio)-3,4-di-*n*-propylisoquinolin-1(2*H*)-one (3ka)



The representive procedure was followed using [Fe(acac)$_3$] (21.1 mg, 0.06 mmol), dppe (29.9 mg, 0.075 mmol), **1k** (99.6 mg, 0.30 mmol) and 4-octyne (**2a**) (66.0 mg, 0.60 mmol). Purification by column chromatography (*n*-hexane/EtOAc 9:1→ 3:1) yielded **3ka** (69.7 mg, 53 %) as a yellowish solid. M. p. = 143-145 °C. R_f (*n*-hexane/EtOAc 1:1) = 0.38. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 8.29 (d, *J* = 8.6 Hz, 1H), 7.67 (s, 1H), 7.34 (d, *J* = 1.7 Hz, 1H), 7.25 (dd, *J* = 8.6, 1.7 Hz, 1H), 5.36 (s, 2H), 4.22 (t, *J* = 7.3 Hz, 2H), 3.01-2.94 (m, 2H), 2.65-2.60 (m, 2H), 2.53 (s, 3H), 1.86-1.78 (m, 2H), 1.70-1.61 (m, 2H), 1.57-1.49 (m, 2H), 1.28-1.20 (m, 6H), 1.12 (t, *J* = 7.3 Hz, 3H), 1.02 (t, *J* = 7.3 Hz, 3H), 0.84-0.79 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 162.4 (C_q), 144.3 (C_q), 141.0 (C_q), 137.1 (C_q), 129.6 (C_q), 128.5 (CH), 123.8 (CH), 123.4 (CH), 121.8 (C_q), 118.4 (CH), 113.7 (C_q), 50.3 (CH_2), 39.8 (CH_2), 31.4 (CH_2), 31.0 (CH_2), 30.0 (CH_2), 29.8 (CH_2), 26.1 (CH_2), 23.4 (CH_2), 23.2 (CH_2), 22.3 (CH_2), 15.1 (CH_3), 14.5 (CH_3), 14.2 (CH_3), 13.8 (CH_3). IR (ATR): 1638, 1601, 1581, 1425, 789, 729 cm^{-1}. MS (ESI) *m/z* (relative intensity): 903 (66) $[2M+Na]^+$, 463 (68) $[M+Na]^+$, 441 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{25}H_{37}N_4OS$ $[M+H]^+$ 441.2688 found 441.2686.

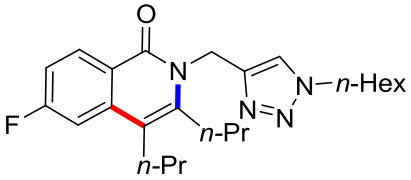
6-(Dimethylamino)-2-[(1-*n*-hexyl-1*H*-1,2,3-triazol-4-yl)methyl]-3,4-di-*n*-propylisoquinolin-1(2*H*)-one (3la)



The representive procedure was followed using [Fe(acac)$_3$] (21.1 mg, 0.06 mmol), dppe (29.9 mg, 0.075 mmol), **1l** (98.7 mg, 0.30 mmol) and 4-octyne (**2a**) (66.0 mg, 0.60 mmol). Purification by column chromatography (*n*-hexane/EtOAc 9:1→ 3:1→1:1) yielded **3la** (84.7 mg, 65 %) as a yellowish solid. M. p. = 143-145 °C. R_f (*n*-hexane/EtOAc 1:1) = 0.22. ^{1}H-

NMR (300 MHz, $CDCl_3$): δ = 8.26 (d, J = 9.1 Hz, 1H), 7.65 (s, 1H), 6.86 (dd, J = 9.1, 2.5 Hz, 1H), 6.61 (d, J = 2.5 Hz, 1H), 5.35 (s, 2H), 4.21 (t, J = 7.2 Hz, 2H), 3.06 (s, 6H), 2.97-2.88 (m, 2H), 2.64-2.60 (m, 2H), 1.87-1.78 (m, 2H), 1.68-1.52 (m, 4H), 1.30-1.20 (m, 6H), 1.11 (t, J = 7.2 Hz, 3H), 1.02 (t, J = 7.2 Hz, 3H), 0.85-0.80 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 162.5 (C_q), 152.9 (C_q), 145.0 (C_q), 140.1 (C_q), 138.3 (C_q), 129.5 (CH), 123.7 (CH), 114.8 (C_q), 113.8 (C_q), 112.1 (CH), 102.1 (CH), 50.3 (CH_2), 40.2 (CH_3), 39.5 (CH_2), 31.5 (CH_2), 31.1 (CH_2), 30.1 (CH_2), 30.0 (CH_2), 26.1 (CH_2), 23.3 (CH_2), 23.1 (CH_2), 22.3 (CH_2), 14.6 (CH_3), 14.3 (CH_3), 13.9 (CH_3). IR (ATR): 1629, 1606, 1574, 1507, 1047, 788 cm^{-1}. MS (ESI) *m/z* (relative intensity): 897 (13) $[2M+Na]^+$, 460 (28) $[M+Na]^+$, 438 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{26}H_{40}N_5O$ $[M+H]^+$ 438.3233 found 438.3228.

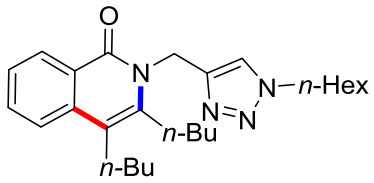
6-Fluoro-2-[(1-*n*-hexyl-1*H*-1,2,3-triazol-4-yl)methyl]-3,4-di-*n*-propylisoquinolin-1(2*H*)-one (3ma)



The representive procedure was followed using [Fe(acac)$_3$] (21.1 mg, 0.06 mmol), dppe (29.9 mg, 0.075 mmol), **1m** (91.2 mg, 0.30 mmol) and 4-octyne (**2a**) (66.0 mg, 0.60 mmol). Purification by column chromatography (*n*-hexane/EtOAc 9:1→ 3:1) yielded **3ma** (49.3 mg, 40%) as a viscous yellow oil. R_f (*n*-hexane/EtOAc 1:1) = 0.55. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 8.41 (dd, J = 10.6, 6.1 Hz, 1H), 7.68 (s, 1H), 7.20 (dd, J = 10.6, 2.4 Hz, 1H), 7.09 (dd, J = 6.1, 2.4, 1H), 5.35 (s, 2H), 4.23 (t, J = 7.3 Hz, 2H), 3.03-2.97 (m, 2H), 2.62-2.57 (m, 2H), 1.90-1.77 (m, 2H), 1.71-1.63 (m, 2H), 1.57-1.47 (m, 2H), 1.31-1.19 (m, 6H), 1.13 (t, J = 7.3 Hz, 3H), 1.03 (t, J = 7.3 Hz, 3H), 0.87-0.79 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 165.4 (d, $^1J_{C\text{-}F}$ = 249 Hz, C_q), 161.9 (C_q), 144.0 (C_q), 141.5 (C_q), 139.2 (d, $^5J_{C\text{-}F}$ = 10 Hz, C_q), 131.2 (d, $^4J_{C\text{-}F}$ = 10 Hz, CH), 123.7 (CH), 121.3 (C_q), 114.2 (d, $^2J_{C\text{-}F}$ = 24 Hz, CH), 113.8 (d, $^6J_{C\text{-}F}$ = 4 Hz, C_q), 107.9 (d, $^3J_{C\text{-}F}$ = 22 Hz, CH), 50.4 (CH_2), 39.9 (CH_2), 31.6 (CH_2), 31.1 (CH_2), 30.1 (CH_2), 30.0 (CH_2), 26.2 (CH_2), 23.4 (CH_2), 23.3 (CH_2), 22.4 (CH_2), 14.5 (CH_3), 14.4 (CH_3), 13.9 (CH_3). ^{19}F-NMR (285 MHz, $CDCl_3$): δ = -106.4 (ddd, J = 11, 9, 8 Hz). IR (ATR): 1646, 1615, 1597, 1487, 1170, 788 cm^{-1}. MS (ESI) *m/z* (relative intensity): 847 (42) $[2M+Na]^+$, 431

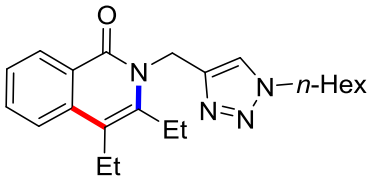
(100) $[M+Na]^+$, 413 (63) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{24}H_{34}FN_4O$ $[M+H]^+$ 413.2717 found 413.2711.

3,4-Di-*n*-butyl-2-[(1-*n*-hexyl-1*H*-1,2,3-triazol-4-yl)methyl]isoquinolin-1(2*H*)-one (3bb)



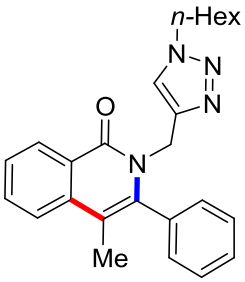
The representive procedure was followed using **1b** (85.8 mg, 0.30 mmol) and 5-decyne (**2b**) (82.8 mg, 0.60 mmol). Purification by column chromatography (*n*-hexane/EtOAc 9:1→ 3:1) yielded **3bb** (106.3 mg, 84%) as a yellow oil. R_f (*n*-hexane/EtOAc 1:1) = 0.55. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 8.42 (d, J = 8.5 Hz, 1H), 7.69 (s, 1H), 7.65-7.60 (m, 2H), 7.42-7.36 (m, 1H), 5.39 (s, 2H), 4.23 (t, J = 7.3 Hz, 2H), 3.08-2.97 (m, 2H), 2.73-2.64 (m, 2H), 1.89-1.76 (m, 2H), 1.68-1.41 (m, 8H), 1.35-1.18 (m, 6H), 1.07-0.98 (m, 3H), 0.96-0.92 (m, 3H), 0.85-0.79 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 162.7 (C_q), 144.4 (C_q), 140.1 (C_q), 136.9 (C_q), 132.2 (CH), 128.1 (CH), 125.6 (CH), 124.8 (C_q), 123.8 (CH), 122.7 (CH), 114.4 (C_q), 50.3 (CH_2), 39.8 (CH_2), 32.4 (CH_2), 32.0 (CH_2), 31.1 (CH_2), 30.1 (CH_2), 29.1 (CH_2), 27.4 (CH_2), 26.1 (CH_2), 23.1 (CH_2), 22.9 (CH_2), 22.3 (CH_2), 13.9 (CH_2), 13.9 (CH_2), 13.9 (CH_3). IR (ATR): 2957, 2859, 1642, 1590, 1260, 1087, 1020, 800, 734, 703 cm^{-1}. MS (ESI) *m/z* (relative intensity): 867 (24) $[2M+Na]^+$, 445 (26) $[M+Na]^+$, 423 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{26}H_{39}N_4O$ $[M+H]^+$ 423.3124 found 423.3118.

3,4-Diethyl-2-[(1-*n*-hexyl-1*H*-1,2,3-triazol-4-yl)methyl]isoquinolin-1(2*H*)-one (3bc)



The representive procedure was followed using **1b** (85.8 mg, 0.30 mmol) and 3-hexyne (**2c**) (51.6 mg, 0.60 mmol). Purification by column chromatography (*n*-hexane/EtOAc 9:1→ 3:1) yielded **3bc** (82.9 mg, 76%) as a viscous orange oil. R_f (*n*-hexane/EtOAc 2:1) = 0.41. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 8.43 (dd, *J* = 8.0, 1.1 Hz, 1H), 7.71 (s, 1H), 7.67-7.59 (m, 2H), 7.40 (ddd, *J* = 8.0, 5.4, 2.8 Hz, 1H), 4.23 (t, *J* = 7.4 Hz, 2H), 3.09 (q, *J* = 7.2 Hz, 2H), 2.75 (q, *J* = 7.2 Hz, 2H), 1.89-1.75 (m, 2H), 1.31 (t, *J* = 7.2 Hz, 3H), 1.27-1.21 (m, 8H), 1.17 (t, *J* = 7.2 Hz, 3H), 0.83-0.79 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 162.6 (C_q), 144.3 (C_q), 140.8 (C_q), 136.7 (C_q), 132.2 (CH), 128.1 (CH), 125.6 (CH), 124.8 (C_q), 123.9 (CH), 122.6 (CH), 115.3 (C_q), 50.3 (CH_2), 39.6 (CH_2), 31.0 (CH_2), 30.0 (CH_2), 26.1 (CH_2), 22.4 (CH_2), 22.3 (CH_2), 20.5 (CH_2), 14.7 (CH_3), 14.1 (CH_3), 13.8 (CH_3). IR (ATR): 1640, 1588, 1463, 1049, 771, 702 cm^{-1}. MS (ESI) *m/z* (relative intensity): 755 (23) $[2M+Na]^+$, 389 (31) $[M+Na]^+$, 367 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{22}H_{31}N_4O$ $[M+H]^+$ 367.2498 found 367.2493.

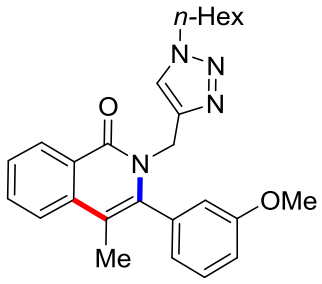
2-[(1-*n*-Hexyl-1*H*-1,2,3-triazol-4-yl)methyl]-4-methyl-3-phenylisoquinolin-1(2*H*)-one (5ba)



The representive procedure was followed using **1b** (85.8 mg, 0.30 mmol) and prop-1-yn-1-ylbenzene (**4a**) (69.6 mg, 0.60 mmol). Purification by column chromatography (*n*-hexane/EtOAc 9:1→ 3:1) yielded **5ba** (88.4 mg, 74%) as a yellowish solid. M. p. = 88-91 °C. R_f (*n*-hexane/EtOAc 1:1) = 0.33. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 8.49 (d,

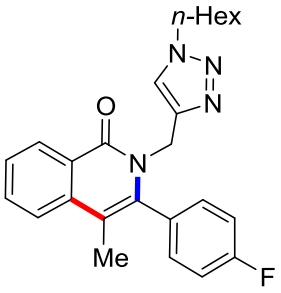
J = 8.0 Hz, 1H), 7.72-7.66 (m, 2H), 7.55 (s, 1H), 7.53-7.49 (m, 1H), 7.48-7.43 (m, 3H), 7.27-7.22 (m, 2H), 5.02 (s, 2H), 4.20 (t, *J* = 7.2 Hz, 2H), 1.97 (s, 3H), 1.86-1.75 (m, 2H), 1.30-1.19 (m, 6H), 0.86-0.79 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 162.1 (C_q), 143.9 (C_q), 140.1 (C_q), 137.2 (C_q), 134.7 (C_q), 132.3 (CH), 130.0 (CH), 128.9 (CH), 128.6 (CH), 128.6 (CH), 128.0 (CH), 126.4 (CH), 125.2 (C_q), 123.2 (CH), 110.9 (C_q), 50.2 (CH_2), 42.1 (CH_2), 31.1 (CH_2), 30.1 (CH_2), 26.1 (CH_2), 22.4 (CH_2), 15.0 (CH_3), 13.9 (CH_2). IR (ATR): 1651, 1613, 1321, 762, 726, 696 cm^{-1}. MS (ESI) *m/z* (relative intensity): 823 (61) $[2M+Na]^+$, 423 (33) $[M+Na]^+$, 401 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{25}H_{29}N_4O$ $[M+H]^+$ 401.2341 found 401.2340.

2-[(1-*n*-Hexyl-1*H*-1,2,3-triazol-4-yl)methyl]-3-(3-methoxyphenyl)-4-methylisoquinolin-1(2*H*)-one (5bb)



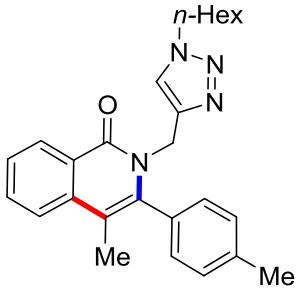
The representive procedure was followed using **1b** (85.8 mg, 0.30 mmol) and 1-methoxy-3-(prop-1-yn-1-yl)benzene (**4b**) (87.6 mg, 0.60 mmol). Purification by column chromatography (*n*-hexane/EtOAc 9:1→ 3:1) yielded **5bb** (52.7 mg, 41%) as a viscous yellow oil. R_f (*n*-hexane/EtOAc 1:1) = 0.37. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 8.51 (dd, *J* = 8.2, 2.6 Hz, 1H), 7.73-7.69 (m, 2H), 7.60 (s, 1H), 7.52 (ddd, *J* = 8.2, 5.2, 2.6 Hz, 1H), 7.41-7.35 (m, 1H), 7.01 (ddd, *J* = 8.2, 2.6, 1.1 Hz, 1H), 6.87-6.81 (m, 2H), 5.15-4.96 (m, 2H), 4.23 (t, *J* = 7.2 Hz, 2H), 3.82 (s, 3H), 2.03 (s, 3H), 1.88-1.73 (m, 2H), 1.31-1.19 (m, 6H), 0.84 (t, *J* = 7.2 Hz, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 162.1 (C_q), 159.6 (C_q), 144.0 (C_q), 140.0 (C_q), 137.2 (C_q), 135.9 (C_q), 132.3 (CH), 129.7 (CH), 128.0 (CH), 126.4 (CH), 125.2 (C_q), 123.3 (CH), 123.2 (CH), 122.2 (CH), 115.4 (CH), 115.1 (CH), 110.8 (C_q), 55.4 (CH_3), 50.2 (CH_2), 42.1 (CH_2), 31.1 (CH_2), 30.2 (CH_2), 26.2 (CH_2), 22.4 (CH_2), 15.0 (CH_3), 13.9 (CH_3). IR (ATR): 1645, 1589, 1285, 1045, 766, 708, 697 cm^{-1}. MS (ESI) *m/z* (relative intensity): 883 (44) $[2M+Na]^+$, 453 (35) $[M+Na]^+$, 431 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{26}H_{31}N_4O_2$ $[M+H]^+$ 431.2447 found 431.2442.

3-(4-Fluorophenyl)-2-[(1-*n*-hexyl-1*H*-1,2,3-triazol-4-yl)methyl]-4-methylisoquinolin-1(2*H*)-one (5bc)



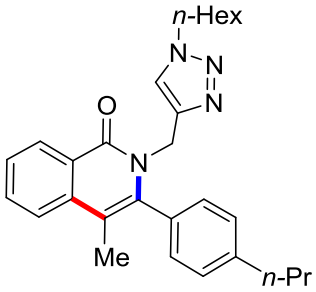
The representive procedure was followed using **1b** (85.8 mg, 0.30 mmol) and 1-fluoro-3-(prop-1-yn-1-yl)benzene (**4c**) (80.4 mg, 0.60 mmol). Purification by column chromatography (*n*-hexane/EtOAc 9:1→ 3:1) yielded **5bc** (63.8 mg, 51%) as viscous yellow oil. R_f (*n*-hexane/EtOAc 1:1) = 0.41. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 8.50-8.48 (m, 1H), 7.72-7.66 (m, 2H), 7.61 (s, 1H), 7.53-7.49 (m, 1H), 7.29-7.26 (m, 2H), 7.20-7.14 (m, 2H), 5.03 (s, 2H), 4.23 (t, J = 7.2 Hz, 2H), 1.97 (s, 3H), 1.85-1.78 (m, 2H), 1.28-1.22 (m, 6H), 0.85-0.80 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 163.9 (d, $^1J_{C-F}$ = 249 Hz, C_q), 161.6 (C_q), 139.1 (C_q), 137.2 (C_q), 132.5 (CH), 132.1 (d, $^3J_{C-F}$ = 8 Hz, CH), 130.8, (d, $^4J_{C-F}$ = 4 Hz, C_q), 128.1 (CH), 126.7 (CH), 125.9 (C_q), 125.5 (C_q) 123.6 (CH), 123.4 (CH), 115.9 (d, $^2J_{C-F}$ = 22 Hz, CH), 111.5 (C_q), 50.2 (CH_2), 41.9 (CH_2), 31.1 (CH_2), 30.1 (CH_2), 26.1 (CH_2), 22.4 (CH_2), 14.9 (CH_3), 13.9 (CH_3). ^{19}F-NMR (285 MHz, $CDCl_3$): δ = -111.6 (tt, J = 9, 6 Hz). IR (ATR): 1645, 1509, 1220, 849, 766, 729 cm^{-1}. MS (ESI) *m/z* (relative intensity): 859 (58) $[2M+Na]^+$, 441 (33) $[M+Na]^+$, 419 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{25}H_{28}FN_4O$ $[M+H]^+$ 419.2247 found 419.2242.

2-[(1-*n*-Hexyl-1*H*-1,2,3-triazol-4-yl)methyl]-4-methyl-3-phenylisoquinolin-1(2*H*)-one (5bd)



The representive procedure was followed using **1b** (85.8 mg, 0.30 mmol) and 1-methyl-4-(prop-1-yn-1-yl)benzene (**4d**) (78.2 mg, 0.60 mmol). Purification by column chromatography (*n*-hexane/EtOAc 9:1→ 3:1) yielded **5bd** (87.7 mg, 71%) as viscous yellow oil. R_f (*n*-hexane/EtOAc 1:1) = 0.39. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 8.51 (d, *J* = 8.1 Hz, 1H), 7.74-7.66 (m, 2H), 7.57 (s, 1H), 7.51 (ddd, *J* = 8.1, 5.6, 2.1 Hz, 1H), 7.28 (d, *J* = 7.8 Hz, 2H), 7.13 (d, *J* = 7.8 Hz, 2H), 5.07 (s, 2H), 4.22 (t, *J* = 7.2 Hz, 2H), 2.43 (s, 3H), 1.99 (s, 3H), 1.89-1.76 (m, 2H), 1.32-1.20 (m, 6H), 0.88-0.81 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 162.3 (C_q), 144.1 (C_q), 140.3 (C_q), 138.8 (C_q), 137.3 (C_q), 132.4 (CH), 131.8 (C_q), 129.9 (CH), 129.4 (CH), 128.0 (CH), 126.4 (CH), 125.2 (C_q), 123.4 (CH), 123.3 (CH), 111.1 (C_q), 50.1 (CH_2), 42.0 (CH_2), 31.0 (CH_2), 30.0 (CH_2), 26.0 (CH_2), 22.3 (CH_2), 21.4 (CH_3), 14.9 (CH_3) 13.9 (CH_3). IR (ATR): 1645, 1613, 1592, 1322, 804, 765, 696 cm^{-1}. MS (ESI) *m/z* (relative intensity): 851 (40) $[2M+Na]^+$, 437 (45) $[M+Na]^+$, 415 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{26}H_{31}N_4O$ $[M+H]^+$ 415.2498 found 415.2492.

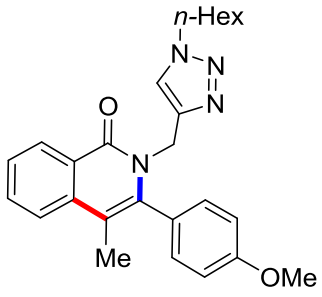
2-[(1-*n*-Hexyl-1*H*-1,2,3-triazol-4-yl)methyl]-4-methyl-3-(4-*n*-propylphenyl)isoquinolin-1(2*H*)-one (5be)



The representive procedure was followed using **1b** (85.8 mg, 0.30 mmol) and 1-(prop-1-yn-1-yl)-4-propylbenzene (**4e**) (194.8 mg, 0.60 mmol). Purification by column chromatography (*n*-hexane/EtOAc 9:1→ 3:1) yielded **5be** (87.3 mg, 66%) as a viscous yellow oil. R_f (*n*-

hexane/EtOAc 1:1) = 0.47. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 8.51-8.48 (m, 1H), 7.69-7.66 (m, 2H), 7.55 (s, 1H), 7.51-7.48 (m, 1H), 7.25 (d, *J* = 7.8 Hz, 2H), 7.14 (d, *J* = 7.8 Hz, 2H), 5.05 (s, 2H), 4.21 (t, *J* = 7.2 Hz, 2H), 2.64 (t, *J* = 7.8 Hz, 2H), 1.98 (s, 3H), 1.85-1.76 (m, 2H), 1.73-1.63 (m, 2H), 1.27-1.21 (m, 6H), 0.97 (t, *J* = 7.8 Hz, 3H), 0.83 (t, *J* = 6.8 Hz, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 162.2 (C_q), 144.3 (C_q), 143.5 (C_q), 140.4 (C_q), 137.4 (C_q), 132.4 (CH), 132.1 (CH), 129.9 (CH), 128.8 (CH), 128.7 (C_q), 128.0 (CH), 126.4 (CH), 125.1 (C_q), 123.3 (CH), 111.1 (C_q), 50.2 (CH_2), 42.0 (CH_2), 37.9 (CH_2), 31.1 (CH_2), 30.1 (CH_2), 26.0 (CH_2), 24.2 (CH_2), 22.4 (CH_2), 15.0 (CH_3), 13.9 (CH_3), 13.9 (CH_3). IR (ATR): 1646, 1613, 1592, 1322, 1047, 765, 697 cm^{-1}. MS (ESI) *m/z* (relative intensity): 907 (35) $[2M+Na]^+$, 443 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{28}H_{35}N_4O$ $[M+H]^+$ 443.2811 found 443. 2805.

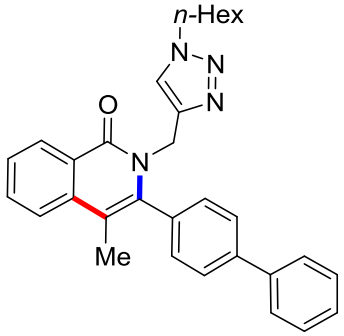
2-[(1-*n*-Hexyl-1*H*-1,2,3-triazol-4-yl)methyl]-3-(4-methoxyphenyl)-4-methylisoquinolin-1(2*H*)-one (5bf)



The representive procedure was followed using **1b** (85.8 mg, 0.30 mmol) and 1-methoxy-4-(prop-1-yn-1-yl)benzene (**4f**) (87.6 mg, 0.60 mmol). Purification by column chromatography (*n*-hexane/EtOAc 9:1→ 3:1) yielded **5bf** (99.0 mg, 77%) as a yellowish solid. M. p. = 101-103 °C. R_f (*n*-hexane/EtOAc 1:1) = 0.25. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 8.48 (d, *J* = 8.0 Hz, 1H), 7.70-7.65 (m, 2H), 7.57 (s, 1H), 7.49 (ddd, *J* = 8.0, 5.9, 2.0 Hz, 1H), 7.16 (d, *J* = 7.6 Hz, 2H), 6.97 (d, *J* = 7.6 Hz, 2H), 5.06 (s, 2H), 4.21 (t, *J* = 7.2 Hz, 2H), 3.85 (s, 3H), 1.98 (s, 3H), 1.85-1.77 (m, 2H), 1.29-1.20 (m, 6H), 0.83-0.80 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 159.8 (C_q), 140.0 (C_q), 137.3 (C_q), 133.3 (CH), 131.3 (CH), 130.4 (C_q), 128.0 (CH), 127.0 (C_q), 126.4 (CH), 125.2 (C_q), 123.4 (C_q), 123.3 (CH), 114.0 (CH), 113.3 (CH), 111.5 (C_q), 55.2 (CH_3), 50.1 (CH_2), 42.0 (CH_2), 31.0 (CH_2), 30.0 (CH_2), 26.1 (CH_2), 22.3 (CH_2), 15.0 (CH_3), 13.9 (CH_3). IR (ATR): 1644, 1508, 1245, 843, 766, 694 cm^{-1}. MS (ESI) *m/z* (relative

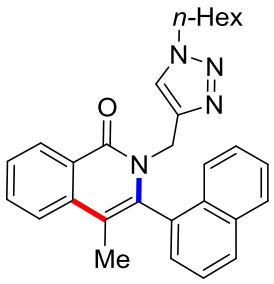
intensity): 833 (33) $[2M+Na]^+$, 453 (40) $[M+Na]^+$, 431 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{26}H_{31}N_4O_2$ $[M+H]^+$ 431.2447 found 431.2449.

3-{[1,1'-Biphenyl]-4-yl}-2-[(1-*n*-hexyl-1*H*-1,2,3-triazol-4-yl)methyl]-4-methylisoquinolin-1(2*H*)-one (5bg)



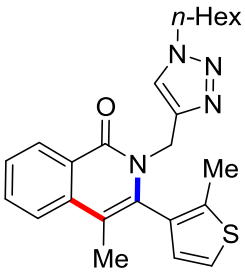
The representive procedure was followed using **1b** (85.8 mg, 0.30 mmol) and 4-(prop-1-yn-1-yl)-1,1'-biphenyl (**4g**) (115.2 mg, 0.60 mmol). Purification by column chromatography (*n*-hexane/EtOAc 9:1→ 3:1) yielded **5bg** (76.7 mg, 54%) as a yellowish solid. M. p. = 125-128 °C. R_f (*n*-hexane/EtOAc 1:1) = 0.38. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 8.53 (d, *J* = 8.0 Hz, 1H), 7.76-7.65 (m, 6H), 7.60 (s, 1H), 7.57-7.51 (m, 1H), 7.50-7.44 (m, 2H), 7.41-7.37 (m, 3H), 5.12 (s, 2H), 4.24 (t, *J* = 7.2 Hz, 2H), 2.05 (s, 3H), 1.89-1.79 (m, 2H), 1.30-1.23 (m, 6H), 0.87-0.82 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 162.1 (C_q), 143.9 (C_q), 141.6 (C_q), 140.1 (C_q), 139.9 (C_q), 137.2 (C_q), 133.7 (C_q), 132.3 (CH), 130.5 (CH), 128.7 (CH), 128.0 (CH), 127.6 (CH), 127.2 (CH), 127.0 (CH), 126.4 (CH), 125.3 (C_q), 123.3 (CH), 123.2 (CH), 111.1 (C_q), 50.2 (CH_2), 42.1 (CH_2), 31.1 (CH_2), 30.2 (CH_2), 26.2 (CH_2), 22.4 (CH_2), 15.1 (CH_3), 13.9 (CH_3). IR (ATR): 1644, 1591, 1484, 764, 728, 696 cm^{-1}. MS (ESI) *m/z* (relative intensity): 975 (71) $[2M+Na]^+$, 499 (41) $[M+Na]^+$, 477 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{31}H_{33}N_4O$ $[M+H]^+$ 477.2654 found 477.2649.

2-[(1-*n*-Hexyl-1*H*-1,2,3-triazol-4-yl)methyl]-4-methyl-3-(naphthalen-1-yl)isoquinolin-1(2*H*)-one (5bh)



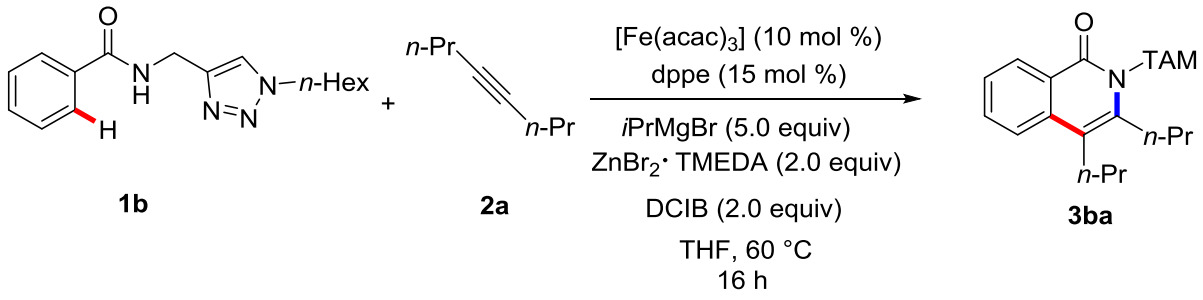
The representive procedure was followed using [Fe(acac)$_3$] (21.1 mg, 0.06 mmol), dppe (29.9 mg, 0.075 mmol), **1b** (85.8 mg, 0.30 mmol) and 1-(prop-1-yn-1-yl)naphthalene (**4h**) (99.6 mg, 0.60 mmol). Purification by column chromatography (*n*-hexane/EtOAc 9:1→ 3:1) yielded **5bh** (75.1 mg, 56%) as a viscous brownish oil. R_f (*n*-hexane/EtOAc 1:1) = 0.35. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 8.59 (d, *J* = 8.1 Hz, 1H), 7.99 (d, *J* = 8.1 Hz, 1H), 7.94 (d, *J* = 8.1 Hz, 1H), 7.78-7.72 (m, 2H), 7.62-7.54 (m, 2H), 7.53-7.45 (m, 2H), 7.45-7.32 (m, 3H), 5.27 (d, *J* = 14.5 Hz, 1H), 4.57 (d, *J* = 14.5 Hz, 1H), 4.22-4.11 (m, 2H), 1.92 (s, 3H), 1.83-1.72 (m, 2H), 1.34-1.16 (m, 6H), 0.90-0.78 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 162.5 (C_q), 144.0 (C_q), 138.0 (C_q), 137.3 (C_q), 133.5 (C_q), 132.5 (CH), 131.8 (C_q), 131.7 (C_q), 129.7 (CH), 129.1 (CH), 128.7 (CH), 128.2 (CH), 127.0 (CH), 126.7 (CH), 126.3 (CH), 125.6 (C_q), 125.5 (CH), 124.6 (CH), 123.4 (CH), 123.1 (CH), 112.2 (C_q), 50.1 (CH_2), 41.9 (CH_2), 31.0 (CH_2), 30.1 (CH_2), 26.1 (CH_2), 22.4 (CH_2), 14.6 (CH_3), 13.9 (CH_3). IR (ATR): 1645, 1592, 799, 775, 727, 696 cm^{-1}. MS (ESI) *m/z* (relative intensity): 923 (76) $[2M+Na]^+$, 473 (18) $[M+Na]^+$, 451 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{29}H_{31}N_4O$ $[M+H]^+$ 451.2498 found 451.2492.

2-[(1-*n*-Hexyl-1*H*-1,2,3-triazol-4-yl)methyl]-4-methyl-3-(2-methylthiophen-3-yl)isoquinolin-1(2*H*)-one (5bi)



The representive procedure was followed using **1b** (85.8 mg, 0.30 mmol) and 2-methyl-3-(prop-1-yn-1-yl)thiophene (**4i**) (81.6 mg, 0.60 mmol). Purification by column chromatography (*n*-hexane/EtOAc 9:1→ 3:1) yielded **5bi** (85.4 mg, 68%) as a yellowish oil. R_f (*n*-hexane/EtOAc 1:1) = 0.39. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 8.50 (ddd, *J* = 8.1, 1.4, 0.6 Hz, 1H), 7.72-7.67 (m, 2H), 7.56 (s, 1H), 7.51 (ddd, *J* = 8.1, 6.4, 2.0 Hz, 1H), 7.22 (d, *J* = 5.1 Hz, 1H), 6.85 (dd, *J* = 5.1, 0.6 Hz, 1H), 5.13-5.02 (m, 2H), 4.20 (t, *J* = 7.2 Hz, 2H), 2.07 (s, 3H), 2.00 (s, 3H), 1.84-1.77 (m, 2H), 1.28-1.19 (m, 6H), 0.85-0.79 (m, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 162.5 (C_q), 143.9 (C_q), 139.1 (C_q), 137.1 (C_q), 134.7 (C_q), 132.4 (CH), 131.6 (C_q), 128.6 (CH), 128.0 (CH), 126.6 (CH), 125.4 (C_q), 123.4 (CH), 123.3 (CH), 123.2 (CH), 112.5 (C_q), 50.1 (CH_2), 41.6 (CH_2), 31.0 (CH_2), 30.1 (CH_2), 26.0 (CH_2), 22.3 (CH_2), 14.2 (CH_3), 13.9 (CH_3), 13.4 (CH_3). IR (ATR): 1644, 1612, 1593, 1046, 766, 679 cm^{-1}. MS (ESI) *m/z* (relative intensity): 863 (28) $[2M+Na]^+$, 443 (38) $[M+Na]^+$, 421 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{24}H_{29}N_4OS$ $[M+H]^+$ 421.2062 found 421.2057.

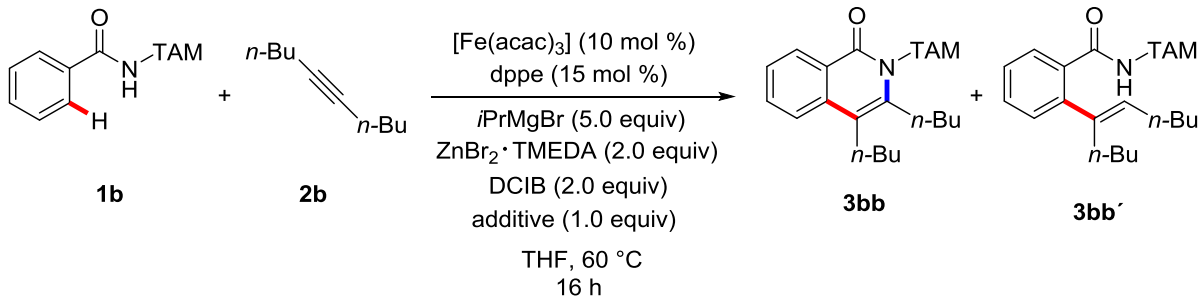
Reaction on one gram-scale



1b **2a** **3ba**

To a stirred solution of **1** (1.00 g, 3.5 mmol), $ZnBr_2$•TMEDA (2.40 g, 7.0 mmol), dppe (209 mg, 0.52 mmol) and the 4-octyne (1.1 ml, 7.0 mmol) in THF (4.0 ml), *i*PrMgBr (3.0 M in 2-MeTHF, 5.9 ml, 17.5 mmol) was added dropwise and the reaction mixture was stirred for 5 min at ambient temperature. Then, $Fe(acac)_3$ (124 mg, 0.35 mmol) was added in a single portion. After stirring the solution for additional 5 min, DCIB (0.8 ml, 7.0 mmol) was added. Then, the mixture was placed in a pre-heated oil bath at 60 °C. After stirring for 16 h, sat. aqueous NH_4Cl (15 ml) was added to the reaction mixture, which was extracted with CH_2Cl_2 (3 x15 ml). The combined organic extracts were dried over Na_2SO_4, filtered and concentrated. Purification by column chromatography (*n*-hexane/EtOAc 9:1→ 3:1) yielded **3ba** (952 mg, 69%) as a yellowish oil. R_f (*n*-hexane/EtOAc 1:1) = 0.57.

Probing SET-type mechanism

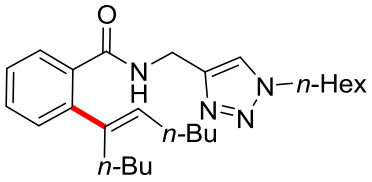


To a stirred solution of **1b** (0.30 mmol), $ZnBr_2$•TMEDA (204.6 mg, 0.60 mmol), dppe (17.9 mg, 0.045 mmol) and the alkyne **2b** (0.60 mmol) in THF (0.50 ml), *i*PrMgBr (3.0 M in 2-MeTHF, 500 µl, 1.5 mmol) was added dropwise and the reaction mixture was stirred for 5 min at ambient temperature. Then, $Fe(acac)_3$ (10.6 mg, 0.03 mmol) was added in a single portion. After stirring the solution for additional 5 min, an additive (0.30 mmol, 1.0 equiv) and DCIB (70 µl, 0.60 mmol) were added. Then, the mixture was placed in a pre-heated oil bath at 60 °C. After stirring for 16 h, sat. aqueous NH_4Cl (15 ml) was added to the reaction mixture, which was extracted with CH_2Cl_2 (3 x15 ml). The combined organic extracts were dried over Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography (*n*hexane/EtOAc 9:1→ 3:1→1:1).

Entry	Additive (1.0 equiv)	**3ab** (%)	**3ab´**
1	none	84	--
2	TEMPO[b]	58	15
3	BHT[c]	46	24
4	Galvinoxyl[d]	43	20

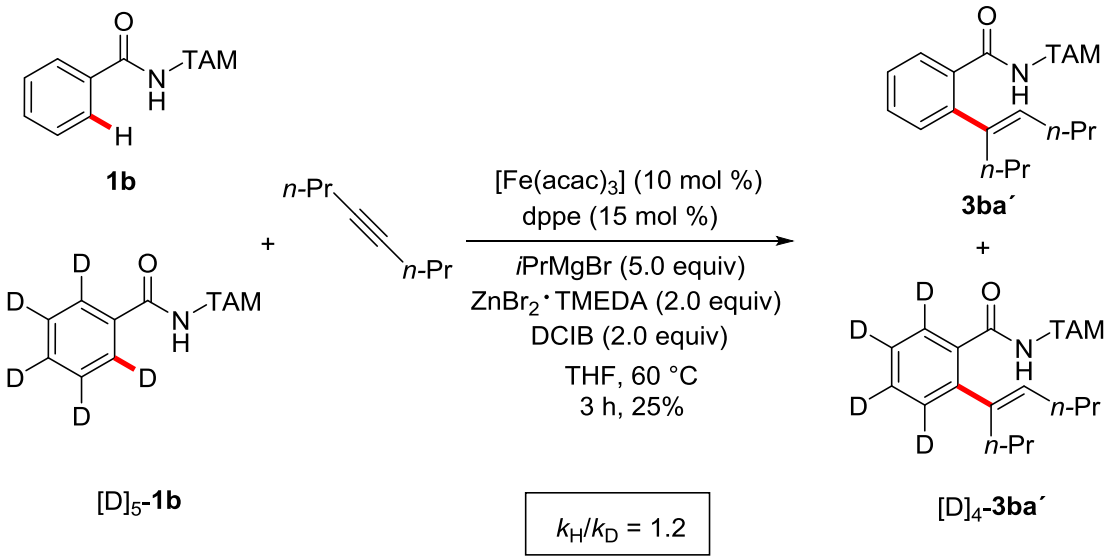
[a] Reaction conditions: **1b** (0.30 mmol), **2b** (0.60 mmol), $Fe(acac)_3$ (0.03 mmol), dppen (0.045 mmol), additive (0.30 mmol), $ZnBr_2$•TMEDA (0.60 mmol, 2.0 equiv), *i*PrMgBr (1.50 mmol, 5.0 equiv), DCIB (0.60 mmol), THF (0.50 ml), 60 °C, 16 h, isolated yields; [b] TEMPO = (2,2,6,6-tetramethypiperidin-1-yl)oxidanyl; [c] BHT = 2,6-bis(1,1-dimethylethyl)-4-methylphenol; [d] Galvinoxyl = 2,6-di-*tert*-butyl-α-(3,5-di-*tert*-butyl-4-oxo-2,5-cyclohexadien-1-ylidene)-*p*-tolyloxy.

(*E*)-2-(Dec-5-en-5-yl)-*N*-[(1-*n*-hexyl-1*H*-1,2,3-triazol-4-yl)methyl]benzamide (3bb´)

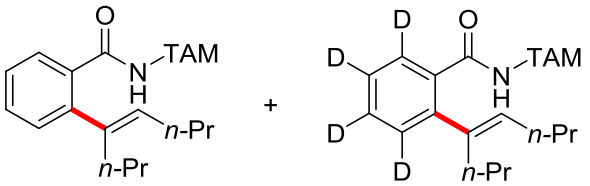


The representive procedure was followed using **1b** (85.8 mg, 0.30 mmol) and 5-decyne (**2b**) (82.8 mg, 0.60 mmol). Purification by column chromatography (*n*-hexane/EtOAc 9:1→ 3:1) yielded **3bb´** (68.6 mg, 54%) as a white solid. M. p. = 76-79 °C. R_f (*n*-hexane/EtOAc 1:1) = 0.35. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 7.71 (dd, *J* = 7.4, 1.8 Hz, 1H), 7.58 (s, 1H), 7.39-7.24 (m, 2H), 7.09 (dd, *J* = 7.4, 1.8 Hz, 1H), 6.90 (t, *J* = 5.8 Hz, 1H), 5.46 (t, *J* = 7.0 Hz, 1H), 4.62 (t, *J* = 5.8 Hz, 2H), 4.31 (t, *J* = 7.0 Hz, 2H), 2.15-2.08 (m, 4H), 1.93-1.82 (m, 2H), 1.41-1.24 (m, 10H), 1.18-1.06 (m, 4H), 0.96-0.83 (m, 6H), 0.77 (t, *J* = 6.8 Hz, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 169.0 (C_q), 144.2 (C_q), 142.6 (C_q), 141.5 (C_q), 133.2 (C_q), 131.8 (CH), 131.6 (CH), 130.3 (CH), 128.9 (CH), 127.0 (CH), 122.1 (CH), 50.3 (CH_2), 35.4 (CH_2), 31.9 (CH_2), 31.7 (CH_2), 31.1 (CH_2), 30.4 (CH_2), 30.2 (CH_2), 27.9 (CH_2), 26.2 (CH_2), 22.6 (CH_2), 22.5 (CH_2), 22.3 (CH_2), 14.0 (CH_3), 13.9 (CH_3), 13.8 (CH_3). IR (ATR): 2925, 1653, 1260, 1060, 1021, 797 cm^{-1}. MS (ESI) *m/z* (relative intensity): 871 (26) $[2M+Na]^+$, 447 (92) $[M+Na]^+$, 425 (100) $[M+H]^+$. HR-MS (ESI) *m/z* calcd for $C_{26}H_{41}N_4O$ $[M+H]^+$ 425.3280 found 425.3275.

Intermolecular KIE

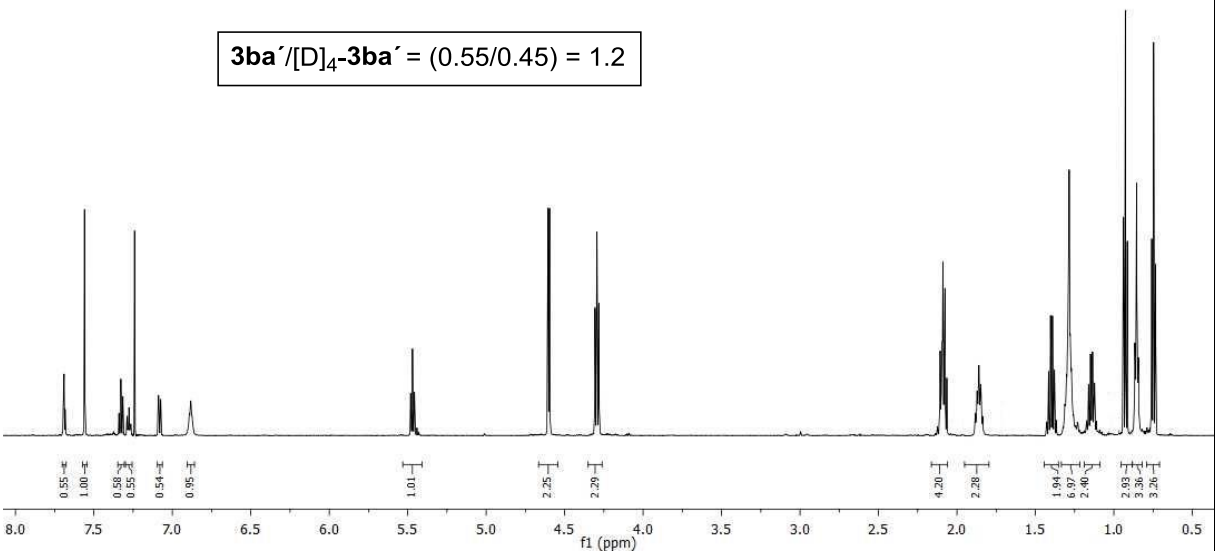


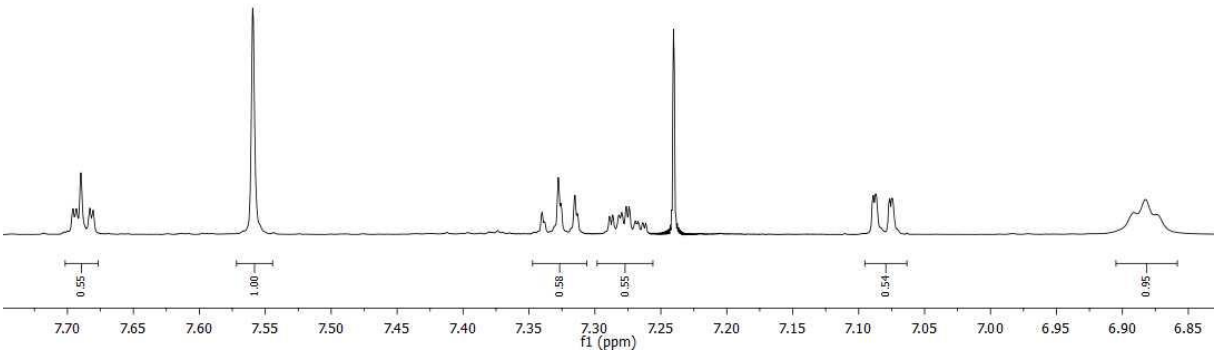
To a stirred solution of **1b** (0.15 mmol), [D]$_5$-**1b** (0.15 mmol), $ZnBr_2$•TMEDA (204.6 mg, 0.60 mmol), dppe (17.9 mg, 0.045 mmol) and 4-octyne (**2a**) (0.60 mmol) in THF (0.50 ml), *i*PrMgBr (3.0 M in 2-MeTHF, 500 µl, 1.5 mmol) was added dropwise and the reaction mixture was stirred for 5 min at ambient temperature. Then, $Fe(acac)_3$ (10.6 mg, 0.03 mmol) was added in a single portion. After stirring the solution for additional 5 min, DCIB (70 µl, 0.60 mmol) was added. Then, the mixture was placed in a pre-heated oil bath at 60 °C. After stirring for 3 h, sat. aqueous NH_4Cl (15 ml) was added to the reaction mixture, which was extracted with CH_2Cl_2 (3 x15 ml). The combined organic extracts were dried over Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography (*n*-hexane/EtOAc 9:1→ 1:1). The mixture was analyzed by 600 MHz ^{1}H-NMR spectroscopy to determine the ratio of **3ba´**/[D]$_4$-**3ba´**.



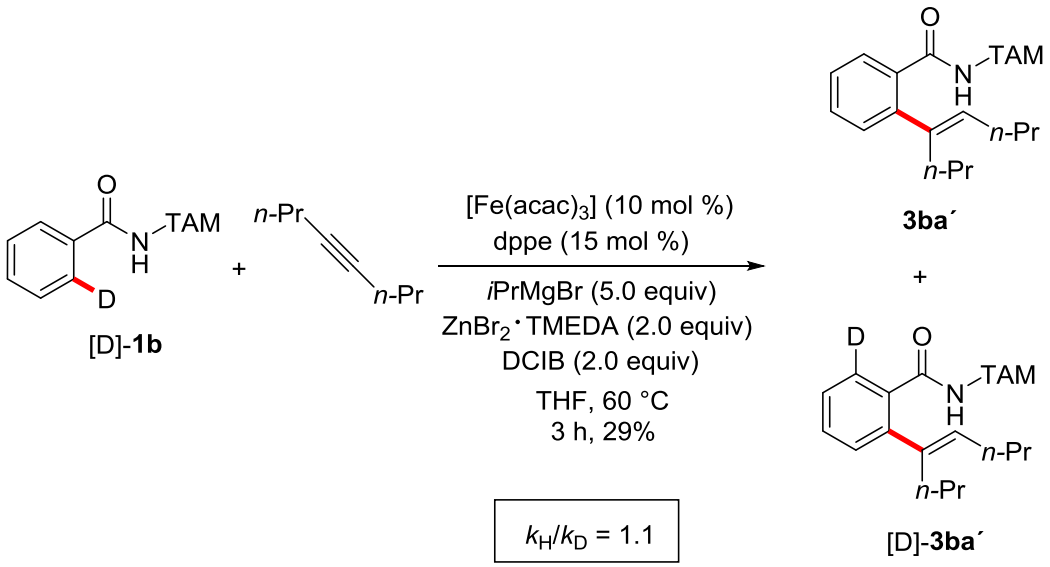
3ba´ [D]$_4$-**3ba´**

3ba´/[D]$_4$-**3ba´** = (0.55/0.45) = 1.2

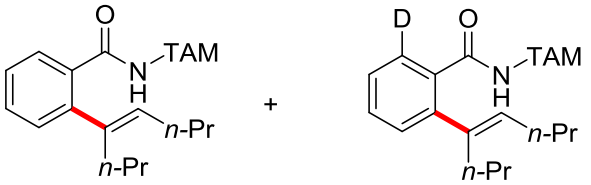




Intramolecular KIE



To a stirred solution of [D]-**1b** (0.30 mmol), $ZnBr_2$•TMEDA (204.6 mg, 0.60 mmol), dppe (17.9 mg, 0.045 mmol) and 4-octyne (**2a**) (0.60 mmol) in THF (0.50 ml), *i*PrMgBr (3.0 M in 2-MeTHF) (500 µl, 1.5 mmol) was added dropwise and the reaction mixture was stirred for 5 min at ambient temperature. Then, $Fe(acac)_3$ (10.6 mg, 0.03 mmol) was added in a single portion. After stirring the solution for additional 5 min, DCIB (70 µl, 0.60 mmol) was added. Then, the mixture was placed in a pre-heated oil bath at 60 °C. After stirring for 3 h, sat. aqueous NH_4Cl (15 ml) was added to the reaction mixture, which was extracted with CH_2Cl_2 (3 x15 ml). The combined organic extracts were dried over Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography (*n*hexane/EtOAc 9:1→ 1:1). The mixture was analyzed by ^{1}H-NMR spectroscopy to determine the ratio of [D]-**3ba´/3ba´**.

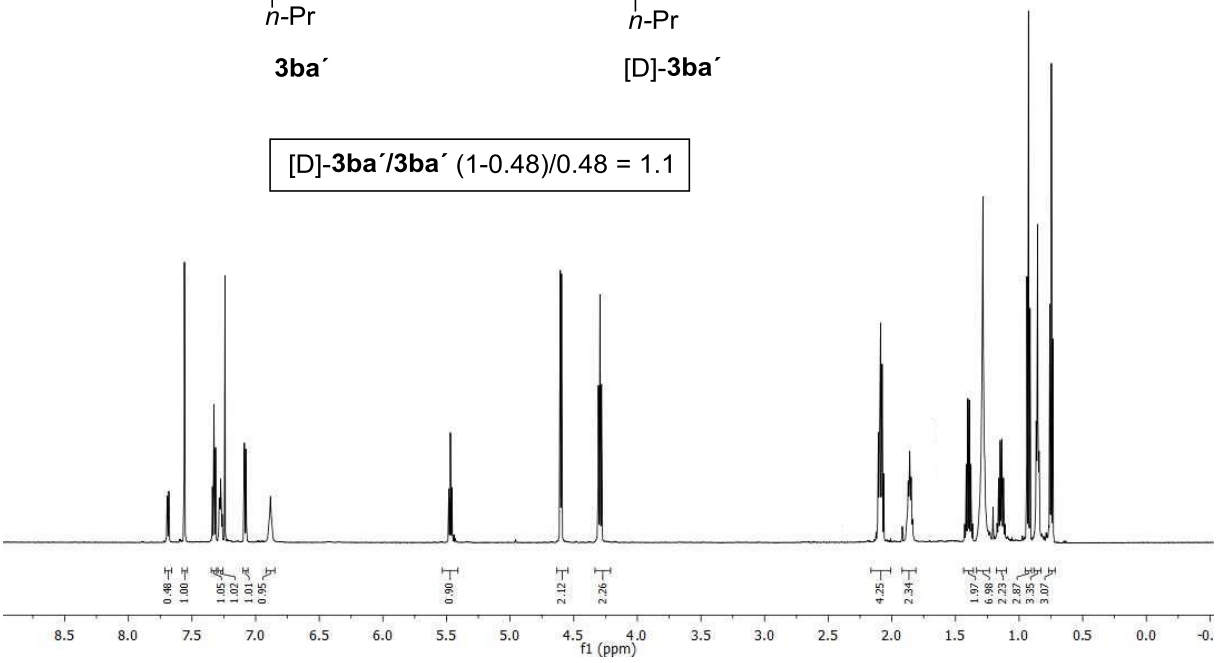


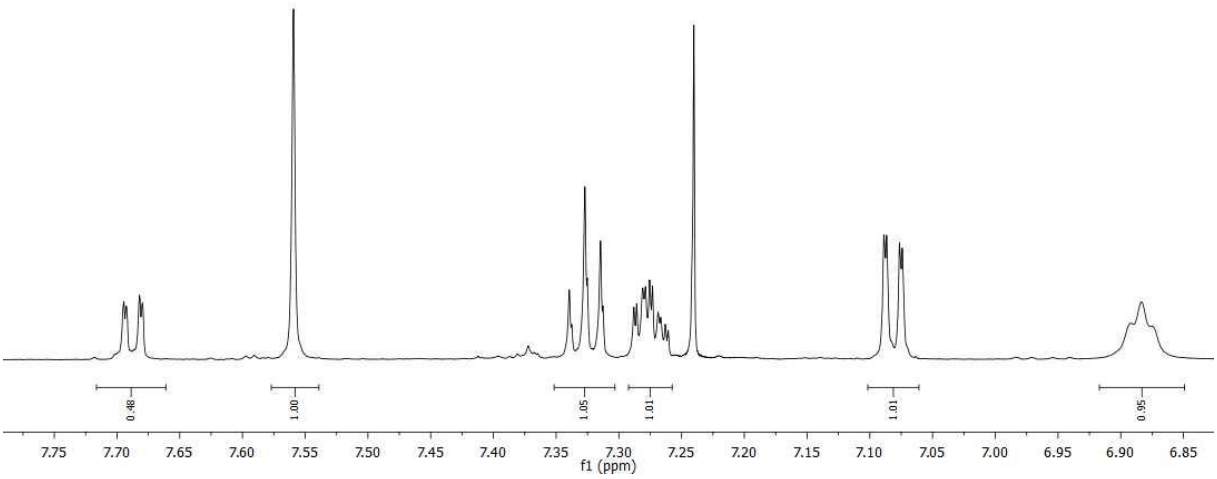
3ba´

+

[D]-**3ba´**

[D]-**3ba´**/**3ba´** (1-0.48)/0.48 = 1.1





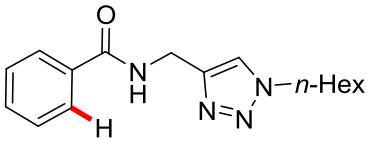
References

[1] H. H. Al Mamari, E. Diers and L. Ackermann, *Chem. Eur. J.* 2014, **20**, 9739–9743.

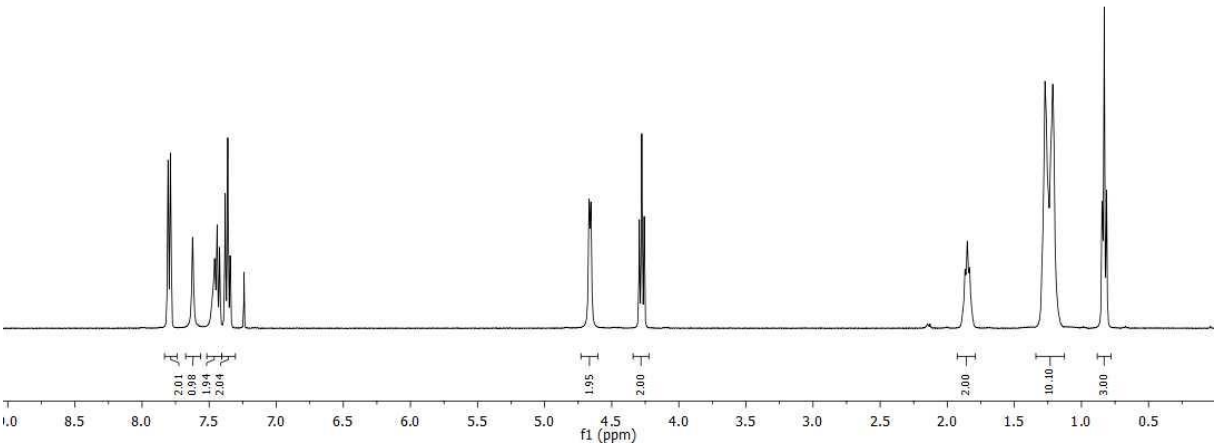
[2] B.–Y. Lee, S. R. Park, H. B. Yeon and K. S. Kim, *Tetrahedron Lett.* 2006, **47**, 5105–5109.

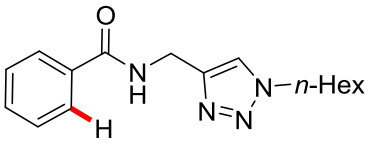
[3] D. R. Stuart, M. Bertrand-Laperle, K. M. N. Burgess and K. Fagnou, *J. Am. Chem. Soc.* 2008, **130**, 16474–16475.

^{1}H-, ^{13}C-, ^{19}F-NMR Spectra

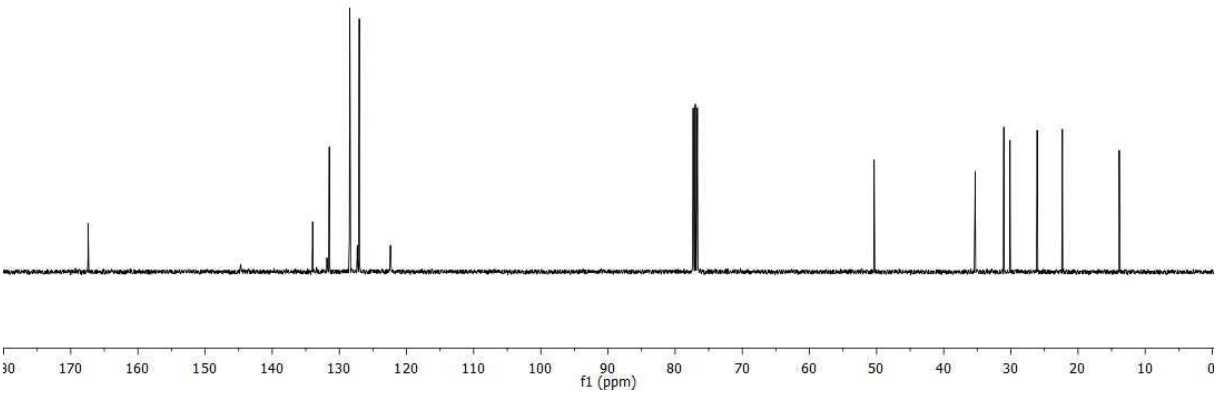


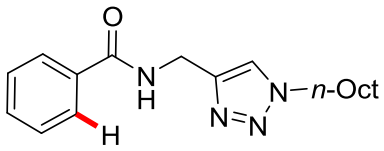
1b (300 MHz, $CDCl_3$)





1b (125 MHz, $CDCl_3$)





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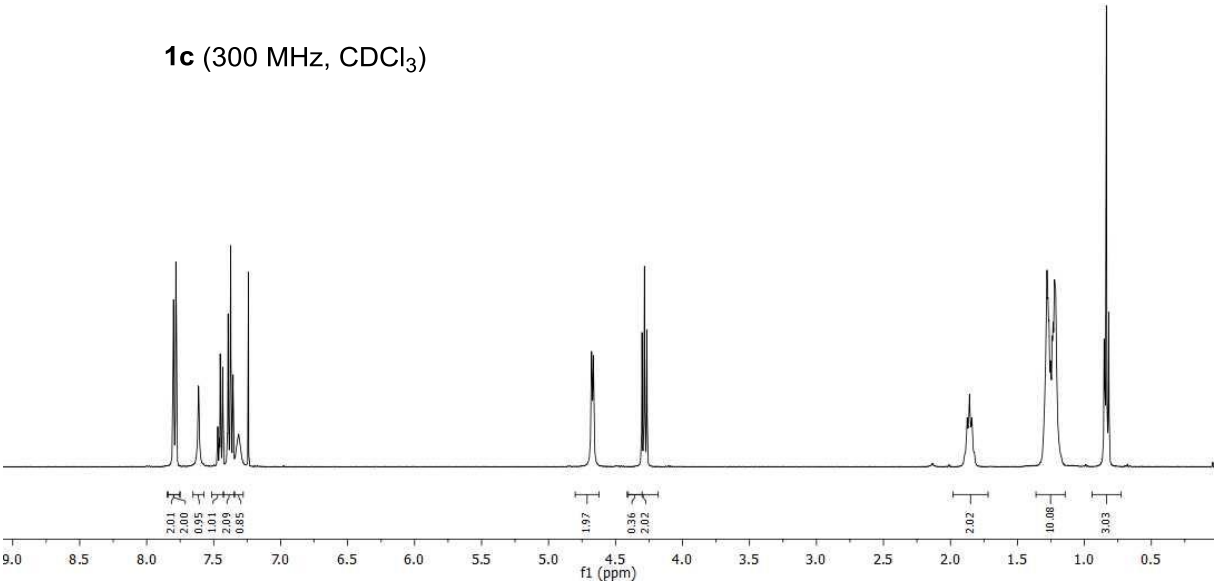
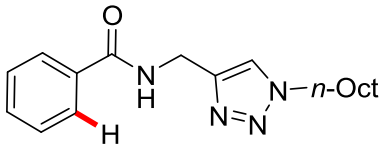
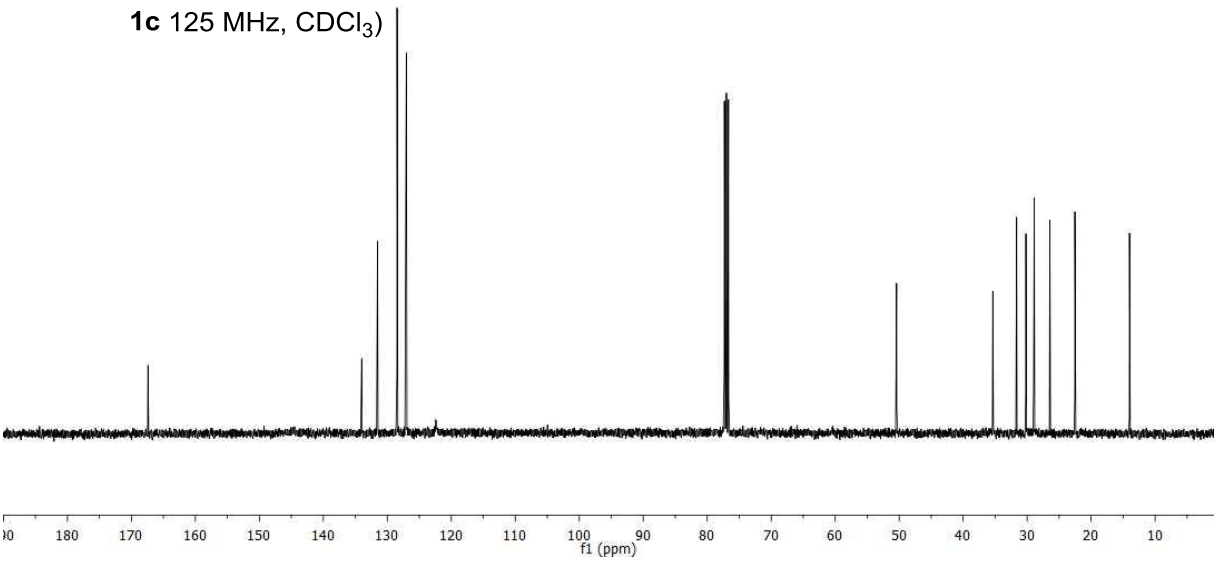
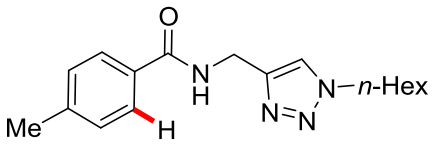


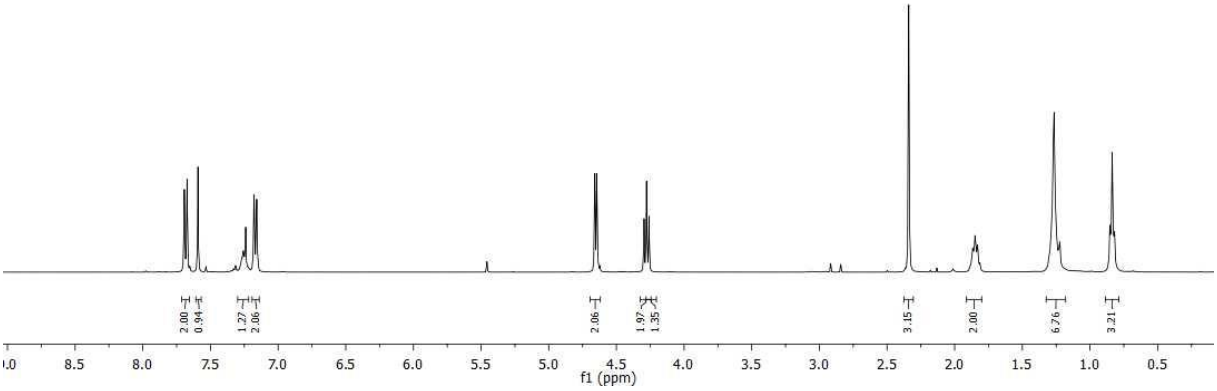


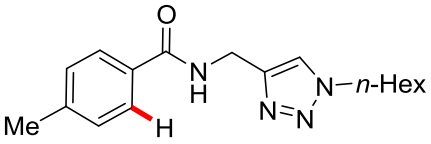
1c 125 MHz, $CDCl_3$)





1f (300 MHz, $CDCl_3$)





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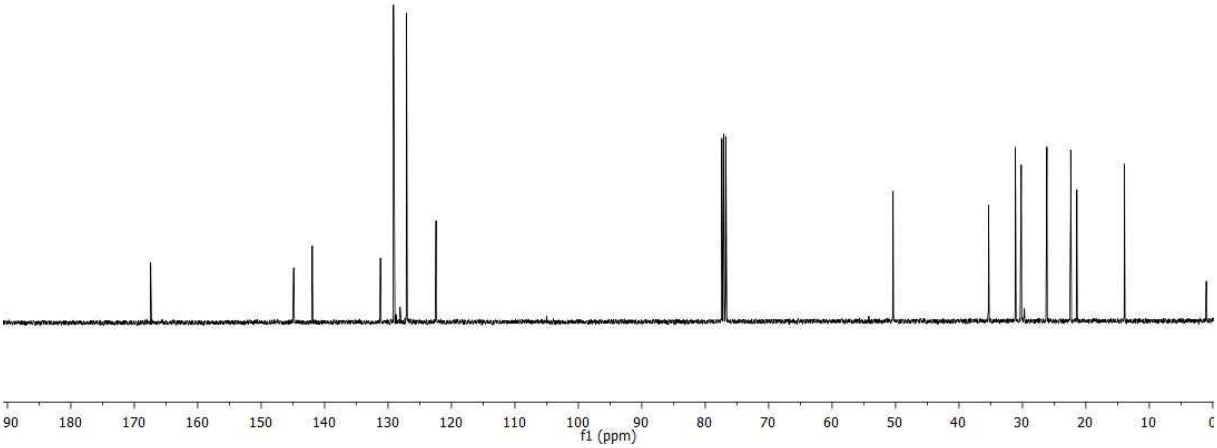

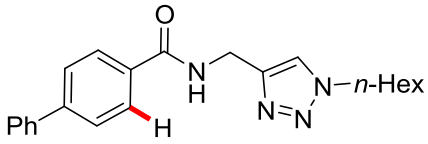

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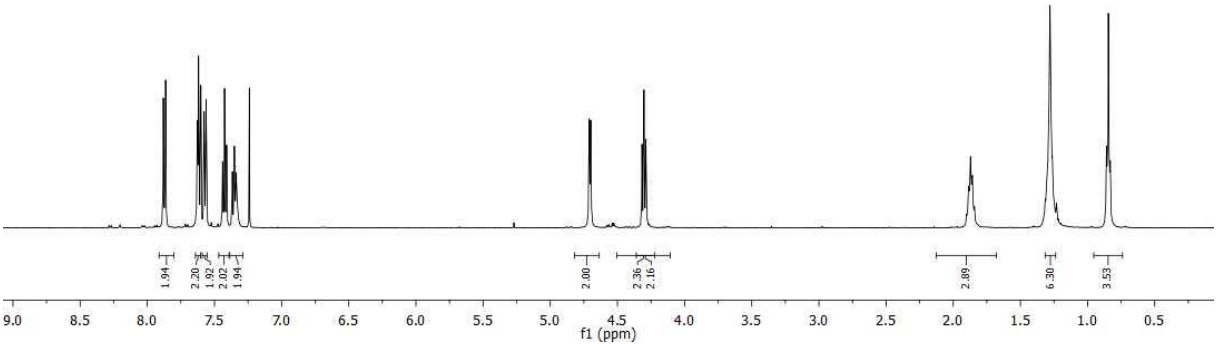

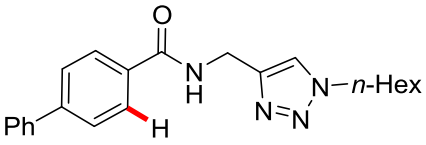

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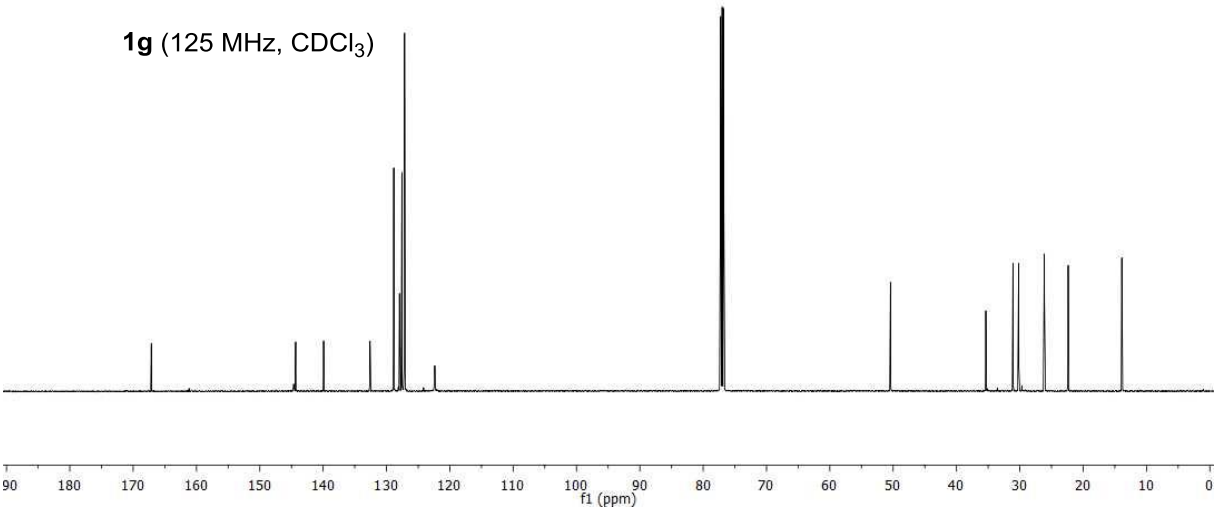

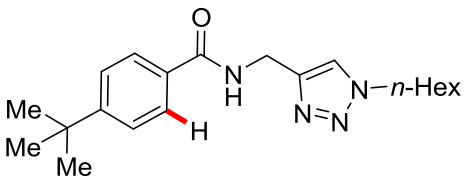

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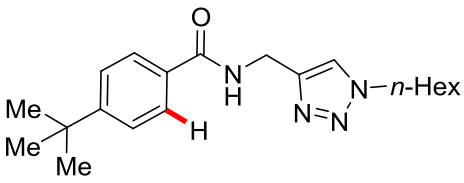

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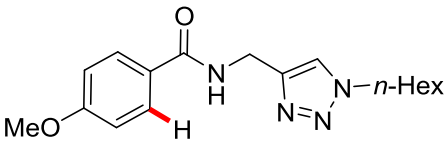

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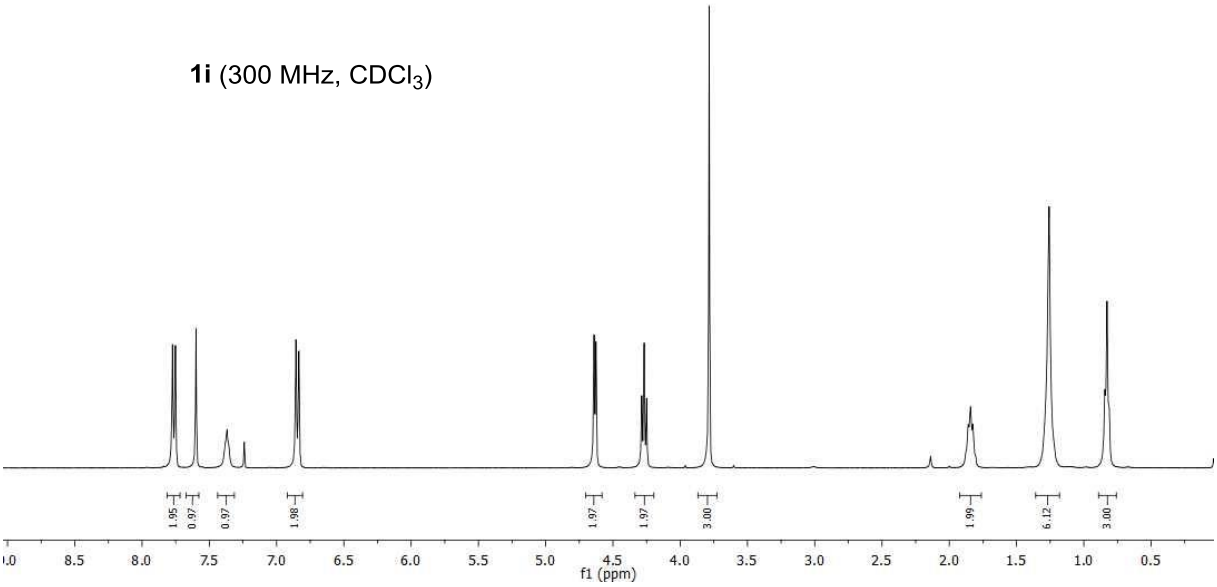

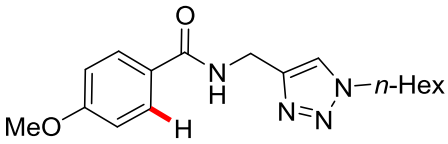

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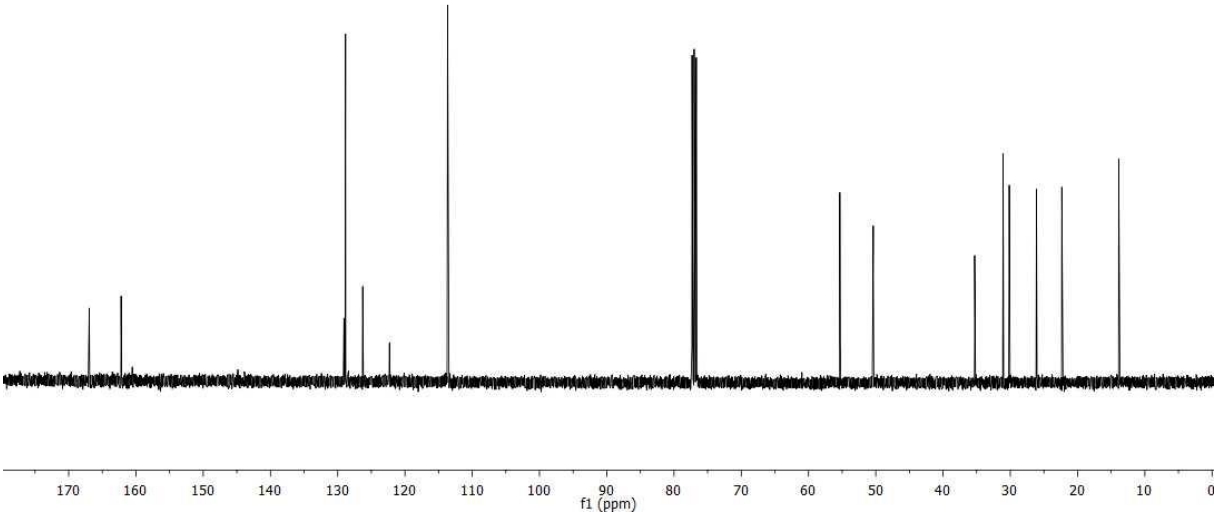

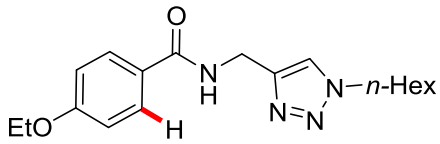

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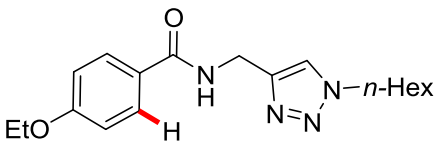

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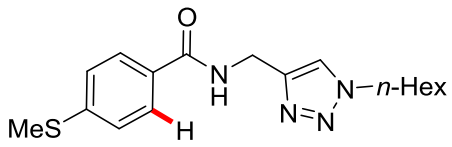

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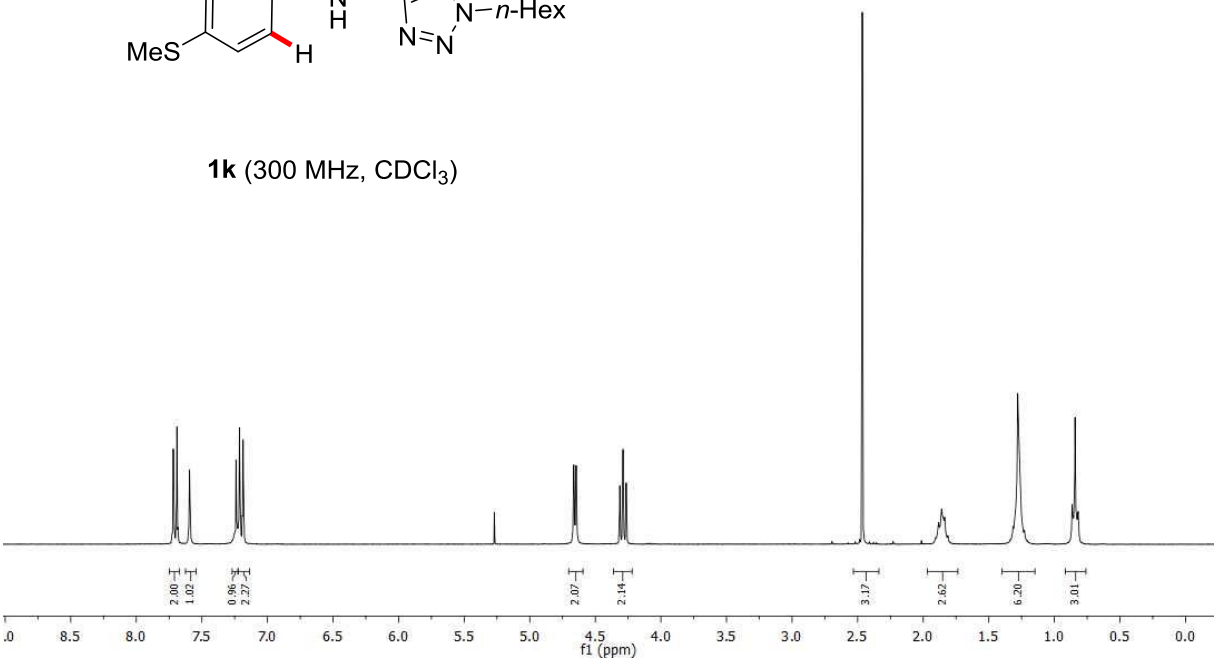

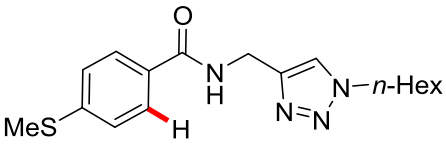

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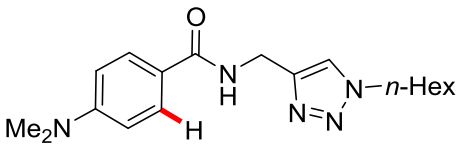

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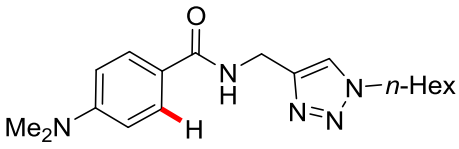

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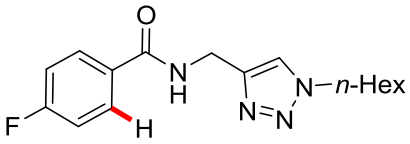

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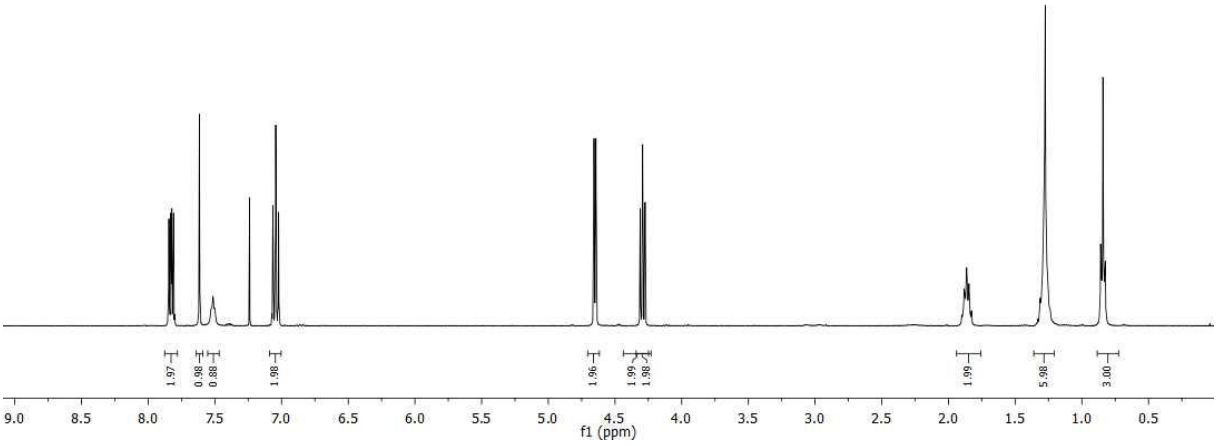

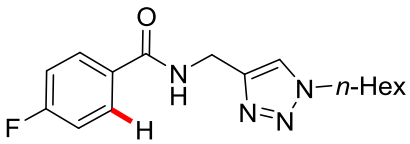

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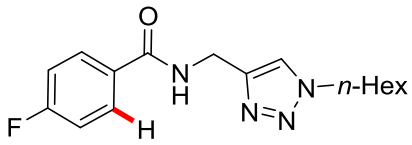

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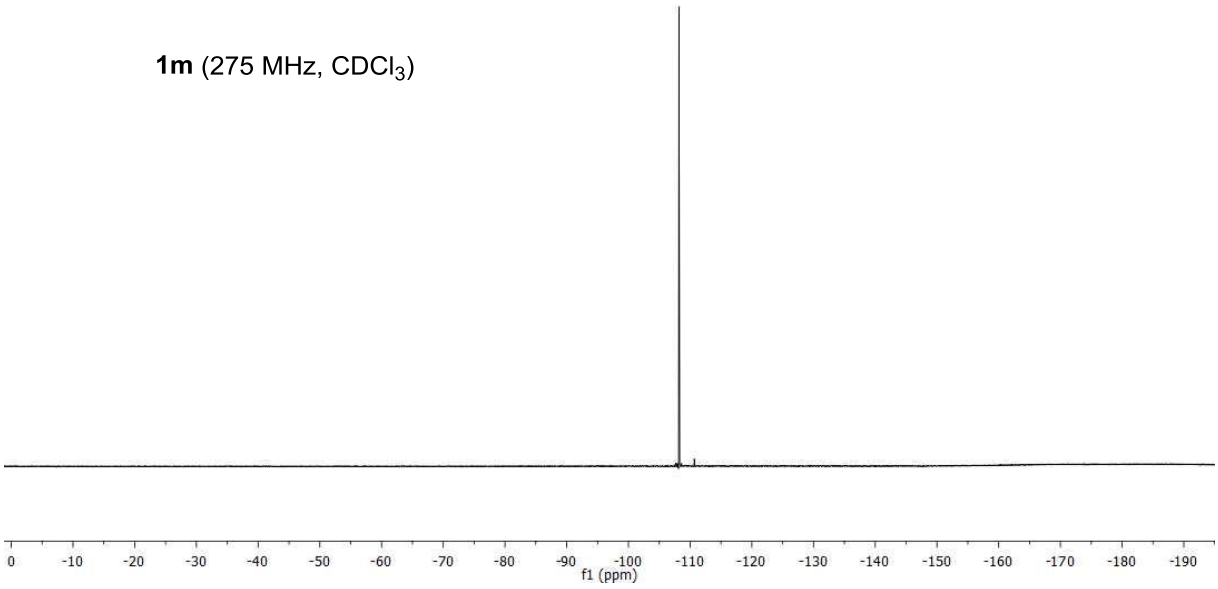
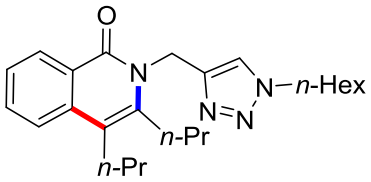
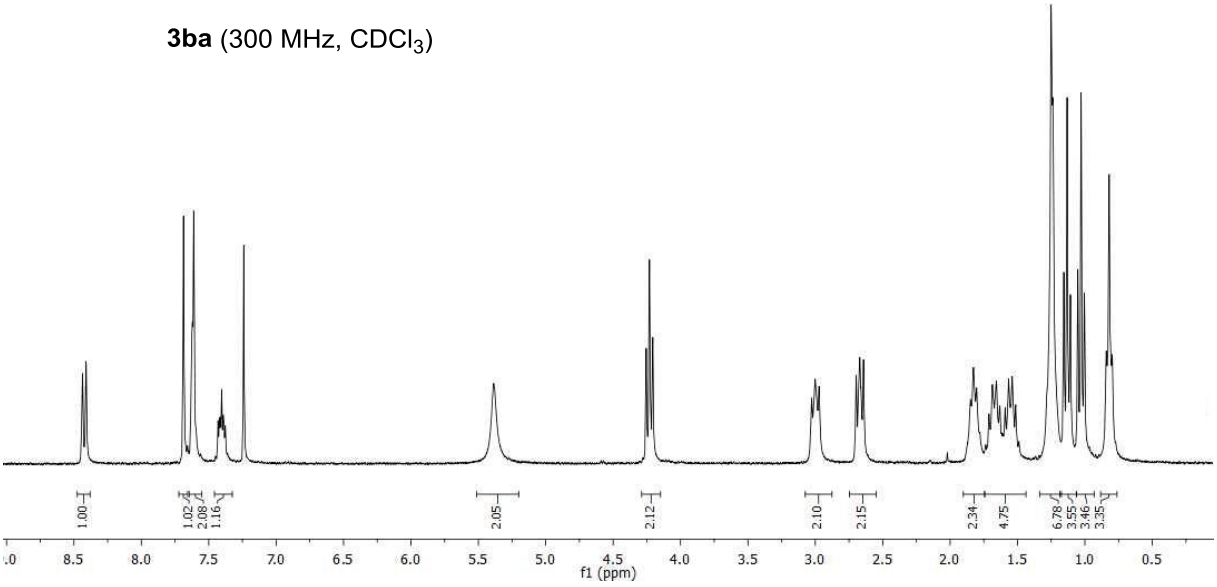
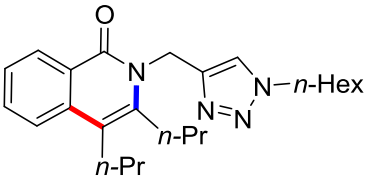


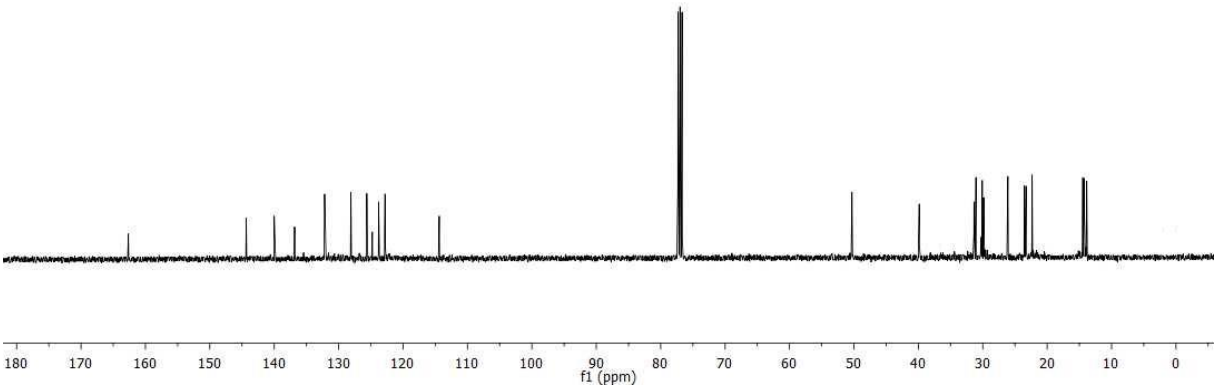


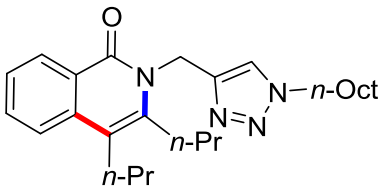
3ba (300 MHz, $CDCl_3$)





3ba (125 MHz, $CDCl_3$)





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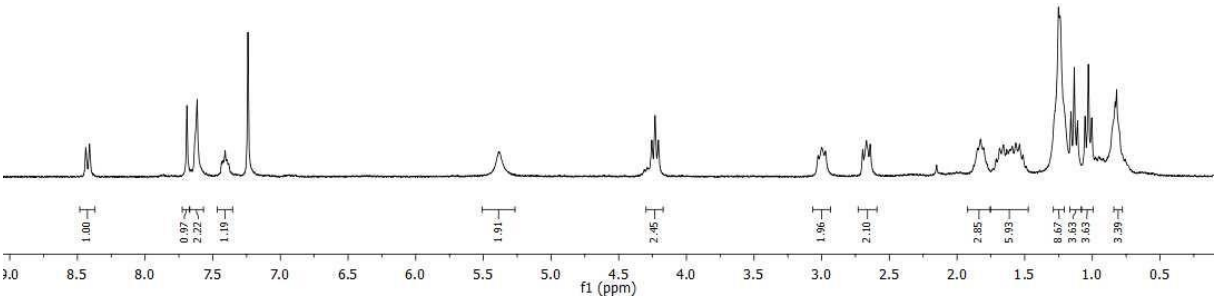

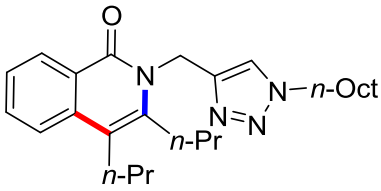

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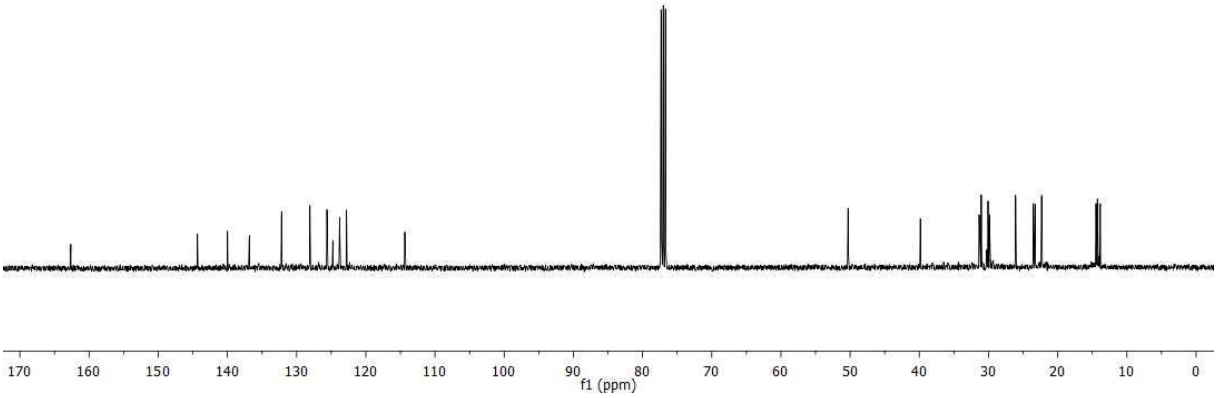
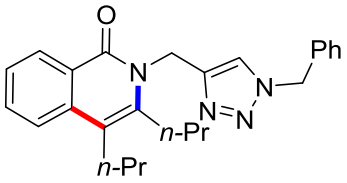
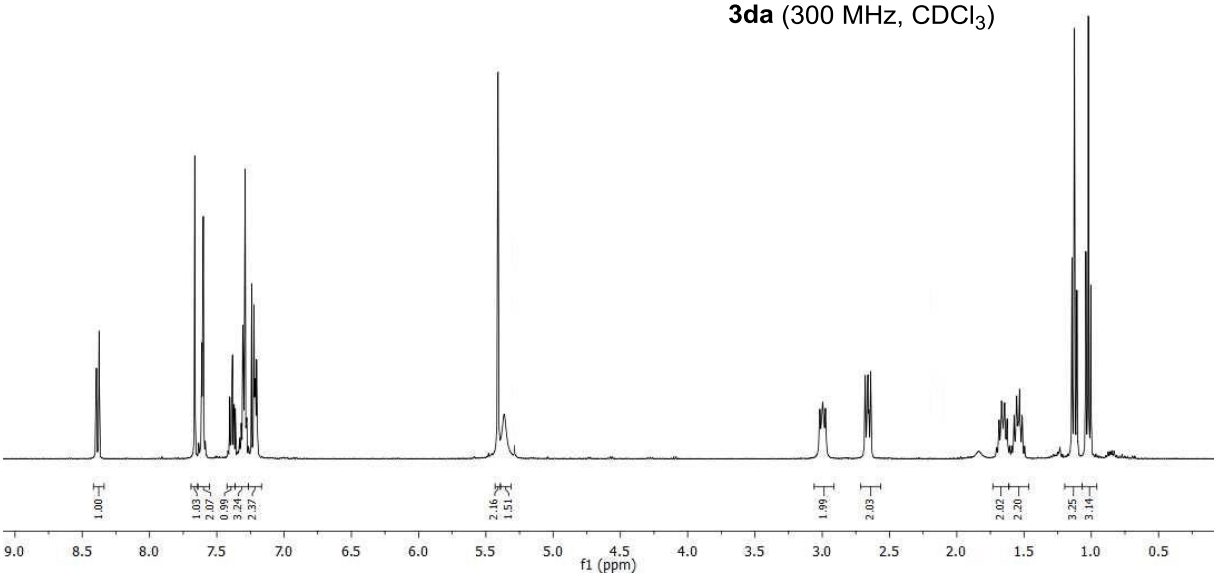
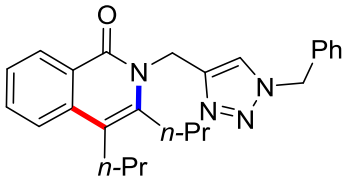




3da (300 MHz, $CDCl_3$)





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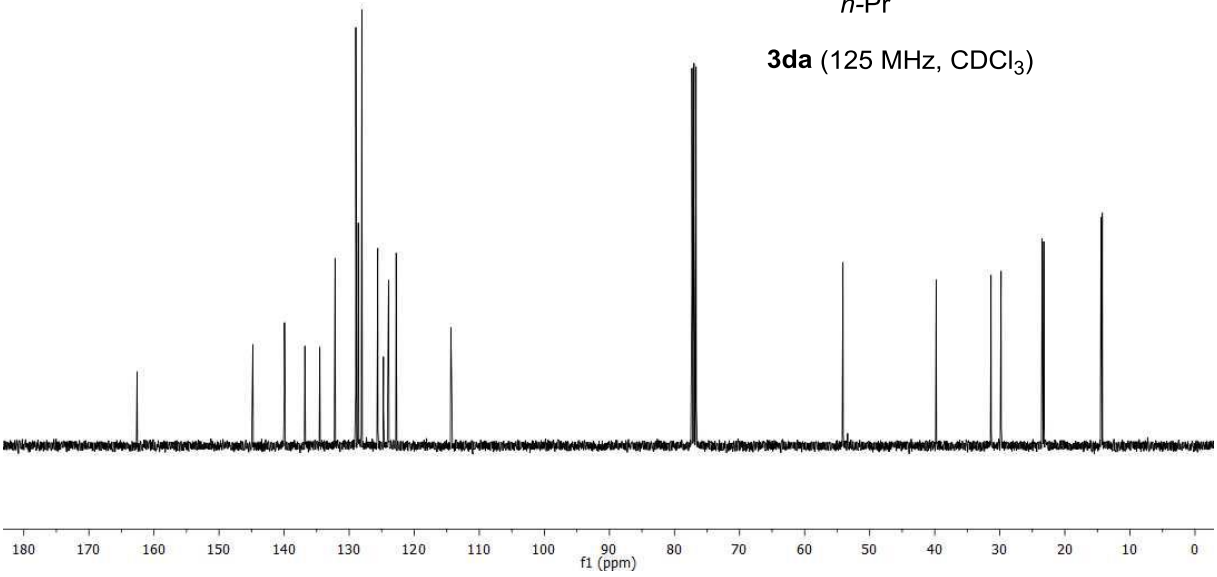

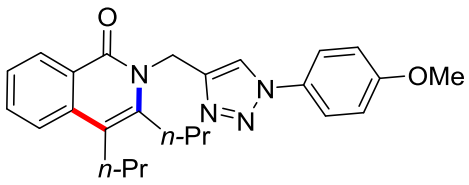

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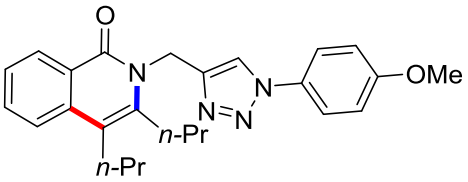

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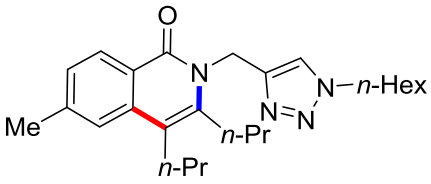
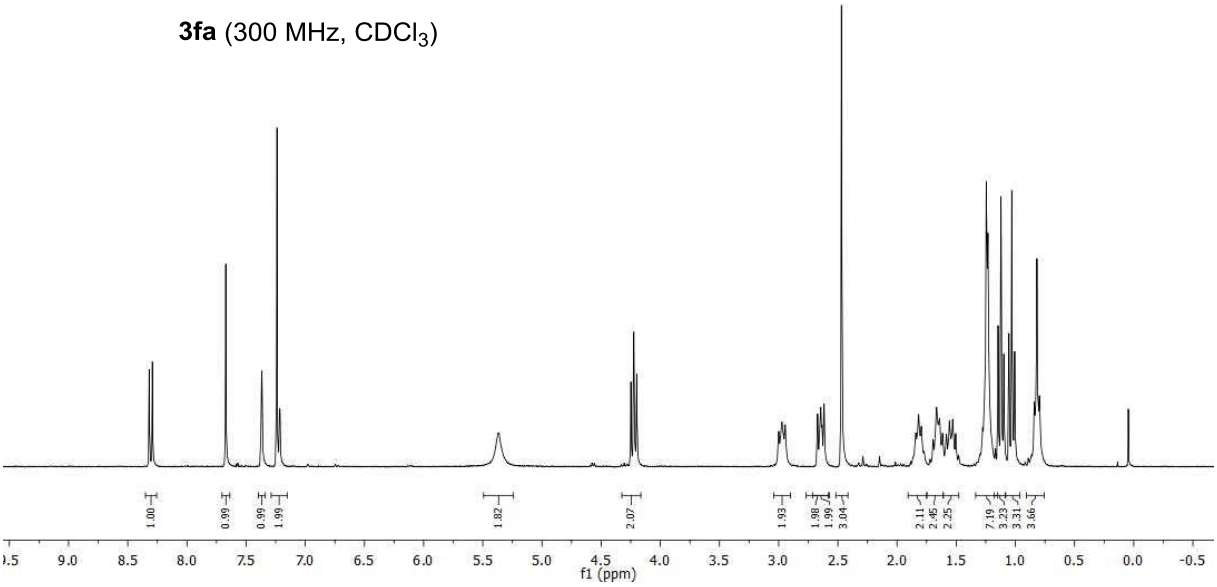
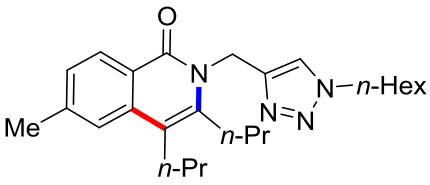
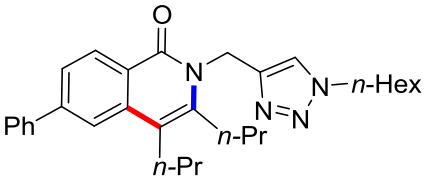


3fa (300 MHz, $CDCl_3$)

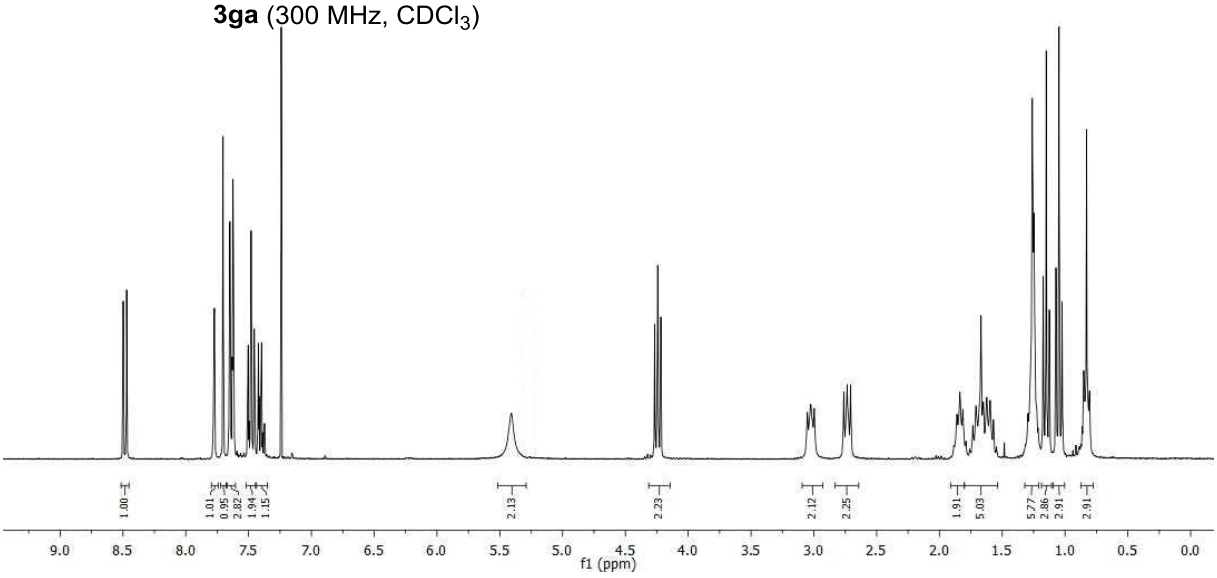


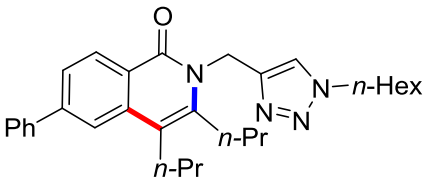


3fa (125 MHz, $CDCl_3$)

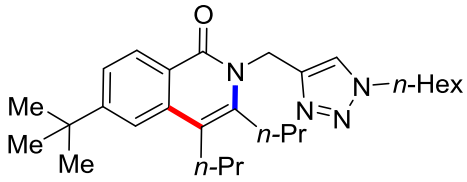


3ga (300 MHz, $CDCl_3$)

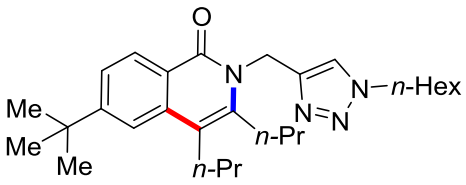




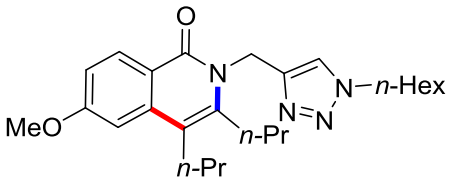
3ga (125 MHz, $CDCl_3$)



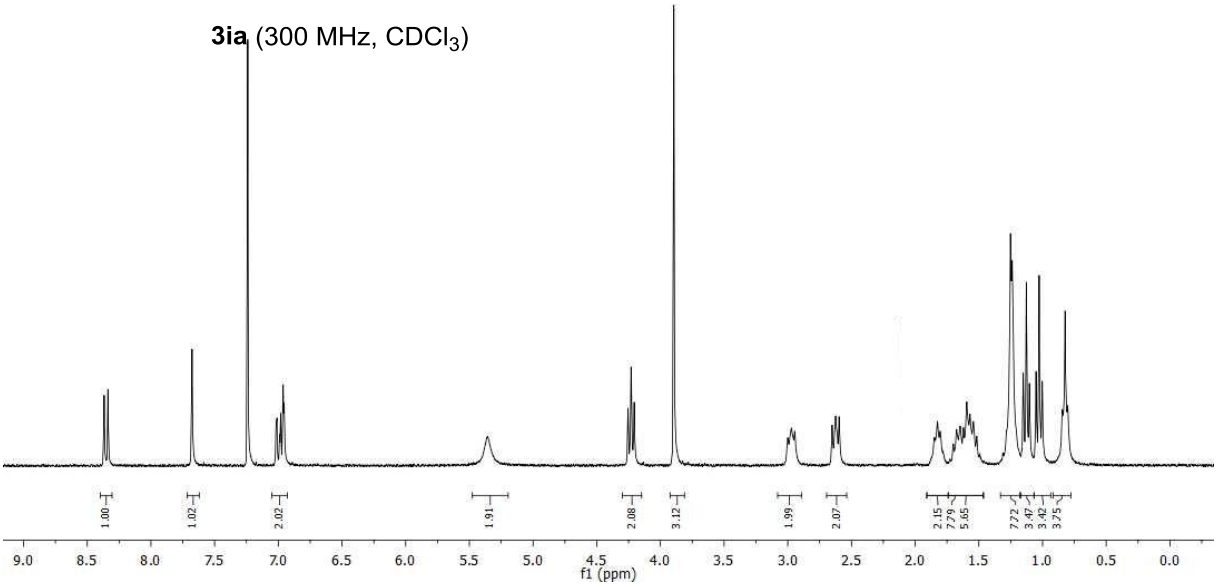
3ha (300 MHz, $CDCl_3$)

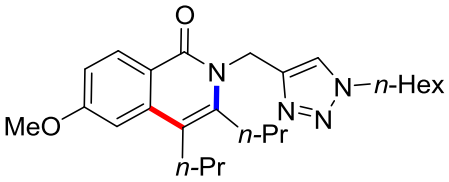


3ha (125 MHz, $CDCl_3$)

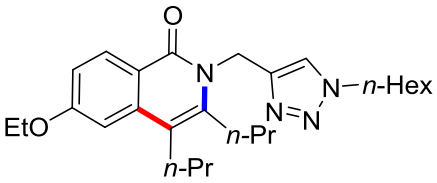


3ia (300 MHz, $CDCl_3$)

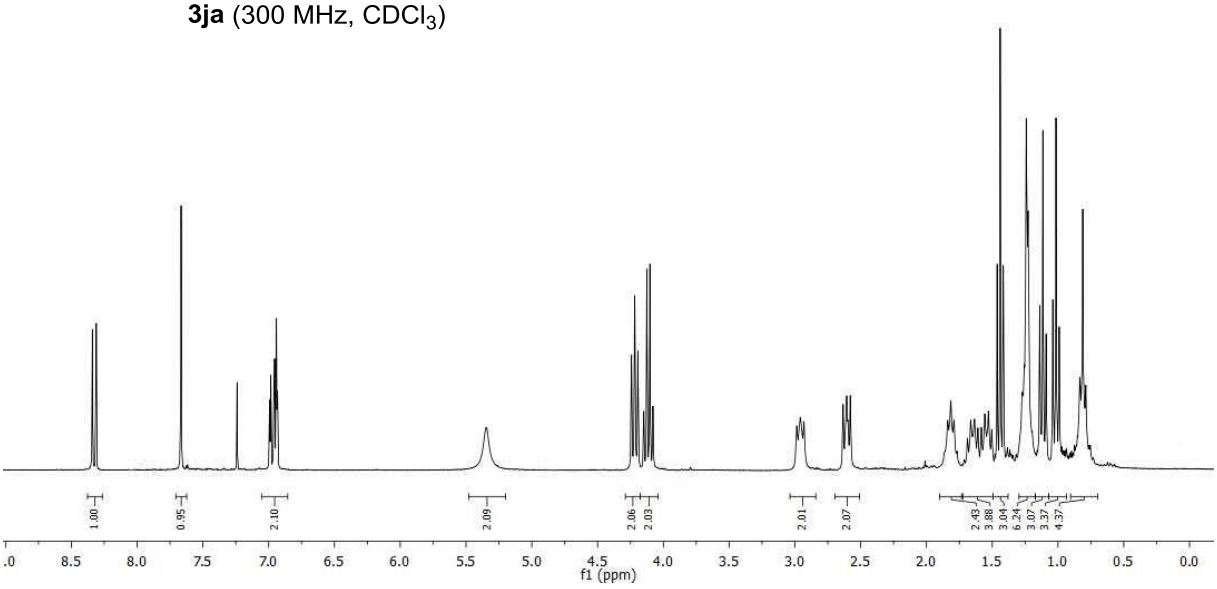


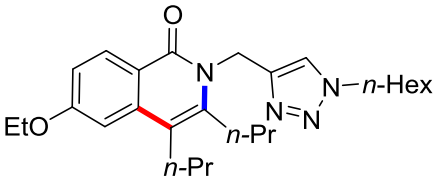


3ia (125 MHz, $CDCl_3$)

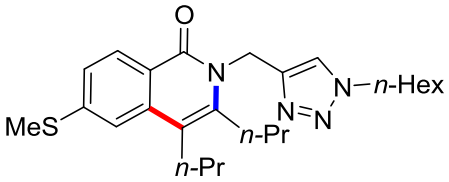


3ja (300 MHz, $CDCl_3$)

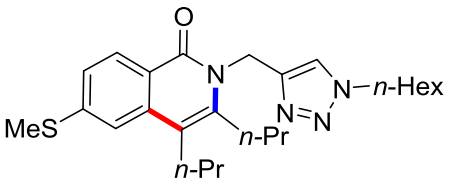




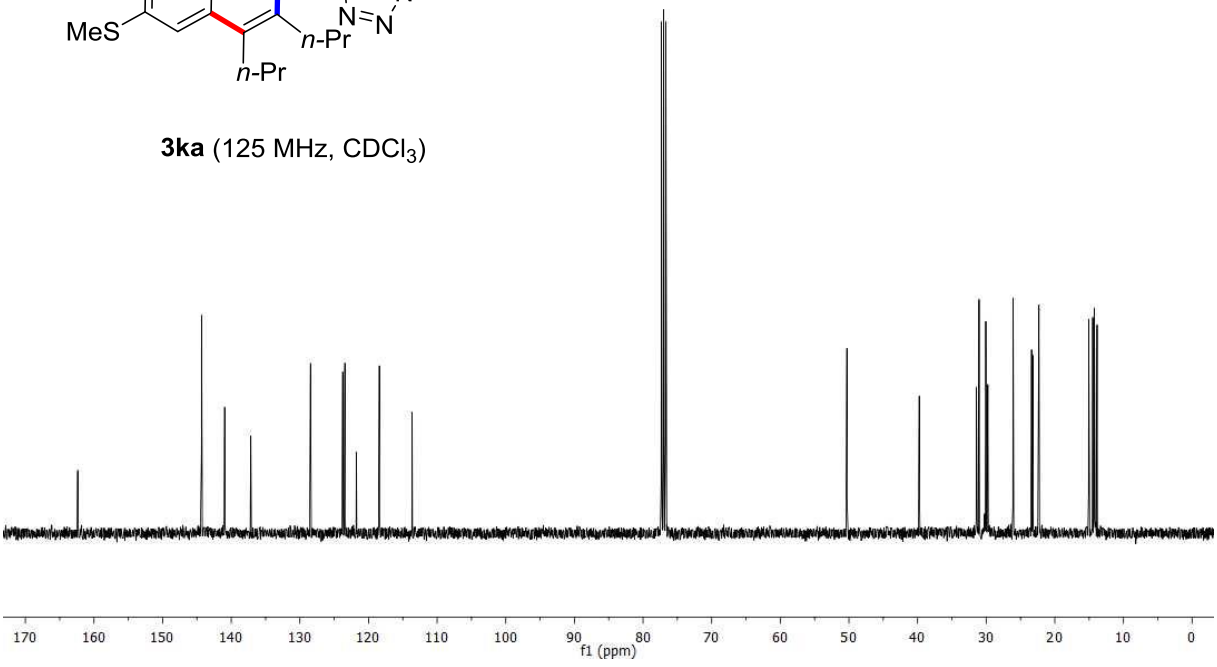
3ja (125 MHz, $CDCl_3$)

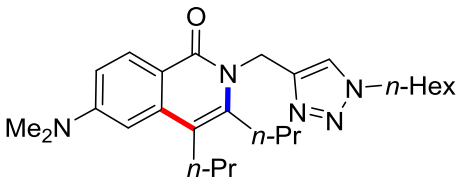


3ka (300 MHz, $CDCl_3$)

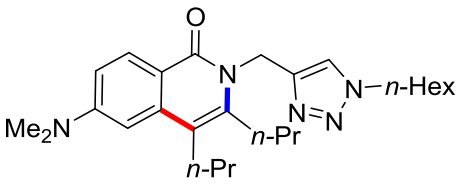


3ka (125 MHz, $CDCl_3$)

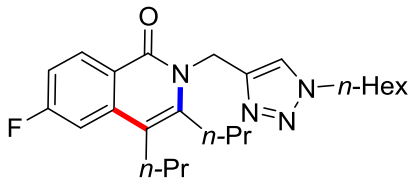




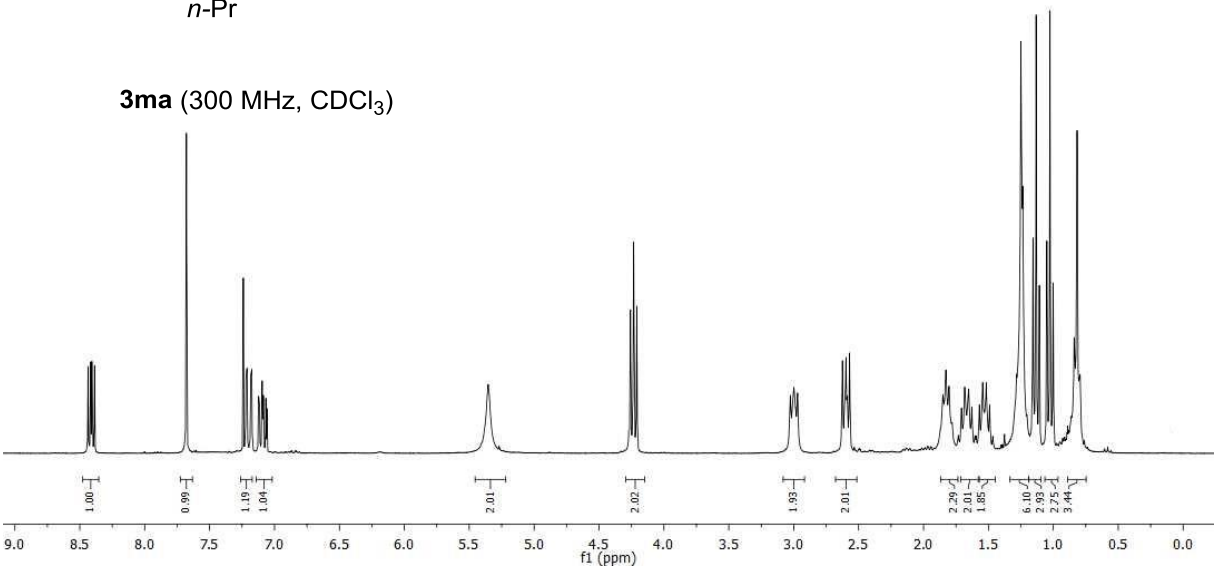
3la (300 MHz, $CDCl_3$)

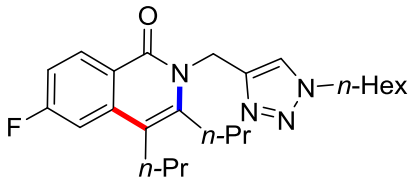


3la (125 MHz, $CDCl_3$)

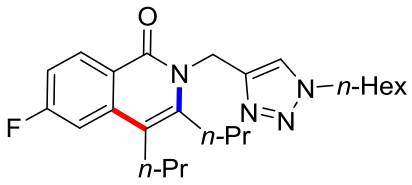


3ma (300 MHz, $CDCl_3$)

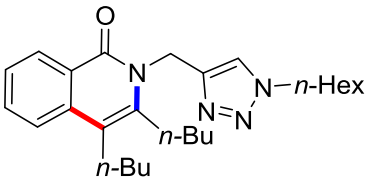




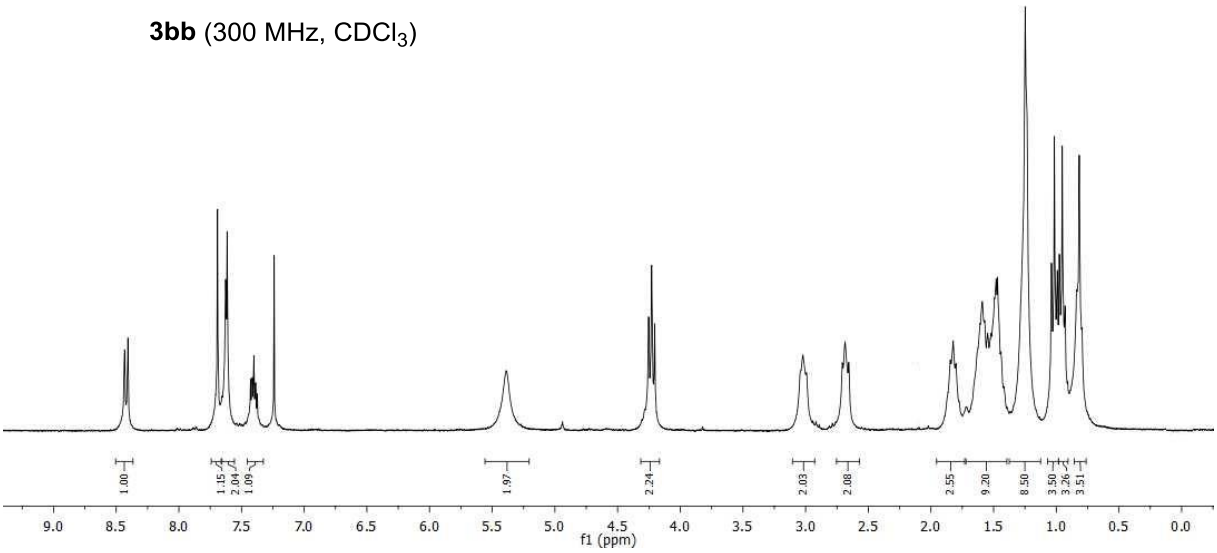
3ma (125 MHz, $CDCl_3$)

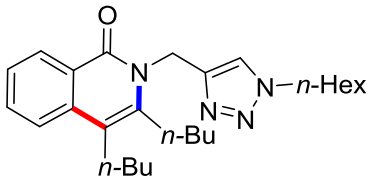


3ma (275 MHz, $CDCl_3$)

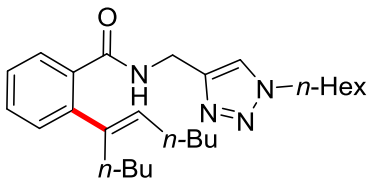


3bb (300 MHz, $CDCl_3$)

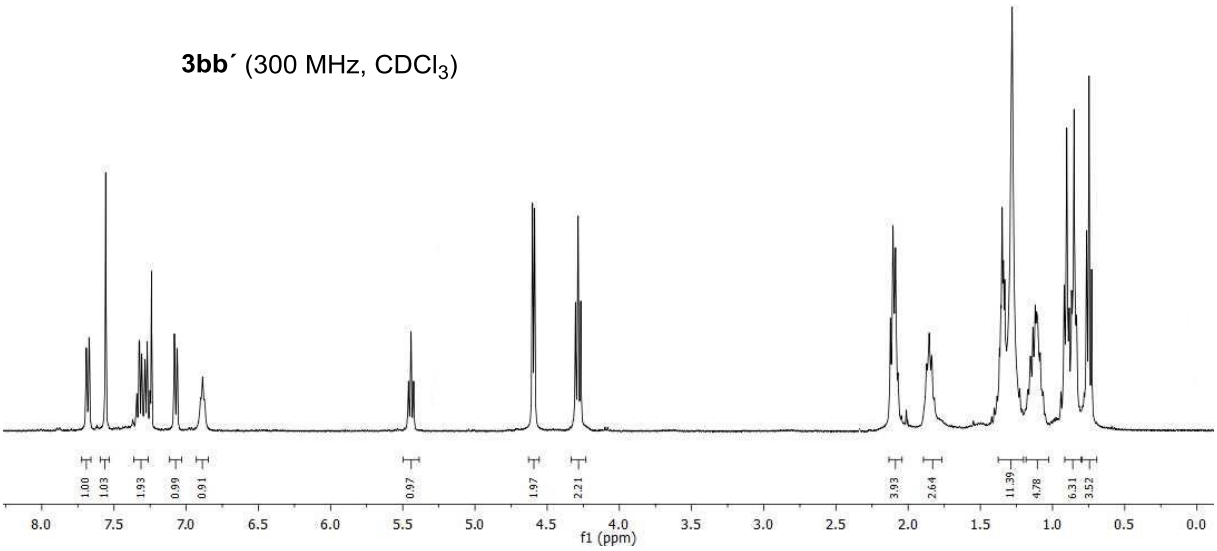


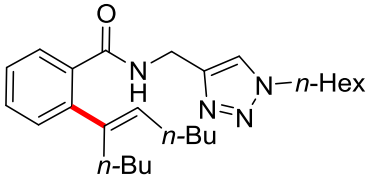


3bb (125 MHz, $CDCl_3$)

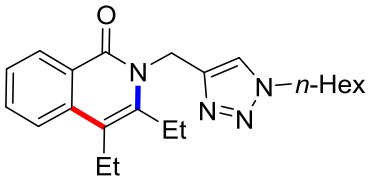


3bb´ (300 MHz, $CDCl_3$)

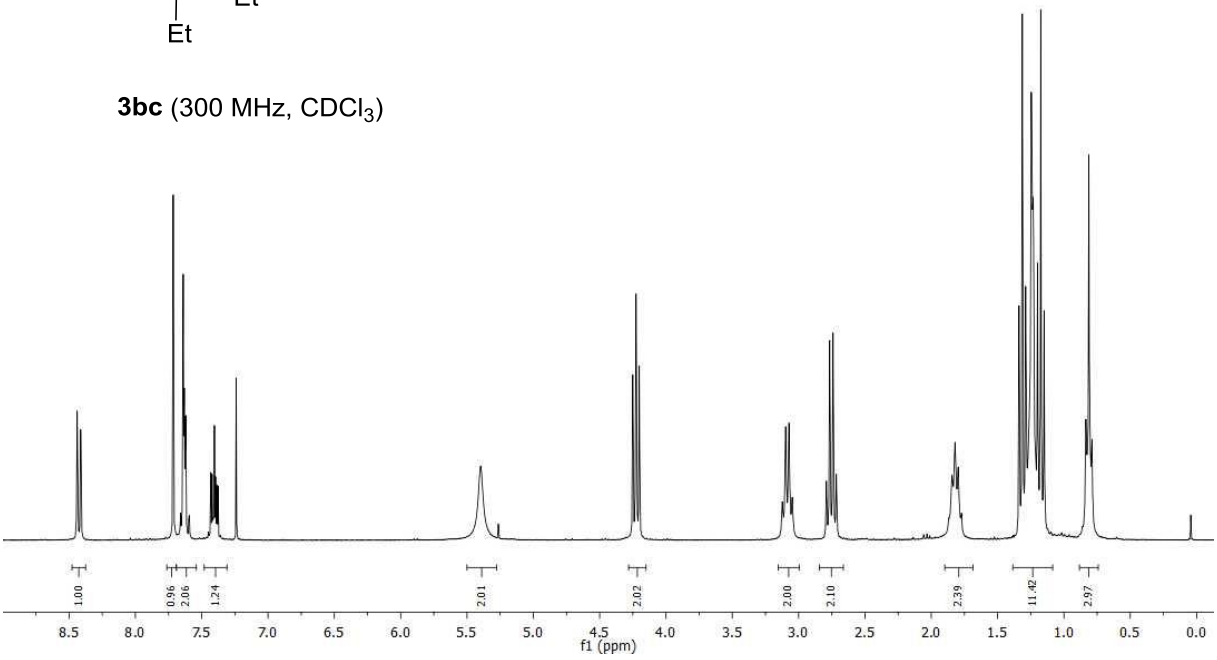


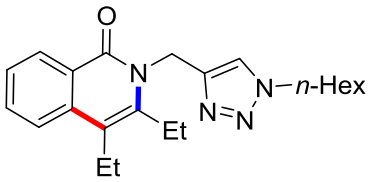


3bb´ (125 MHz, $CDCl_3$)

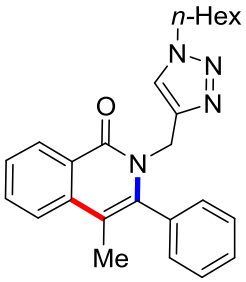


3bc (300 MHz, $CDCl_3$)

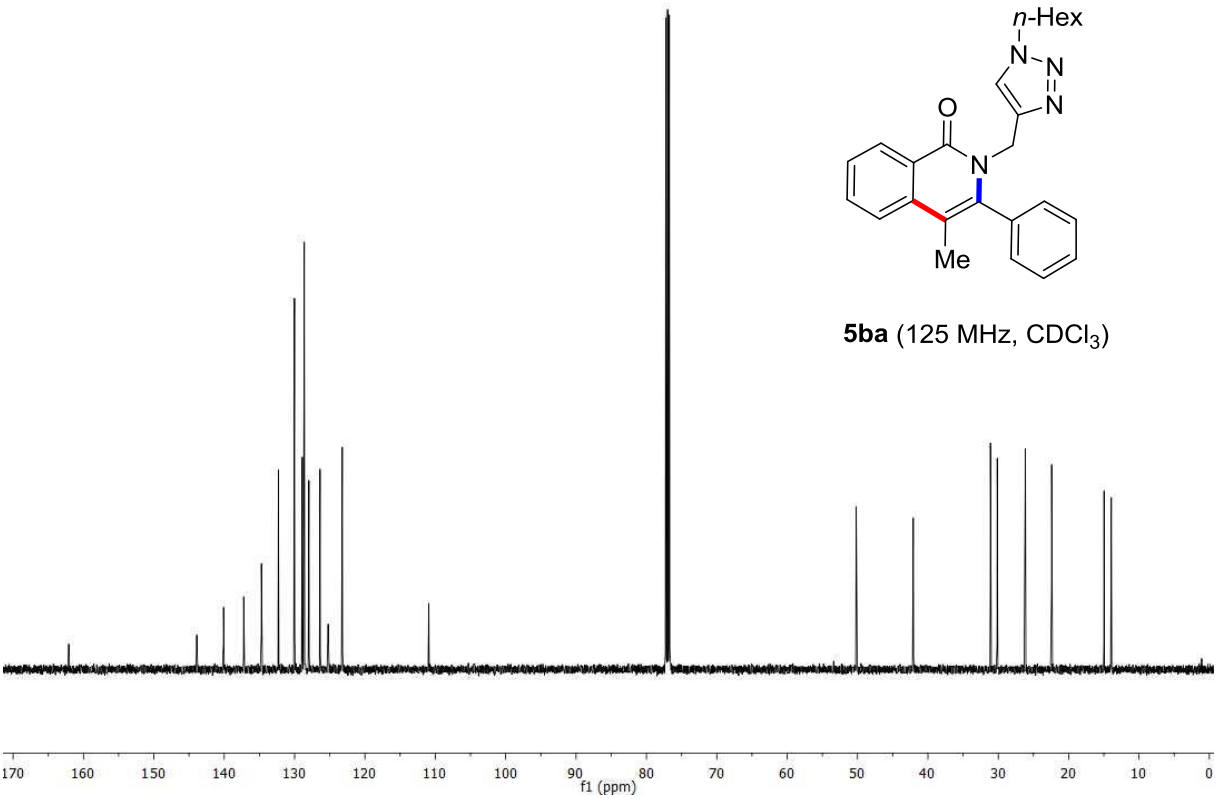




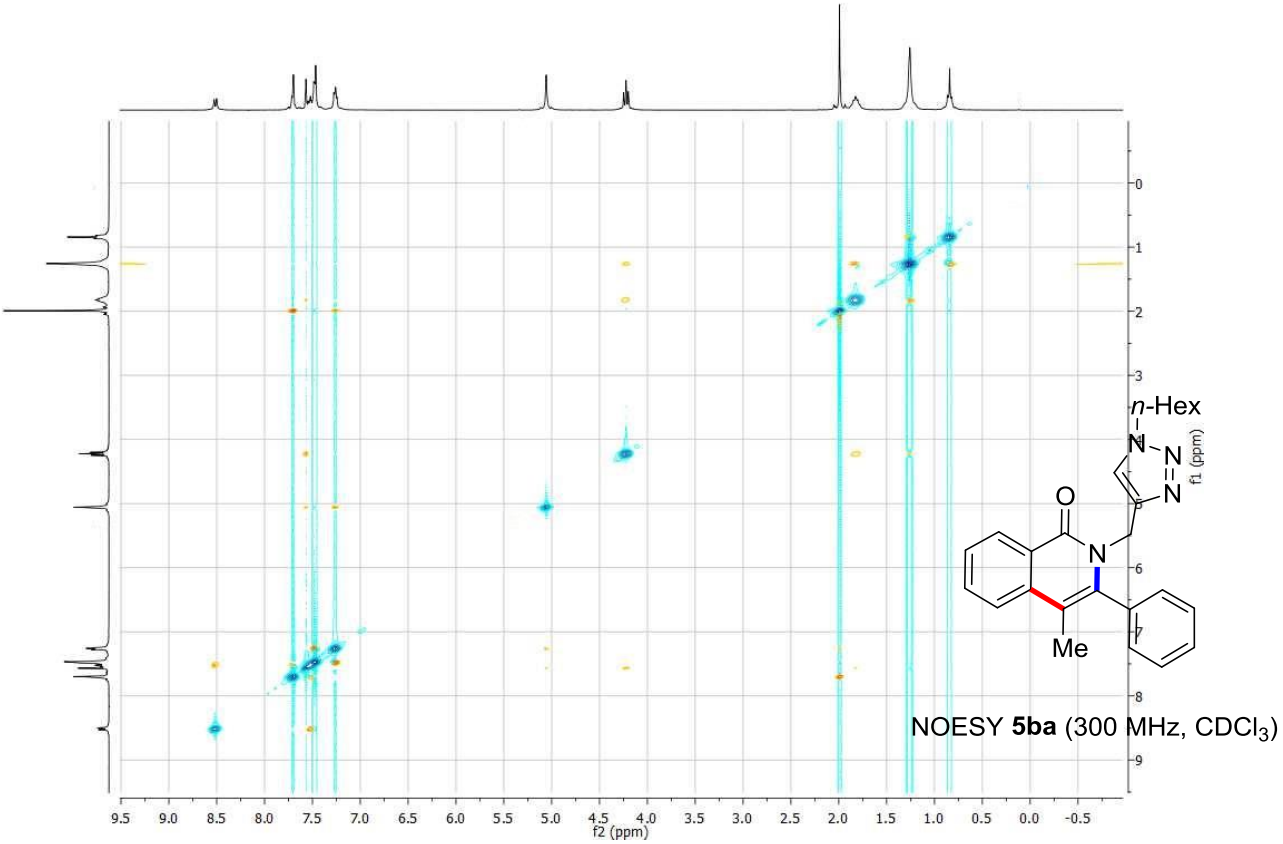
3bc (125 MHz, $CDCl_3$)



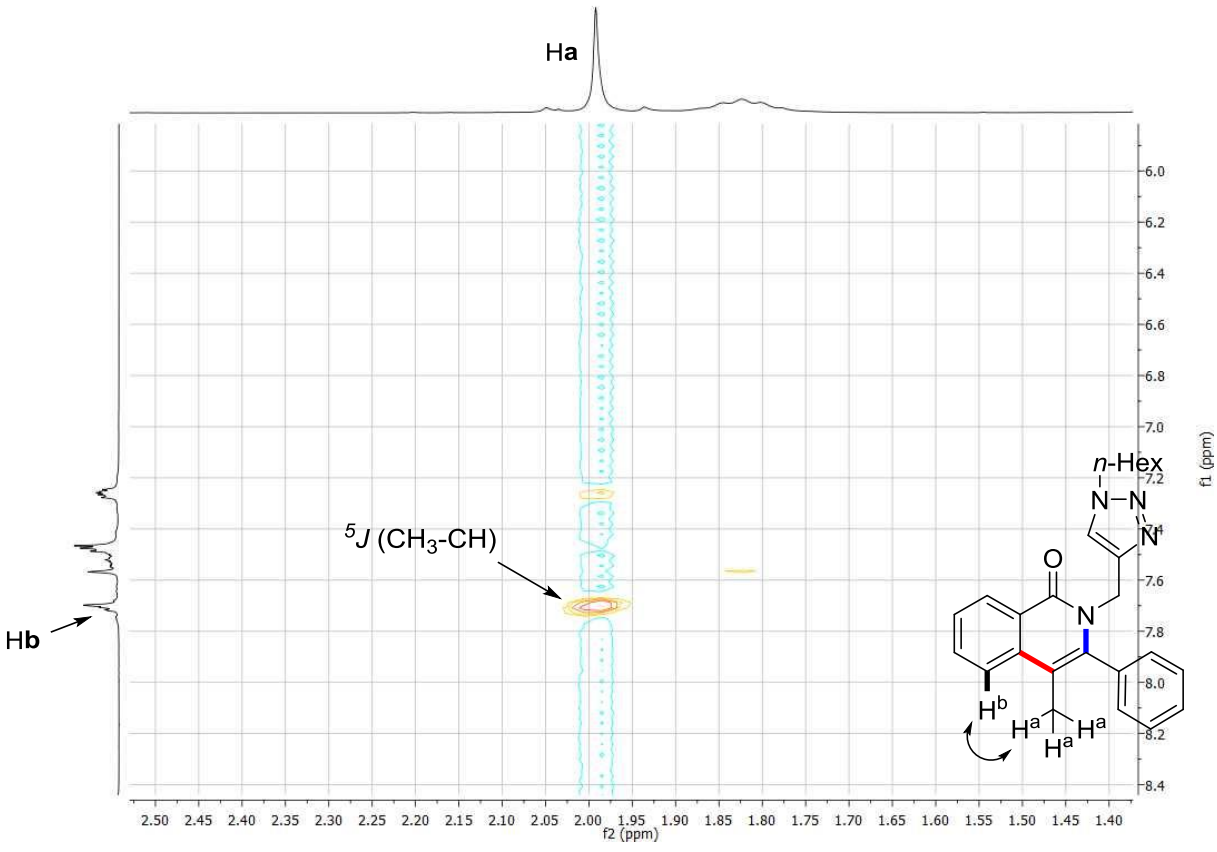
5ba (300 MHz, $CDCl_3$)

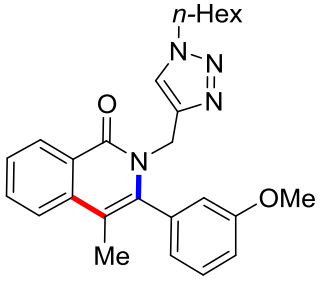


5ba (125 MHz, $CDCl_3$)

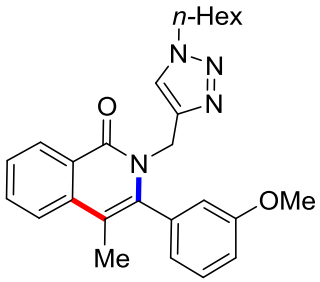


NOESY **5ba** (300 MHz, $CDCl_3$)

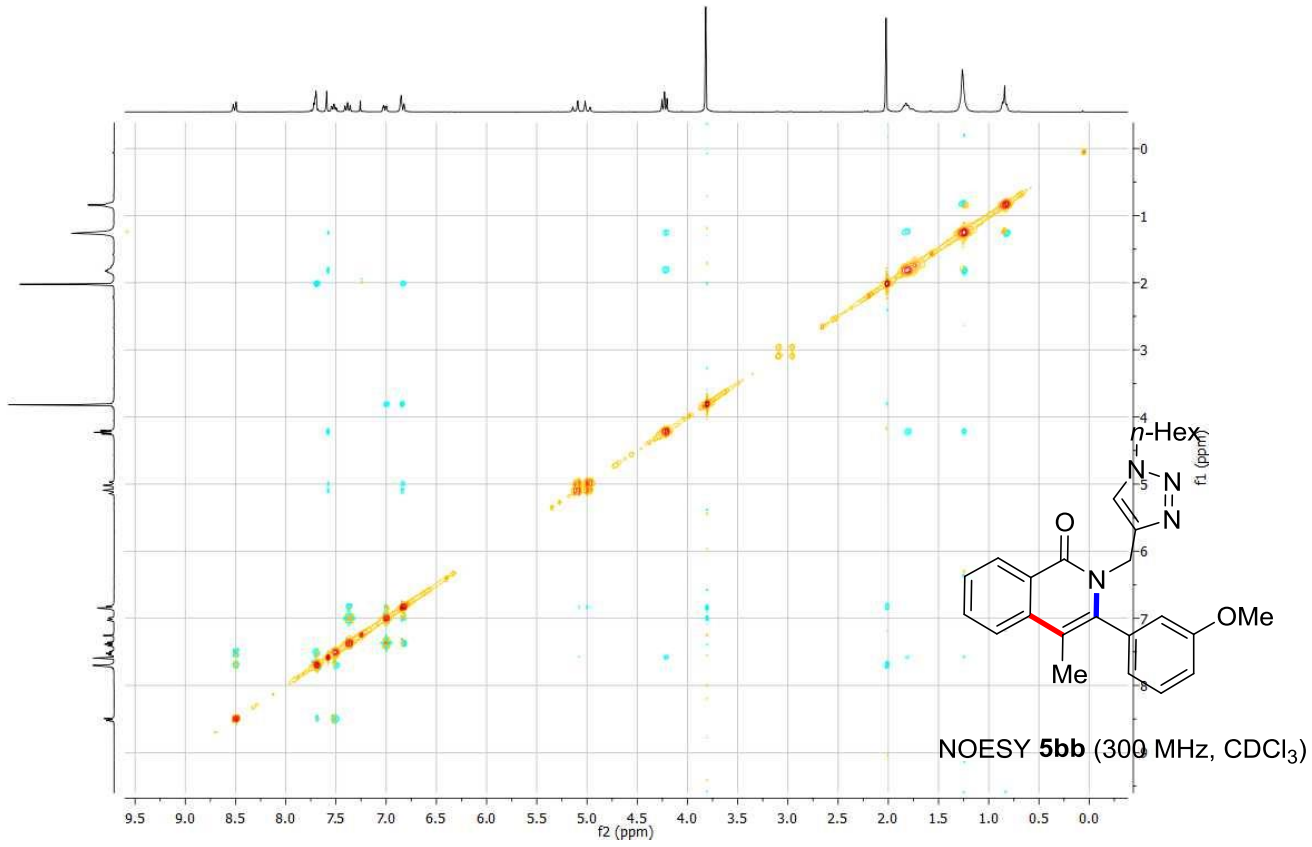




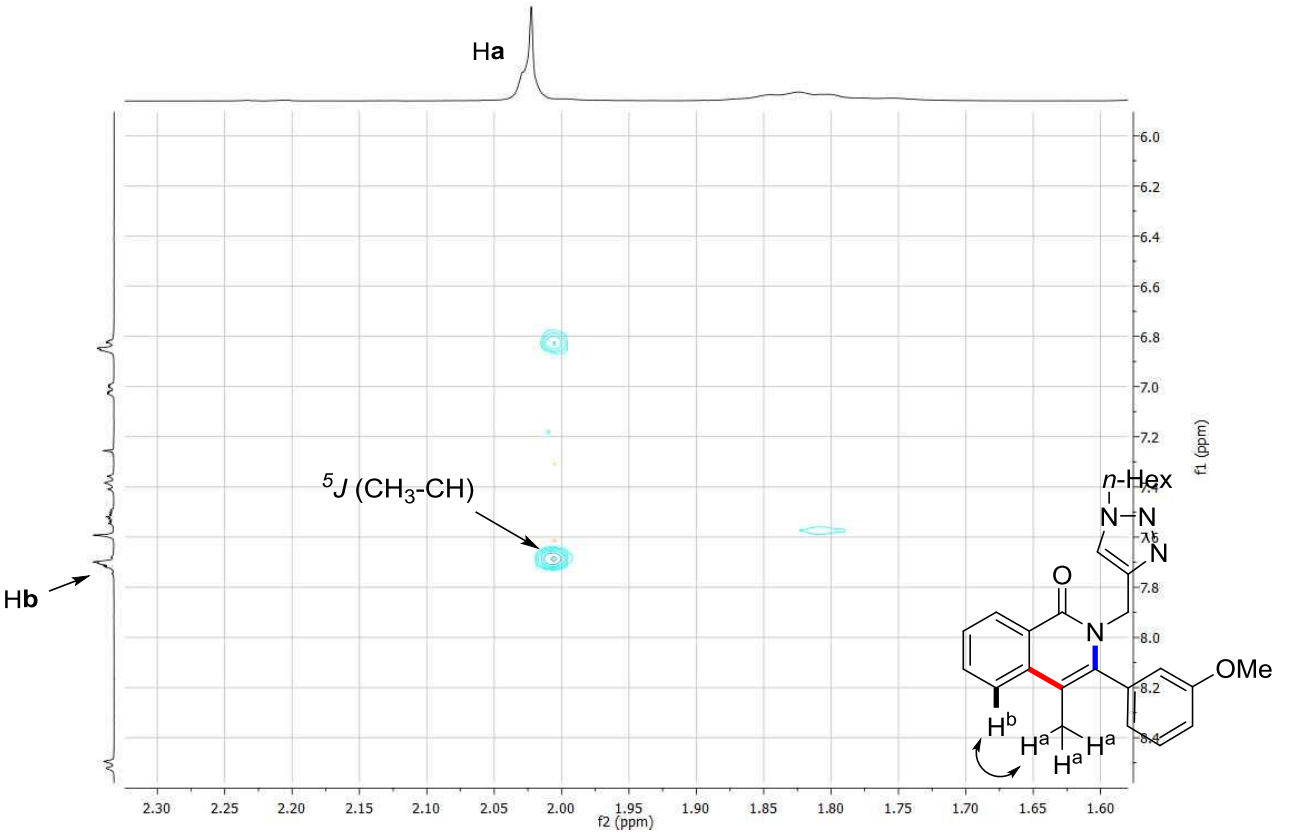
5bb (300 MHz, $CDCl_3$)

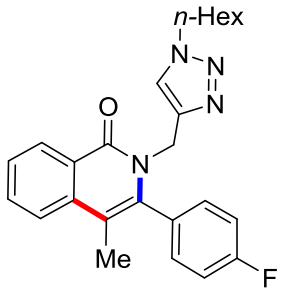


5bb (125 MHz, $CDCl_3$)

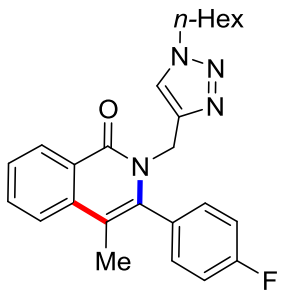


NOESY **5bb** (300 MHz, $CDCl_3$)

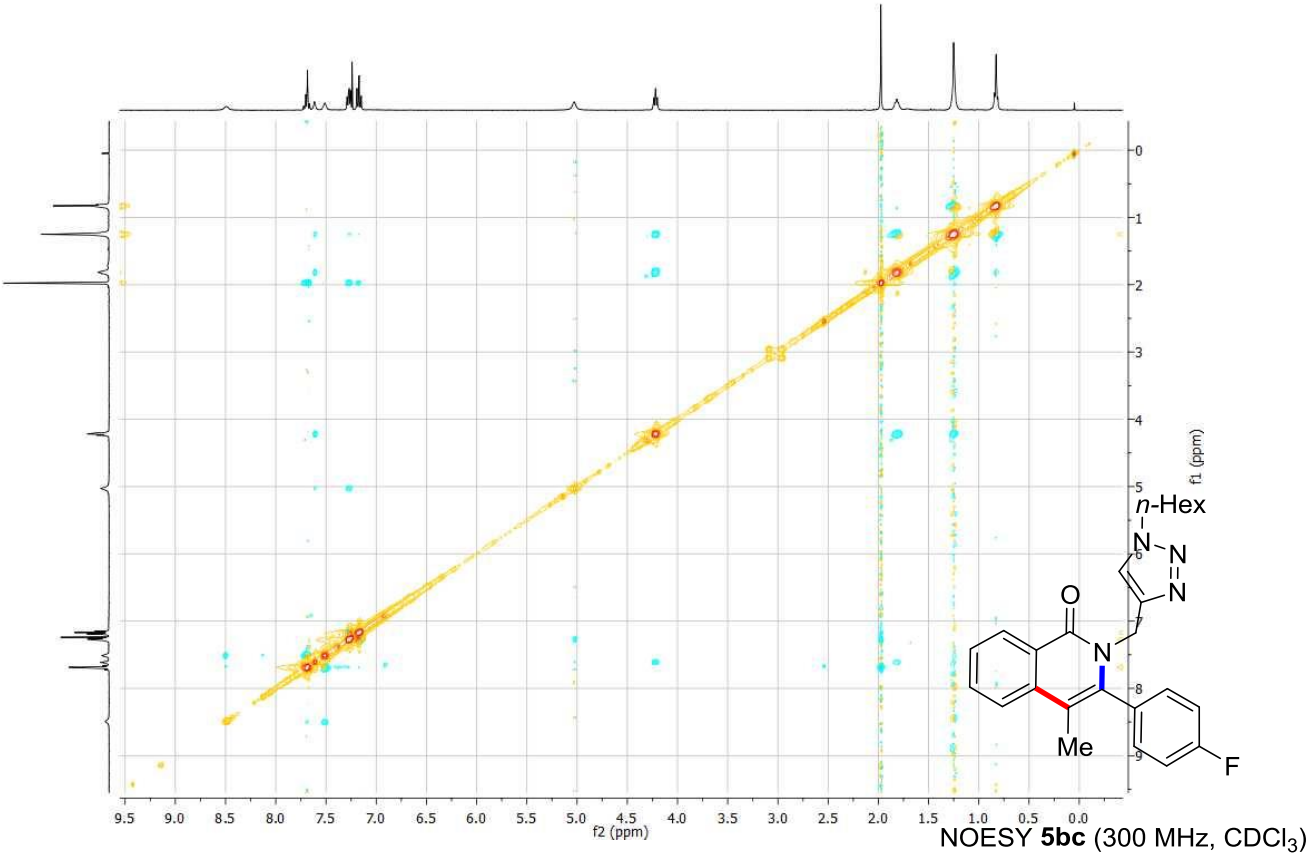




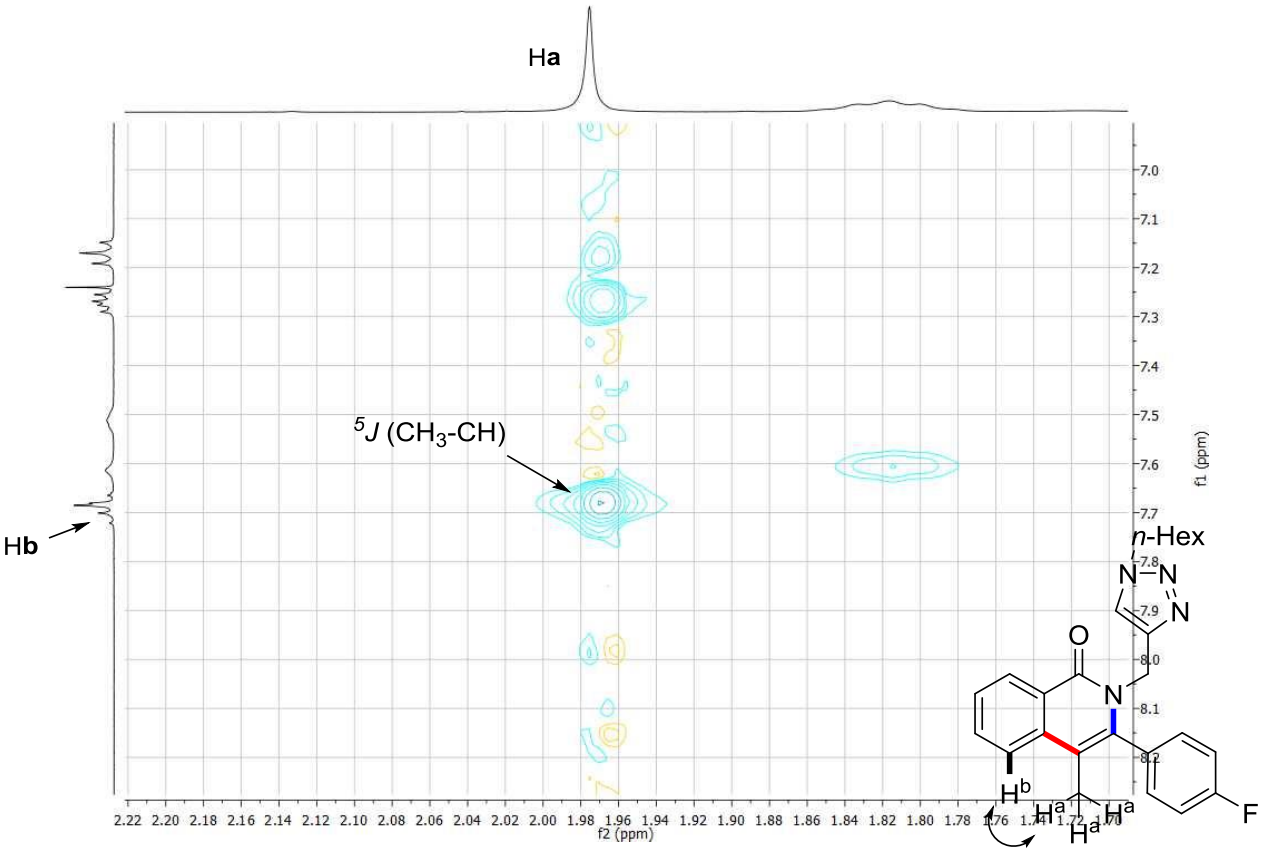
5bc (300 MHz, $CDCl_3$)

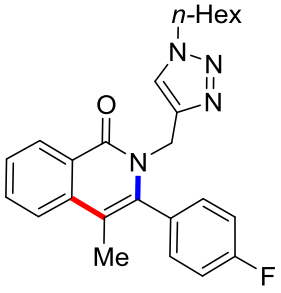


5bc (125 MHz, $CDCl_3$)

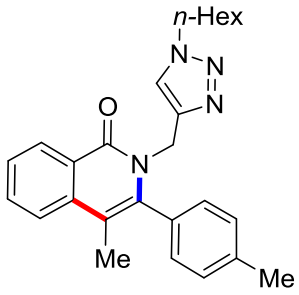


NOESY **5bc** (300 MHz, $CDCl_3$)

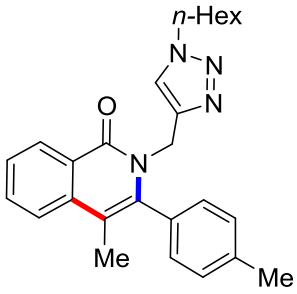




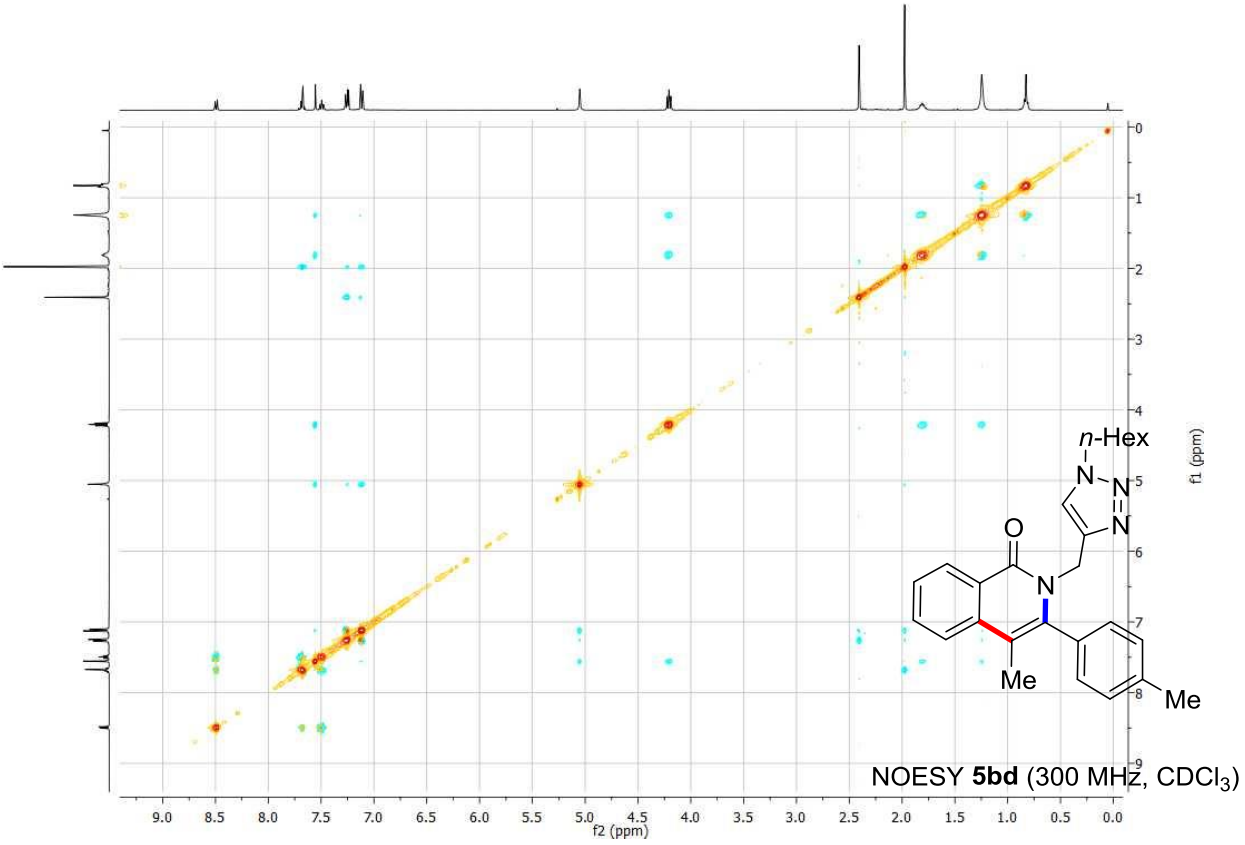
5bc (275 MHz, $CDCl_3$)



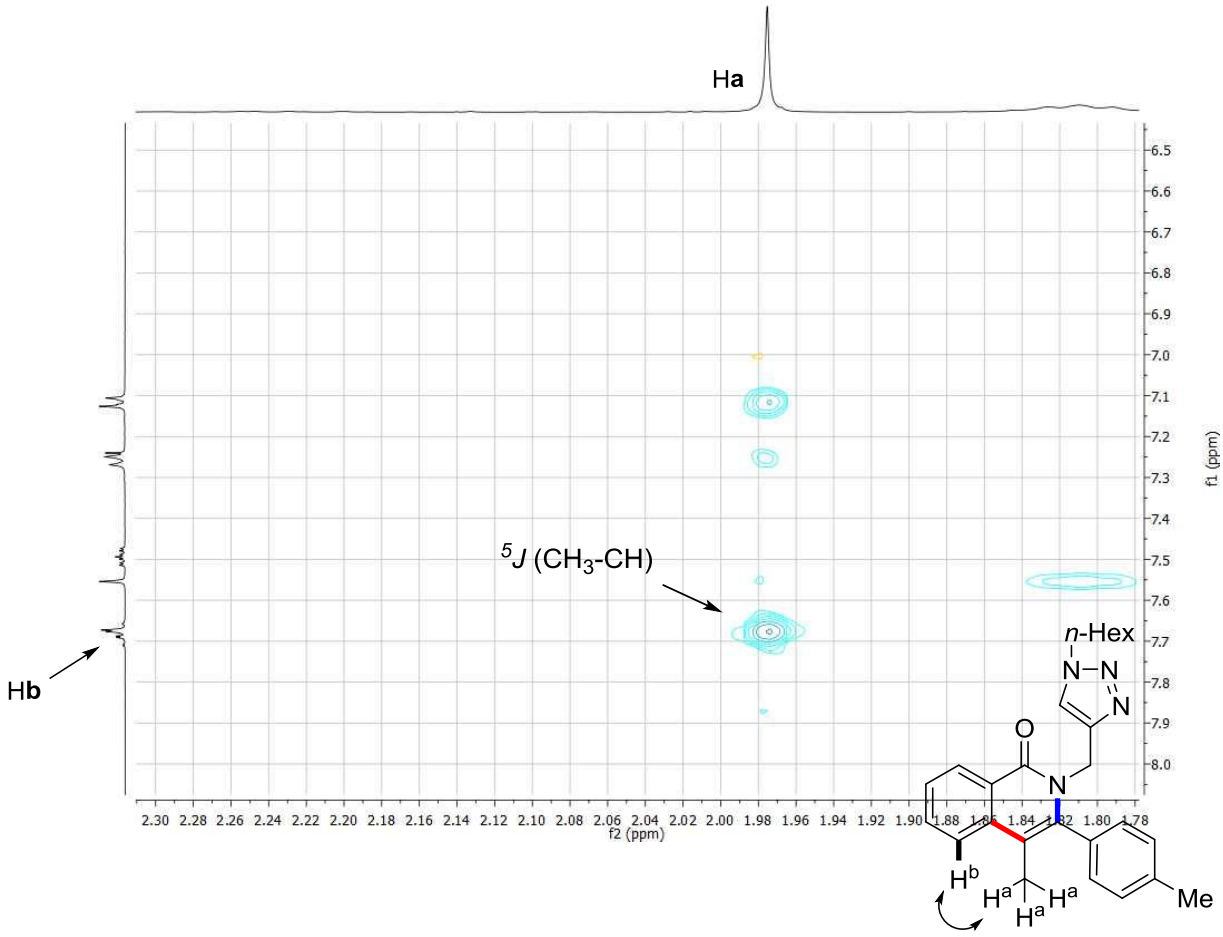
5bd (300 MHz, $CDCl_3$)

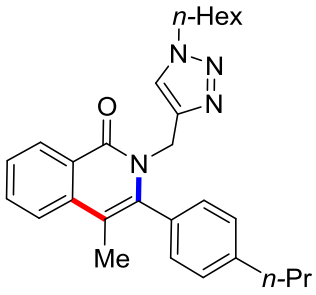


5bd (125 MHz, $CDCl_3$)

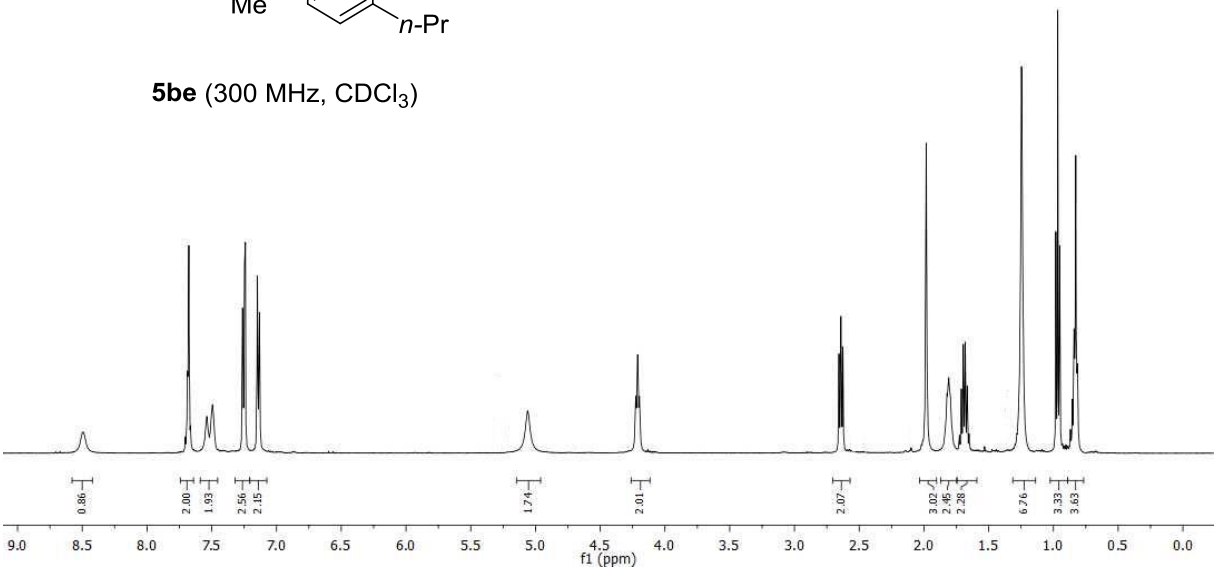


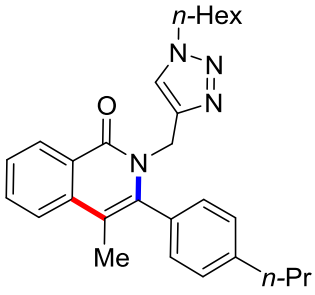
NOESY **5bd** (300 MHz, $CDCl_3$)



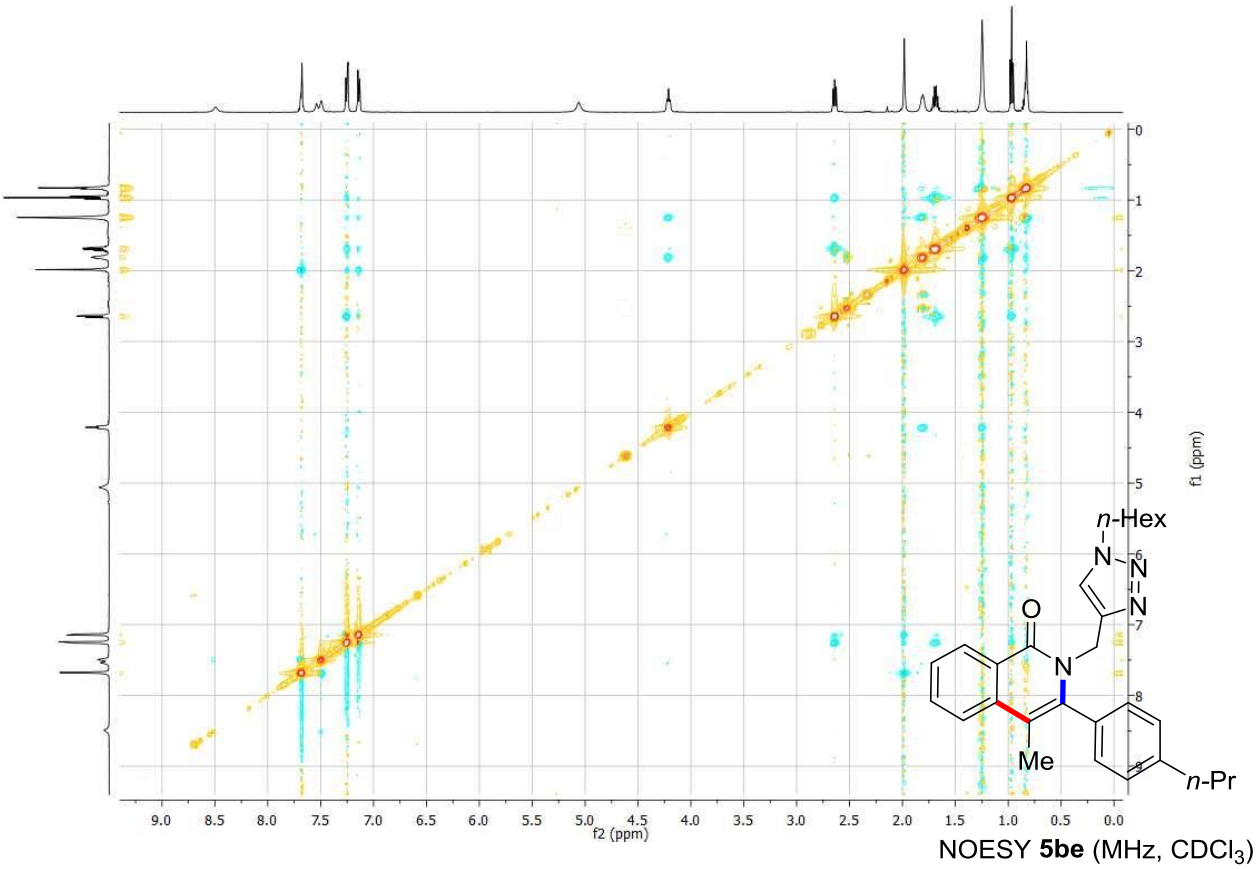


5be (300 MHz, $CDCl_3$)

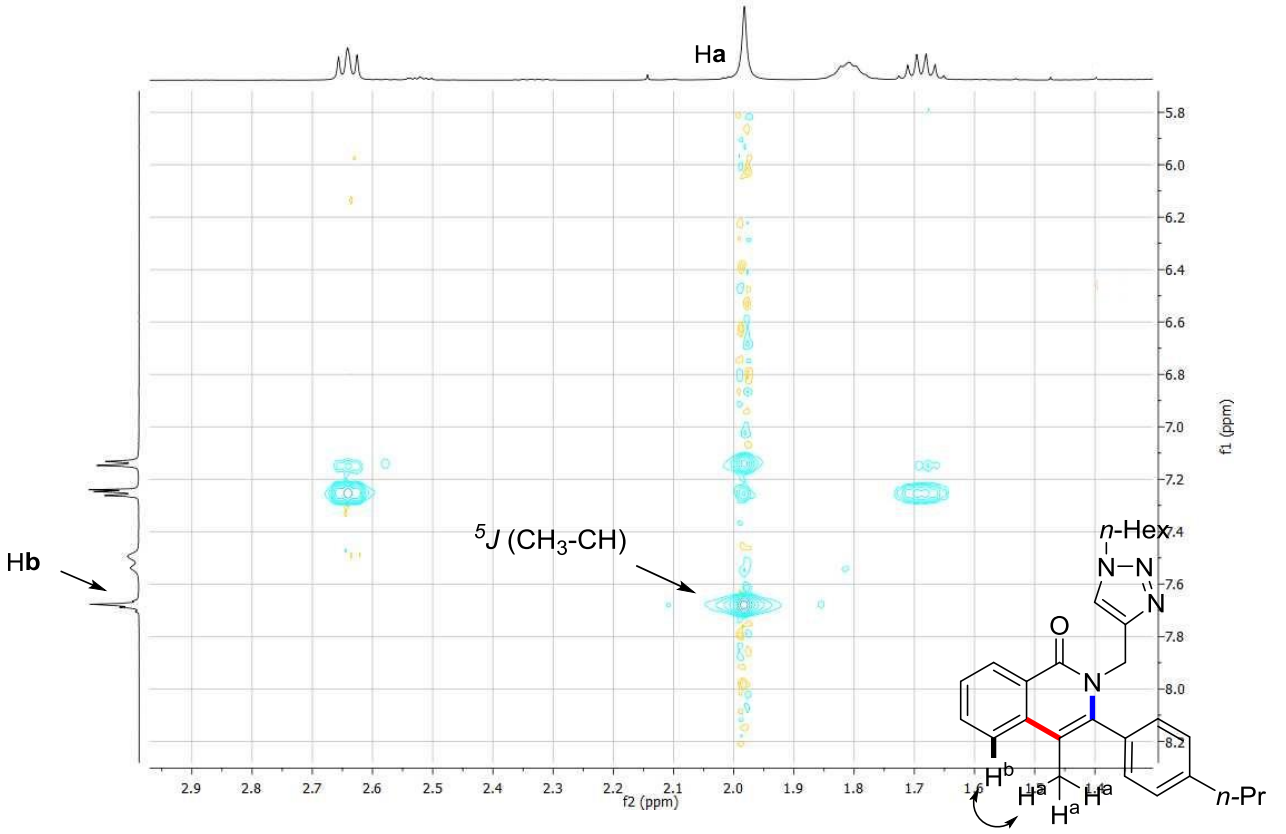


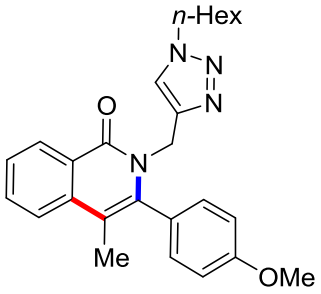


5be (125 MHz, $CDCl_3$)

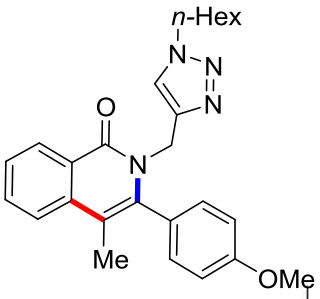


NOESY **5be** (MHz, $CDCl_3$)

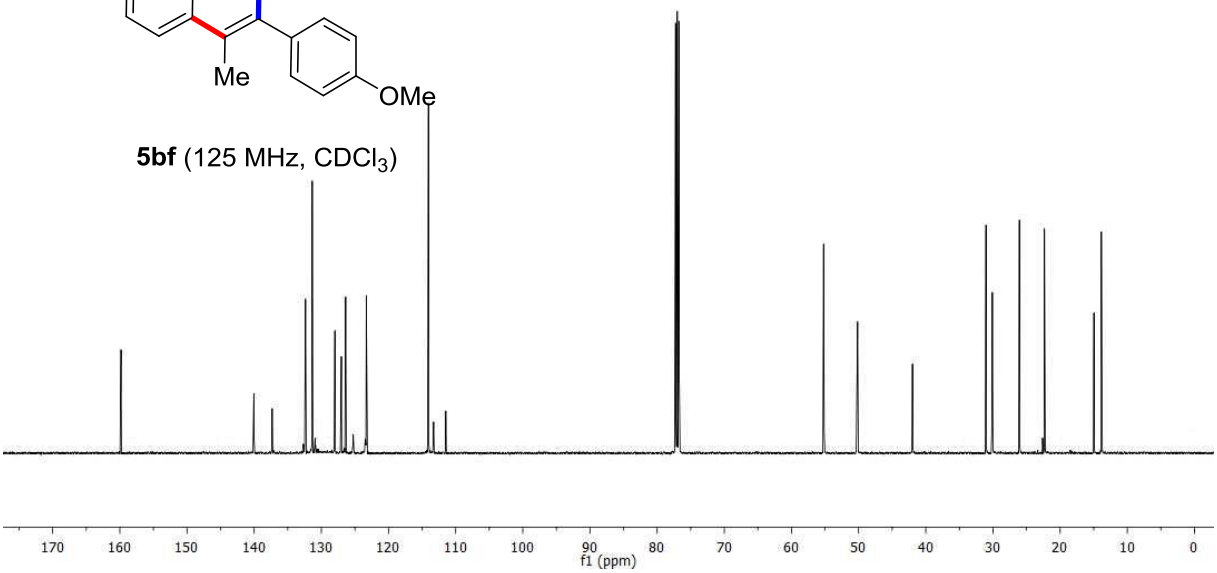


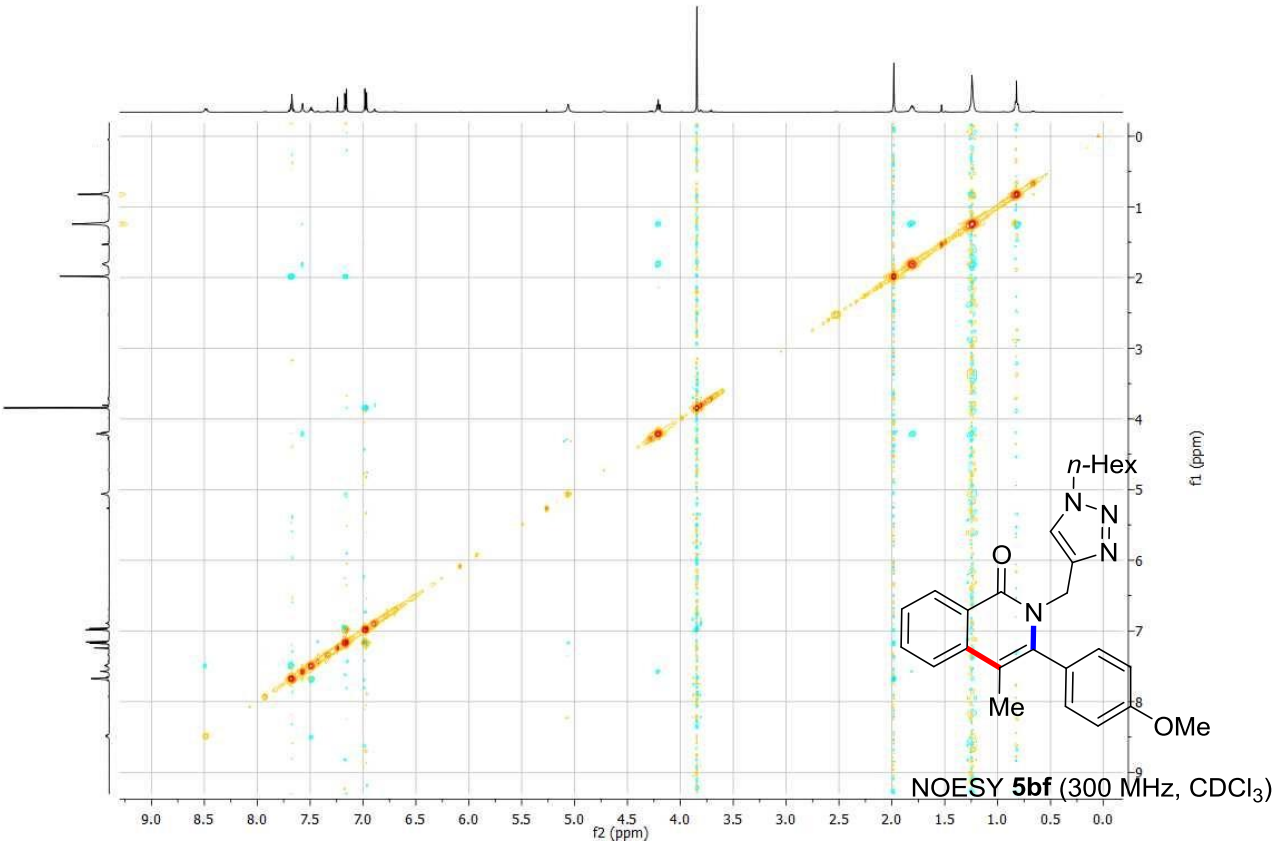


5bf (300 MHz, $CDCl_3$)

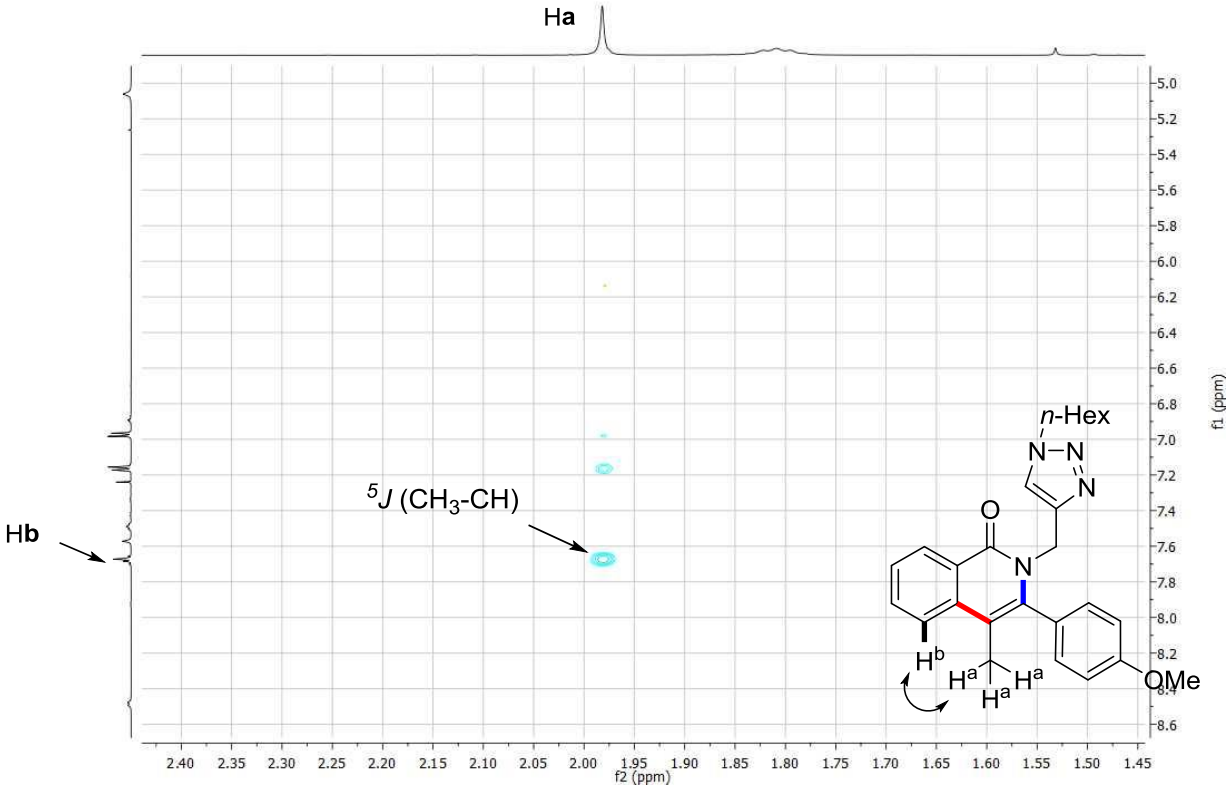


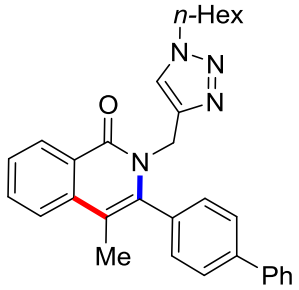
5bf (125 MHz, $CDCl_3$)



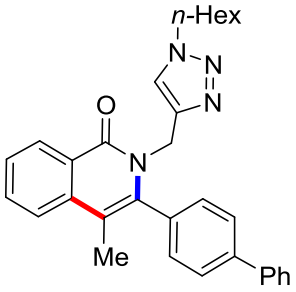


NOESY **5bf** (300 MHz, $CDCl_3$)

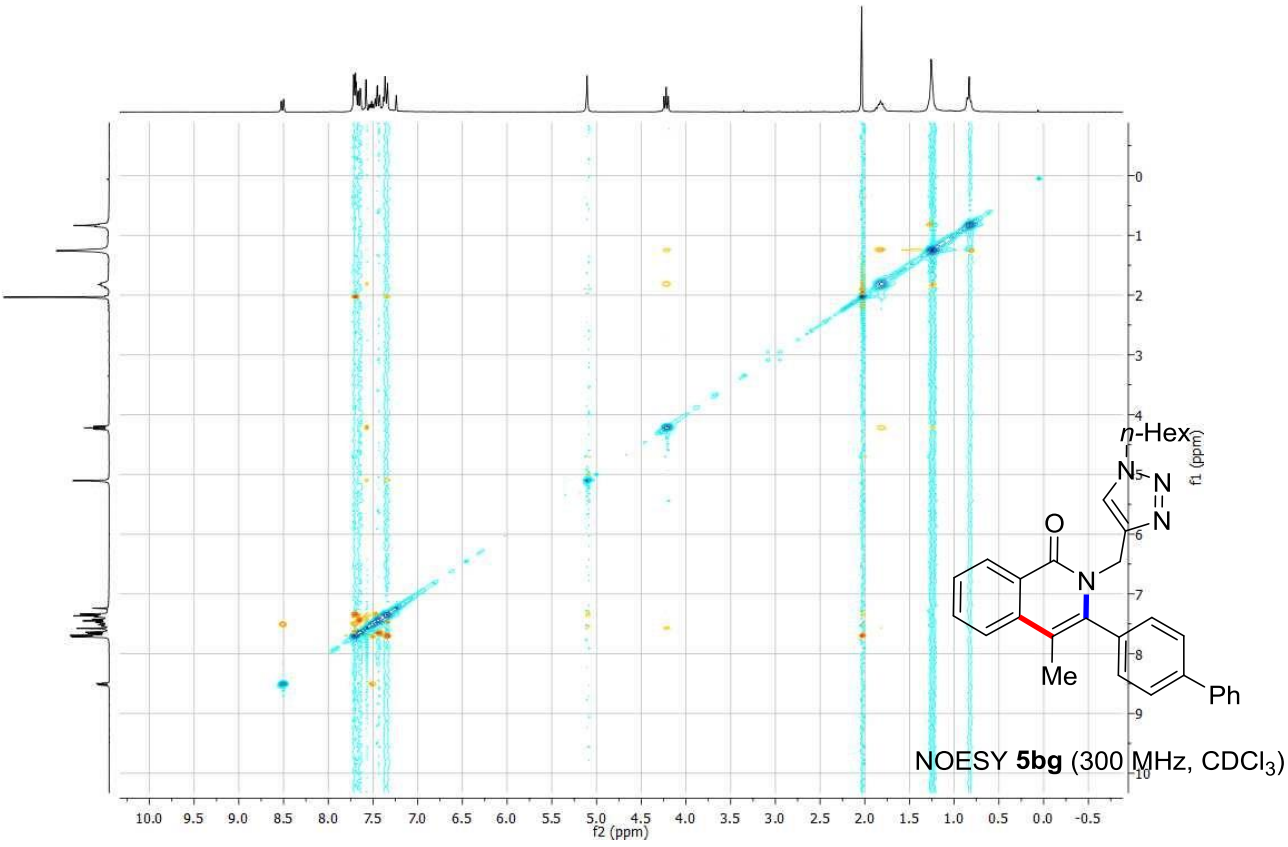




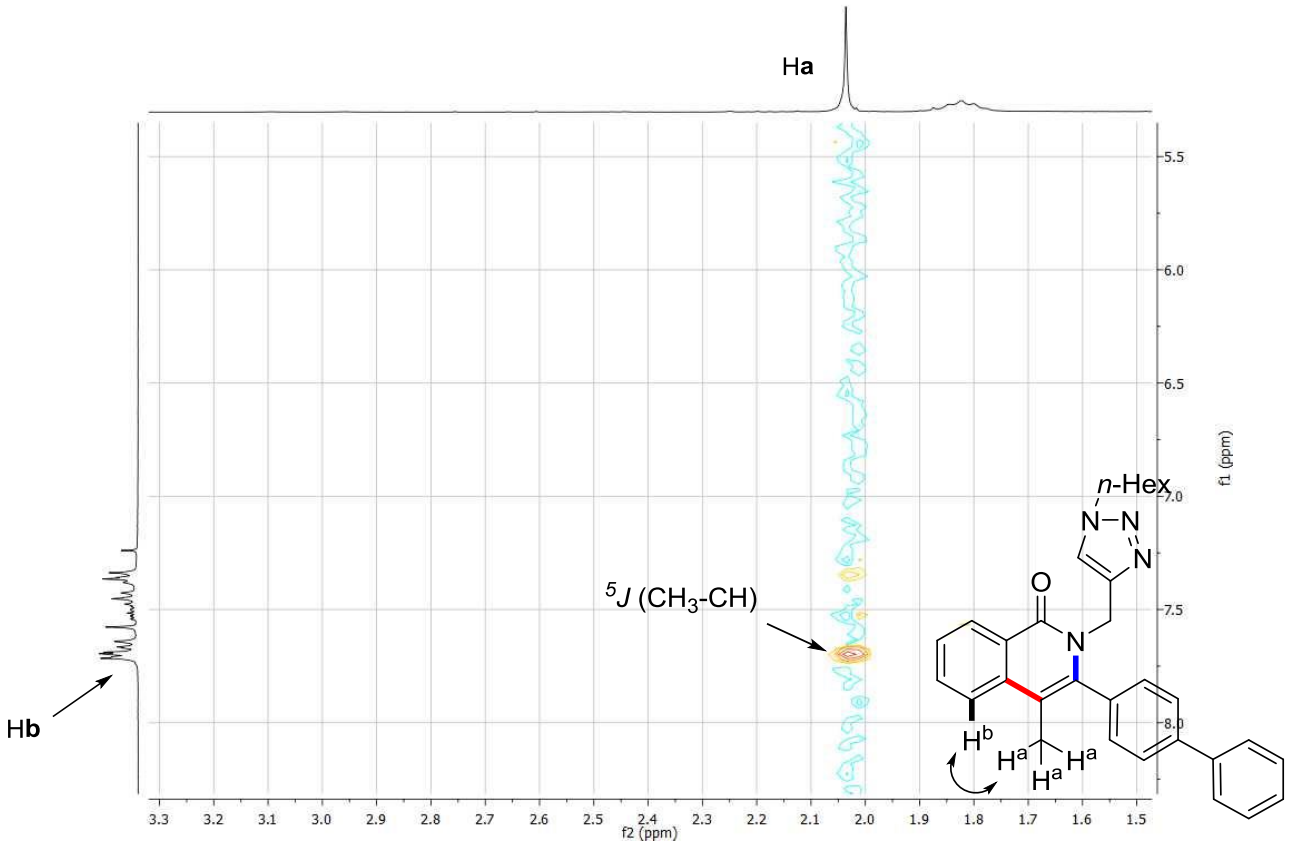
5bg (300 MHz, $CDCl_3$)

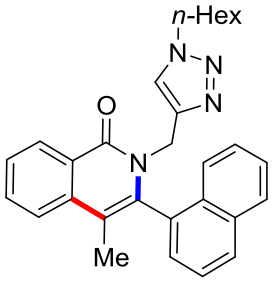


5bg (125 MHz, $CDCl_3$)

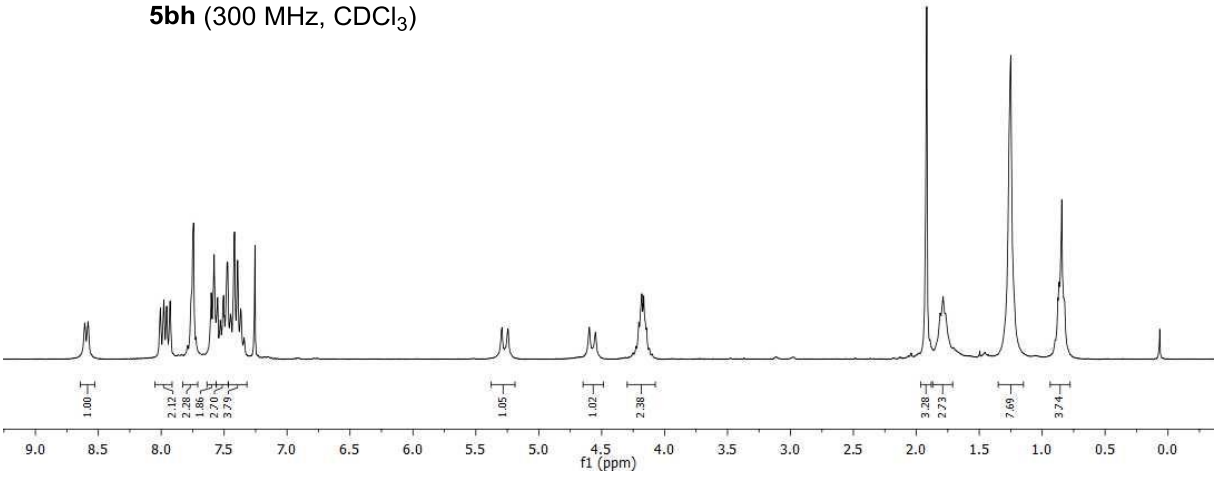


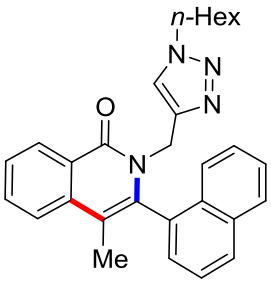
NOESY **5bg** (300 MHz, $CDCl_3$)



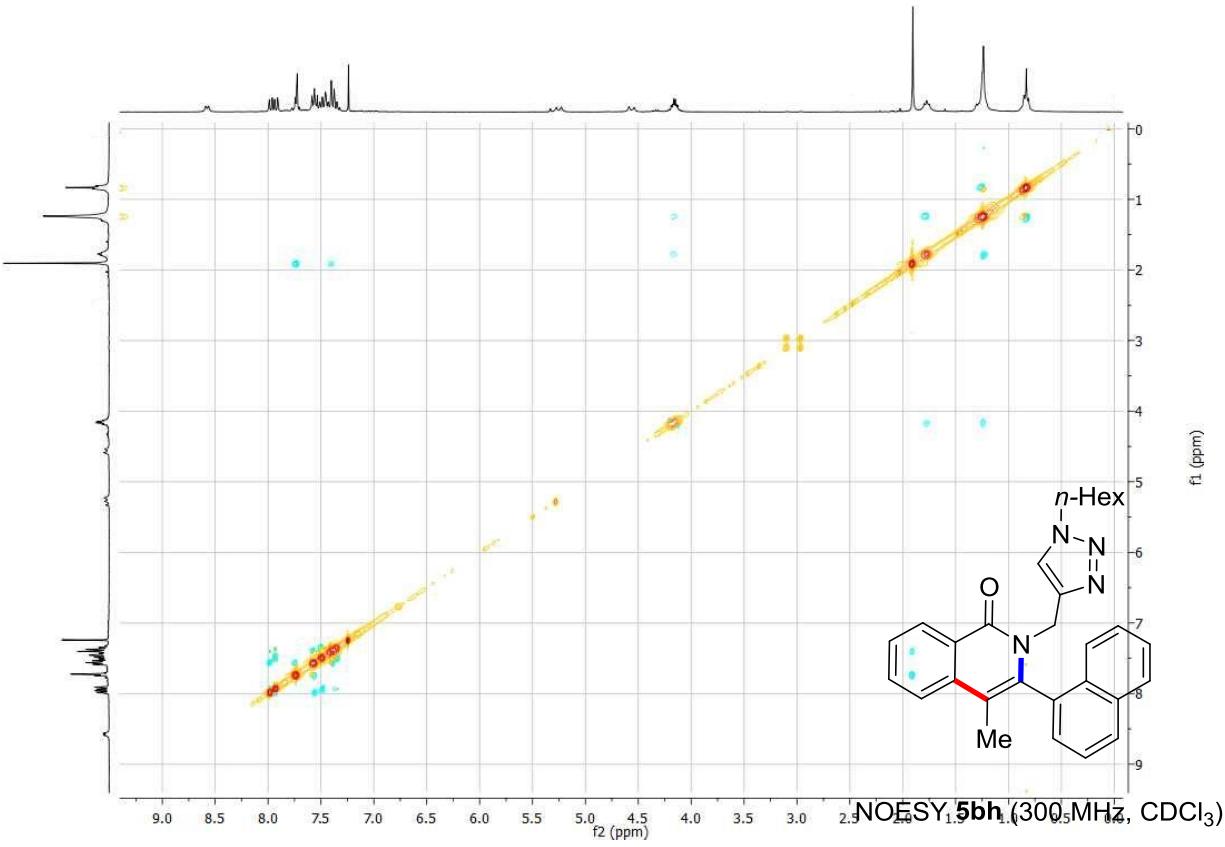


5bh (300 MHz, $CDCl_3$)

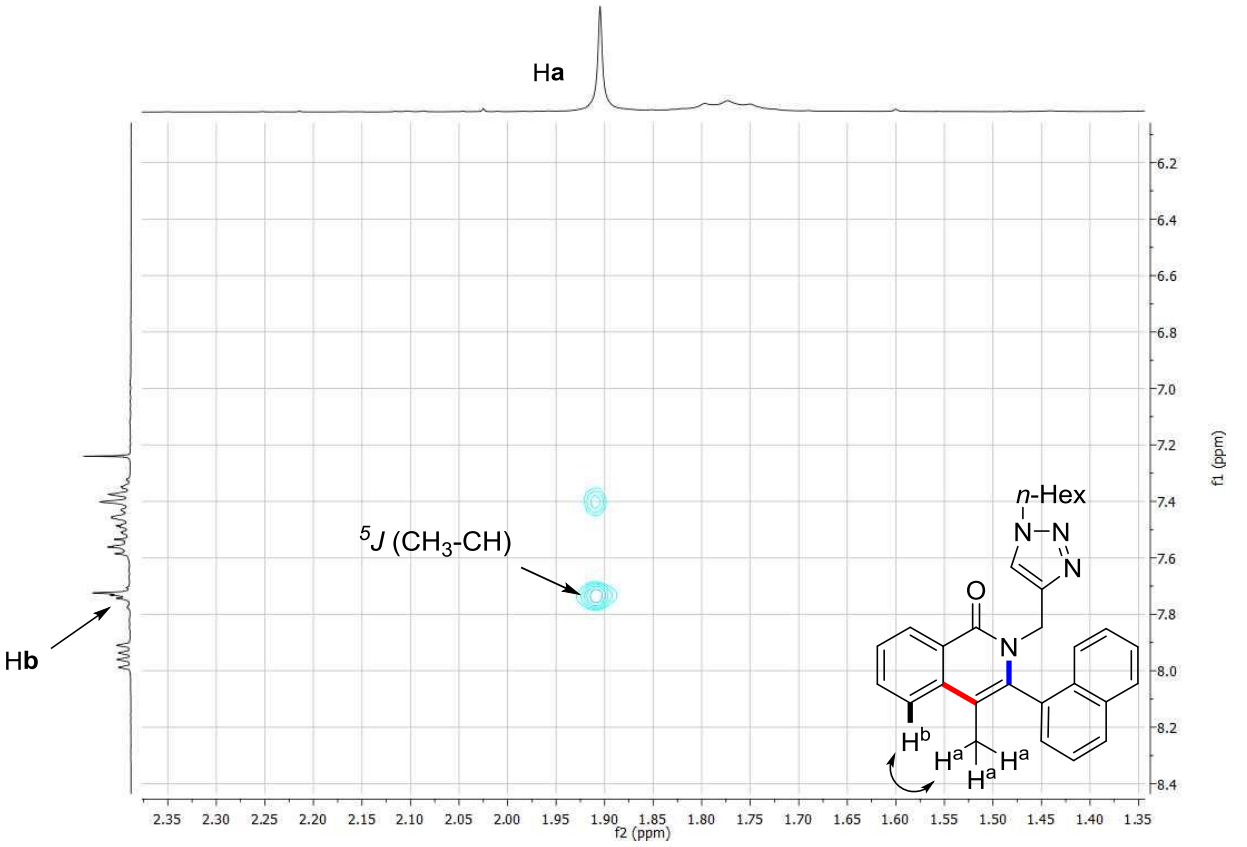


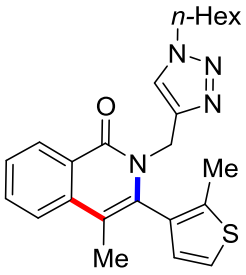


5bh (125 MHz, $CDCl_3$)

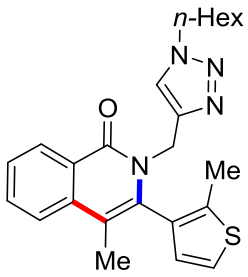


NOESY **5bh** (300 MHz, $CDCl_3$)

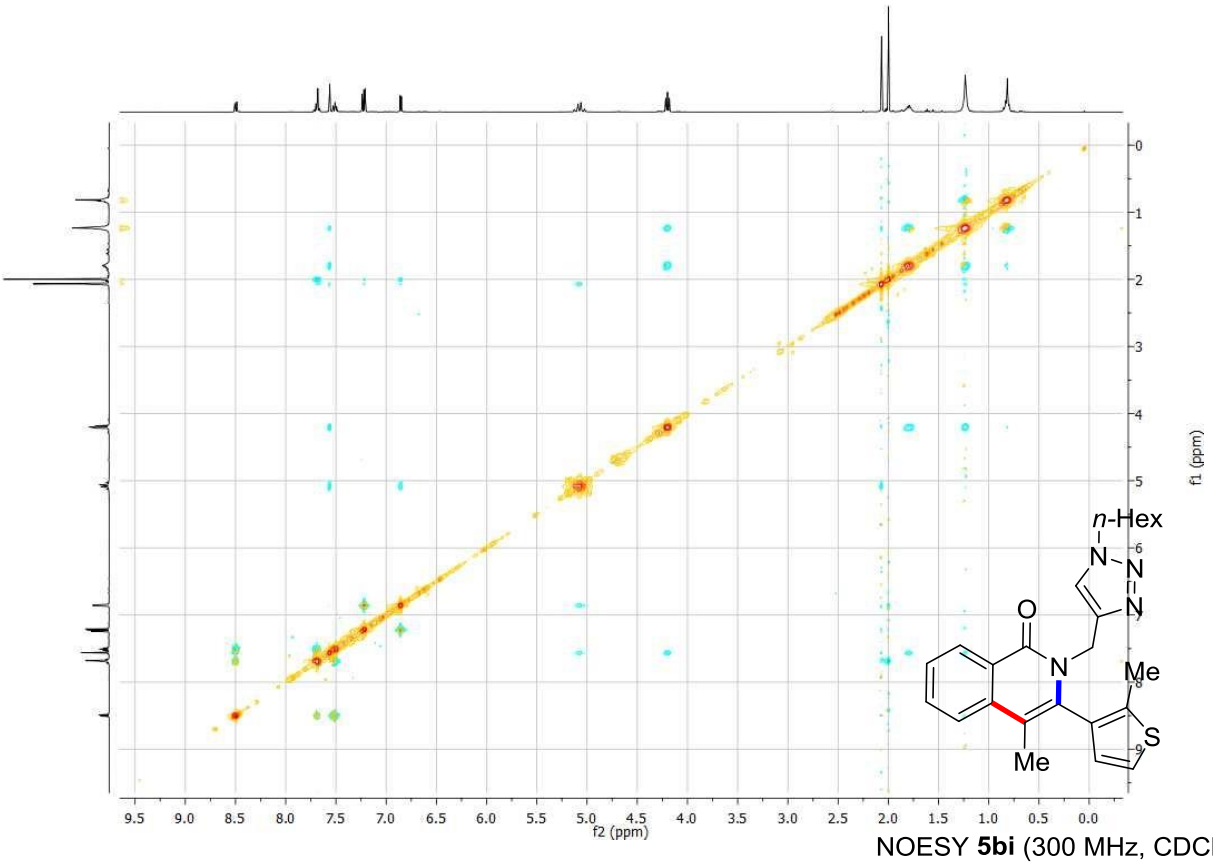




5bi (300 MHz, $CDCl_3$)



5bi (125 MHz, $CDCl_3$)



NOESY **5bi** (300 MHz, $CDCl_3$)