Supporting Information

A Novel Isocyanide-Based Three-Component Reaction: Synthesis of Highly Substituted 1,6- Dihydropyrazine-2,3-dicarbonitrile Derivatives

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EXPERIMENTAL PROCEDURES

General

Melting points are uncorrected. Mass spectra were recorded at an ionization potential of 70 eV. ^1H NMR Spectra were recorded on 300.13 MHz instrument; chemical shifts (δ scale) are reported in parts per million (ppm). ^1H NMR Spectra are reported in order: multiplicity, approximate coupling constant (J value) in hertz (Hz) and number of protons; signals were characterized as s (singlet), d (doublet), t (triplet), m (multiplet), br s (broad signal). The ^{13}C NMR spectra were recorded at 75.47 MHz; chemical shifts (δ scale) are reported in parts per million (ppm). All the products are new compounds, which were characterized by IR, ^1H NMR and ^{13}C NMR spectra and mass spectral data.

General procedure for the synthesis of 1,6-dihydropyrazine-2,3-dicarbonitriles (4a-o): To a solution of DAMN (0.108 g, 1 mmol), ketone (1 mmol) and isocyanide (1 mmol), in 3 mL of EtOH, was added *p*-TsOH·H₂O (0.095 g, 5 mol%). The resulting mixture was stirred for appropriate time at ambient temperature. After completion of the reaction, as indicated by TLC (ethyl acetate/*n*-hexane, 3/1), the product was precipitated by addition of 10 mL of water. The precipitate was filtered off and washed with water, and then crystallized from acetone to give **4**.

Experimental procedure for the synthesis of 2-amino-3-(benzylideneamino)maleonitrile (8):

To a solution of DAMN (0.108 g, 1 mmol), benzaldehyde (0.106 g, 1 mmol) and cyclohexyl isocyanide (0.109 g, 1 mmol), in 3 mL of EtOH, was added *p*-TsOH·H₂O (0.095 g, 5 mol%). The resulting mixture was stirred for 2 h at ambient temperature. After completion of the reaction, as indicated by TLC (ethyl acetate/*n*-hexane, 2/1), the product was precipitated by addition of 10

mL of water. The precipitate was filtered off and washed with water, and then crystallized from acetone to give **8**.

5-(Cyclohexylamino)-1,6-dihydro-6,6-dimethylpyrazine-2,3-dicarbonitrile (4a). Colorless crystals (0.230 g, 90%); mp 255–258°C (dec.). IR (KBr) cm^{-1} : 3343, 2935, 2852, 2219, 1572, 1542. ^1H NMR (300.13 MHz, $\text{DMSO-}d_6$) δ : 1.00–1.77 (10H, m), 1.19 (6H, s), 3.69 (1H, m), 6.88 (1H, d, $^3J_{\text{HH}} = 5.1$ Hz), 7.14 (1H, br s). ^{13}C NMR (75.47 MHz, $\text{DMSO-}d_6$) δ : 24.3, 25.2, 25.7, 32.0, 49.7, 50.0, 110.2, 110.8, 115.0, 118.4, 155.8. MS m/z : 257 (M^+ , 20), 242 (25), 175 (25), 160 (100), 133 (22), 57 (45), 41 (75). Anal. Calcd for $\text{C}_{14}\text{H}_{19}\text{N}_5$: C, 65.34; H, 7.44; N, 27.22; found C, 65.32; H, 7.43; N, 27.20.

5-(Cyclohexylamino)-6-ethyl-1,6-dihydro-6-methylpyrazine-2,3-dicarbonitrile (4b). Light yellow crystals (0.252 g, 94%). mp 222–225°C. IR (KBr) cm^{-1} : 3416, 3257, 3054, 2217, 1542. ^1H NMR (300.13 MHz, $\text{DMSO-}d_6$) δ : 0.79 (3H, t, $^3J_{\text{HH}} = 6.8$ Hz), 1.09–1.85 (12H, m), 1.28 (3H, s), 3.73 (1H, m), 6.87 (1H, d, $^3J_{\text{HH}} = 7.5$ Hz), 7.11 (1H, br s). ^{13}C NMR (75.47 MHz, $\text{DMSO-}d_6$) δ : 7.9, 22.8, 25.3, 25.3, 25.7, 25.9, 29.5, 32.1, 50.0, 53.4, 109.9, 110.5, 114.9, 118.6, 154.8. MS m/z : 271 (M^+ , 60), 242 (70), 160 (100), 133 (15), 55 (40), 41 (45). Anal. Calcd for $\text{C}_{15}\text{H}_{21}\text{N}_5$: C, 66.39; H, 7.80; N, 25.81; found C, 66.38; H, 7.81; N, 25.79.

5-(Cyclohexylamino)-1,6-dihydro-6-methyl-6-propylpyrazine-2,3-dicarbonitrile (4c). Light yellow crystals (0.281 g, 98%). mp 197–204°C. IR (KBr) cm^{-1} : 3352, 2931, 2854, 2213, 1580, 1546. ^1H NMR (300.13 MHz, $\text{DMSO-}d_6$) δ : 0.84 (3H, t, $^3J_{\text{HH}} = 7.1$ Hz), 1.00–1.85 (14H, m), 1.29 (3H, s), 3.71 (1H, m), 6.87 (1H, d, $^3J_{\text{HH}} = 7.8$ Hz), 7.14 (1H, br s). ^{13}C NMR (75.47 MHz, $\text{DMSO-}d_6$) δ : 14.5, 16.5, 23.3, 24.0, 25.2, 25.3, 25.7, 32.0, 32.1, 50.0, 53.1, 109.8, 110.5, 114.9,

118.6, 154.9. MS m/z : 285 (M^+ , 10), 242 (70), 160 (100), 133 (20), 57 (45), 41 (75). Anal. Calcd for $C_{16}H_{23}N_5$: C, 67.34; H, 8.12; N, 24.54; found C, 67.31; H, 8.10; N, 24.52.

5-(Cyclohexylamino)-6-spiro(cyclopentan)-1,6-dihydro-pyrazine-2,3-dicarbonitrile (4d).

Light yellow crystals (0.253 g, 88%). mp 230–233°C. IR (KBr) cm^{-1} : 3359, 2930, 2852, 2214, 1566, 1535. 1H NMR (300.13 MHz, DMSO- d_6) δ : 1.00-1.85 (18H, m), 3.65 (1H, m), 6.81 (1H, d, $^3J_{HH} = 7.2$ Hz), 7.10 (1H, br s). ^{13}C NMR (75.47 MHz, DMSO- d_6) δ : 23.3, 25.3, 25.6, 31.8, 35.1, 50.5, 60.7, 110.4, 110.9, 114.8, 118.1, 154.0. MS m/z : 283 (M^+ , 45), 236 (15), 201 (75), 172 (85), 160 (75), 145 (25), 107 (25), 91 (60), 65 (45), 54 (60), 41 (100). Anal. Calcd for $C_{16}H_{21}N_5$: C, 67.82; H, 7.47; N, 24.71; found C, 67.80; H, 7.46; N, 24.72.

5-(Cyclohexylamino)-6-spiro(cyclohexan)-1,6-dihydro-pyrazine-2,3-dicarbonitrile (4e).

Colorless crystals (0.240 g, 82%). mp 284–285°C. IR (KBr) cm^{-1} : 3382, 3295, 2933, 2852, 2202, 1633, 1617, 1543. 1H NMR (300.13 MHz, DMSO- d_6) δ : 1.00-1.90 (20H, m), 3.55 (1H, m), 6.85 (1H, d, $^3J_{HH} = 7.3$ Hz), 6.99 (1H, br s). ^{13}C NMR (75.47 MHz, DMSO- d_6) δ : 21.0, 21.5, 25.2, 25.6, 32.3, 32.6, 48.2, 55.3, 108.5, 110.6, 116.2, 119.4, 152.7. MS m/z : 297 (M^+ , 90), 269 (100), 208 (15), 98 (20), 82 (26), 55 (48), 41 (54). Anal. Calcd for $C_{17}H_{23}N_5$: C, 68.66; H, 7.80; N, 23.55; found C, 68.63; H, 7.81; N, 23.54.

5-(Cyclohexylamino)-6-spiro(2-methylcyclohexan)-1,6-dihydro-pyrazine-2,3-dicarbonitrile (4f).

Light yellow crystals (0.262 g, 85%). mp 237–240°C. IR (KBr) cm^{-1} : 3378, 3197, 2932, 2856, 2213, 1570. 1H NMR (300.13 MHz, DMSO- d_6) δ : 0.81 (3H, d, $^3J_{HH} = 6.5$ Hz), 1.00-1.92 (18H, m), 2.34-2.42 (1H, m), 3.72 (1H, m), 6.81 (1H, d, $^3J_{HH} = 7.5$ Hz), 6.94 (1H, br s). ^{13}C NMR (75.47 MHz, DMSO- d_6) δ : 14.2, 19.1, 20.8, 25.2, 25.4, 25.7, 26.1, 26.3, 28.4, 31.8, 32.0,

50.9, 54.5, 107.6, 112.5, 115.3, 118.3, 152.5. MS m/z : 311 (M^+ , 70), 254 (40), 240 (25), 229 (54), 188 (25), 172 (35), 160 (25), 82 (15), 67 (25), 55 (70), 41 (100). Anal. Calcd for $C_{18}H_{25}N_5$: C, 69.42; H, 8.09; N, 22.49; found C, 69.40; H, 8.07; N, 22.46.

5-(Cyclohexylamino)-1,6-dihydro-6-methyl-6-phenylpyrazine-2,3-dicarbonitrile (4g). Light yellow crystals (0.262 g, 82%). mp 230–233°C. IR (KBr) cm^{-1} : 3404, 3358, 2934, 2854, 2215, 1568. 1H NMR (300.13 MHz, DMSO- d_6) δ : 1.11–2.05 (10H, m), 1.67 (3H, s), 3.85 (1H, m), 7.23–7.42 (6H, m), 8.03 (1H, br s). ^{13}C NMR (75.47 MHz, DMSO- d_6) δ : 25.2, 25.3, 25.7, 25.9, 31.8, 32.2, 50.8, 55.6, 110.3, 111.9, 114.7, 118.0, 125.1, 128.3, 129.0, 143.0, 153.3. MS m/z : 319 (M^+ , 30), 304 (40), 337 (23), 222 (100), 195 (15), 160 (45), 103 (30), 77 (25), 55 (50), 41 (60). Anal. Calcd for $C_{19}H_{21}N_5$: C, 71.45; H, 6.63; N, 21.93; found C, 71.41; H, 6.62; N, 21.94.

5-(Cyclohexylamino)-1,6-dihydro-6-methyl-6-*p*-tolylpyrazine-2,3-dicarbonitrile (4h). Light yellow crystals (0.310 g, 92%). mp 250–253°C. IR (KBr) cm^{-1} : 3350, 2932, 2854, 2225, 2209, 1564, 1536. 1H NMR (300.13 MHz, DMSO- d_6) δ : 1.00–2.01 (10H, m), 1.64 (3H, s), 2.26 (3H, s), 3.85 (1H, m), 7.11, 7.16 (4H, AB-q, $^3J_{AB} = 7.5$ Hz), 7.36 (1H, d, $^3J_{HH} = 7.1$ Hz), 7.97 (1H, br s). ^{13}C NMR (75.47 MHz, DMSO- d_6) δ : 20.5, 24.7, 24.8, 25.2, 25.4, 31.3, 31.6, 50.2, 54.9, 109.8, 111.4, 114.2, 117.5, 124.6, 129.0, 137.0, 139.4, 153.0. MS m/z : 333 (M^+ , 24), 318 (47), 251 (20), 236 (100), 209 (20), 160 (20), 117 (27), 91 (25), 65 (15), 55 (54), 41 (70). Anal. Calcd for $C_{20}H_{23}N_5$: C, 72.04; H, 6.95; N, 21.00; found C, 72.02; H, 6.96; N, 21.02.

6-(4-Bromophenyl)-5-(cyclohexylamino)-1,6-dihydro-6-methylpyrazine-2,3-dicarbonitrile (4i). White powder (0.372 g, 94%). mp 275–278°C. IR (KBr) cm^{-1} : 3351, 2931, 2855, 2219, 1568. 1H NMR (300.13 MHz, DMSO- d_6) δ : 1.10–2.05 (10H, m), 1.66 (3H, s), 3.86 (1H, m), 7.18

(2H, d, $^3J_{\text{HH}} = 8.5$ Hz), 7.42 (2H, d, $^3J_{\text{HH}} = 7.9$ Hz), 7.59 (2H, d, $^3J_{\text{HH}} = 8.5$ Hz), 8.04 (1H, br s). ^{13}C NMR (75.47 MHz, DMSO- d_6) δ : 25.2, 25.3, 25.5, 25.7, 31.8, 32.1, 50.8, 55.4, 110.0, 112.0, 114.5, 117.8, 121.5, 127.5, 132.0, 142.4, 152.8. MS m/z : 399 (M^+ , ^{81}Br , 18), 397 (M^+ , ^{79}Br , 16), 384 (20), 382 (18), 317 (16), 315 (14), 302 (50), 300 (48), 269 (50), 220 (27), 183 (17), 160 (20), 102 (33), 77 (23), 55 (95), 41 (100). Anal. Calcd for $\text{C}_{19}\text{H}_{20}\text{BrN}_5$: C, 57.29; H, 5.06; N, 17.58; found C, 57.26; H, 5.04; N, 17.57.

5-(*tert*-Butylamino)-1,6-dihydro-6,6-dimethylpyrazine-2,3-dicarbonitrile (4j). Light yellow crystals (0.221 g, 93%). mp 225–228°C. IR (KBr) cm^{-1} : 3412, 3324, 2977, 2212, 1616, 1560. ^1H NMR (300.13 MHz, DMSO- d_6) δ : 1.19 (6H, s), 1.34 (9H, s), 6.15 (1H, br s), 7.11 (1H, br s). ^{13}C NMR (75.47 MHz, DMSO- d_6) δ : 24.0, 28.5, 49.4, 52.6, 110.1, 110.2, 114.9, 118.4, 154.9. MS m/z : 231 (M^+ , 30), 175 (20), 160 (100), 133 (15), 57 (35), 41 (55). Anal. Calcd for $\text{C}_{12}\text{H}_{17}\text{N}_5$: C, 62.31; H, 7.41; N, 30.28; found C, 62.32; H, 7.40; N, 30.26.

5-(Cyclohexylamino)-1,6-dihydro-6-spiro(cyclopentan)-pyrazine-2,3-dicarbonitrile (4k). Violet crystals (0.220 g, 84%). mp 190–194°C. IR (KBr) cm^{-1} : 3399, 2971, 2879, 2214, 1572, 1547. ^1H NMR (300.13 MHz, DMSO- d_6) δ : 1.33 (9H, s, $\text{C}(\text{CH}_3)_3$), 1.53–1.81 (8H, m), 6.18 (1H, br s), 7.09 (1H, br s). ^{13}C NMR (75.47 MHz, DMSO- d_6) δ : 23.0, 28.5, 34.6, 52.7, 60.4, 109.8, 111.2, 114.9, 118.5, 152.7. MS m/z : 257 (M^+ , 100), 201 (32), 172 (45), 160 (20), 148 (20), 57 (30), 41 (50). Anal. Calcd for $\text{C}_{14}\text{H}_{19}\text{N}_5$: C, 65.34; H, 7.44; N, 27.22; found C, 65.30; H, 7.41; N, 27.24.

5-(*tert*-Butylamino)-1,6-dihydro-6-methyl-6-*p*-tolylpyrazine-2,3-dicarbonitrile (4l). Light yellow crystals (0.251 g, 82%). mp 215–216°C. IR (KBr) cm^{-1} : 3390, 3333, 2970, 2920, 2220, 1564, 1542. ^1H NMR (300.13 MHz, DMSO- d_6) δ : 1.42 (9H, s), 1.66 (3H, s), 2.27 (3H, s), 6.64

(1H, br s), 7.13, 7.17 (4H, AB-q, $^3J_{AB} = 8.3$ Hz), 7.99 (1H, br s). ^{13}C NMR (75.47 MHz, DMSO- d_6) δ : 21.0, 25.6, 28.5, 53.2, 55.2, 110.2, 111.2, 114.7, 118.0, 125.3, 129.4, 137.5, 139.9, 152.5. MS m/z : 307 (M^+ , 20), 251 (15), 236 (100), 117 (20), 91 (17), 57 (25), 41 (35). Anal. Calcd for $\text{C}_{18}\text{H}_{21}\text{N}_5$: C, 70.33; H, 6.89; N, 22.78; found C, 70.35; H, 6.87; N, 22.76.

5-(2,6-Dimethylphenylamino)-1,6-dihydro-6-spiro(2-methyl-cyclohexan)-pyrazine-2,3-dicarbonitrile (4m). Orange powder (0.266 g, 80%). mp 260–263°C. IR (KBr) cm^{-1} : 3358, 2931, 2861, 2214, 1559, 1536. ^1H NMR (300.13 MHz, DMSO- d_6) δ : 1.04 (3H, d, $^3J_{\text{HH}} = 6.2$ Hz), 1.20–2.60 (9H, m), 2.13 (6H, s), 7.05–7.20 (3H, m), 7.23 (1H, br s), 8.48 (1H, br s). ^{13}C NMR (75.47 MHz, DMSO- d_6) δ : 14.3, 17.1, 21.1, 25.3, 26.3, 28.7, 35.7, 55.2, 108.1, 112.7, 114.6, 115.0, 117.8, 127.3, 128.3, 135.5, 151.3. MS m/z : 333 (M^+ , 100), 264 (26), 248 (38), 226 (15), 203 (16), 187 (24), 159 (52), 145 (70), 105 (30), 77 (45), 55 (27), 41 (60). Anal. Calcd for $\text{C}_{20}\text{H}_{23}\text{N}_5$: C, 72.04; H, 6.95; N, 21.00; found C, 72.05; H, 6.93; N, 21.02.

5-(2,4,4-Trimethylpentan-2-ylamino)-1,6-dihydro-6,6-dimethylpyrazine-2,3-dicarbonitrile (4n). White powder (0.251 g, 87%). mp 150–151°C. IR (KBr) cm^{-1} : 3358, 2931, 2861, 2214, 1559, 1536. ^1H NMR (300.13 MHz, DMSO- d_6) δ : 0.93 (9H, s), 1.17 (6H, s), 1.36 (6H, s), 1.83 (2H, s), 6.07 (1H, br s), 7.15 (1H, br s). ^{13}C NMR (75.47 MHz, DMSO- d_6) δ : 23.9, 29.3, 31.7, 31.8, 49.3, 49.8, 56.5, 109.8, 110.1, 115.0, 118.4, 154.1. MS m/z : 287 (M^+ , 17), 176 (48), 160 (100), 133 (15), 97 (17), 57 (60), 41 (63). Anal. Calcd for $\text{C}_{16}\text{H}_{25}\text{N}_5$: C, 66.86; H, 8.77; N, 24.37; found C, 66.85; H, 8.78; N, 24.34.

5-(2,4,4-Trimethylpentan-2-ylamino)-1,6-dihydro-6-spiro(cyclohexan)-pyrazine-2,3-dicarbonitrile (4o). Light yellow crystals (0.250 g, 80%). mp 204–205°C. IR (KBr) cm^{-1} :

3435, 3360, 2943, 2869, 2212, 1574, 1551. ^1H NMR (300.13 MHz, $\text{DMSO-}d_6$) δ : 0.92 (9H, s), 1.15-1.62 (10H, m), 1.36 (6H, s), 1.83 (2H, s), 6.04 (1H, br s), 6.82 (1H, br s). ^{13}C NMR (75.47 MHz, $\text{DMSO-}d_6$) δ : 20.4, 25.0, 29.3, 29.6, 31.7, 31.8, 49.7, 51.6, 56.5, 108.1, 111.4, 115.2, 118.3, 152.8. MS m/z : 327 (M^+ , 10), 215 (85), 172 (77), 97 (20), 81 (20), 57 (77), 41 (100). Anal. Calcd for $\text{C}_{19}\text{H}_{29}\text{N}_5$: C, 69.69; H, 8.93; N, 21.39; found C, 69.66; H, 8.90; N, 21.41.

2-Amino-3-(benzylideneamino)maleonitrile (8). Yellow solid (0.188 g, 96%). mp 205–207°C. IR (KBr) cm^{-1} : 3415, 3310, 3165, 2235, 2210, 1611, 1576. ^1H NMR (300.13 MHz, $\text{DMSO-}d_6$) δ : 7.41-7.52 (3H, m), 7.92 (2H, br s), 8.00-8.06 (2H, m), 8.27 (1H, s). ^{13}C NMR (75.47 MHz, $\text{DMSO-}d_6$) δ : 103.0, 114.2, 114.9, 127.4, 129.2, 129.5, 132.0, 136.0, 155.5. MS m/z : 196 (M^+ , 70), 118 (47), 106 (100), 91 (65), 77 (20). Anal. Calcd for $\text{C}_{11}\text{H}_8\text{N}_4$: C, 67.34; H, 4.11; N, 28.55; found C, 67.31; H, 4.12; N, 28.54.

Current Data Parameters
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PROCNO 1

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***** CHANNEL f2 *****
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4a

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4a

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DI/MOGHIMI-169/85.10.30

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PROCNO: 1

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F2 - Processing parameters
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Current Data Parameters
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EXPNO 112
PROCNO 1

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DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.0002000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 6.75 usec
PL1 -2.00 dB
SFO1 75.4752553 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 87.00 usec
PL2 -2.00 dB
PL12 12.00 dB
PL13 16.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 65536
SF 75.467460 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 27.30 cm
FIP 219.155 ppm
F1 16539.10 Hz
F2 -1445.51 Hz
PNUC 11.91665 ppm/cg
NUC1 699.28056 Hz/cg

File : DI_49.X50 Date 8/ 3/10 Time 14:39:42
 S=[88->96] Bp=41 Bi=105250, RT=1.59 CT=264

251 271 291 311 331 351 371 391 411 431 451 471 491

SB=30 SE=480 DB=1 DE=480 N=0 Z=2 T=0.0 FactI -> 1 *1

S List > S=[88->96] B=0 Pos=4 Tot=4

File : DI_49.X68 Date 8/ 3/10 Time 18:46:58
 S=[71->101] Bp=41 Bi=103060. RT=1.67 CT=238

280 300 320 340 360 380 400 420 440 460 480 500 520

SB=30 SE=470 DB=30 DE=470 N=0 Z=2 T=0.0 FactI -> 1 *1

S List > S=[71->101] B=0 Pos=6 Tot=6

File : DI_49.X65 Date 8/ 3/10 Time 18:23:04
 S=72 Bp=236 Bi=209700, RT=1.19 CI=201

280 300 320 340 360 380 400 420 440 460 480 500 520

SB=30 SE=336 DB=30 DE=510 N=0 Z=2 T=0.0 FactI -> 1 *1

S List > S=72 B=0 Pos=3 Tot=3

DI/MOGHIMI-173/85.11.04

File : DI_50.X02 Date 8/ 4/10 Time 02:53:15

S=[111->128] Bp=41 Bi=87950. RT=2.12 CT=301

SB=30 SE=520 DB=30 DE=520 N=0 Z=2 T=0.0 FactI -> 1 *1

S List > S=[111->128] B=0 Pos=7 Tot=7

Current Data Parameters
NAME MATEL-PRO
EXPNO 52
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070103
Time 12.45
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 799
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 9195.2
DM 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec
d11 0.0000000 sec
d12 0.0000000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 8.75 usec
PL1 -2.00 dB
SF01 75.4752953 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 87.00 usec
PL2 -2.00 dB
PL12 12.00 dB
PL13 18.00 dB
SF02 300.1332005 MHz

F2 - Processing parameters
SI 65536
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

ID NMR plot parameters
CX 20.00 cm
CY 10.24 cm
FIP 219.155 ppm
F1 16536.11 Hz
F2 -15.167 ppm
F2 -1446.51 Hz
PPMCM 11.91609 ppm/cm
HSCM 899.28058 Hz/cm

File : DI_49.X69 Date 8/ 3/10 Time 18:54:17
 S=[67->96] Bp=160 Bi=406670. RT=1.59 CT=239

4i

280 300 320 340 360 380 400 420 440 460 480 500 520

S8=30 SE=510 DB=30 DE=510 N=0 Z=2 T=0.0 FactI -> 1 *1

S List > S=[67->96] B=0 Pos=3 Tot=3

Current Data Parameters
NAME Maleki-PHO
EXPNO 46
PROCNO 1

F2 - Acquisition Parameters
Date_ 20061217
Time 11.12
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TO 32768
SOLVENT DMSO
NS 10
DS 1
SWH 6172.838 Hz
FIDRES 0.169360 Hz
AQ 2.6542580 sec
RG 228.1
DM B1.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 15.50 usec
PL1 -2.00 dB
SF01 300.1318534 MHz

F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 33.61 cm
FIP 11.068 ppm
F1 3327.68 Hz
F2P -0.926 ppm
F2 -278.04 Hz
PPHCH 0.50073 ppm/cm
HZCM 180.29562 Hz/cm

DI/MOGHIMI-163/85.10.30

File : DI_49.X99 Date 8/ 4/10 Time 02:39:32

S=[58->83] Bp=258 Bi=199220. RT=1.37 CT=227

4k

280 300 320 340 360 380 400 420 440 460 480 500 520

SB=30 SE=530 DB=30 DE=530 N=0 Z=2 T=0.0 FactI -> 1 *1

S List > S=[58->83] B=0 Pos=3 Tot=3

DI/MOGHIMI-197/85.11.04

File : DI_50.X04 Date 8/ 4/10 Time 03: 8:01

S=83 Bp=160 Bi=409400. RT=1.37 CT=221

4n

SB=30 SE=520 DB=30 DE=520 N=0 Z=2 T=0.0 FactI -> 1 *1

S List > S=83 B=0 Pos=5 Tot=5

Current Data Parameters
NAME: hogh1a1
CNO: 100
PROG: 1

F2 - Acquisition Parameters
Date_: 20061216
Time: 13.00
INSTRUM: spect
PROBHD: 5 mm BBO BB-1H
PULPROG: zgpg30
TD: 32768
SOLVENT: DMSO
NS: 10
DS: 1
SWH: 6172.830 Hz
FIDRES: 0.160360 Hz
AQ: 2.6542590 sec
RG: 80.0
DM: 81.000 MHz
DE: 5.00 MHz
TE: 300.0 K
OI: 2.00000000 sec

===== CHANNEL f1 =====
NUC1: 1H
P1: 15.50 MHz
PL1: -2.00 dB
SFO1: 300.1318334 MHz

F2 - Processing parameters
SI: 65536
SF: 300.1300000 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.00

1D NMR plot parameters
CX: 20.00 cm
CY: 10.00 cm
F1P: 11.080 mm
F1: 3327.80 Hz
F2P: -0.505 mm
F2: -278.04 Hz
PH0H: 0.00073 ppm/cm
H0H0: 180.29593 Hz/cm

40

File : DI_49.X52 Date 8/ 3/10 Time 16:39:04
 S=[87->95] Bp=41 Bi=214670. RI=1.57 CT=224

40

280 300 320 340 360 380 400 420 440 460 480 500 520

SB=30 SE=520 DB=30 DE=520 N=0 Z=2 T=0.0 FactI -> 1 *1
 S List > S=[87->95] B=0 Pos=3 Tot=3

1H NMR

8

7.48007
7.96332
8.01811
8.03480
8.04097
8.26822

2.49566
3.36458

2.9472
1.7932
2.0644
1.0000

Integral

ppm

Current Data Parameters
NAME Maleki-PHO
EXPNO 71
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070317
Time 13.19
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 10
DS 1
SWH 7812.500 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 228.1
DW 64.000 usec
DE 6.00 usec
TE 380.0 K
D1 2.0000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 15.50 usec
PL1 -2.00 dB
SF01 300.1323986 MHz

F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 9.69 cm
F1P 11.212 ppm
F1 3365.00 Hz
F2P -0.956 ppm
F2 -286.93 Hz
PPMCM 0.60839 ppm/cm
HZCM 182.59663 Hz/cm

¹³C {¹H} NMR

Current Data Parameters
NAME Maleki-PHO
EXPNO 70
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070317
Time 13.14
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 72
DS 2
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 2048
DM 27.800 usec
DE 6.00 usec
TE 300.2 K
D1 2.0000000 sec
d11 0.0300000 sec
d12 0.0002600 sec

===== CHANNEL f1 =====
NUC1 ¹³C
P1 8.75 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
CPOPRG2 waltz16
NUC2 ¹H
PCPD2 87.00 usec
P2 -2.00 dB
P12 12.00 dB
P13 18.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 65536
SF 75.4677490 MHz
WDW EN
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 17.09 cm
FIP 229.155 ppm
F1 16539.10 Hz
F2P -19.167 ppm
F2 -1446.51 Hz
PPHCK 11.91509 ppm/cm
HZCM 899.28058 Hz/cm