Photochemical protection of amines with the Cbz and Fmoc groups

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Supporting Information

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Characterization data for compounds not described in the Experimental Section

***N*-Cbz-cyclohexylamine 1b:** Brown solid (mp 87-91°C). ¹H NMR (CDCl₃) δ 7.39-7.30 (5H), 5.09 (s, 2H), 4.65 (br s, 1H), 3.51 (1H), 1.99-1.90 (2H), 1.76-1.66 (2H), 1.65-1.56 (1H), 1.42-1.25 (2H), 1.23-1.08 (3H); ¹³C NMR (CDCl₃) δ 155.5 (C), 136.7 (C), 128.5 (CH), 128.1 (CH), 128.0 (CH), 66.4 (CH₂), 49.9 (CH), 33.4 (CH₂), 25.5 (CH₂), 24.7 (CH₂). IR (CHCl₃) 3441.5, 3019.6, 2936.9, 1712.8, 1509.8, 1453.4, 1339.3, 1316.3, 1252.7, 1227.2, 1044.2. MS *m/z* (%) 233 (<1, M⁺), 146 (10), 142 (6), 108 (57), 91 (100); HR-MS 233.1419 (C₁₄H₁₉NO₂ calcd 233.1416).

***N*-Cbz-(±)-1-phenylethylamine 1c:** Brown oil. ¹H NMR (CDCl₃) δ 7.40-7.28 (10H), 5.15-5.02 (m, 3H), 4.88 (1H), 1.50 (d, *J* = 6.8 Hz, 3H); ¹³C NMR (CDCl₃) δ 155.5 (C), 143.4 (C), 136.4 (C), 128.6 (CH), 128.5 (CH), 128.1 (CH), 127.3 (CH), 125.9 (CH), 66.7 (CH₂), 50.7 (CH), 22.4 (CH₃). IR (CHCl₃) 3443.8, 3019.2, 1717.1, 1505.9, 1454.4, 1340.4, 1056.5. MS *m/z* (%) 255 (<1, M⁺), 240 (2), 196 (5), 164 (40), 120 (18), 108 (13), 105 (19), 91 (100); HR-MS 255.1240 (C₁₆H₁₇NO₂ calcd 255.1259).

***N*-Cbz-dipentylamine 1d:** Brown oil. ¹H NMR (CDCl₃) δ 7.39-7.29 (5H), 5.13 (s, 2H), 3.22 (4H), 1.52 (4H), 1.27 (8H), 0.88 (6H); ¹³C NMR (CDCl₃) δ 156.1 (C), 137.1 (C), 128.3 (CH), 127.71 (CH), 127.67 (CH), 66.7 (CH₂), 47.5 (CH₂), 46.8 (CH₂), 28.9 (CH₂), 28.3 (CH₂), 27.8 (CH₂), 22.4 (CH₂), 14.0 (CH₃). IR (CHCl₃) 3011.7, 2960.3, 2933.0, 2862.4, 1685.7, 1477.2, 1426.4, 1274.5, 1159.7, 1089.6. MS *m/z* (%) 291 (5, M⁺), 234 (7), 200 (4), 190 (29), 108 (7), 100 (9), 91 (100); HR-MS 291.2192 (C₁₈H₂₉NO₂ calcd 291.2198).

***N*-Cbz-piperidine 1e:** Brown oil. ¹H NMR (CDCl₃) δ 7.39-7.29 (5H), 5.13 (s, 2H), 3.45 (t, *J* = 5.5 Hz, 4H), 1.59 (2H), 1.54 (4H); ¹³C NMR (CDCl₃) δ 155.3 (C), 137.0 (C), 128.4 (CH), 127.84 (CH), 127.76 (CH), 66.9 (CH₂), 44.8 (CH₂), 25.7 (CH₂), 24.4 (CH₂). IR (CHCl₃) 3013.5, 2943.4,

2859.9, 1687.7, 1435.2, 1265.8, 1237.5, 1150.5, 1088.9, 1026.2. MS m/z (%) 219 (7, M^+), 174 (7), 128 (13), 108 (12), 91 (100), 79 (17), 77 (10), 65 (12); HR-MS 219.1270 ($C_{13}H_{17}NO_2$ calcd 219.1259).

N-Cbz-dicyclohexylamine 1f: Brown oil, isolated by FC, TLC R_f 0.60 (cyclohexane:AcOEt 2:1). 1H NMR ($CDCl_3$) δ 7.42-7.28 (5H), 5.19-5.13 (2H), 3.77-3.22 (2H), 1.86-1.70 (6H), 1.66-1.56 (8H), 1.39-1.23 (4H), 1.18-1.01 (2H); ^{13}C NMR ($CDCl_3$) δ 155.7 (C), 137.3 (C), 128.6 (CH), 128.4 (CH), 127.6 (CH), 66.4 (CH_2), 55.4 (CH), 31.5 (CH_2), 26.3 (CH_2), 25.5 (CH_2). IR ($CHCl_3$) 2934.9, 2857.2, 1674.2, 1453.9, 1294.0, 1014.4. MS m/z (%) 315 (2, M^+), 224 (26), 180 (3), 142 (24), 91 (100), 55 (13); HR-MS 315.2218 ($C_{20}H_{29}NO_2$ calcd 315.2198).

N-Cbz-(+)-ephedrine 1g: Brown oil, isolated by FC, TLC R_f 0.32 (cyclohexane:AcOEt 2:1). 1H NMR ($CDCl_3$) (rotamers) δ 7.4-7.2 (10H), 5.2-5.0 (2H), 5.0-4.6 (1H), 4.4-4.1 (1H), 2.9-2.7 (3H), 2.2-1.7 (br s, 1H), 1.25 (d, $J = 7.1$ Hz, 3H); ^{13}C NMR ($CDCl_3$) (rotamers) δ 157.2 (C), 157.0 (C), 141.9 (C), 136.7 (C), 136.6 (C), 128.50 (CH), 128.46 (CH), 128.2 (CH), 128.1 (CH), 128.0 (CH), 127.7 (CH), 127.6 (CH), 126.2 (CH), 77.2 (CH), 77.1 (CH), 67.1 (CH_2), 66.7 (CH_2), 59.2 (CH), 56.8 (CH), 32.0 (CH_3), 30.3 (CH_3), 27.6 (CH_3), 14.5 (CH_3), 13.3 (CH_3), 12.1 (CH_3). IR ($CHCl_3$) 3465.5, 3012.7, 2949.0, 1685.5, 1454.5, 1404.5, 1322.7, 1151.8, 1011.0. MS m/z (%) 300 (<1), 282 (<1), 192 (18), 148 (24), 108 (22), 107 (13), 91 (100), 79 (12); HR-MS 192.1021 ($C_{11}H_{14}NO_2$ calcd 192.1025).

N-Cbz-allylamine 1i: Brown oil. 1H NMR ($CDCl_3$) δ 7.39-7.30 (m, 5H), 5.86 (ddt, $J = 16.6$, 10.6, 5.5 Hz, 1H), 5.24-5.11 (m, 4H), 4.82 (br s, 1 H), 3.84 (t, $J = 5.3$ Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 156.2 (C), 136.5 (C), 134.4 (CH), 128.5 (CH), 128.1 (CH), 116.1 (CH_2), 66.8 (CH_2), 43.5 (CH_2). IR ($CHCl_3$) 3452.5, 3020.6, 1718.3, 1513.0, 1455.7, 1341.7, 1227.3, 1133.9, 990.0, 931.0. MS m/z (%) 191 (<1, M^+), 130 (8), 108 (48), 107 (10), 91 (100), 77 (8), 56 (6); HR-MS 191.0935 ($C_{11}H_{13}NO_2$ calcd 191.0946).

***N*-Cbz-*L*-leucine allyl ester 1j:** Yellow oil, isolated by FC, TLC R_f 0.55 (cyclohexane:AcOEt 2:1). ^1H NMR (CDCl_3) δ 7.41-7.29 (5H), 5.91 (ddt, $J = 16.8, 11.1, 5.6$ Hz, 1H), 5.34 (d, $J = 17.2$ Hz, 1H), 5.26 (d, $J = 10.4$ Hz, 1H), 5.16 (br d, $J = 8.6$ Hz, 1H), 5.12 (s, 2H), 4.64 (d, $J = 5.8$ Hz, 2H), 4.43 (m, 1H), 1.78-1.49 (m, 3H), 0.96 (t, $J = 6.8$ Hz, 6H); ^{13}C NMR (CDCl_3) δ 172.8 (C), 155.9 (C), 136.2 (C), 131.6 (CH), 128.5 (CH), 128.2 (CH), 128.1 (CH), 118.7 (CH_2), 67.0 (CH_2), 65.9 (CH_2), 52.5 (CH), 41.8 (CH_2), 24.7 (CH), 22.8 (CH_3), 21.8 (CH_3). IR (CHCl_3) 3438.5, 3020.5, 2962.2, 1721.4, 1511.8, 1455.2, 1332.8, 1050.4, 987.3. MS m/z (%) 305 (<1, M^+), 220 (10), 176 (18), 108 (6), 91 (100), 65 (5); HR-MS 305.1629 ($\text{C}_{17}\text{H}_{23}\text{NO}_4$ calcd 305.1627).

***N*-Cbz-*L*-tyrosine *tert*-butyl ester 1k:** Yellow oil, isolated by FC, TLC R_f 0.30 (cyclohexane:AcOEt 2:1). ^1H NMR (CDCl_3) δ 7.40-7.29 (5H), 7.01 (d, $J = 8.3$ Hz, 2H), 6.72 (d, $J = 8.3$ Hz, 2H), 5.25 (1H), 5.10 (m, 2H), 4.49 (m, 1H), 3.01 (m, 2H), 1.42 (s, 9H); ^{13}C NMR (CDCl_3) δ 170.7 (C), 155.7 (C), 154.7 (C), 136.3 (C), 130.7 (CH), 128.5 (CH), 128.14 (CH), 128.09 (CH), 128.0 (C), 115.3 (CH), 82.3 (C), 66.9 (CH_2), 55.3 (CH), 37.6 (CH_2), 28.0 (CH_3). IR (CHCl_3) 3596.9, 3430.4, 3020.5, 2983.1, 1717.6, 1514.9, 1455.3, 1370.3, 1355.5, 1257.3, 1155.7, 1059.4. MS m/z (%) 371 (<1, M^+), 270 (1), 220 (26), 164 (27), 108 (15), 107 (86), 91 (100), 57 (27); HR-MS 270.1146 ($\text{C}_{16}\text{H}_{16}\text{NO}_3$ calcd 270.1130).

***N*-Cbz-*L*-phenylalanine allyl ester 1l:** Brown oil. ^1H NMR (CDCl_3) δ 7.39-7.24 (8H), 7.14-7.10 (2H), 5.87 (ddt, $J = 16.8, 11.2, 5.6$ Hz, 1H), 5.35-5.23 (m, 3H), 5.14-5.07 (2H), 4.73-4.68 (m, 1H), 4.66-4.54 (2H), 3.20-2.98 (2H); ^{13}C NMR (CDCl_3) δ 171.2 (C), 155.6 (C), 136.2 (C), 135.6 (C), 131.4 (CH), 129.3 (CH), 128.6 (CH), 128.5 (CH), 128.2 (CH), 128.1 (CH), 127.1 (CH), 119.1 (CH_2), 67.0 (CH_2), 66.0 (CH_2), 54.8 (CH), 38.2 (CH_2). IR (CHCl_3) 3433.6, 3019.2, 2953.9, 1721.1, 1508.5, 1455.4, 1341.2, 1203.7, 1058.1. MS m/z (%) 339 (<1, M^+), 254 (1), 188 (18), 131 (8), 107 (7), 104 (7), 91 (100); HR-MS 254.1139 ($\text{C}_{16}\text{H}_{16}\text{NO}_2$ calcd 254.1181).

N-Boc-cyclohexylamine 3b: Pale brown solid (mp 75-79°C), TLC R_f 0.58 (cyclohexane:AcOEt 2:1). ^1H NMR (CDCl_3) δ 4.42 (br s, 1H), 3.42 (1H), 1.93 (dd, $J = 12.5, 3.2$ Hz, 2H), 1.70 (dt, $J = 13.6, 3.9$ Hz, 2H), 1.64-1.55 (1H), 1.45 (s, 9H), 1.40-1.25 (2H), 1.21-1.04 (3H); ^{13}C NMR (CDCl_3) δ 155.2 (C), 79.0 (C), 49.4 (CH), 33.5 (CH_2), 28.4 (CH_3), 25.6 (CH_2), 24.9 (CH_2). IR (neat) 3325.5, 2930.8, 2853.3, 1677.8, 1520.7, 1449.1, 1365.4, 1317.2, 1235.7, 1164.1, 1046.0. MS m/z (%) 199 (<1, M^{+}), 143 (23), 100 (9), 82 (9), 57 (100); HR-MS 199.1567 ($\text{C}_{11}\text{H}_{21}\text{NO}_2$ calcd 199.1572).

N-Boc-(±)-1-phenylethylamine 3c: Pale yellow solid (mp 72-76°C), TLC R_f 0.49 (cyclohexane:AcOEt 2:1). ^1H NMR (CDCl_3) δ 7.37-7.22 (5H), 4.89-4.56 (2H), 1.51-1.31 (12H); ^{13}C NMR (CDCl_3) δ 155.1 (C), 143.8 (C), 128.5 (CH), 127.1 (CH), 125.9 (CH), 79.4 (C), 50.2 (CH), 28.4 (CH_3), 22.7 (CH_3). IR (neat) 3388.1, 2971.4, 1682.4, 1509.0, 1456.3, 1365.5, 1318.0, 1244.4, 1155.9, 1056.0, 861.0, 755.5. MS m/z (%) 165 (22), 150 (38), 120 (7), 105 (44), 79 (10), 77 (13), 57 (100).

N-Boc-L-leucine allyl ester 3j: Brown oil, TLC R_f 0.56 (cyclohexane:AcOEt 2:1). ^1H NMR (CDCl_3) δ 5.92 (ddt, $J = 16.6, 11.1, 5.6$ Hz, 1H), 5.34 (dd, $J = 17.2, 1.5$ Hz, 1H), 5.26 (d, $J = 10.6$ Hz, 1H), 4.89 (1H), 4.63 (m, 2H), 4.34 (m, 1H), 1.78-1.47 (3H), 1.45 (s, 9H), 0.96 (d, $J = 6.6$ Hz, 3H), 0.95 (d, $J = 6.6$ Hz, 3H); ^{13}C NMR (CDCl_3) δ 173.2 (C), 155.4 (C), 131.7 (CH), 118.6 (CH_2), 79.8 (C), 65.7 (CH_2), 52.1 (CH), 41.8 (CH_2), 28.3 (CH_3), 24.8 (CH), 22.8 (CH_3), 21.9 (CH_3). IR (CHCl_3) 3443.1, 2962.9, 2873.3, 1737.5, 1711.9, 1501.8, 1368.9, 1162.2. MS m/z (%) 186 (7), 170 (5), 130 (40), 86 (64), 57 (100); HR-MS 186.1489 ($\text{C}_{10}\text{H}_{20}\text{NO}_2$ calcd 186.1494).

N-Fmoc-cyclohexylamine 2b: White solid (mp 181-184°C), TLC R_f 0.49 (cyclohexane:AcOEt 2:1). ^1H NMR (CDCl_3) δ 7.78 (d, $J = 7.3$ Hz, 2H), 7.61 (d, $J = 7.6$ Hz, 2H), 7.41 (t, $J = 7.2$ Hz, 2H), 7.33 (td, $J = 7.5, 1.3$ Hz, 2H), 4.66 (br s, 1H), 4.40 (d, $J = 6.6$ Hz, 2H), 4.23 (t, $J = 6.7$ Hz, 1H), 3.57-3.45 (1H), 2.01-1.91 (2H), 1.78-1.57 (4H), 1.44-1.09 (4H); ^{13}C

NMR (CDCl₃) δ 155.6 (C), 144.1 (C), 141.3 (C), 127.6 (CH), 127.0 (CH), 125.0 (CH), 119.9 (CH), 66.3 (CH₂), 49.9 (CH), 47.3 (CH), 33.4 (CH₂), 25.5 (CH₂), 24.8 (CH₂). IR (neat) 3338.9, 2935.5, 1683.6, 1530.6, 1446.9, 1233.4, 1039.7, 733.7. MS m/z (%) 321 (<1, M⁺), 196 (1), 179 (22), 178 (100), 165 (16); HR-MS 178.0766 (C₁₄H₁₀ calcd 178.0783).

N-Fmoc-dipentylamine 2d: Yellow oil, TLC R_f 0.68 (cyclohexane:AcOEt 2:1). ¹H NMR (CDCl₃) δ 7.77 (d, J = 7.6 Hz, 2H), 7.60 (d, J = 7.3 Hz, 2H), 7.40 (t, J = 7.4 Hz, 2H), 7.32 (td, J = 7.5, 1.2 Hz, 2H), 4.50 (d, J = 6.0 Hz, 2H), 4.24 (t, J = 6.1 Hz, 1H), 3.19 (t, J = 7.2 Hz, 2H), 3.04 (t, J = 7.4 Hz, 2H), 1.56-1.45 (2H), 1.39-1.20 (m, 8H), 1.17-1.06 (2H), 0.90 (t, J = 7.2 Hz, 6H); ¹³C NMR (CDCl₃) δ 156.1 (C), 144.2 (C), 141.4 (C), 127.5 (CH), 126.9 (CH), 124.8 (CH), 119.9 (CH), 66.6 (CH₂), 47.53 (CH₂), 47.48 (CH), 47.1 (CH₂), 29.0 (CH₂), 28.3 (CH₂), 27.8 (CH₂), 22.5 (CH₂), 14.0 (CH₃). IR (CHCl₃) 2959.9, 2932.8, 1682.4, 1478.9, 1451.3, 1426.8, 1274.1, 1160.5, 1090.2. MS m/z (%) 179 (26), 178 (100), 165 (3), 133 (4), 71 (4).

N-Fmoc-piperidine 2e: Yellow solid (mp 96-99°C), TLC R_f 0.60 (cyclohexane:AcOEt 2:1). ¹H NMR (CDCl₃) δ 7.78 (d, J = 7.6 Hz, 2H), 7.60 (d, J = 8.1 Hz, 2H), 7.41 (t, J = 7.3 Hz, 2H), 7.33 (td, J = 7.5, 1.1 Hz, 2H), 4.41 (d, J = 7.0 Hz, 2H), 4.27 (t, J = 6.9 Hz, 1H), 3.46 (t, J = 5.6 Hz, 4H), 1.70-1.50 (m, 6H); ¹³C NMR (CDCl₃) δ 155.3 (C), 144.2 (C), 141.3 (C), 127.6 (CH), 127.0 (CH), 125.0 (CH), 119.9 (CH), 67.2 (CH₂), 47.4 (CH), 44.9 (CH₂), 25.7 (CH₂), 24.4 (CH₂). IR (CHCl₃) 3012.0, 2943.6, 2859.5, 1684.6, 1450.7, 1265.1, 1236.6, 1150.8. MS m/z (%) 307 (<1, M⁺), 178 (100), 165 (4), 112 (5), 84 (3), 69 (7); HR-MS 178.0759 (C₁₄H₁₀ calcd 178.0783).

N-Fmoc-dicyclohexylamine 2f: Yellow solid (mp 193-195°C), TLC R_f 0.57 (cyclohexane:AcOEt 2:1). ¹H NMR (CDCl₃) δ 7.76 (d, J = 7.5 Hz, 2H), 7.59 (d, J = 7.3 Hz, 2H), 7.39 (t, J = 7.4 Hz, 2H), 7.32 (td, J = 7.4, 1.1 Hz, 2H), 4.65 (d, J = 4.4 Hz, 2H), 4.23 (t, J = 4.7 Hz, 1H), 3.63-3.41 (1H), 3.08-2.85 (1H), 1.83-0.68 (20H); ¹³C NMR (CDCl₃) δ 155.8 (C), 144.4 (C), 141.5 (C), 127.4 (CH), 127.0 (CH), 124.5 (CH), 119.9 (CH), 65.6 (CH₂), 55.2 (CH), 47.5

(CH), 30.7 (CH₂), 26.2 (CH₂), 25.4 (CH₂). IR (neat) 2926.2, 2846.9, 1671.1, 1440.1, 1287.4, 1236.4, 1167.1, 1119.4, 1045.7, 741.9. MS *m/z* (%) 178 (100), 165 (5), 152 (5), 138 (5), 84 (18), 55 (13).

***N*-Fmoc-L-leucine allyl ester 2j**: Pale yellow solid (mp 72-76°C), TLC *R_f* 0.53 (cyclohexane:AcOEt 2:1). ¹H NMR (CDCl₃) δ 7.78 (d, *J* = 7.6 Hz, 2H), 7.61 (m, 2H), 7.41 (t, *J* = 7.5 Hz, 2H), 7.33 (t, *J* = 7.4 Hz, 2H), 5.93 (ddt, *J* = 16.8, 11.1, 5.6 Hz, 1H), 5.35 (d, *J* = 17.2 Hz, 1H), 5.27 (d, *J* = 10.4 Hz, 1H), 5.19 (d, *J* = 8.9 Hz, 1H), 4.65 (d, *J* = 5.4 Hz, 2H), 4.52-4.37 (3H), 4.24 (t, *J* = 7.0 Hz, 1H), 1.78-1.53 (3H), 1.03-0.83 (6H); ¹³C NMR (CDCl₃) δ 172.9 (C), 156.0 (C), 143.9 (C), 143.7 (C), 141.3 (C), 131.6 (CH), 127.7 (CH), 127.0 (CH), 125.0 (CH), 120.0 (CH), 119.95 (CH), 118.8 (CH₂), 66.9 (CH₂), 65.9 (CH₂), 52.5 (CH), 47.2 (CH), 41.8 (CH₂), 24.7 (CH), 22.9 (CH₃), 21.8 (CH₃). IR (neat) 3311.5, 2957.4, 1745.1, 1691.5, 1537.6, 1449.3, 1255.8, 1175.7, 1046.6, 986.6, 738.7. MS *m/z* (%) 355 (<1), 308 (<1), 214 (<1), 178 (100), 165 (24), 69 (5).

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PROCNO : 1

*** Processing Parameters ***

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Celine □ C13CPD CDCl3 u guest 2 □

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PROCNO : 1

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*** Processing Parameters ***

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PROCNO : 1

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*** Current Data Parameters ***

NAME : ch309g

EXPNO : 10

PROCNO : 1

*** Processing Parameters ***

SF : 400.1300170 MHz

*** Current Data Parameters ***

NAME : ch309g

EXPNO : 11

PROCNO : 1

*** Processing Parameters ***

SF : 125.7577960 MHz

— 151.2331

└─ 142.4387

└─ 140.9242

└─ 138.3516

└─ 137.6309

— 123.1711

— 120.3156

— 84.4078

└─ 77.2554

└─ 77.0000

└─ 76.7446

— 50.6895

└─ 27.9643

└─ 27.6724

PROTON CDCl₃ u guest 2

*** Current Data Parameters ***

NAME : ch397c

EXPNO : 10

PROCNO : 1

*** Processing Parameters ***

SF : 400.1300050 MHz

7.3912
7.3723
7.3609
7.3395
7.3312
7.3186
7.2700

5.1019

4.7420

3.2173
3.2002
3.1838
3.1674

1.5126
1.4968
1.4797
1.2613
0.9058
0.8888
0.8711

10a

C13CPD CDCl3 u guest 2

*** Current Data Parameters ***

NAME : ch397c

EXPNO : 11

PROCNO : 1

*** Processing Parameters ***

SF : 100.6127710 MHz

156.3488

136.6331

128.4897

128.0941

128.0529

77.3132

77.0000

76.6786

66.5488

41.0965

31.8898

29.9364

29.6149

29.5985

29.5490

29.5160

29.3182

29.2440

26.7136

22.6667

S21

10a

*** Current Data Parameters ***

NAME : ch393a~1

EXPNO : 10

PROCNO : 1

*** Processing Parameters ***

SF : 300.1300120 MHz

HELGEN C13CPD cdc13

*** Current Data Parameters ***

NAME : ch393a

EXPNO : 11

PROCNO : 1

*** Processing Parameters ***

SF : 125.7577970 MHz

155.5044

136.6934

128.4806

128.1009

128.0266

77.2476

77.0000

76.7441

66.4431

49.8689

33.3773

25.4616

24.7435

S23

*** Current Data Parameters ***

NAME : ch406a~1

EXPNO : 10

PROCNO : 1

*** Processing Parameters ***

SF : 400.1300050 MHz

7.3508
7.3331
7.2700

5.1467
5.1158
5.0842
5.0533
4.8967
4.8778
4.8626

1.5056
1.4886

*** Current Data Parameters ***

NAME : ch406a

EXPNO : 11

PROCNO : 1

*** Processing Parameters ***

SF : 125.7577940 MHz

— 155.4961

— 143.4287

136.4375

128.6209

128.4806

128.1092

127.3168

125.8971

77.2559

77.0000

76.7441

— 66.7072

— 50.6943

— 22.4324

*** Current Data Parameters ***

NAME : ch324a

EXPNO : 20

PROCNO : 1

*** Processing Parameters ***

SF : 400.1300180 MHz

*** Current Data Parameters ***

NAME : ch324a

EXPNO : 21

PROCNO : 1

*** Processing Parameters ***

SF : 100.6127750 MHz

10d

*** Current Data Parameters ***

NAME : ch346a

EXPNO : 10

PROCNO : 1

*** Processing Parameters ***

SF : 500.1300080 MHz

7.3721
7.3633
7.3324
7.3242
7.3154
7.2700

5.1337

3.4657
3.4544
3.4437 1.6156
1.6005
1.5929
1.5841
1.5810
1.5375 **10e**

Integral

9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5

(ppm)

*** Current Data Parameters ***

NAME : ch346a

EXPNO : 11

PROCNO : 1

*** Processing Parameters ***

SF : 125.7577930 MHz

155.3174

137.0115

128.4197

127.8440

127.7638

77.2551

77.0000

76.7449

66.8487

25.6605

24.3415

*** Current Data Parameters ***

NAME : ch418c

EXPNO : 10

PROCNO : 1

*** Acquisition Parameters ***

SOLVENT : CDCl₃

*** Processing Parameters ***

SF : 500.1300080 MHz

7.3813
7.3721
7.3630
7.3053
7.27005.1848
5.1403

3.6243

3.3337

1.7839
1.7594
1.6276
1.6027
1.5851
1.2989
1.2645
1.0852 **10f**

Celine HELGEN □ C13CPD CDC13 u guest 40 □

*** Current Data Parameters ***

NAME : ch418c

EXPNO : 11

PROCNO : 1

*** Processing Parameters ***

SF : 125.7577900 MHz

155.7273

137.2712
135.1581
128.5714
128.5219
128.3815
128.3155
127.6304

77.2559
77.0000
76.7441

69.7199
66.4183

55.4321

31.5284
30.6804
29.6877
29.3740
26.2623
25.4699

*** Current Data Parameters ***

NAME : ch420e~1

EXPNO : 10

PROCNO : 1

*** Processing Parameters ***

SF : 400.1300050 MHz

*** Current Data Parameters ***

NAME : ch420e

EXPNO : 11

PROCNO : 1

*** Processing Parameters ***

SF : 125.7577920 MHz

157.1552
156.9984
141.8604
136.7016
136.6026
128.4971
128.4641
128.2330
128.0844
127.9523
127.6965
127.5892
126.2355

77.2559
77.0000
76.7441
67.1034
66.6742
59.1795
56.7528

31.9988
30.2655
27.5747

14.5002
13.2456
12.1065

*** Current Data Parameters ***

NAME : ch425a

EXPNO : 10

PROCNO : 1

*** Processing Parameters ***

SF : 400.1300050 MHz

— 1.6521

— 1.2657

10i

*** Current Data Parameters ***

NAME : ch425a

EXPNO : 11

PROCNO : 1

*** Processing Parameters ***

SF : 100.6127710 MHz

*** Current Data Parameters ***

NAME : ch430c

EXPNO : 10

PROCNO : 1

*** Processing Parameters ***

SF : 400.1300050 MHz

*** Current Data Parameters ***

NAME : ch431d

EXPNO : 10

PROCNO : 1

*** Processing Parameters ***

SF : 400.1300050 MHz

7.3514
7.2700
7.0168
6.9960
6.7314
6.7106

5.2566
5.2364
5.1398
5.1088
5.0956
5.0646

4.5185
4.5040
4.4838
4.4686
4.3556

3.0594
3.0443
3.0241
3.0108
2.9975
2.9767
2.9622

1.6445
1.4191

*** Current Data Parameters ***

NAME : ch431d

EXPNO : 11

PROCNO : 1

*** Processing Parameters ***

SF : 100.6127700 MHz

7.3690
7.3658
7.3548
7.3453
7.3334
7.3274
7.2965
7.2832
7.2700
7.2615
7.2479
7.1307
7.1275
7.1146

5.9081
5.8961
5.8860
5.8750
5.8624
5.8517
5.8410
5.8293

5.3291
5.2948
5.2721
5.2516
5.2390
5.1879
5.1356
5.1110
5.1050
5.0798
4.7195
4.7073
4.6912
4.6792
4.6250
4.6133
4.5623

3.1843
3.1729
3.1569
3.1452
3.1313
3.1190
3.1036
3.0913

1.6692

*** Current Data Parameters ***

NAME : ch434a

EXPNO : 10

PROCNO : 1

*** Processing Parameters ***

SF : 500.1300080 MHz

*** Current Data Parameters ***

NAME : ch434a

EXPNO : 11

PROCNO : 1

*** Processing Parameters ***

SF : 125.7577940 MHz

*** Current Data Parameters ***

NAME : ch319c

EXPNO : 10

PROCNO : 1

*** Processing Parameters ***

SF : 500.1300093 MHz

*** Current Data Parameters ***

NAME : ch319c

EXPNO : 11

PROCNO : 1

*** Processing Parameters ***

SF : 125.7578486 MHz

151.8942

138.7664
136.2849

122.3999

47.7380
40.0839
39.9926
39.9196
39.8284
39.7554
39.6642
39.5821
39.4909
39.3266
39.1624
38.9891
25.3868

*** Current Data Parameters ***

NAME : ch389d

EXPNO : 10

PROCNO : 1

*** Processing Parameters ***

SF : 500.1300080 MHz

7.2700

4.4926

3.1165
3.1033

1.5936
1.4498
1.2841
1.2620
0.8882

11a

*** Current Data Parameters ***

NAME : ch389d

EXPNO : 21

PROCNO : 1

*** Processing Parameters ***

SF : 100.6127670 MHz

— 155.9861

78.9782
77.3132
77.0000
76.6786 40.6514
31.9063
30.0930
29.6314
29.6149
29.5655
29.3265
29.2935
28.4363
26.8126
22.6667
14.0782

1H Spectrum

*** Current Data Parameters ***

NAME : ch419c~1

EXPNO : 10

PROCNO : 1

*** Processing Parameters ***

SF : 400.1300050 MHz

S46

11b

*** Current Data Parameters ***

NAME : ch419c

EXPNO : 13

PROCNO : 1

*** Processing Parameters ***

SF : 125.7577890 MHz

155.1990

78.9645
77.2559
77.0000
76.7441

49.4232

33.5423

28.4331

25.5524

24.8921

*** Current Data Parameters ***

NAME : ch426f

EXPNO : 10

PROCNO : 1

*** Processing Parameters ***

SF : 400.1300050 MHz

7.3603
7.3413
7.3243
7.3148
7.2978
7.2700
7.2548
7.2378

4.8020

1.6344
1.6111
1.4671
1.4513
1.4317

11c

*** Current Data Parameters ***

NAME : ch426f

EXPNO : 11

PROCNO : 1

*** Processing Parameters ***

SF : 100.6127710 MHz

— 155.0630

— 143.7463

— 128.5392

— 127.0968

— 125.8522

— 79.4150

— 77.3214

— 77.0000

— 76.6868

— 50.1878

— 28.3703

— 22.6667

*** Current Data Parameters ***

NAME : ch432c

EXPNO : 10

PROCNO : 1

*** Processing Parameters ***

SF : 400.1300050 MHz

— 173.1961

— 155.4091

— 131.7207

— 118.5578

79.8271
77.3215
77.0000
76.6868

— 65.7080

— 52.1329

— 41.8383

— 28.3044
— 24.7849
— 22.8315
— 21.8754

*** Current Data Parameters ***

NAME : ch432c

EXPNO : 11

PROCNO : 1

*** Processing Parameters ***

SF : 100.6127690 MHz

Celine PROTON CDCl3 u guest 8

7.7852
7.7663
7.6179
7.6160
7.5989
7.5977
7.4278
7.4095
7.3919
7.3432
7.3401
7.3243
7.3211
7.3060
7.3028
7.2700

6.0900

4.7344

4.4263
4.4093
4.2508
4.2338
4.2173

3.2223
3.2078
3.1914
3.1762

1.5126

1.2790

0.9140
0.8976
0.8799

*** Current Data Parameters ***

NAME : ch390d

EXPNO : 20

PROCNO : 1

*** Acquisition Parameters ***

SOLVENT : CDCl3

*** Processing Parameters ***

SF : 400.1300050 MHz

12a

Integral

Celine C13CPD CDCl3 u guest 8

*** Current Data Parameters ***

NAME : ch390d

EXPNO : 21

PROCNO : 1

*** Acquisition Parameters ***

SOLVENT : CDCl3

*** Processing Parameters ***

SF : 100.6127680 MHz

156.4065

144.0677

141.3477

127.6325

127.0061

125.0197

119.9425

77.3132

77.0000

76.6786

66.4993

47.3689

41.1377

31.9063

30.0023

29.6397

29.6149

29.5820

29.5490

29.3265

29.2852

26.7466

22.6667

14.0782

S53

12a

7.7871
7.7688
7.6173
7.5983
7.4304
7.4121
7.3944
7.3483
7.3451
7.3293
7.3262
7.3110
7.3079
7.2700

4.6707
4.6542
4.4086
4.3922
4.2458
4.2281
4.2123

3.5235
3.5134
3.5039

1.9716
1.9463
1.7392
1.7058
1.6426
1.6041
1.3806
1.3490
1.3187
1.2689
1.1868
1.1634
1.1401

*** Current Data Parameters ***

NAME : ch427d

EXPNO : 10

PROCNO : 1

*** Processing Parameters ***

SF : 400.1300050 MHz

*** Current Data Parameters ***

NAME : ch427d

EXPNO : 11

PROCNO : 1

*** Processing Parameters ***

SF : 100.6127710 MHz

— 155.5657

— 144.0512

— 141.3065

— 127.6160

— 126.9896

— 125.0197

— 119.9260

— 77.3215

— 77.0000

— 76.6868

— 66.3427

— 49.8993

— 47.3277

— 33.4229

— 25.4691

— 24.8179

7.7808
7.7618
7.6103
7.5920
7.4234
7.4045
7.3862
7.3407
7.3376
7.3218
7.3192
7.3035
7.3003
7.2700

4.5027
4.4876
4.2552
4.2401
4.2249

3.2128
3.1945
3.1768
3.0556
3.0373
3.0184

1.6363
1.5069
1.4898
1.3415
1.3244
1.3074
1.2689
1.2537
1.1293
1.1116
0.9147
0.8963
0.8787

*** Current Data Parameters ***

NAME : ch398d

EXPNO : 10

PROCNO : 1

*** Processing Parameters ***

SF : 400.1300050 MHz

12d

*** Current Data Parameters ***

NAME : ch398d

EXPNO : 21

PROCNO : 1

*** Processing Parameters ***

SF : 100.612720 MHz

156.1097

144.2325

141.3725

127.5171

126.9319

124.7972

119.8600

77.3132

77.0000

76.6785

66.5652

47.4843

47.0474

28.9720

28.3044

27.7769

22.4524

14.0370

12d

*** Current Data Parameters ***

NAME : ch392d

EXPNO : 11

PROCNO : 1

*** Acquisition Parameters ***

SOLVENT : CDC13

*** Processing Parameters ***

SF : 125.7577920 MHz

155.2650

144.1881

141.3322

127.5974

126.9866

125.0304

119.9376

77.2559

77.0000

76.7524

67.1529

47.4174

44.8834

25.6762

24.3803

7.7674
7.7525
7.6671
7.5965
7.5820
7.5316
7.4979
7.4021
7.3872
7.3724
7.3356
7.3334
7.3207
7.3185
7.3059
7.3037
7.2700

4.6581
4.6493

4.2389
4.2294
4.2203

3.5323

2.9545

1.7143
1.5838
1.4877
1.4366
1.2639
1.1797
1.0253

*** Current Data Parameters ***

NAME : ch428e

EXPNO : 10

PROCNO : 1

*** Processing Parameters ***

SF : 500.1300080 MHz

*** Current Data Parameters ***

NAME : ch428e

EXPNO : 11

PROCNO : 1

*** Processing Parameters ***

SF : 125.7577900 MHz

*** Current Data Parameters ***

NAME : ch435a

EXPNO : 10

PROCNO : 1

*** Processing Parameters ***

SF : 500.1300080 MHz

172.8483
155.9460
143.9136
143.7238
141.2909
131.5656
127.6717
127.0341
125.0634
119.9624
119.9459
118.7821

77.2538
77.0000
76.7462

66.9424
65.8838

52.4978

47.1677

41.7902

24.7332
22.8533
21.7906

*** Current Data Parameters ***

NAME : ch435a

EXPNO : 11

PROCNO : 1

*** Processing Parameters ***

SF : 125.7577930 MHz