Supporting Information for:

Room Temperature Highly Diastereoselective Zn-Mediated Allylation of Chiral *N*-*tert*-Butanesulfinyl Imines: Remarkable Reaction Condition Controlled Stereoselectivity Reversal

Xing-Wen Sun, Ming-Hua Xu,* and Guo-Qiang Lin*

*Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, 354 Fenglin Road,
Shanghai 200032, and Shanghai Institute of Materia Medica, Chinese Academy of Sciences, 555
Zuchongzhi Road, Shanghai 201203, China.*

xumh@mail.sioc.ac.cn

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1. General Procedure for the Allylation of Chiral *N*-*tert*-Butanesulfinyl Aldimines in THF System.

Under argon, to a suspension of activated zinc powder (32 mg, 0.5 mmol), In(OTf)₃ (154 mg, 0.275 mmol) and (*R*)-*N*-*tert*-butanesulfinyl imines (0.25 mmol) in freshly distilled dry THF (5 mL) was added allyl bromide (0.04 mL, 0.5 mmol) at room temperature. The resultant mixture was then stirred at rt for 10 hours. The reaction was quenched with 5 mL of saturated aqueous NaCl. Extraction with ethyl acetate, dried over anhydrous Na₂SO₄. After concentrated, the residue was purified by flash column chromatography to afford the desired allylation product.

2. General Procedure for the Allylation of Chiral *N*-*tert*-Butanesulfinyl Aldimines in HMPA System.

Under argon, to a suspension of activated zinc powder (32 mg, 0.5 mmol), (*R*)-*N*-*tert*-butanesulfinyl imines (0.25 mmol) in dry HMPA (5 mL) was added 10 μL of H₂O and allyl bromide (0.04 mL, 0.5 mmol) at room temperature. The resultant mixture was stirred at rt for 12 hours. The reaction was quenched with 5 mL of 1N aqueous HCl. The solution was extracted with ethyl acetate, washed with brine and dried over anhydrous Na₂SO₄. After concentrated, the residue was purified by flash column chromatography to afford the desired allylation product.

[Caution]: HMPA is a suspected human carcinogen.

3. General Procedure for the Allylation of Chiral *N*-*tert*-Butanesulfinyl Ketimines in THF System.

Under argon, to a suspension of activated zinc powder (48 mg, 0.75 mmol), In(OTf)₃ (183 mg, 0.325 mmol) and (*R*)-*N*-*tert*-butanesulfinyl imines (0.25 mmol) in freshly distilled dry THF (5 mL) was added allyl bromide (0.06 mL, 0.75 mmol) at room temperature. The resultant mixture was then stirred at rt for 24 hours. The

reaction was quenched with 5 mL of saturated aqueous NaCl. Extraction with ethyl acetate, dried over anhydrous Na₂SO₄. After concentrated, the residue was purified by flash column chromatography to afford the desired allylation product.

4. Determination of the Diastereoselectivity / Enantiomeric Excess.

The diastereoselectivity of the allylation product was determined according to ¹H NMR of the crude materials. In the case of product **2a**, the enantiomeric excess was further measured by chiral HPLC of its acetate derivative to ensure the accuracy.

HPLC: Daicel Chiralpak AD column, detected at 254 nm, eluent: *n*-hexane/*iso*-propanol 9:1.

¹H NMR (300 MHz, CDCl₃): δ 1.92 (s, 9H), 2.52 (t, 3H, J = 6.9 Hz), 5.00-5.10 (m, 3H), 5.60-5.74 (m, 1H), 6.52 (d, 1H, J = 7.5 Hz), 7.20-7.33 (m, 5H) ppm; ESI-MS (m/z, %): 190.2 (M⁺+H).

5. The Configuration Assignment

The configuration of the allylation product was assigned by comparison with literature data for the free homoallylic amines:

a)

Lit: Yanada, R.; Negoro, N.; Okaniwa, M.; Ibuka, T.; *Tetrahedron*; **1999**, 55, 13947.

b)

Lit: Hua, D. H.; Miao, S. W.; Chen, J. S.; Iguchi, S. *J. Org. Chem.* **1991**, 56, 4.

6. The Influence of the Amount of H₂O in HMPA System.

entry ^a	solvent ^b	time (h)	yield (%) ^c	dr ^d
1	HMPA	1	99	26:74
2	HMPA + 5 μL H ₂ O	12	98	3:97
3	HMPA + 10 μL H ₂ O	12	97	1:99
4	HMPA + 15 μL H ₂ O	14	98	2:98
5	HMPA + 20 μL H ₂ O	16	95	2:98
6	HMPA + 25 μL H ₂ O	18	88	3:97
7	HMPA + 50 μL H ₂ O	18	60	3:97
8	HMPA + 100 μL H ₂ O	18	43	2:98

^a Reactions were carried out with 0.25 mmol of imine **1a**, 0.5 mmol of Zn/allyl bromide, and H₂O (additive) in 5 mL of dry solvent at rt. ^b HMPA was first distilled under vacuum from CaH₂, then distilled from sodium carefully, see *Purification of Laboratory Chemicals*, 5th Edition, Elsevier, **2002**, 428. ^c isolated yield. ^d determined by ¹H NMR of the crude materials.

7. Characterization Data of the Obtained Enantioenriched Homoallylic Amines.

$[\alpha]_D^{26}$ -119.8 (*c* 1.90, CHCl₃) ; ¹H NMR (400 MHz, CDCl₃): δ 1.19 (s, 9H), 2.44-2.52 (m, 1H), 2.57-2.63 (m, 1H), 3.69 (s, 1H), 4.477 (ddd, 1H, *J* = 2.35, 5.47, 8.00 Hz), 5.15-5.20(m, 2H), 5.68-5.74 (m, 1H), 7.27-7.33 (m, 5H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 22.51, 43.30, 55.53, 57.06, 119.14, 127.40, 127.55, 128.36, 134.07, 141.64 ppm; FT-IR (film, cm⁻¹) ν 3033, 2927, 2869, 2235, 1640, 1456, 1058, 911, 734, 701; ESI-MS (*m/z*, %): 252.2 (M⁺+H), 274.2 (M⁺+Na).; HRMS (EI) for C₁₄H₂₁NOS: calcd. 251.1344, found 251.1348.

$[\alpha]_D^{26}$ -40.3 (*c* 2.46, CHCl₃); ¹H NMR (300 MHz, CDCl₃): δ 1.22 (s, 9H), 2.50-2.60 (m, 1H), 2.70-2.78 (m, 1H), 3.52 (s, 1H), 4.45(d, 1H, *J* = 1.8 Hz), 5.00-5.06 (m, 2H), 5.611 (dt, 1H, *J* = 7.2, 24.3 Hz), 7.28-7.33 (m, 5H) ppm; ¹³C NMR (75 MHz, CDCl₃): δ 22.66, 41.23, 55.97, 58.52, 118.14, 127.23, 127.82, 128.61, 133.82, 141.90 ppm; ESI-MS (*m/z*, %): 252.2 (M⁺+H), 274(M⁺+Na).

$[\alpha]_D^{26}$ -124.6 (*c* 1.52, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 1.19 (s, 9H), 2.42-2.49 (m, 1H), 2.53-2.60 (m, 1H), 3.69 (s, 1H), 4.45 (ddd, 1H, *J* = 1.89, 5.57, 7.81 Hz), 5.15-5.19(m, 2H), 5.66-5.74 (m, 1H), 7.02 (t, 2H, *J* = 8.67Hz), 7.29 (dt, 2H, *J* = 2.09, 5.32 Hz) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 22.49, 43.32, 55.54, 56.33, 115.17, 115.38, 119.32, 129.00, 129.08, 133.84, 137.29, 137.32, 160.94, 163.38 ppm; FT-IR (film, cm⁻¹) ν 3428, 3206, 3078, 2959, 2937, 2870, 1641, 1605, 1511, 1058, 836, 795; ESI-MS (*m/z*, %): 270.2 (M⁺+H), 292.2 (M⁺+Na); HRMS (EI) for C₁₄H₂₀NOS: calcd. 269.1250, found 269.1259.

$[\alpha]_D^{26}$ -39.6 (*c* 2.65, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 1.22 (s, 9H), 2.48-2.56 (m, 1H), 2.69-2.75 (m, 1H), 3.49 (d, 1H, *J* = 3.39 Hz), 4.44 (dd, 1H, *J* = 5.99, 11.6 Hz), 5.02-5.06 (m, 2H), 5.55-5.64 (m, 1H), 7.03 (t, 2H, *J* = 8.6 Hz), 7.30 (dt, 2H, *J* = 2.87, 8.15 Hz) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 22.54, 41.22, 55.93, 57.89, 115.30, 115.51, 118.34, 128.78, 128.86, 133.48, 137.59, 137.62, 160.96, 163.41 ppm; ESI-MS (*m/z*, %): 270.2 (M⁺+H), 292.2 (M⁺+Na).

$[\alpha]_D^{26}$ -129.1 (c 2.30, CHCl_3); ^1H NMR (400 MHz, CDCl_3): δ 1.19 (s, 9H), 2.29-2.49 (m, 1H), 2.54-2.60 (m, 1H), 3.67 (s, 1H), 4.45 (ddd, 1H, $J = 2.04, 5.53, 7.86$ Hz), 5.14-5.19 (m, 2H), 5.67-5.72 (m, 1H), 7.24-7.32 (m, 5H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 22.52, 43.22, 55.64, 55.41, 119.51, 128.63, 128.84, 133.35, 133.69, 140.20 ppm; FT-IR (KBr, cm^{-1}) ν 3201, 3079, 2981, 2956, 2867, 1489, 1408, 1900, 827, 647; ESI-MS (m/z , %): 286.2 ($\text{M}^+ + \text{H}$); HRMS (EI) for $\text{C}_{14}\text{H}_{20}\text{ClNOS}$: calcd. 285.0954, found 285.0949.

$[\alpha]_D^{26}$ -24.3 (c 3.10, CHCl_3); ^1H NMR (400 MHz, CDCl_3): δ 1.22 (s, 9H), 2.50-2.55 (m, 1H), 2.68-2.74 (m, 1H), 3.50 (d, 1H, $J = 4.00$ Hz), 4.43 (ddd, 1H, $J = 0.63, 2.97, 8.9$ Hz), 5.01-5.06 (m, 2H), 5.55-5.63 (m, 1H), 7.25-7.33 (m, 4H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 22.55, 41.13, 56.00, 57.91, 118.51, 128.57, 128.72, 133.31, 133.50, 140.35 ppm; ESI-MS (m/z , %): 286.2 ($\text{M}^+ + \text{H}$).

$[\alpha]_D^{26}$ -94.5 (c 2.75, CHCl_3); ^1H NMR (400 MHz, CDCl_3): δ 1.19 (s, 9H), 2.43-2.48 (m, 1H), 2.53-2.58 (m, 1H), 3.68 (d, 1H, $J = 1.22$ Hz), 4.43 (ddd, 1H, $J = 2.06, 5.55, 7.87$ Hz), 5.15-5.19 (m, 2H), 5.65-5.73 (m, 1H), 7.20 (dd, 2H, $J = 1.67, 6.63$ Hz), 7.46 (dd, 2H, $J = 1.86, 6.59$ Hz) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 22.47, 43.11, 55.59, 55.43, 119.47, 121.41, 129.15, 131.52, 133.60, 140.69 ppm; FT-IR (KBr, cm^{-1}) ν 3198, 3077, 2923, 1643, 1484, 1055, 842, 779; ESI-MS (m/z , %): 330.1 ($\text{M}^+ + \text{H}$); HRMS (EI) for $\text{C}_{14}\text{H}_{20}\text{BrNOS}$: calcd. 329.0449, found 329.0441.

$[\alpha]_D^{26}$ -21.7 (c 3.95, CHCl_3); ^1H NMR (400 MHz, CDCl_3): δ 1.21 (s, 9H), 2.50-2.55 (m, 1H), 2.67-2.74 (m, 1H), 3.54 (d, 1H, $J = 4.33$ Hz), 4.41 (dd, 1H, $J = 5.96, 11.95$ Hz), 5.02-5.06 (m, 2H), 5.55-5.63 (m, 1H), 7.21 (dd, 2H, $J = 1.63, 6.69$ Hz), 7.47 (dd, 2H, $J = 1.88, 6.61$ Hz) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 22.52, 41.05, 56.00, 57.96, 118.51, 121.59, 128.88, 131.64, 133.25, 140.84 ppm; ESI-MS (m/z , %): 330.1 ($\text{M}^+ + \text{H}$).

$[\alpha]_D^{26}$ -139.3 (c 1.38, CHCl_3); ^1H NMR (400 MHz, CDCl_3): δ 1.19 (s, 9H), 2.34 (s, 3H), 2.44-2.50 (m, 1H), 2.55-2.60 (m, 1H), 3.66 (s, 1H), 4.43 (ddd, 1H, $J = 2.16, 5.44, 7.9$ Hz), 5.14-5.20 (m, 2H), 5.68-5.74 (m, 1H), 7.14 (d, 2H, $J = 7.9$ Hz), 7.18 (d, 2H, $J = 8.1$ Hz) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ

21.09, 22.56, 43.41, 55.54, 56.81, 119.07, 127.37, 129.13, 134.28, 137.26, 138.64 ppm; FT-IR (film, cm^{-1}) ν 3204, 2976, 2863, 1643, 1053, 816; ESI-MS (m/z , %): 266.2 ($M^+ + H$), 288.2 ($M^+ + Na$); HRMS (EI) for $C_{15}H_{23}NOS$: calcd. 265.1500, found 265.1512.

$[\alpha]_D^{26}$ -45.3 (c 2.27, $CHCl_3$); 1H NMR (400 MHz, $CDCl_3$): δ 1.21 (s, 9H), 2.33 (s, 3H), 2.50-2.57 (m, 1H), 2.69-2.75 (m, 1H), 3.47 (d, 1H, $J = 3.61$ Hz), 4.39-4.43 (m, 1H), 5.00-5.06 (m, 2H), 5.57-5.67 (m, 1H), 7.14 (d, 2H, $J = 7.99$ Hz), 7.21 (d, 2H, $J = 8.1$ Hz) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.02, 22.58, 41.10, 55.83, 58.24, 117.94, 127.07, 129.23, 133.90, 137.43, 138.82 ppm; ESI-MS (m/z , %): 266.2 ($M^+ + H$), 288.2 ($M^+ + Na$).

$[\alpha]_D^{26}$ -85.6 (c 1.85, $CHCl_3$); 1H NMR (400 MHz, $CDCl_3$): δ 1.18 (s, 9H), 2.44-2.60 (m, 2H), 3.65 (d, 1H, $J = 1.19$ Hz), 3.80 (s, 3H), 4.41 (ddd, 1H, $J = 2.08, 5.58, 7.91$ Hz), 5.14-5.19 (m, 2H), 5.69-5.74 (m, 1H), 6.87 (dd, 2H, $J = 2.01, 6.7$ Hz), 7.24 (dd, 2H, $J = 2.01, 6.81$ Hz) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 22.55, 43.38, 55.14, 55.46, 56.49, 137.79, 118.99, 128.58, 133.56, 134.28, 159.03 ppm; FT-IR (KBr, cm^{-1}) ν 3450, 3283, 3216, 3076, 2958, 2869, 1612, 1514, 7247, 1056, 832; ESI-MS (m/z , %): 282.2 ($M^+ + H$), 304.2 ($M^+ + Na$); HRMS (EI) for $C_{15}H_{23}NO_2S$: calcd. 281.1449, found 281.1452.

$[\alpha]_D^{26}$ -37.0 (c 2.28, $CHCl_3$); 1H NMR (400 MHz, $CDCl_3$): δ 1.21 (s, 9H), 2.50-2.56 (m, 1H), 2.70-2.74 (m, 1H), 3.43 (d, 1H, $J = 3.36$ Hz), 3.79 (s, 3H), 4.38-4.42 (m, 1H), 5.00-5.06 (m, 2H), 5.56-5.65 (m, 1H), 6.86-6.89 (m, 2H), 7.25 (dd, 2H, $J = 2.03, 6.71$ Hz) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 22.60, 41.12, 55.19, 55.80, 57.94, 113.93, 117.96, 128.35, 133.91, 133.95, 159.12 ppm. ESI-MS (m/z , %): 282.2 ($M^+ + H$).

$[\alpha]_D^{26}$ -110.5 (c 3.45, $CHCl_3$); 1H NMR (400 MHz, $CDCl_3$): δ 1.21 (s, 9H), 2.45-2.52 (m, 1H), 2.66-2.73 (m, 1H), 3.72 (d, 1H, $J = 2.68$ Hz), 5.01 (ddd, 1H, $J = 3.45, 5.15, 8.28$ Hz), 5.17-5.21 (m, 2H), 5.72-5.78 (m, 1H), 7.20-7.28 (m, 2H), 7.35-7.42 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 22.54, 42.37, 53.70, 55.80, 119.44, 126.73, 128.46, 129.74, 133.14, 133.72, 133.44, 139.21 ppm; FT-IR (film, cm^{-1}) ν 3219, 3073, 2924, 2866, 1640, 1573, 1474, 1440, 1364, 915, 754, 648; ESI-MS (m/z , %): 286.2 ($M^+ + H$); HRMS (EI) for $C_{14}H_{20}ClNOS$: calcd. 285.0954, found 285.0961.

$[\alpha]_D^{26}$ -10.9 (c 2.35, CHCl_3); ^1H NMR (400 MHz, CDCl_3): δ 1.21 (s, 9H), 2.60-2.69 (m, 2H), 3.78 (d, 1H, J = 6.05 Hz), 4.92 (dd, 1H, J = 6.35, 12.68 Hz), 5.06-5.11 (m, 2H), 5.63-5.71 (m, 1H), 7.22 (dd, 1H, J = 1.73, 7.52 Hz), 7.27 (dd, 1H, J = 1.38, 7.83 Hz), 7.35 (dd, 1H, J = 1.32, 7.86 Hz), 7.42 (dd, 1H, J = 1.55, 7.69 Hz) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 22.54, 40.23, 55.17, 56.29, 118.60, 126.94, 128.46, 128.66, 129.80, 132.80, 133.44, 139.30 ppm; ESI-MS (m/z , %): 286.2 (M^+ +H).

$[\alpha]_D^{26}$ -127.5 (c 2.20, CHCl_3); ^1H NMR (400 MHz, CDCl_3): δ 1.19 (s, 9H), 2.40 (s, 3H), 2.44-2.49 (m, 1H), 2.56-2.60 (m, 1H), 3.67 (d, 1H, J = 1.46 Hz), 4.74 (ddd, 1H, J = 2.56, 5.48, 8.14 Hz), 5.15-5.22 (m, 2H), 5.73-5.75 (m, 1H), 7.14-7.19 (m, 3H), 7.33-7.35 (m, 1H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 19.36, 22.54, 42.25, 52.62, 55.57, 119.10, 126.05, 126.84, 127.20, 130.46, 134.23, 135.80, 139.54 ppm; FT-IR (film, cm^{-1}) ν 3216, 3075, 2924, 2866, 1639, 1605, 1463, 1364, 1180, 1064, 911, 765, 727; ESI-MS (m/z , %): 266.2 (M^+ +H), 288.2 (M^+ +Na); HRMS (EI) for $\text{C}_{15}\text{H}_{22}\text{NNaOS}$: calcd. 288.1398, found 288.1406.

$[\alpha]_D^{26}$ -59.7 (c 1.98, CHCl_3); ^1H NMR (400 MHz, CDCl_3): δ 1.21 (s, 9H), 2.38 (s, 3H), 2.56-2.61 (m, 1H), 2.68-2.75 (m, 1H), 3.44 (d, 1H, J = 2.85 Hz), 4.69-4.74 (m, 1H), 5.01-5.08 (m, 2H), 5.58-5.66 (m, 1H), 7.16-7.34 (m, 3H), 7.36 (d, 1H, J = 1.19 Hz) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 19.39, 22.58, 40.55, 53.60, 55.78, 118.14, 126.25, 126.27, 127.46, 130.61, 133.80, 135.59, 139.78 ppm; ESI-MS (m/z , %): 266.2 (M^+ +H).

$[\alpha]_D^{26}$ -123.2 (c 2.95, CHCl_3); ^1H NMR (400 MHz, CDCl_3): δ 1.20 (s, 9H), 2.43-2.49 (m, 1H), 2.55-2.61 (m, 1H), 3.68 (s, 1H), 4.44 (ddd, 1H, J = 2.08, 5.38, 7.86 Hz), 5.16-5.20 (m, 2H), 5.67-5.74 (m, 1H), 7.20-7.24 (m, 2H), 7.40 (dd, 1H, J = 1.89, 3.31 Hz), 7.48 (dd, 1H, J = 0.3, 1.86 Hz) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 22.52, 43.21, 55.74, 56.49, 119.66, 122.54, 126.17, 130.01, 130.49, 130.71, 133.58, 144.18 ppm; FT-IR (film, cm^{-1}) ν 3281, 3201, 3076, 2957, 2923, 2867, 1604, 1570, 1056, 996, 916, 784, 696; ESI-MS (m/z , %): 330.1 (M^+ +H); HRMS (EI) for $\text{C}_{14}\text{H}_{20}\text{BrNOS}$: calcd. 329.0449, found 329.0456.

[α]_D²⁶ -23.6 (c 3.79, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 1.22 (s, 9H), 2.49-2.56 (m, 1H), 2.67-2.74 (m, 1H), 3.54 (d, 1H, J = 3.38 Hz), 4.41 (dd, 1H, J = 5.93, 10.93 Hz), 5.25-5.07 (m, 2H), 5.55-5.64 (m, 1H), 7.19-7.28 (m, 2h), 7.40-7.46 (m, 1H), 7.47 (s, 1H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 22.55, 41.11, 56.05, 57.99, 118.59, 122.60, 125.96, 130.12, 130.87, 133.19, 144.22 ppm; ESI-MS (m/z, %): 330.1 (M⁺+H).

[α]_D²⁶ -111.0 (c 1.51, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 1.19 (s, 9H), 2.53-2.60 (m, 1H), 2.64-2.69 (m, 1H), 3.76 (d, 1H, J = 1.47 Hz), 4.64 (ddd, 1H, J = 2.01, 5.59, 7.88 Hz), 5.15-5.22 (m, 2H), 5.72-5.76 (m, 1H), 7.43-7.47 (m, 3H), 7.77-7.83 (m, 4H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 22.51, 43.09, 55.53, 57.24, 119.20, 124.97, 125.88, 126.10, 126.79, 127.59, 127.84, 128.25, 132.95, 133.17, 134.03, 138.90 ppm; FT-IR (KBr, cm⁻¹) ν 3254, 3057, 2980, 2962, 2924, 2866, 1366, 1053, 925, 827, 756, 650, 598; ESI-MS (m/z, %): 302.2 (M⁺+H), 324.2 (M⁺+Na).; HRMS (EI) for C₁₈H₂₃NOS: calcd. 301.1500, found 301.1506.

[α]_D²⁶ -48.7 (c 1.03, CHCl₃); ¹H NMR (500 MHz, CDCl₃): δ 1.22 (s, 9H), 2.63-2.68 (m, 1H), 2.80-2.85 (m, 1H), 3.63 (d, 1H, J = 3.73 Hz), 4.59 (m, 1H), 5.00-5.08 (m, 2H), 5.60-5.68 (m, 1H), 7.44-7.49 (m, 3H), 7.77 (s, 1H), 7.79-7.83 (m, 3H) ppm; ¹³C NMR (125 MHz, CDCl₃): δ 22.59, 41.00, 55.97, 58.58, 118.21, 125.00, 126.01, 126.18, 126.22, 127.60, 127.96, 128.46, 132.98, 133.19, 133.72, 139.12 ppm; ESI-MS (m/z, %): 302.2 (M⁺+H), 324.2 (M⁺+Na).

[α]_D²⁶ -26.2 (c 0.45, CHCl₃); ¹H NMR (300 MHz, CDCl₃): δ 0.10-0.34 (m, 1H), 0.40-0.595 (m, 3H), 0.68-0.80 (m, 1H), 0.88-0.97 (m, 1H), 1.15 (s, 9H), 2.24-2.62 (m, 3H), 3.35 (d, 1H, J = 3.0 Hz), 5.07-5.12 (m, 2H), 5.71-5.85 (m, 1H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 4.21, 4.73, 16.08, 22.65, 41.29, 55.48, 58.77, 118.54, 134.61 ppm; FT-IR (film, cm⁻¹) ν 3223, 3078, 2979, 2924, 2668, 1641, 1473, 1363, 1060, 909, 822; ESI-MS (m/z, %): 216.2 (M⁺⁺H); HRMS (EI) for C₁₁H₂₁NOS: calcd. 215.1344, found 215.1353.

$[\alpha]_D^{26}$ -117.4 (c 1.55, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 0.28-0.33 (m, 1H), 0.36-0.41 (m, 1H), 0.55-0.65 (m, 2H), 0.88-0.97 (m, 1H), 1.21 (s, 9H), 2.35-2.45 (m, 2H), 2.45-2.55 (m, 1H), 3.24 (d, 1H, J = 3.73 Hz), 5.06-5.13 (m, 2H), 5.79-5.94 (m, 1H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 4.15, 4.68, 16.87, 22.60, 40.28, 55.61, 60.88, 117.50, 134.44 ppm; ESI-MS (m/z, %): 216.2 (M⁺+H).

$[\alpha]_D^{26}$ -29.5 (c 3.83, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 1.07-1.26 (m, 14H), 1.38-1.56 (m, 1H), 1.66-1.77 (m, 5H), 2.25-2.45 (m, 2H), 3.12-3.19 (m, 2H), 5.13-5.17 (m, 2H), 5.74-5.83 (m, 1H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 22.70, 26.20, 26.24, 28.52, 29.05, 29.60, 37.14, 41.20, 55.88, 59.64, 118.62, 134.61 ppm; FT-IR (film, cm⁻¹) ν 3244, 3071, 3977, 2925, 2853, 1449, 1055, 909, 505; ESI-MS (m/z, %): 258.2 (M⁺⁺+H); HRMS (EI) for C₁₄H₂₇NOS: calcd. 257.1813, found 257.1824.

$[\alpha]_D^{26}$ -46.0 (c 2.65, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 1.01-1.28 (m, 15H), 1.54-1.58 (m, 1H), 1.65-1.79 (m, 5H), 2.21-2.34 (m, 2H), 3.10-3.19 (m, 2H), 5.04-5.09 (m, 2H), 5.73-5.82 (m, 1H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 22.66, 26.16, 26.18, 26.34, 28.81, 28.89, 37.12, 41.95, 55.96, 60.93, 117.12, 135.26 ppm; ESI-MS (m/z, %): 258.2 (M⁺+H).

$[\alpha]_D^{26}$ -18.2 (c 0.45, CHCl₃); ¹H NMR (500 MHz, CDCl₃): δ 0.92 (t, 3H, J = 7.41 Hz), 1.21 (s, 9H), 1.56-1.64 (m, 2H), 2.21-2.28 (m, 1H), 2.21-2.34 (m, 1H), 2.35-2.43 (m, 1H), 3.23-3.29 (m, 1H), (d, 1H, J = 5.87 Hz), 3.24 (dt, 1H, J = 6.26, 12.44 Hz), 5.15-5.19 (m, 2H), 5.75-5.83 (m, 1H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 9.81, 22.68, 27.68, 39.88, 55.77, 56.05, 118.79, 134.29 ppm; FT-IR (film, cm⁻¹) ν 3236, 3076, 2958, 2927, 2871, 1640, 1457, 1364, 1055, 910; ESI-MS (m/z, %): 204.2 (M⁺+H); HRMS (EI) for C₁₀H₂₁NOS: calcd. 203.1344, found 203.1329.

$[\alpha]_D^{26}$ -53.8 (c 1.61, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ .97 (t, 3H, J = 7.42 Hz), 1.21 (s, 9H), 1.62 (dt, 2H, J = 7.37, 13.97Hz), 2.17-2.37 (m, 2H), 3.09 (d, 1H, J = 5.87 Hz), 3.24 (dt, 1H, J = 6.26, 12.44 Hz), 5.07-5.12 (m, 2H), 5.71-5.85 (m, 1H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 10.06, 22.64, 28.46, 39.29, 55.80, 57.30, 117.66, 134.45 ppm; ESI-MS (m/z, %): 204.2 (M⁺+H).

$[\alpha]_D^{26}$ -50.5 (c 1.56, CHCl_3); ^1H NMR (500 MHz, CDCl_3): δ 0.89 (d, 6H, $J = 10.01$ Hz), 1.22 (s, 9H), 1.86-1.90 (m, 1H), 2.26-2.32 (m, 1H), 2.40-2.36 (m, 1H), 3.14-3.19 (m, 2H), 5.14-5.17 (m, 2H), 5.76-5.84 (m, 1H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 17.82, 18.40, 31.01, 37.00, 55.94, 59.99, 118.61, 134.73 ppm; FT-IR (film, cm^{-1}) ν 3234, 3076, 2956, 2926, 2871, 1640, 1465, 1389, 1056, 908; ESI-MS (m/z , %): 218.2 ($\text{M}^+ + \text{H}$); HRMS (EI) for $\text{C}_{11}\text{H}_{23}\text{NOS}$: calcd. 217.1500, found 217.1489.

$[\alpha]_D^{26}$ -46.5 (c 1.15, CHCl_3); ^1H NMR (500 MHz, CDCl_3): δ 0.96 (dd, 6H, $J = 6.85, 14.41$ Hz), 1.21 (s, 9H), 1.88-2.02 (m, 1H), 2.17-2.34 (m, 2H), 3.12-3.22 (m, 2H), 5.05-5.09 (m, 2H), 5.75-5.83 (m, 1H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 18.13, 18.34, 31.87, 36.88, 56.04, 61.40, 117.18, 135.30 ppm; ESI-MS (m/z , %): 218.2 ($\text{M}^+ + \text{H}$).

$[\alpha]_D^{26}$ -24.2 (c 3.95, CHCl_3); ^1H NMR (400 MHz, CDCl_3): δ 1.23 (s, 9H), 1.79-1.88 (m, 2H), 2.35-2.48 (m, 2H), 2.60-2.76 (m, 2H), 3.30 (d, 1H, $J = 6.48$ Hz), 3.36 (dt, 1H, $J = 5.86, 12.24$ Hz), 5.14-5.20 (m, 2H), 5.74-5.84 (m, 1H), 7.16-7.20 (m, 3H), 7.26-7.30 (m, 2H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 22.66, 31.81, 36.86, 40.41, 54.60, 55.84, 119.04, 125.88, 128.27, 128.39, 133.84, 141.71 ppm; FT-IR (film, cm^{-1}) ν 3215, 3063, 3026, 2856, 1673, 1640, 1455, 1363, 1054, 911, 745, 699; ESI-MS (m/z , %): 280.2 ($\text{M}^+ + \text{H}$); HRMS (EI) for $\text{C}_{16}\text{H}_{25}\text{NOS}$: calcd. 279.1657, found 279.1649.

$[\alpha]_D^{26}$ -64.6 (c 2.75, CHCl_3); ^1H NMR (400 MHz, CDCl_3): δ 1.19 (s, 9H), 1.89-1.94 (m, 2H), 2.33 (dd, 2H, $J = 6.15, 6.92$ Hz), 2.68-2.78 (m, 2H), 3.16 (d, 1H, $J = 6.08$ Hz), 3.35 (dt, 1H, $J = 6.17, 12.37$ Hz), 5.08-5.12 (m, 2H), 5.72-5.83 (m, 1H), 7.17-7.22 (m, 3H), 7.28 (dd, 2H, $J = 1.82, 7.31$ Hz) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 22.58, 31.85, 37.04, 40.02, 55.47, 55.78, 117.92, 125.87, 128.37, 128.40, 134.16, 141.38 ppm; ESI-MS (m/z , %): 280.2 ($\text{M}^+ + \text{H}$).

$[\alpha]_D^{26}$ -134.0 (c 1.75, CHCl_3); ^1H NMR (300 MHz, CDCl_3): δ 1.21 (s, 9H), 2.38-2.56 (m, 2H), 3.34 (d, 1H, $J = 5.4$ Hz), 4.02 (dt, 1H, $J = 6.9, 12.9$ Hz), 5.08-5.15 (m, 2H), 5.74-5.85 (m, 1H), 6.20 (dd, 1H, $J = 7.2, 15.9$ Hz), 6.60 (d, 1H, $J = 16.2$ Hz), 7.18-7.39 (m, 5H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 22.63, 41.29, 55.55, 55.87, 119.18, 126.50, 127.72, 128.55, 129.88,

132.36, 133.75, 136.55 ppm; FT-IR (KBr, cm⁻¹) ν 3199, 3073, 2976, 2867, 1640, 1053, 964, 910, 752. 694; ESI-MS (m/z, %): 278.2 (M⁺+H); HRMS (EI) for C₁₆H₂₃NOS: calcd. 277.1500, found 277.1509.

[α]_D²⁶ -16.2 (c 2.55, CHCl₃); ¹H NMR (300 MHz, CDCl₃): δ 1.22 (s, 9H), 2.34-2.55 (m, 2H), 3.49 (d, 1H, 2.1 Hz), 4.07 (dd, 1H, J = 7.5, 15.0 Hz), 5.16-5.22 (m, 2H), 5.73-5.87 (m, 1H), 6.02 (dd, 1H, J = 7.8, 15.9 Hz), 6.61 (d, 1H, J = 15.9 Hz), 7.21-7.40 (m, 5H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 22.55, 40.11, 55.88, 57.31, 118.17, 126.52, 127.72, 128.46, 130.32, 131.98, 133.76, 136.39 ppm; ESI-MS (m/z, %): 278.2 (M⁺+H).

[α]_D²⁶ -85.6 (c 3.15, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 1.24 (s, 9H), 1.77 (s, 3H), 2.05 (s, 1H), 2.69 (d, 2H, J = 7.35 Hz), 3.77 (s, 1H), 5.11-5.20 (m, 2H), 5.52-5.60 (m, 1H), 7.21-7.30 (m, 1H), 7.34 (dd, 2H, J = 6.45, 7.89 Hz), 7.44 (dd, 2H, 0.66, 7.9 Hz) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 22.81, 27.77, 49.04, 56.20, 59.93, 120.33, 126.31, 126.99, 128.20, 133.10, 145.32 ppm; FT-IR (film, cm⁻¹) ν 3215, 3066, 2977, 2926, 2854, 1447, 1378, 1268, 1070, 913, 763, 700, 663; ESI-MS (m/z, %): 266.2 (M⁺+H); HRMS (EI) for C₁₅H₂₃NOS: calcd. 265.1500, found 265.1489.

[α]_D²⁶ -80.1 (c 1.70, CHCl₃); ¹H NMR (300 MHz, CDCl₃): δ 1.22 (s, 9H), 1.75 (s, 3H), 2.34 (s, 3H), 2.66 (d, 2H, J = 7.3 Hz), 3.74 (s, 1H), 5.10-5.20 (m, 2H), 5.51-5.65 (m, 1H), 7.15 (d, 2H, J = 7.8 Hz), 7.19 (d, 2H, J = 8.1 Hz) ppm; ¹³C NMR (75 MHz, CDCl₃): δ 20.90, 22.81, 27.80, 30.13, 49.08, 56.13, 59.73, 120.15, 126.25, 128.92, 133.26, 136.60, 142.34 ppm; FT-IR (film, cm⁻¹) ν 2977, 2955, 2923, 2867, 1455, 1069, 935, 915, 817, 653; ESI-MS (m/z, %): 280.2 (M⁺+H); HRMS (EI) for C₁₆H₂₅NOS: calcd. 279.1657, found 279.1649.

[α]_D²⁶ -76.2 (c 3.15, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 1.24 (s, 9H), 1.80 (s, 3H), 2.69 (d, 2H, J = 7.35 Hz), 3.82 (s, 1H), 5.15-5.22 (m, 2H), 5.49-5.60 (m, 1H), 7.59 (dd, 4H, J = 8.57, 15.69 Hz) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 22.76, 27.68, 29.64, 48.94, 56.40, 59.91, 121.00, 125.13, 125.17, 125.20, 125.24, 126.82, 129.44, 132.37, 149.50 ppm; FT-IR (film, cm⁻¹) ν 2979, 2868, 1411, 1328, 1167, 1124, 1070, 1015, 841, 600; ESI-MS (m/z, %): 334.2 (M⁺+H), 356.2 (M⁺+Na); HRMS (EI) for C₁₆H₂₂F₃NNaOS: calcd. 356.1272, found 356.1261.

$[\alpha]_D^{26}$ -41.8 (c 1.03, CHCl_3); ^1H NMR (400 MHz, CDCl_3): δ 1.21 (s, 9H), 1.74 (s, 3H), 2.65 (d, 1H, $J=7.35$ Hz), 3.70 (s, 1H), 3.80 (d, 3H, $J=2.19$ Hz), 5.11-5.18 (m, 2.0 Hz), 5.53-5.62 (m, 1H), 6.86-6.88 (m, 2H), 7.35 (dd, 2H, $J=2.16$, 6.82 Hz) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 22.85, 27.829, 49.24, 55.21, 56.16, 59.60, 113.51, 120.11, 127.67, 133.32, 137.28, 158.52 ppm; FT-IR (film, cm^{-1}) ν 2926, 2854, 1611, 1513, 1378, 1251, 1182, 1070, 1036, 830, 668; ESI-MS (m/z , %): 296.2 ($\text{M}^+\text{+H}$); HRMS (EI) for $\text{C}_{16}\text{H}_{25}\text{NO}_2\text{S}$: calcd. 296.1606, found 295.1601.

$[\alpha]_D^{26}$ -75.0 (c 2.55, CHCl_3); ^1H NMR (400 MHz, CDCl_3): δ 1.22 (s, 9H), 1.76 (s, 3H), 2.04 (s, 1H), 2.64 (d, 2H, $J=7.29$ Hz), 3.73 (s, 1H), 5.13-5.20 (m, 2H), 5.50-5.61 (m, 1H), 7.30-7.39 (m, 4H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 22.77, 27.66, 49.04, 56.27, 59.66, 120.65, 127.93, 128.33, 132.65, 132.90, 143.85 ppm; FT-IR (film, cm^{-1}) ν 3286, 3219, 3076, 2978, 2926, 2867, 1636, 1492, 1095, 1058, 1012, 828, 752, 609; ESI-MS (m/z , %): 300.2 ($\text{M}^+\text{+H}$), 322.2 ($\text{M}^+\text{+Na}$); HRMS (EI) for $\text{C}_{15}\text{H}_{22}\text{ClNOS}$: calcd. 299.2111, found 299.1101.

$[\alpha]_D^{26}$ -68.7 (c 2.81, CHCl_3); ^1H NMR (400 MHz, CDCl_3): δ 1.22 (s, 9H), 1.75 (s, 3H), 2.03 (s, 1H), 2.64 (d, 2H, $J=7.36$ Hz), 5.13-5.20 (m, 2H), 5.50-5.60 (m, 1H), 7.31 (dd, 2H, $J=2.04$, 6.71 Hz), 7.46 (dd, 2H, $J=2.08$, 6.74 Hz) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 22.77, 27.60, 48.97, 56.27, 59.71, 120.69, 121.07, 128.29, 131.29, 132.61, 144.41 ppm; FT-IR (film, cm^{-1}) ν 3216, 2977, 2925, 2867, 1487, 1396, 1364, 1059, 1008, 825, 600; ESI-MS (m/z , %): 344.1 ($\text{M}^+\text{+H}$), 368.1 ($\text{M}^+\text{+Na}$); HRMS (EI) for $\text{C}_{15}\text{H}_{22}\text{BrNOS}$: calcd. 343.0605, found 343.0617.

$[\alpha]_D^{26}$ -84.7 (c 2.87, CHCl_3); ^1H NMR (400 MHz, CDCl_3): δ 1.22 (s, 9H), 1.76 (s, 3H), 2.65 (d, 2H, $J=7.41$ Hz), 3.73 (s, 1H), 5.13-5.19 (m, 2H), 5.51-5.60 (m, 1H), 7.00-7.04 (m, 2H), 7.39-7.43 (m, 2H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 22.75, 27.79, 49.16, 56.18, 59.60, 114.81, 115.02, 120.46, 128.13, 128.20, 132.80, 140.95, 140.98, 160.56, 162.94 ppm; FT-IR (film, cm^{-1}) ν 3209, 3076, 2958, 2925, 2855, 1603, 1510, 1456, 1376, 1227, 1163, 1055, 918, 836, 817, 594; ESI-MS (m/z , %): 284.2 ($\text{M}^+\text{+H}$); HRMS (EI) for $\text{C}_{15}\text{H}_{22}\text{FNOS}$: calcd. 283.1406, found 283.1402.

$[\alpha]_D^{26}$ -86.9 (c 2.02, CHCl_3); ^1H NMR (400 MHz, CDCl_3): δ 1.23 (s, 9H), 1.75 (s, 3H), 2.35 (s, 3H), 2.67 (d, 2H, $J = 7.31$ Hz), 3.75 (s, 1H), 5.11-5.20 (m, 2H), 5.52-5.61 (m, 1H), 7.06-7.08 (m, 1H), 7.22-7.24 (m, 3H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 21.64, 22.82 (22.73), 27.75, 49.12, 56.21, 59.87, 120.22, 123.41, 127.10, 127.72, 128.08, 133.25, 137.67, 145.32 ppm; FT-IR (film, cm^{-1}) ν 3208, 2923, 2853, 1606, 1456, 1377, 1184, 1056, 914, 787, 706, 643; ESI-MS (m/z , %): 280.2 ($\text{M}^+ + \text{H}$); HRMS (EI) for $\text{C}_{16}\text{H}_{25}\text{NOS}$: calcd. 279.1657, found 279.1663.

$[\alpha]_D^{26}$ -80.9 (c 4.68, CHCl_3); ^1H NMR (400 MHz, CDCl_3): δ 1.24 (s, 9H), 1.77 (s, 3H), 2.65 (d, 2H, $J = 7.35$ Hz), 3.76 (s, 1H), 5.15-5.21 (m, 2H), 5.51-5.61 (m, 1H), 7.22-7.32 (m, 3H), 7.43 (t, 1H, $J = 1.61$ Hz) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 22.75, 27.60, 48.99, 56.30, 59.72, 120.79, 124.53, 126.84, 127.12, 129.45, 132.53, 134.16, 147.62 ppm; FT-IR (film, cm^{-1}) ν 3209, 3079, 2977, 2955, 2922, 2851, 1595, 1573, 1474, 1056, 918, 785, 698; ESI-MS (m/z , %): 300.1 ($\text{M}^+ + \text{H}$); HRMS (EI) for $\text{C}_{15}\text{H}_{22}\text{ClNOS}$: calcd. 299.1111, found 299.1104.

Hz

2932.76
2929.77
2926.94
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2921.16
2914.69
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2908.41
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475.26

Current Data Parameters
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EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
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Time 4 54
INSTRUM drx400
PROBHD 5 mm GNP 13C
PULPROG zg
TD 65536
SOLVENT DMSO
NS 4
DS 0
SWH 12019.230
FIDRES 0.183399
AQ 2.7263477
RG 16
DW 41.600
DE 20.00
TE 300.0
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S15

integral

ppm

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7

6

5

4

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127.396

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77.018

76.700

57.056

55.532

43.301

22.505

S16

Hz

Integral

ppm

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Current Data Parameters
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PROCNO 1

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AQ 2.7263477
RG 16
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PL1 0.00
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LB 0.00
GB 0
PC 1.00

1D NMR plot parameters
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F2P 0.000
F2 0.00
PPMCM 0.50000
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S19

ppm

ppm

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129.075
128.996

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115.169

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55.543

43.316

22.494

S20

Hz

Current Data Parameters

NAME h1
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters

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PL1 0.00
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F2 - Processing parameters

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1D NMR plot parameters

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S21

Hz

Integr. a1

ppm

Current Data Parameters
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PROCNO 1

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PL1 0.00
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WDW no
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LB 0.00
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00
F1P 10.000
F1 4001.30
F2P 0.000
F2 0.00
PPMCM 0.50000
HZCM 200.06500

S23

ppm

Current Data Parameters

NAME c13
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters

Date_ 20060707
Time 4.46
INSTRUM drx400
PROBHD 5 mm GNP 13C
PULPROG zgdc
TD 65536
SOLVENT DMSO
NS 40
DS 0
SWH 35211.270 Hz
FIDRES 0.537281 Hz
AQ 0.9306612 sec
RG 4096
DW 14.200 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====

NUC1 13C
P1 6.00 usec
PL1 -6.00 dB
SF01 100.6224600 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 1H
PCPD2 85.00 usec
PL2 120.00 dB
PL12 20.00 dB
SF02 400.1316820 MHz

F2 - Processing parameters

SI 32768
SF 100.6127723 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 1006.12769 Hz/cm

Current Data Parameters
 NAME h1
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060425
 Time 7.54
 INSTRUM drx400
 PROBHD 5 mm QNP 13C
 PULPROG zg
 TD 65536
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 12019.230
 FIDRES 0.183399
 AQ 2.7263477
 RG 8
 DW 41.600
 DE 20.00
 TE 300.0
 D1 2.00000000

===== CHANNEL f1
 NUC1 1H
 P1 5.00
 PL1 0.00
 SF01 400.1315926

F2 - Processing parameters
 SI 65536
 SF 400.1300010
 WDW no
 SSB 0
 LB 0.00
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00
 F1P 10.000
 F1 4001.30
 F2P 0.000
 F2 0.00
 PPMCM 0.50000
 HZCM 200.06500

S25

ppm

140.352
133.498
133.308
128.724
128.566

118.511

77.340
77.022
76.703

57.906
56.002

41.126

22.551

ppm

180

160

140

120

100

80

60

40

20

HZ

Integral

ppm

2988.26
2986.40
2981.67
2979.81
2883.64
2881.94
2876.98
2875.31

2076.65
2075.12
2073.82
2061.46
2061.11
2059.39
2058.14
1773.44

1474.16
1472.94

1016.63
984.64

503.34
485.28
475.20

Current Data Parameters
NAME n1
EXPNO 10
PROCNO 1

F2 - Acquisition Paramet
Date_ 20060712
Time 5.10
INSTRUM drx400
PROBHD 5 mm GNP 13C
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 4
DS 0
SWH 12019.230
FIDRES 0.183399
AQ 2.7263477
RG 16
DW 41.600
DE 20.00
TE 300.0
D1 1.00000000

===== CHANNEL f1
NUC1 1H
P1 5.00
PL1 0.00
SF01 400.1331253

F2 - Processing paramete
SI 65536
SF 400.1299998
WDW no
SSB 0
LB 0.00
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00
F1P 10.000
F1 4001.30
F2P 0.000
F2 0.00
PPMCM 0.50000
FZCM 200.06500

ppm

Hz

Integral

ppm

2990.32
2988.46
2983.73
2981.85
2887.99
2886.31
2881.25
2879.62

2225.95
2024.55
2023.19
2021.46
2009.70
2008.97
2007.50
2006.00
1766.66
1760.67
1416.33
1412.00
1074.39
1073.31
1013.33
1006.28

485.13

2d'

Current Data Parameters

NAME n1
EXPNO 10
PROCNO 1

F2 - Acquisition Parameter

Date_ 20060724
Time 12.08
INSTRUM drx400
PROBHD 5 mm QNP 13C
PULPROG zg
TD 65536
SOLVENT DMSO
NS 4
DS 0
SWH 12019.230
FIDRES 0.183399
AQ 2.7263477
RG 8
DW 41.600
DE 20.00
TE 300.0
D1 1.00000000

===== CHANNEL f1

NUC1 1H
P1 5.00
PL1 0.00
SF01 400.1315416

F2 - Processing parameter

SI 65536
SF 400.1299983
WDW no
SSB 0
LB 0.00
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00
F1P 10.000
F1 4001.30
F2P 0.000
F2 0.00
PPMCM 0.50000
HZCM 200.06500

S29

ppm

140.843

133.249

131.639

128.883

121.589

118.507

77.336

77.018

76.699

57.956

56.001

41.051

22.520

ppm

180

160

140

120

100

80

60

40

20

S30

Hz

Current Data Parameters

NAME h1
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters

Date_ 20060630
Time 6.47
INSTRUM drx400
PROBHD 5 mm GNP 13C
PULPROG zg
TD 65536
SOLVENT D2O
NS 8
DS 0
SWH 12019.230
FIDRES 0.183399
AQ 2.7263477
RG 8
DW 41.600
DE 20.00
TE 300.0
D1 1.00000000

===== CHANNEL f1

NUC1 1H
P1 5.00
PL1 0.00
SF01 400.1331253

F2 - Processing parameters

SI 65536
SF 400.1300078
WDW no
SSB 0
LB 0.00
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00
F1P 10.000
F1 4001.30
F2P 0.000
F2 0.00
PPMCM 0.50000
HZCM 200.06500

ppm

138.624
137.257
134.276
129.129
127.366
119.068

77.342
77.023
76.707

56.806
55.544

43.410

22.563
21.089

Current Data Parameters

NAME c13
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters

Date_ 20060630
Time 6.49
INSTRUM drx400
PROBHD 5 mm QNP 13C
PULPROG zgdc
TD 65536
SOLVENT MeOH
NS 44
DS 0
SWH 35211.270 Hz
FIDRES 0.537281 Hz
AQ 0.9306612 sec
RG 4096
DW 14.200 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====

NUC1 13C
P1 6.00 usec
PL1 -6.00 dB
SF01 100.6227290 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 1H
PCPD2 85.00 usec
PL2 120.00 dB
PL12 20.00 dB
SF02 400.1318000 MHz

F2 - Processing parameters

SI 32768
SF 100.6127726 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.80

1D NMR plot parameters

CX 20.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 1006.12769 Hz/cm

Hz

Current Data Parameters
 NAME n1
 EXPNO 10
 PROCNO 1

F2 - Acquisition Paramet
 Date_ 20060630
 Time 6.40
 INSTRUM drx400
 PROBHD 5 mm QNP 13C
 PULPROG zg
 TD 65536
 SOLVENT D2O
 NS 8
 DS 0
 SWH 12019.230
 FIDRES 0.183399
 AQ 2.7263477
 RG 8
 DW 41.600
 DE 20.00
 TE 300.0
 D1 1.00000000

===== CHANNEL f1
 NUC1 1H
 P1 5.00
 PL1 0.00
 SF01 400.1331253

F2 - Processing paramete
 SI 65536
 SF 400.1300058
 WDW no
 SSB 0
 LB 0.00
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00
 F1P 10.000
 F1 4001.30
 F2P 0.000
 F2 0.00
 PPMCM 0.50000
 HZCM 200.06500

ppm

ppm

138.816
137.425
133.903
129.229
127.068
117.936

77.340
77.021
76.703

58.235
55.831

41.101

22.575
21.025

Current Data Parameters

NAME c13
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters

Date_ 20060630
Time 6.44
INSTRUM drx400
PROBHD 5 mm GNP 13C
PULPROG zgdc
TD 65536
SOLVENT MeOH
NS 74
DS 0
SWH 35211.270 Hz
FIDRES 0.537281 Hz
AQ 0.9306612 sec
RG 4096
DW 14.200 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====

NUC1 13C
P1 6.00 usec
PL1 -6.00 dB
SF01 100.6227290 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 1H
PCPD2 85.00 usec
PL2 120.00 dB
PL12 20.00 dB
SF02 400.1318000 MHz

F2 - Processing parameters

SI 32768
SF 100.6127759 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.80

1D NMR plot parameters

CX 20.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 1006.12781 Hz/cm

S34

Hz

2909.98
2898.55
2896.66
2891.86
2889.85
2752.05
2750.02
2745.33
2743.32

2075.78
2074.58
2065.46
2064.86
2058.59
2055.68
1765.86

1518.73
1462.04
1460.85

1019.03
989.64

508.36
503.24
501.31
494.11
485.51
472.94

Current Data Parameters

NAME h1
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters

Date_ 20060712
Time 5 20
INSTRUM drx400
PROBHD 5 mm QNP 13C
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 4
DS 0
SWH 12019.230
FIDRES 0.183399
AQ 2.7263477
RG 16
DW 41.600
DE 20.00
TE 300.0
D1 1.00000000

===== CHANNEL f1

NUC1 1H
P1 5.00
PL1 0.00
SF01 400.1331253

F2 - Processing parameters

SI 65536
SF 400.1300044
WDW no
SSB 0
LB 0.00
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00
F1P 10.000
F1 4001.30
F2P 0.000
F2 0.00
PPMCM 0.50000
HZCM 200.06500

S35

ppm

Current Data Parameters

NAME	h1
EXPNO	11
PROCNO	1

F2 - Acquisition Parameters

Date_	20060425
Time	7.41
INSTRUM	drx400
PROBHD	5 mm GNP 13C
PULPROG	zg
TD	65536
SOLVENT	CDC13
NS	4
DS	0
SWH	12019.230
FIDRES	0.183399
AQ	2.7263477
RG	8
DW	41.600
DE	20.00
TE	300.0
D1	2.00000000

===== CHANNEL f1

NUC1	1H
P1	5.00
PL1	0.00
SF01	400.1315926

F2 - Processing parameters

SI	65536
SF	400.1300043
WDW	no
SSB	0
LB	0.00
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00
F1P	10.000
F1	4001.30
F2P	0.000
F2	0.00
PPMCM	0.50000
HZCM	200.06500

ppm

ppm

159.118

133.954
133.905

128.351

117.956

113.932

77.339
77.021
76.70357.944
55.799
55.189

41.118

22.596

Hz

Integral

ppm

Current Data Parameters

NAME h1
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters

Date_ 20060720
Time 6.12
INSTRUM drx400
PROBHD 5 mm QNP 13C
PULPROG zg
TD 65536
SOLVENT MeOH
NS 4
DS 0
SWH 12019.230
FIDRES 0.183399
AQ 2.7263477
RG 32
DW 41.600
DE 20.00
TE 300.0
D1 1.00000000

===== CHANNEL f1

NUC1 1H
P1 5.00
PL1 0.00
SF01 400.1318545

F2 - Processing parameters

SI 65536
SF 400.1300031
WDW no
SSB 0
LB 0.00
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00
F1P 10.000
F1 4001.30
F2P 0.000
F2 0.00
PPMCM 0.50000
HZCM 200.06500

ppm

139.212
133.722
133.135
129.738
128.458
126.727
119.444

77.334
77.017
76.698

55.820
53.701

41.370

22.540

ppm

180

160

140

120

100

80

60

40

20

Current Data Parameters

NAME	h1
EXPNO	11
PROCNO	1

F2 - Acquisition Parameters

Date_	20060425
Time	7.45
INSTRUM	drx400
PROBHD	5 mm QNP 13C
PULPROG	zg
TD	65536
SOLVENT	CDCl ₃
NS	4
DS	0
SWH	12019.230
FIDRES	0.183399
AQ	2.7263477
RG	8
DW	41.600
DE	20.00
TE	300.0
D1	2.00000000

===== CHANNEL f1

NUC1	¹ H
P1	5.00
PL1	0.00
SF01	400.1315926

F2 - Processing parameters

SI	65536
SF	400.1300038
WDW	no
SSB	0
LB	0.00
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00
F1P	10.000
F1	4001.30
F2P	0.000
F2	0.00
PPMCM	0.50000
HZCM	200.06500

S41

Hz

Integral

ppm

Current Data Parameters
NAME h1
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060720
Time 5.49
INSTRUM drx400
PROBHD 5 mm QNP 13C
PULPROG zg
TD 65536
SOLVENT MeOH
NS 4
DS 0
SWH 12019.230
FIDRES 0.183399
AQ 2.7263477
RG 32
DW 41.600
DE 20.00
TE 300.0
D1 1.00000000

===== CHANNEL f1
NUC1 1H
P1 5.00
PL1 0.00
SF01 400.1318545

F2 - Processing parameters
SI 65536
SF 400.1300081
WDW no
SSB 0
LB 0.00
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00
F1P 10.000
F1 4001.30
F2P 0.000
F2 0.00
PPMCM 0.50000
HZCM 200.06500

S43

ppm

139.542
135.803
134.233
130.456
127.197
126.843
126.052
119.096

77.339
77.020
76.701

55.570
52.615

42.246

22.540
19.358

ppm

180

160

140

120

100

80

60

40

20

S44

Integral

ppm

Current Data Parameters

NAME h1
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters

Date_ 20060425
Time 7.49
INSTRUM drx400
PROBHD 5 mm GNP 13C
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 4
DS 0
SWH 12019.230
FIDRES 0.183399
AQ 2.7263477
RG 8
DW 41.600
DE 20.00
TE 300.0
D1 2.00000000

===== CHANNEL f1

NUC1 1H
P1 5.00
PL1 0.00
SF01 400.1315926

F2 - Processing parameters

SI 65536
SF 400.1300067
WDW no
SSB 0
LB 0.00
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00
F1P 10.000
F1 4001.30
F2P 0.000
F2 0.00
PPMCM 0.50000
HZCM S45 200.06500

ppm

139.775
135.591
133.797
130.611
127.462
126.266
126.245
118.139

77.331
77.015
76.697

55.780
53.600

40.549

22.582
19.390

ppm

180

160

140

120

100

80

60

40

20

S46

Current Data Parameters

NAME	h1
EXPNO	10
PROCNO	1

F2 - Acquisition Parameters

Date_	20060720
Time	5.41
INSTRUM	drx400
PROBHD	5 mm GNP 13C
PULPROG	zg
TD	65536
SOLVENT	MeOH
NS	4
DS	0
SWH	12019.230
FIDRES	0.183399
AQ	2.7263477
RG	32
DW	41.600
DE	20.00
TE	300.0
D1	1.00000000

===== CHANNEL f1

NUC1	1H
P1	5.00
PL1	0.00
SF01	400.1318545

F2 - Processing parameters

SI	65536
SF	400.1300029
WDW	no
SSB	0
LB	0.00
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00
F1P	10.000
F1	4001.30
F2P	0.000
F2	0.00
PPMCM	0.50000
HZCM	200.06500

S47

ppm

144.176
133.583
130.709
130.494
130.011
126.173
122.537
119.655

77.342
77.024
76.707

56.493
55.735

43.212

22.522

Current Data Parameters
 NAME h1
 EXPNO 11
 PROCNO 1

F2 - Acquisition Paramet
 Date_ 20060425
 Time 7.17
 INSTRUM drx400
 PROBHD 5 mm QNP 13C
 PULPROG zg
 TD 65536
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 12019.230
 FIDRES 0.183399
 AQ 2.7263477
 RG 16
 DW 41.600
 DE 20.00
 TE 300.0
 D1 2 00000000

===== CHANNEL f1
 NUC1 1H
 P1 5.00
 PL1 0.00
 SF01 400.1315926

F2 - Processing paramete
 SI 65536
 SF 400.1299998
 WDW no
 SSB 0
 LB 0.00
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00
 F1P 10.000
 F1 4001.30
 F2P 0.000
 F2 0.00
 PPMCM 0.50000
 HZCM 200.06500

ppm

ppm

144.217

133.189

130.869

130.123

125.956

122.604

118.585

77.338

77.020

76.702

57.989

56.048

41.111

22.550

S50

Current Data Parameters
 NAME h1
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060712
 Time 5.24
 INSTRUM drx400
 PROBHD 5 mm QNP 13C
 PULPROG zg
 TD 65536
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 12019.230
 FIDRES 0.183399
 AQ 2.7263477
 RG 16
 DW 41.600
 DE 20.00
 TE 300.0
 D1 1.00000000

===== CHANNEL f1
 NUC1 1H
 P1 5.00
 PL1 0.00
 SF01 400.1331253

F2 - Processing parameters
 SI 65536
 SF 400.1300155
 WDW no
 SSB 0
 LB 0.00
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00
 F1P 10.000
 F1 4001.30
 F2P 0.000
 F2 0.00
 PPMCM 0.50000
 HZCM 200.06500

S51

ppm

138.901
134.030
133.165
132.952
128.251
127.837
127.591
126.791
126.095
125.881
124.973
119.199

77.339
77.020
76.702

57.240
55.531

43.089

22.509

Current Data Parameters
NAME txo
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060718
Time 11.15
INSTRUM av500
PROBHD 5 mm TXO 19F/1
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 15015.015 Hz
FIDRES 0.229111 Hz
AQ 2.1824322 sec
RG 64
DW 33.300 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 4.00 usec
PL1 0.00 dB
SFO1 500.1320806 MHz

F2 - Processing parameters
SI 65536
SF 500.1300195 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.20

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 10.000 ppm
F1 5001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.50000 ppm/cm
HZCM 250.06500 Hz/cm

ppm

ppm

180

160

140

120

100

80

60

40

20

134.607

118.543

77.338

77.020

76.701

58.769

55.476

41.290

22.647

16.079

4.727

4.213

Hz

Integral

ppm

2344.09
2052.35
2051.67
2050.29
2037.60
2036.61
2035.40
2034.64
2033.20
2027.43
2026.43

971.29
970.09
968.75
964.16
502.60
485.74
244.34
242.72
240.89
240.10
235.98
234.69
233.02
232.18
153.15
126.90

Current Data Parameters

NAME h1
EXPNO 10
PROCNO 1

F2 - Acquisition Paramet

Date_ 20060720
Time 6.08
INSTRUM drx400
PROBHD 5 mm GNP 13C
PULPROG zg
TD 65536
SOLVENT MeOH
NS 4
DS 0
SWH 12019.230
FIDRES 0.183399
AQ 2.7263477
RG 32
DW 41.600
DE 20.00
TE 300.0
D1 1.00000000

===== CHANNEL f1

NUC1 1H
P1 5.00
PL1 0.00
SF01 400.1318545

F2 - Processing paramete

SI 65536
SF 400.1299996
WDW no
SSB 0
LB 0.00
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00
F1P 10.000
F1 4001.30
F2P 0.000
F2 0.00
PPMCM 0.50000
HZCM 200.06500

S57

ppm

ppm

134.435

117.500

77.338

77.019

76.700

60.884

55.606

40.284

22.603

16.869

4.684

4.150

Hz

Current Data Parameters
NAME h1
EXPNO 10
PROCNO 1

F2 - Acquisition Paramet
Date_ 20060623
Time 5.24
INSTRUM drx400
PROBHD 5 mm QNP 13C
PULPROG zg
TD 65536
SOLVENT MeOH
NS 6
DS 0
SWH 12019.230
FIDRES 0.183399
AQ 2.7263477
RG 16
DW 41.600
DE 20.00
TE 300.0
D1 1.00000000

===== CHANNEL f1
NUC1 1H
P1 5.00
PL1 0.00
SF01 400.1331253

F2 - Processing paramete
SI 65536
SF 400.1299924
WDW no
SSB 0
LB 0.00
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00
F1P 10.000
F1 4001.30
F2P 0.000
F2 0.00
PPMCM 0.50000
HZCM 200.06500

S59

ppm

134.607

118.618

77.341
77.022
76.705

59.643
55.876

41.196
37.138
29.603
29.052
28.522
26.240
26.204
22.701

Current Data Parameters

NAME h1
EXPNO 11
PROCNO 1

F2 - Acquisition Paramet

Date_ 20060425
Time 7.29
INSTRUM drx400
PROBHD 5 mm GNP 13C
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 4
DS 0
SWH 12019.230
FIDRES 0.183399
AQ 2.7263477
RG 8
DW 41.600
DE 20.00
TE 300.0
D1 2.00000000

===== CHANNEL f1

NUC1 1H
P1 5.00
PL1 0.00
SF01 400.1315926

F2 - Processing paramete

SI 65536
SF 400.1299911
WDW no
SSB 0
LB 0.00
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00
F1P 10.000
F1 4001.30
F2P 0.000
F2 0.00
PPMCM 0.50000
HZCM 200.06500

ppm

ppm

180

160

140

120

100

80

60

40

20

135.261

117.124

77.338

77.020

76.701

60.927

55.958

41.946

37.119

28.894

28.805

26.340

26.182

26.159

22.662

Integral

ppm

Current Data Parameters

NAME txo
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20060713
Time 16.46
INSTRUM av500
PROBHD 5 mm TXO 19F/1
PULPROG zg
TD 65536
SOLVENT D2O
NS 16
DS 0
SWH 15015.015 Hz
FIDRES 0.229111 Hz
AQ 2.1824322 sec
RG 256
DW 33.300 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec

===== CHANNEL f1 =====

NUC1 1H
P1 4.00 usec
PL1 0.00 dB
SF01 500.1320806 MHz

F2 - Processing parameters

SI 65536
SF 500.1300128 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
CY 0.00 cm
F1P 10.000 ppm
F1 5001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.50000 ppm/cm
HZCM 250.06500 Hz/cm

ppm

S64

Hz

Integral

ppm

2044.83
2044.50
2043.05
2038.70
2037.68
2028.39
2027.22
2026.02

1300.88
1294.76
918.86
917.59
916.28
911.78
910.60
656.54
649.13
642.53
502.72
483.13
394.55
387.13
379.67

Current Data Parameters
NAME h1
EXPNO 10
PROCNO 1

F2 - Acquisition Paramet
Date_ 20060718
Time 6.26
INSTRUM drx400
PROBHD 5 mm QNP 13C
PULPROG zg
TD 65536
SOLVENT DMSO
NS 4
DS 0
SWH 12019.230
FIDRES 0.183399
AQ 2.7263477
RG 32
DW 41.600
DE 20.00
TE 300.0
D1 1.00000000

===== CHANNEL f1
NUC1 1H
P1 5.00
PL1 0.00
SF01 400.1318545

F2 - Processing paramete
SI 65536
SF 400.1300000
WDW no
SSB 0
LB 0.00
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00
F1P 10.000
F1 4001.30
F2P 0.000
F2 0.00
PPMCM 0.50000
HZCM 200.06500

S65

ppm

Current Data Parameters
NAME txo
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060718
Time 11.07
INSTRUM av500
PROBHD 5 mm TXO 19F/1
PULPROG zg
TD 65536
SOLVENT CDC13
NS 8
DS 0
SWH 15015.015 Hz
FIDRES 0.229111 Hz
AQ 2.1824322 sec
RG 64
DW 33.300 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 4.00 usec
PL1 0.00 dB
SF01 500.1320806 MHz

F2 - Processing parameters
SI 65536
SF 500.1300055 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.20

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 10.000 ppm
F1 5001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.50000 ppm/cm
HZCM 250.06500 Hz/cm

ppm

Current Data Parameters

NAME txo
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20060718
Time 10.34
INSTRUM av500
PROBHD 5 mm TXO 19F/1
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 15015.015 Hz
FIDRES 0.229111 Hz
AQ 2.1824322 sec
RG 64
DW 33.300 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec

===== CHANNEL f1 =====

NUC1 1H
P1 4.00 usec
PL1 0.00 dB
SF01 500.1320806 MHz

F2 - Processing parameters

SI 65536
SF 500.1300046 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.20

1D NMR plot parameters

CX 20.00 cm
CY 0.00 cm
F1P 10.000 ppm
F1 5001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.50000 ppm/cm
HZCM 250.06500 Hz/cm

ppm

Hz

2919.20
2913.76
2911.97
2905.83
2904.53
2880.36
2879.10
2871.15
2869.86
2867.42
2863.03
2301.78
2076.10
2074.77
2073.10
2071.97
2060.34
2059.22
2057.82
1352.32
1346.14
1322.63
1316.15
1094.25
1060.37
977.48
976.34
960.47
959.53
739.59
738.32
735.53
733.01
731.78
731.21
730.15
728.59
724.86
723.22
722.48
503.04
491.72
477.65

Integral

2.032

2.971

1.000

1.989

1.140

0.958

2.221

2.246

2.160

9.206

Current Data Parameters
NAME n1
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060623
Time 5.15
INSTRUM drx400
PROBHD 5 mm QNP 13C
PULPROG zg
TD 65536
SOLVENT MeOH
NS 6
DS 0
SWH 12019.230
FIDRES 0.183399
AQ 2.7263477
RG 16
DW 41.600
DE 20.00
TE 300.0
D1 1.00000000

===== CHANNEL f1
NUC1 1H
P1 5.00
PL1 0.00
SF01 400.1331253

F2 - Processing parameters
SI 65536
SF 400.1300082
WDW no
SSB 0
LB 0.00
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00
F1P 10.000
F1 4001.30
F2P 0.000
F2 0.00
PPMCM 0.50000
HZCM 200.06500

141.706
133.842
128.394
128.273
125.881
119.036

77.335
77.018
76.699

55.842
54.601

40.409
36.860
31.805

22.658

Current Data Parameters
NAME h1
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060623
Time 5.20
INSTRUM drx400
PROBHD 5 mm. QNP 13C
PULPROG zg
TD 65536
SOLVENT MeOH
NS 8
DS 0
SWH 12019.230
FIDRES 0.183399
AQ 2.7263477
RG 16
DW 41.600
DE 20.00
TE 300.0
D1 1.00000000

===== CHANNEL f1
NUC1 1H
P1 5.00
PL1 0.00
SF01 400.1331253

F2 - Processing parameters
SI 65536
SF 400.1300069
WDW no
SSB 0
LB 0.00
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00
F1P 10.000
F1 4001.30
F2P 0.000
F2 0.00
PPMCM 0.50000
HZCM 200.06500

Hz

Current Data Parameters
 NAME n1
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060707
 Time 4.49
 INSTRUM drx400
 PROBHD 5 mm QNP 13C
 PULPROG zg
 TD 65536
 SOLVENT D2O
 NS 4
 DS 0
 SWH 12019.230
 FIDRES 0.183399
 AQ 2.7263477
 RG 16
 DW 41.600
 DE 20.00
 TE 300.0
 D1 1.00000000

===== CHANNEL f1
 NUC1 1H
 P1 5.00
 PL1 0.00
 SF01 400.1331253

F2 - Processing parameters
 SI 65536
 SF 400.1300049
 WDW no
 SSB 0
 LB 0.00
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00
 F1P 10.000
 F1 4001.30
 F2P 0.000
 F2 0.00
 PPMCM 0.50000
 HZCM 200.06500

ppm

145.320
133.100
128.203
126.994
126.307
120.330

77.340
77.021
76.703

59.932
56.203
49.038

27.768
22.805

Current Data Parameters

NAME c13
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters

Date_ 20060707
Time 4.51
INSTRUM drx400
PROBHD 5 mm QNP 13C
PULPROG zgdc
TD 65536
SOLVENT DMSO
NS 56
DS 0
SWH 35211.270 Hz
FIDRES 0.537281 Hz
AQ 0.9306612 sec
RG 4096
DW 14.200 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====

NUC1 13C
P1 6.00 usec
PL1 -6.00 dB
SF01 100.6224600 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 1H
PCPD2 85.00 usec
PL2 120.00 dB
PL12 20.00 dB
SF02 400.1316820 MHz

F2 - Processing parameters

SI 32768
SF 100.6127734 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm

ppm

142.340
136.598
133.263
128.916
126.249
120.152

77.333
77.017
76.699

59.729
56.133
49.081

30.125
27.801
22.808
20.903

cpm

Current Data Parameters
NAME h1
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060628
Time 4.53
INSTRUM drx400
PROBHD 5 mm QNP 13C
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 12019.230
FIDRES 0.183399
AQ 2.7263477
RG 8
DW 41.600
DE 20.00
TE 300.0
D1 1.00000000

===== CHANNEL f1
NUC1 1H
P1 5.00
PL1 0.00
SF01 400.1331253

F2 - Processing parameters
SI 65536
SF 400.1300003
WDW no
SSB 0
LB 0.00
GB 0
PC 1.00

10 NMR plot parameters
CX 20.00
F1P 10.000
F1 4001.30
F2P 0.000
F2 0.00
PPMCM 0.50000
HZCM 200.06500

Hz

Current Data Parameters

NAME	h1
EXPNO	10
PROCNO	1

F2 - Acquisition Paramet

Date_	20060718
Time	6 44
INSTRUM	drx400
PROBHD	5 mm GNP 13C
PULPROG	zg
TD	65536
SOLVENT	DMSO
NS	4
DS	0
SWH	12019.230
FIDRES	0.183399
AQ	2.7263477
RG	32
DW	41.600
DE	20.00
TE	300.0
D1	1.00000000

===== CHANNEL f1

NUC1	1H
P1	5.00
PL1	0.00
SF01	400.1318545

F2 - Processing paramete

SI	65536
SF	400.1300095
WDW	no
SSB	0
LB	0.00
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00
F1P	10.000
F1	4001.30
F2P	0.000
F2	0.00
PPMCM	0.50000
HZCM	200.06500

ppm

—158.520

—137.278

—133.315

—127.665

—120.105

—113.514

77.341

77.024

76.707

59.604

56.161

55.211

49.240

—27.829

—22.850

ppm 180 160 140 120 100 80 60 40 20 S86

Current Data Parameters

NAME	h1
EXPNO	10
PROCNO	1

F2 - Acquisition Parameters

Date_	20060630
Time	12.37
INSTRUM	drx400
PROBHD	5 mm QNP 13C
PULPROG	zg
TD	65536
SOLVENT	D2O
NS	8
DS	0
SWH	12019.230
FIDRES	0.183399
AQ	2.7263477
RG	8
DW	41.600
DE	20.00
TE	300.0
D1	1.00000000

===== CHANNEL f1

NUC1	1H
P1	5.00
PL1	0.00
SFO1	400.1331253

F2 - Processing parameters

SI	65536
SF	400.1300012
WDW	no
SSB	0
LB	0.00
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00
F1P	10.000
F1	4001.30
F2P	0.000
F2	0.00
PPMCM	0.50000
HZCM	200.06500

S87

ppm

— 143.846
 — 132.902
 — 132.652
 — 128.327
 — 127.931
 — 120.651

77.342
 77.025
 76.708

59.662
 56.272
 49.037

27.655
 22.771

Current Data Parameters

NAME c13
 EXPNO 5
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20060630
 Time 12.39
 INSTRUM drx400
 PROBHD 5 mm QNP 13C
 PULPROG zgdc
 TD 65536
 SOLVENT MeOH
 NS 46
 DS 0
 SWH 35211.270 Hz
 FIDRES 0.537281 Hz
 AQ 0.9306612 sec
 RG 4096
 DW 14.200 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.0000000 sec
 d11 0.0300000 sec

===== CHANNEL f1 =====

NUC1 13C
 P1 6.00 usec
 PL1 -6.00 dB
 SF01 100.6227290 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
 NUC2 1H
 PCPD2 85.00 usec
 PL2 120.00 dB
 PL12 20.00 dB
 SF02 400.1318000 MHz

F2 - Processing parameters

SI 32768
 SF 100.6127727 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 0.80

1D NMR plot parameters

CX 20.00 cm
 F1P 200.000 ppm
 F1 20122.55 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 10.00000 ppm/cm

S88

Current Data Parameters
NAME h1
EXPNO 10
PROCNO 1

F2 - Acquisition Paramet
Date_ 20060630
Time 12 42
INSTRUM drx400
PROBHD 5 mm GNP 13C
PULPROG zg
TD 65536
SOLVENT D2O
NS 8
DS 0
SWH 12019 230
FIDRES 0.183399
AQ 2.7263477
RG 8
DW 41.600
DE 20.00
TE 300.0
D1 1.00000000

===== CHANNEL f1
NUC1 1H
P1 5.00
PL1 0.00
SF01 400.1331253

F2 - Processing paramete
SI 65536
SF 400.1300012
WDW no
SSB 0
LB 0.00
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00
F1P 10.000
F1 4001.30
F2P 0.000
F2 0.00
PPMCM 0.50000
HZCM 200.06500

S89

ppm

— 144.406
 — 132.612
 — 131.287
 — 128.286
 — 121.073
 — 120.689

77.339
 77.021
 76.702

59.706
 56.269
 48.974

— 27.604
 — 22.765

Current Data Parameters
 NAME c13
 EXPNO 5
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060630
 Time 12.44
 INSTRUM drx400
 PROBH 5 mm QNP 13C
 PULPROG zgdc
 TD 65536
 SOLVENT MeOH
 NS 36
 DS 0
 SWH 35211.270 Hz
 FIDRES 0.537281 Hz
 AQ 0.9306612 sec
 RG 4096
 DW 14.200 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 6.00 usec
 PL1 -6.00 dB
 SF01 100.6227290 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 85.00 usec
 PL2 120.00 dB
 PL12 20.00 dB
 SF02 400.1318000 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127737 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 0.80

10 NMR plot parameters
 CX 20.00 cm
 F1P 200.000 ppm
 F1 20122.55 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 10.00000 ppm/cm

S90

Current Data Parameters
NAME n1
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060804
Time 5.40
INSTRUM drx400
PROBHD 5 mm QNP 13C
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 4
DS 0
SWH 12019.230
FIDRES 0.183399
AQ 2.7263477
RG 32
DW 41.600
DE 20.00
TE 300.0
D1 2.00000000

===== CHANNEL f1
NUC1 1H
P1 5.00
PL1 0.00
SF01 400.1317776

F2 - Processing parameters
SI 65536
SF 400.1300002
WDW no
SSB 0
LB 0.00
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00
F1P 10.000
F1 4001.30
F2P 0.000
F2 0.00
PPMCM 0.50000
HZCM 200.06500

S91

Current Data Parameters
NAME h1
EXPNO 11
PROCNO 1

F2 - Acquisition Paramet
Date_ 20060804
Time 5.19
INSTRUM drx400
PROBHD 5 mm QNP 13C
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 4
DS 0
SWH 12019.230
FIDRES 0.183399
AQ 2.7263477
RG 32
DW 41.600
DE 20.00
TE 300.0
D1 2.00000000

===== CHANNEL f1
NUC1 1H
P1 5.00
PL1 0.00
SF01 400.1317776

F2 - Processing paramete
SI 65536
SF 400.1300070
WDW no
SSB 0
LB 0.00
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00
F1P 10.000
F1 4001.30
F2P 0.000
F2 0.00
PPMCM 0.50000
HZCM 200.06500

S93

Hz

Integral

Current Data Parameters
 NAME h1
 EXPNO 11
 PROCNO 1

F2 - Acquisition Paramet
 Date_ 20060804
 Time 5.09
 INSTRUM drx400
 PROBHD 5 mm QNP 13C
 PULPROG zg
 TD 65536
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 12019.230
 FIDRES 0.183399
 AQ 2.7263477
 RG 32
 DW 41.600
 DE 20.00
 TE 300.0
 D1 2.00000000

===== CHANNEL f1
 NUC1 1H
 P1 5.00
 PL1 0.00
 SF01 400.1317776

F2 - Processing paramete
 SI 65536
 SF 400.1299971
 WDW no
 SSB 0
 LB 0.00
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00
 F1P 10.000
 F1 4001.30
 F2P 0.000
 F2 0.00
 PPMCM 0.50000
 HZCM 200.06500

ppm

147.624

134.160

132.534

129.452

127.118

126.843

124.525

120.790

77.341

77.023

76.707

59.724

56.300

48.994

27.600

22.746

4i

ppm

180

160

140

120

100

80

60

40

20