**Palladium-Catalyzed Highly Stereoselective Synthesis of
N-Aryl-3-arylmethylisoxazolidines via Tandem Arylation of
O-homoallylhydroxylamines**

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Supporting Information

- 1) General Experimental Method**
- 2) Experimental procedures**
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- 4) Proton and Carbon NMR Spectra of new compounds. NOESY and HPLC spectra of compound 27.**

General Experimental Method:

Experimental procedures and characterization data for new compounds in Table 1 and 2, complete descriptions of stereochemical assignments.

General All reactions were carried out under a nitrogen atmosphere in flame dried glassware. Tris(dibenzylideneacetone)dipalladium (0) and all phosphine ligands were purchased from commercial sources and used without further purification. All reagents except for 4-bromobenzoic acid *tert*-butyl ester and 2-(4-bromophenyl)-1,3-dioxolane were obtained from commercial sources and were used as obtained. 4-bromobenzoic acid *tert*-butyl ester¹ and 2-(4-bromophenyl)-1,3-dioxolane² were prepared according to literature procedures. The hydroxylamine substrate **1** and **11** were prepared according to literature procedures³. Yields refer to isolated yields of compounds estimated to be >95% pure as determined by ¹H NMR. All the solvents were purified using a solvent purification system. The yields reported in the supporting information describe the result of a single experiment.

General procedure for the preparation of *O*-homoallylhydroxylamines

A solution of DEAD (1.5 eq.) in dry THF (0.25 M) was added dropwise to a solution of the homoallylic alcohol³, triphenylphosphine (1.2 eq.) and *N*-hydroxyphthalimide (1.2 eq.) in dry THF (0.25 M) under nitrogen at 0°C. The mixture was allowed to warm to room temperature and stirred for five hours. THF was then evaporated and the residue was purified by flash chromatography on silica gel (H), eluting with 5-10 % ethyl acetate/ petroleum ether. The product was dissolved in dichloromethane (*ca* 0.3 M). Hydrazine hydrate (3 eq.) was added and the mixture was stirred at room temperature for four hours. The mixture was filtered through celite, washing with dichloromethane, and the dichloromethane was removed under reduced pressure. The residue was purified by flash chromatography on silica gel (H), eluting with 5-10% ethyl acetate/ petroleum ether.

hydroxylamine 6

Colorless oil, *R*_f = 0.27 (25% EtOAc : Petroleum ether); ¹H (300 MHz, CDCl₃) δ

7.28-7.30 (m, 1H, thienyl), 6.98-7.03 (m, 2H, thienyl), 5.78-5.80 (m, 1H, =CH), 5.32 (br, 2H, NH₂), 5.05-5.15 (m, 2H, =CH₂), 4.81 (m, 1H, OCH), 2.70-2.75 (m, 1H, CH₂), 2.17-2.56 (m, 1H, CH₂). ¹³C (75 MHz, CDCl₃) δ 144.6, 133.98, 126.5, 125.6, 125.1, 117.4, 81.7, 40.3. HRMS-ESI (m/z): [M + Na]⁺ calcd for C₈H₁₁NNaOS 192.0459; found, 192.0457.

hydroxylamine 7

Colorless oil, R_f = 0.28 (25% EtOAc : Petroleum ether); ¹H (300 MHz, CDCl₃) δ 7.17-7.26 (m, 4H, Ar), 5.73-5.78 (m, 1H, =CH), 5.20 (br, 2H, NH₂), 5.02-5.11 (m, 2H, =CH₂), 4.50-4.55 (m, 1H, OCH), 2.57-2.60 (m, 1H, CH₂), 2.38-2.44 (m, 1H, CH₂), 2.36 (s, 3H, CH₃). ¹³C (75 MHz, CDCl₃) δ 138.2, 137.4, 134.5, 129.1, 126.7, 116.9, 86.4, 40.4, 21.1.

hydroxylamine 8

Colorless oil, R_f = 0.31 (25% EtOAc : Petroleum ether); ¹H (300 MHz, CDCl₃) δ 7.32-7.35 (m, 2H, Ar), 7.24-7.27 (m, 2H, Ar), 5.68-5.77 (m, 1H, =CH), 5.08 (br, 2H, NH₂), 5.01-5.08 (m, 2H, =CH₂), 4.52-4.56 (t, J = 6 Hz, 1H, OCH), 2.52-2.59 (m, 1H, CH₂), 2.37-2.42 (m, 1H, CH₂). ¹³C (75 MHz, CDCl₃) δ 139.98, 133.9, 133.4, 128.6, 128.1, 117.4, 85.7, 40.3.

hydroxylamine 9

Colorless oil, R_f = 0.29 (25% EtOAc : Petroleum ether); ¹H (300 MHz, CDCl₃) δ 7.23-7.26 (m, 2H, Ar), 6.88-6.92 (m, 2H, Ar), 5.70-5.79 (m, 1H, =CH), 5.18 (br, 2H, NH₂), 5.00-5.08 (m, 2H, =CH₂), 4.46-4.51 (t, J = 6 Hz, 1H, OCH), 3.80 (s, 3H, OCH₃), 2.57-2.62 (m, 1H, CH₂), 2.39-2.42 (m, 1H, CH₂). ¹³C (75 MHz, CDCl₃) δ 152.2, 127.4, 126.1, 121.0, 109.9, 106.8, 79.1, 48.1, 33.3.

hydroxylamine 10

Colorless oil, R_f = 0.34 (25% EtOAc : Petroleum ether); ¹H (300 MHz, CDCl₃) δ 7.29-7.44 (m, 5H, Ph), 6.62-6.67 (d, J = 15 Hz, 1H, =CH), 6.12-6.20 (dd, J = 15, 9 Hz, 1H, =CH), 5.74 (m, 1H, =CH), 5.30 (br, 2H, NH₂), 5.08-5.17 (m, 2H, =CH₂), 4.15-4.18 (m, 1H, OCH), 2.35-2.51 (m, 2H, CH₂). ¹³C (75 MHz, CDCl₃) δ 135.2, 134.4, 129.7, 128.7, 128.1, 126.4, 125.5, 116.4, 81.8, 38.5. HRMS-ESI (m/z): [M +

$\text{Na}]^+$ calcd for $\text{C}_{12}\text{H}_{15}\text{NNaO}$ 212.1052; found, 212.1056.

General procedures for the one-pot palladium-catalyzed synthesis of *N*-aryl-3-arylmethylisoxazolidines using the two same aryl bromides (Table 1 and 2)

A flame-dried tube was cooled under a stream of nitrogen and charged with $\text{Pd}_2(\text{dba})_3$ (1 mol% complex, 2 mol% Pd), Xantphos (2 mol%), and NaOt-Bu (2.4 equiv). The tube was purged with nitrogen, and toluene (10 mL/mmol hydroxylamine substrate), the hydroxylamine substrate (1.0 equiv), and the aryl bromide (2.05 equiv) were added via syringe. The mixture was heated to 90°C with stirring until the starting material had been consumed as judged by TLC. The reaction mixture was cooled to room temperature, quenched with saturated aqueous ammonium chloride (2 mL), and diluted with ethyl acetate (10 mL). The layers were separated and the aqueous layer was extracted with (2 × 10 mL) ethyl acetate. The combined organic extracts were dried over anhydrous sodium sulfate, filtered, and concentrated *in vacuo*. The crude product was then purified by flash chromatography on silica gel (H), eluting with 4-30% ethyl acetate/ petroleum ether.

(±)-(3*R*, 5*R*)-3-benzyl-2,5-diphenylisoxazolidine (12). Reaction of 50 mg (0.307 mmol) of 3-phenyl-*o*-homoallylhydroxylamine (**1**) with bromobenzene (98.8 mg, 0.63 mmol) and NaOt-Bu (70.7 mg, 0.74 mmol) following General procedures afforded 79.3 mg (82 %) of the title compound as a light yellow oil. R_f = 0.24 (10% EtOAc : Petroleum ether); ^1H (300 MHz, CDCl_3) δ 7.20-7.45 (m, 12H, Ph), 6.88-6.92 (m, 3H, Ph), 5.07-5.13 (m, 1H, OCH), 4.14 (m, 1H, NCH), 3.26-3.33 (dd, J = 13.5, 7.8 Hz, 1H, ArCH_2), 2.92-2.99 (dd, J = 13.5, 6.2 Hz, 1H, ArCH_2), 2.76-2.80 (m, 1H, CH_2), 2.15-2.20 (m, 1H, CH_2). ^{13}C (75 MHz, CDCl_3) δ 151.9, 138.9, 138.6, 129.5, 128.9, 128.5, 128.1, 127.4, 126.6, 126.4, 121.3, 113.9, 79.9, 69.6, 43.8, 43.0. HRMS-ESI (m/z): $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{22}\text{H}_{21}\text{NNaO}$ 338.1515; found, 338.1517.

(±)-3-[(3*R*, 5*R*)-5-phenyl-2-(pyridine-3-yl)isoxazolidin-3-yl]methylpyridine (13a). Reaction of 50 mg (0.307 mmol) of 3-phenyl-*o*-homoallylhydroxylamine (**1**) with 3-bromopyridine (99.4 mg, 0.63 mmol) and NaOt-Bu (70.7 mg, 0.74 mmol) following General procedures afforded 43.8 mg (45 %) of the title compound as a

light yellow oil. R_f = 0.23 (30% EtOAc : Petroleum ether); ^1H (300 MHz, CDCl_3) δ 8.52-8.58 (br, 2H, Py), 8.17 (br, 2H, Py), 7.68-7.70 (m, 1H, Py), 7.33-7.42 (m, 6H, Ar), 7.10-7.12 (m, 2H, Py), 5.04-5.09 (m, 1H, OCH), 4.08-4.13 (m, 1H, NCH), 3.18-3.25 (dd, J = 13.5, 8.7 Hz, 1H, ArCH_2), 2.93-3.00 (dd, J = 13.5, 5.3 Hz, 1H, ArCH_2), 2.85-2.89 (m, 1H, CH_2), 2.19 (m, 1H, CH_2). ^{13}C (75 MHz, CDCl_3) δ 148.9, 148.7, 145.1, 139.2, 138.9, 138.4, 137.6, 135.4, 128.6, 126.0, 124.7, 123.7, 122.8, 77.1, 68.7, 42.6, 37.1. HRMS-ESI (m/z): $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{20}\text{H}_{20}\text{N}_3\text{O}$ 318.1601; found, 318.1601.

3-(3-methylene-5-phenylisoxazolidin-2-yl)pyridine (13b). Reaction of 50 mg (0.307 mmol) of 3-phenyl-*o*-homoallylhydroxylamine (**1**) with 3-bromopyridine (99.4 mg, 0.63 mmol) and NaOt-Bu (70.7 mg, 0.74 mmol) following General procedures afforded 22 mg (30 %) of the title compound as a light yellow oil. R_f = 0.26 (10% EtOAc : Petroleum ether); ^1H (300 MHz, CDCl_3) δ 8.54 (br, 2H, Py), 7.57-7.59 (d, J = 6 Hz, 1H, Py), 7.26-7.37 (m, 6H, Ar), 5.53-5.59 (dd, J = 10.8, 8.4 Hz, 1H, OCH), 3.67-3.79 (q, J = 19 Hz, 2H, $=\text{CH}_2$), 3.21-3.30 (dd, J = 17.1, 10.8 Hz, 1H, CH_2), 2.77-2.85 (dd, J = 17.1, 8.4 Hz, 1H, CH_2). ^{13}C (75 MHz, CDCl_3) δ 156.3, 149.9, 148.7, 140.6, 136.2, 128.7, 128.1, 125.6, 123.8, 81.9, 44.4, 31.4. HRMS-ESI (m/z): $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{15}\text{H}_{14}\text{N}_2\text{NaO}$ 261.0998; found, 261.1008.

($\pm$)-2-[(3*R*, 5*R*)-5-phenyl-3-[(pyridin-2-yl)methyl]isoxazolidin-2-yl]pyridine (14). Reaction of 50 mg (0.307 mmol) of 3-phenyl-*o*-homoallylhydroxylamine (**1**) with 2-bromopyridine (99.4 mg, 0.63 mmol) and NaOt-Bu (70.7 mg, 0.74 mmol) following General procedures afforded 72 mg (74 %) of the title compound as a light yellow oil. R_f = 0.28 (35% EtOAc : Petroleum ether); ^1H (300 MHz, CDCl_3) δ 8.60 (m, 1H, Py), 8.29-8.30 (m, 1H, Py), 7.60-7.63 (m, 2H, Py), 7.38-7.42 (m, 6H, Ar), 7.25-7.30 (m, 1H, Py), 7.10-7.15 (m, 1H, Py), 6.86-6.87 (m, 1H, Py), 5.34 (m, 1H, OCH), 4.89-4.95 (m, 1H, NCH), 3.57-3.63 (dd, J = 13.5, 6.4 Hz, 1H, ArCH_2), 3.22-3.30 (dd, J = 13.5, 7.7 Hz, 1H, ArCH_2), 2.72-2.77 (m, 1H, CH_2), 2.25-2.30 (m, 1H, CH_2). ^{13}C (75 MHz, CDCl_3) δ 162.8, 158.9, 149.1, 147.7, 138.2, 137.8, 136.2, 128.5, 128.2, 126.7, 124.1, 121.4, 116.8, 109.2, 81.2, 62.6, 44.4, 42.7. HRMS-ESI (m/z): $[\text{M} + \text{H}]^+$ calcd for

C₂₀H₂₀N₃O 318.1601; found, 318.1604.

(±)-[(3*R*, 5*R*)-5-phenyl-2-(pyridine-4-yl)isoxazolidin-3-yl]methylpyridine (15).

Reaction of 50 mg (0.307 mmol) of 3-phenyl-*o*-homoallylhydroxylamine (**1**) with 4-bromopyridine hydrochloride (122.1 mg, 0.63 mmol) and NaOt-Bu (141.4 mg, 1.48 mmol) following General procedures afforded 72 mg (74 %) of the title compound as a white solid. *R*_f = 0.26 (35% EtOAc : Petroleum ether); ¹H (300 MHz, CDCl₃) δ 8.55-8.57 (m, 2H, Py), 8.27-8.29 (m, 2H, Py), 7.37-7.41 (m, 5H, Ph), 7.25-7.27 (m, 2H, Py), 6.61-6.63 (m, 2H, Py), 4.94-4.99 (m, 1H, OCH), 4.15 (m, 1H, NCH), 3.15-3.23 (dd, *J* = 13.5, 8.7 Hz, 1H, ArCH₂), 2.92-2.98 (dd, *J* = 13.5, 5 Hz, 1H, ArCH₂), 2.87-2.91 (m, 1H, CH₂), 2.15-2.17 (m, 1H, CH₂). ¹³C (75 MHz, CDCl₃) δ 157.1, 150.2, 150.0, 149.8, 147.1, 137.1, 128.7, 126.8, 124.8, 107.8, 80.7, 67.0, 43.8, 41.8. HRMS-ESI (*m/z*): [M + H]⁺ calcd for C₂₀H₂₀N₃O 318.1601; found, 318.1596.

(±)-(3*R*, 5*R*)-3-(4-methylbenzyl)-5-phenyl-2-(4-methylphenyl)isoxazolidine (17).

Reaction of 50 mg (0.307 mmol) of 3-phenyl-*o*-homoallylhydroxylamine (**1**) with 1-bromo-4-methylbenzene (107.6 mg, 0.63 mmol) and NaOt-Bu (70.7 mg, 0.74 mmol) following General procedures afforded 82.1 mg (78 %) of the title compound as a light yellow oil. *R*_f = 0.31 (10% EtOAc : Petroleum ether); ¹H (300 MHz, CDCl₃) δ 7.34-7.44 (m, 5H, Ph), 7.19-7.22 (d, *J* = 9 Hz, 2H, *p*-Me-Ph), 7.11-7.14 (d, *J* = 9 Hz, 2H, *p*-Me-Ph), 7.03-7.06 (d, *J* = 9 Hz, 2H, *p*-Me-Ph), 6.82-6.85 (d, *J* = 9 Hz, 2H, *p*-Me-Ph), 5.07-5.12 (m, 1H, OCH), 4.06-4.08 (m, 1H, NCH), 3.21-3.28 (dd, *J* = 13.5, 7.5 Hz, 1H, ArCH₂), 2.86-2.93 (dd, *J* = 13.5, 6.2 Hz, 1H, ArCH₂), 2.70-2.76 (m, 1H, CH₂), 2.34 (s, 3H, CH₃), 2.28 (s, 3H, CH₃), 1.80-2.12 (m, 1H, CH₂). ¹³C (75 MHz, CDCl₃) δ 147.1, 139.2, 135.7, 135.5, 133.1, 128.9, 128.6, 128.2, 128.1, 126.9, 126.0, 116.5, 79.8, 69.4, 43.7, 42.9, 24.5, 24.3. HRMS-ESI (*m/z*): [M + Na]⁺ calcd for C₂₄H₂₅NNaO 366.1834; found, 366.1836.

(±)-(3*R*, 5*R*)-3-(4-phenylbenzyl)-5-phenyl-2-(4-biphenyl)isoxazolidine (18).

Reaction of 50 mg (0.307 mmol) of 3-phenyl-*o*-homoallylhydroxylamine (**1**) with 4-biphenyl bromide (146.6 mg, 0.63 mmol) and NaOt-Bu (70.7 mg, 0.74 mmol) following General procedures afforded 123.3 mg (86 %) of the title compound as a

white solid. $R_f = 0.32$ (10% EtOAc : Petroleum ether); ^1H (300 MHz, CDCl_3) δ 7.41-7.66 (m, 21H, Ar), 7.01-7.04 (d, $J = 9$ Hz, 2H, Ar), 5.15-5.21 (m, 1H, OCH), 4.25 (m, 1H, NCH), 3.34-3.41 (dd, $J = 13.5, 7.8$ Hz, 1H, ArCH_2), 3.02-3.08 (dd, $J = 13.5, 6$ Hz, 1H, ArCH_2), 2.86-2.90 (m, 1H, CH_2), 2.23-2.28 (m, 1H, CH_2). ^{13}C (75 MHz, CDCl_3) δ 151.3, 140.9, 140.7, 139.4, 138.5, 138.0, 134.3, 130.0, 128.9, 128.7, 128.65, 128.6, 128.2, 127.7, 127.2, 127.1, 127.0, 126.7, 126.6, 114.3, 80.1, 69.5, 43.9, 42.6. HRMS-ESI (m/z): $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{34}\text{H}_{29}\text{NNaO}$ 490.2147; found, 490.2144.

($\pm$)-(3*R*, 5*R*)-2-(naphthalen-1-yl) -3-[(naphthalen-1-yl)methyl]-5-phenyl isoxazolidine (19).

Reaction of 50 mg (0.307 mmol) of 3-phenyl-*o*-homoallylhydroxylamine (**1**) with 1-bromonaphthalene (130.3 mg, 0.63 mmol) and NaOt-Bu (70.7 mg, 0.74 mmol) following General procedures afforded 96.8 mg (76 %) of the title compound as a light yellow oil. $R_f = 0.27$ (10% EtOAc : Petroleum ether); ^1H (300 MHz, CDCl_3) δ 7.77 (m, 1H, Ar), 7.61-7.66 (m, 3H, Ar), 7.51 (m, 1H, Ar), 7.29-7.31 (m, 4H, Ar), 7.10-7.19 (m, 9H, Ar), 6.72-6.75 (m, 1H, Ar), 5.12-5.18 (m, 1H, OCH), 4.19 (m, 1H, NCH), 3.30-3.37 (dd, $J = 13.5, 7.8$ Hz, 1H, ArCH_2), 2.97-3.03 (dd, $J = 13.5, 6$ Hz, 1H, ArCH_2), 2.80-2.84 (m, 1H, CH_2), 2.20-2.25 (m, 1H, CH_2). ^{13}C (75 MHz, CDCl_3) δ 142.1, 139.2, 137.5, 135.3, 134.3, 132.6, 128.8, 128.7, 128.6, 128.2, 126.9, 126.6, 126.5, 126.2, 126.0, 125.8, 125.6, 125.0, 124.7, 124.3, 124.2, 121.0, 119.2, 109.4, 80.2, 69.7, 43.6, 41.8. HRMS-ESI (m/z): $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{30}\text{H}_{25}\text{NNaO}$ 483.1834; found, 483.1837.

($\pm$)-(3*R*, 5*R*)-3-(4-chlorobenzyl)-2-(4-chlorophenyl) -5-phenylisoxazolidine (20).

Reaction of 50 mg (0.307 mmol) of 3-phenyl-*o*-homoallylhydroxylamine (**1**) with 1-bromo-4-chlorobenzene (120.2 mg, 0.63 mmol) and NaOt-Bu (70.7 mg, 0.74 mmol) following General procedures afforded 101.4 mg (86 %) of the title compound as a light yellow oil. $R_f = 0.19$ (10% EtOAc : Petroleum ether); ^1H (300 MHz, CDCl_3) δ 7.38-7.42 (m, 5H, Ph), 7.24-7.32 (m, 4H, *p*-Cl-Ph), 7.15-7.18 (m, 2H, *p*-Cl-Ph), 6.75-6.78 (m, 2H, *p*-Cl-Ph), 5.03-5.08 (m, 1H, OCH), 4.03 (m, 1H, NCH), 3.15-3.22

(dd, $J = 13.5, 8.4$ Hz, 1H, ArCH₂), 2.86-2.93 (dd, $J = 13.5, 5.5$ Hz, 1H, ArCH₂), 2.79-2.83 (m, 1H, CH₂), 2.12-2.17 (m, 1H, CH₂). ¹³C (75 MHz, CDCl₃) δ 150.4, 138.2, 137.3, 132.5, 131.3, 130.9, 128.9, 128.7, 128.4, 126.6, 126.4, 115.1, 80.0, 69.5, 43.8, 42.1. HRMS-ESI (m/z): $[M + Na]^+$ calcd for C₂₂H₁₉Cl₂NNaO 406.0741; found, 406.0744.

(±)-(3R, 5R)-3-(4-nitrobenzyl)-2-(4-nitrophenyl) -5-phenylisoxazolidine (21).

Reaction of 50 mg (0.307 mmol) of 3-phenyl-*o*-homoallylhydroxylamine (**1**) with 1-bromo-4-nitrobenzene (127.2 mg, 0.63 mmol) and NaO*t*-Bu (70.7 mg, 0.74 mmol) following General procedures afforded 104.4 mg (84 %) of the title compound as a yellow solid. $R_f = 0.22$ (20% EtOAc : Petroleum ether); ¹H (300 MHz, CDCl₃) δ 8.19-8.22 (d, $J = 9$ Hz, 2H, *p*-NO₂-Ph), 8.04-8.07 (d, $J = 9$ Hz, 2H, *p*-NO₂-Ph), 7.51-7.54 (d, $J = 9$ Hz, 2H, *p*-NO₂-Ph), 7.37-7.46 (m, 5H, Ph), 6.74-6.77 (d, $J = 9$ Hz, 2H, *p*-NO₂-Ph), 4.99-5.05 (m, 1H, OCH), 4.24-4.28 (m, 1H, NCH), 3.28-3.36 (dd, $J = 13.5, 8.9$ Hz, 1H, ArCH₂), 3.09-3.15 (dd, $J = 13.5, 4.8$ Hz, 1H, ArCH₂), 2.92-2.99 (m, 1H, CH₂), 2.21-2.27 (m, 1H, CH₂). ¹³C (75 MHz, CDCl₃) δ 156.0, 147.0, 145.7, 141.4, 136.9, 130.5, 128.8, 126.5, 125.5, 123.9, 112.3, 81.0, 67.7, 43.7, 42.2. HRMS-ESI (m/z): $[M + Na]^+$ calcd for C₂₂H₁₉N₃NaO₅ 428.1217; found, 428.1226.

(±)-(3R, 5R)-3-(3-nitrobenzyl)-2-(3-nitrophenyl) -5-phenylisoxazolidine (22).

Reaction of 50 mg (0.307 mmol) of 3-phenyl-*o*-homoallylhydroxylamine (**1**) with 1-bromo-3-nitrobenzene (127.2 mg, 0.63 mmol) and NaO*t*-Bu (70.7 mg, 0.74 mmol) following General procedures afforded 93.3 mg (75 %) of the title compound as a yellow solid. $R_f = 0.23$ (20% EtOAc : Petroleum ether); ¹H (300 MHz, CDCl₃) δ 8.26 (s, 1H, *m*-NO₂-Ph), 8.25 (m, 1H, *m*-NO₂-Ph), 7.74-7.76 (m, 2H, *m*-NO₂-Ph), 7.57-7.60 (m, 2H, *m*-NO₂-Ph), 7.29-7.48 (m, 6H, Ar), 7.11 (m, 1H, *m*-NO₂-Ph), 5.08-5.14 (m, 1H, OCH), 4.23 (m, 1H, NCH), 3.32-3.39 (dd, $J = 13.5, 9$ Hz, 1H, ArCH₂), 3.13-3.19 (dd, $J = 13.5, 4.8$ Hz, 1H, ArCH₂), 2.95-2.99 (m, 1H, CH₂), 2.25-2.27 (m, 1H, CH₂). ¹³C (75 MHz, CDCl₃) δ 152.6, 149.0, 148.5, 140.4, 137.4, 135.9, 129.9, 129.7, 128.8, 128.7, 126.9, 126.5, 124.3, 122.1, 119.2, 108.3, 80.5, 69.0, 43.7, 42.1. HRMS-ESI (m/z): $[M + Na]^+$ calcd for C₂₂H₁₉N₃NaO₅ 428.1217; found,

428.1202.

(±)-(3*R*, 5*R*)-3-(4-cyanobenzyl)-2-(4-cyanophenyl) -5-phenylisoxazolidine (23).

Reaction of 50 mg (0.307 mmol) of 3-phenyl-*o*-homoallylhydroxylamine (**1**) with 4-bromobenzonitrile (114.5 mg, 0.63 mmol) and NaOt-Bu (70.7 mg, 0.74 mmol) following General procedures afforded 100.9 mg (90 %) of the title compound as a white solid. R_f = 0.24 (20% EtOAc : Petroleum ether); ^1H (300 MHz, CDCl_3) δ 7.61-7.64 (d, J = 9 Hz, 2H, *p*-CN-Ph), 7.34-7.47 (m, 9H, Ar), 6.75-6.78 (d, J = 9 Hz, 2H, *p*-CN-Ph), 4.97-5.02 (m, 1H, OCH), 4.14-4.18 (m, 1H, NCH), 3.22-3.29 (dd, J = 13.5, 9 Hz, 1H, ArCH₂), 3.01-3.07 (dd, J = 13.5, 4.8 Hz, 1H, ArCH₂), 2.89-2.93 (m, 1H, CH₂), 2.04-2.23 (m, 1H, CH₂). ^{13}C (75 MHz, CDCl_3) δ 154.5, 143.8, 137.1, 133.3, 132.3, 130.4, 128.7, 128.6, 126.5, 119.2, 118.6, 113.2, 110.7, 103.8, 80.6, 68.0, 43.6, 42.5. HRMS-ESI (m/z): $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{24}\text{H}_{19}\text{N}_3\text{NaO}$ 388.1420; found, 388.1423.

(±)-(3*R*, 5*R*)-3-(4-*tert*-butoxycarbonylbenzyl)-2-(4-*tert*-butoxycarbonylphenyl) -5-phenylisoxazolidine (24). Reaction of 50 mg (0.307 mmol) of 3-phenyl-*o*-homoallylhydroxylamine (**1**) with *tert*-butyl 4-bromobenzoate (161.7 mg, 0.63 mmol) and NaOt-Bu (70.7 mg, 0.74 mmol) following General procedures afforded 107.5 mg (68 %) of the title compound as a white solid. This compound was obtained as a ca. 4 : 1 mixture of inseparable diastereomers as judged by ^1H NMR analysis; data are for the major isomer. R_f = 0.25 (15% EtOAc : Petroleum ether); ^1H (300 MHz, CDCl_3) δ 7.94-7.97 (d, J = 9 Hz, 2H, *p*-CO₂*t*Bu-Ph), 7.82-7.85 (d, J = 9 Hz, 2H, *p*-CO₂*t*Bu-Ph), 7.36-7.43 (m, 7H, Ar), 6.79-6.82 (d, J = 9 Hz, 2H, *p*-CO₂*t*Bu-Ph), 5.00-5.05 (m, 1H, OCH), 4.16-4.21 (m, 1H, NCH), 3.26-3.33 (dd, J = 13.5, 8.5 Hz, 1H, ArCH₂), 2.97-3.03 (dd, J = 13.5, 5.5 Hz, 1H, ArCH₂), 2.80-2.85 (m, 1H, CH₂), 2.16 (m, 1H, CH₂), 1.60 (s, 9H, *t*-Bu), 1.56 (s, 9H, *t*-Bu). ^{13}C (75 MHz, CDCl_3) δ 165.6, 165.5, 154.9, 143.4, 137.8, 130.8, 130.4, 129.8, 129.4, 128.7, 128.5, 126.6, 124.7, 113.8, 112.6, 80.9, 80.4, 68.7, 43.8, 42.8, 28.22, 28.19. HRMS-ESI (m/z): $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{32}\text{H}_{37}\text{NNaO}_5$ 538.2564; found, 538.2565. data are for the minor isomer. R_f = 0.25 (15% EtOAc : Petroleum ether); ^1H (300 MHz, CDCl_3)

δ 7.94-7.97 (d, $J = 9$ Hz, 2H, *p*-CO₂*t*Bu-Ph), 7.82-7.85 (d, $J = 9$ Hz, 2H, *p*-CO₂*t*Bu-Ph), 7.36-7.43 (m, 7H, Ar), 6.86-6.89 (d, $J = 9$ Hz, 2H, *p*-CO₂*t*Bu-Ph), 5.29 (m, 1H, OCH), 4.16-4.21 (m, 1H, NCH), 3.26-3.33 (dd, $J = 13.5, 8.5$ Hz, 1H, ArCH₂), 2.97-3.03 (dd, $J = 13.5, 5.5$ Hz, 1H, ArCH₂), 2.50-2.55 (m, 1H, CH₂), 2.43-2.48 (m, 1H, CH₂), 1.60 (s, 9H, *t*-Bu), 1.56 (s, 9H, *t*-Bu). ¹³C (75 MHz, CDCl₃) δ 165.6, 165.5, 153.7, 142.9, 138.9, 130.8, 130.4, 129.8, 129.4, 128.6, 128.3, 126.8, 124.5, 113.8, 112.6, 80.9, 80.0, 67.2, 40.9, 40.0, 28.22, 28.19. HRMS-ESI (m/z): $[M + Na]^+$ calcd for C₃₂H₃₇NNaO₅ 538.2564; found, 538.2565.

(±)-(3*R*, 5*R*)-2-[4-(1,3-dioxolan-2-yl)phenyl]-3-[4-(1,3-dioxolan-2-yl)benzyl]-5-phenylisoxazolidine (25). Reaction of 50 mg (0.307 mmol) of 3-phenyl-*o*-homoallylhydroxylamine (**1**) with 2-(4-bromophenyl)-1,3-dioxolane (144.1 mg, 0.63 mmol) and NaOt-Bu (70.7 mg, 0.74 mmol) following General procedures afforded 87.4 mg (62 %) of the title compound as a light yellow oil. $R_f = 0.21$ (15% EtOAc : Petroleum ether); ¹H (300 MHz, CDCl₃) δ 7.33-7.46 (m, 11H, Ar), 6.89-6.92 (d, $J = 9$ Hz, 2H, *p*-(1,3-dioxolan-2-yl)ph), 5.81 (s, 1H, OCHO), 5.74 (s, 1H, OCHO), 5.03-5.08 (m, 1H, OCH), 4.10-4.14 (m, 5H, OCH₂ and NCH), 4.00-4.04 (m, 4H, OCH₂), 3.25-3.32 (dd, $J = 13.5, 9$ Hz, 1H, ArCH₂), 2.95-3.01 (dd, $J = 13.5, 4.8$ Hz, 1H, ArCH₂), 2.72-2.78 (m, 1H, CH₂), 2.07-2.23 (m, 1H, CH₂). ¹³C (75 MHz, CDCl₃) δ 149.1, 139.2, 137.8, 134.5, 132.0, 128.6, 128.2, 128.1, 127.3, 126.0, 125.3, 116.5, 109.7, 109.2, 80.6, 68.7, 66.7, 66.2, 43.6, 42.3. HRMS-ESI (m/z): $[M + Na]^+$ calcd for C₂₈H₂₉NNaO₅ 482.1943; found, 482.1946.

(±)-(3*R*, 5*R*)-3-(4-methoxybenzyl)-2-(4-methoxyphenyl) -5-phenylisoxazolidine (26). Reaction of 50 mg (0.307 mmol) of 3-phenyl-*o*-homoallylhydroxylamine (**1**) with 1-bromo-4-methoxybenzene (117.7 mg, 0.63 mmol) and NaOt-Bu (70.7 mg, 0.74 mmol) following General procedures afforded 48.4 mg (42 %) of the title compound as a light yellow oil. $R_f = 0.23$ (10% EtOAc : Petroleum ether); ¹H (300 MHz, CDCl₃) δ 7.34-7.44 (m, 5H, Ph), 7.00-7.03 (d, $J = 9$ Hz, 2H, *p*-MeO-Ph), 6.71-6.74 (d, $J = 9$ Hz, 2H, *p*-MeO-Ph), 6.54-6.57 (d, $J = 9$ Hz, 2H, *p*-MeO-Ph), 6.31-6.34 (d, $J = 9$ Hz, 2H, *p*-MeO-Ph), 4.94-4.99 (m, 1H, OCH), 4.18-4.23 (m, 1H,

NCH), 3.80 (s, 3H, OMe), 3.78 (s, 3H, OMe), 3.17-3.24 (dd, $J = 13.5, 7.2$ Hz, 1H, ArCH₂), 2.93-3.00 (dd, $J = 13.5, 6.8$ Hz, 1H, ArCH₂), 2.87-2.91 (m, 1H, CH₂), 2.01-2.20 (m, 1H, CH₂). ¹³C (75 MHz, CDCl₃) δ 157.9, 155.4, 142.3, 139.2, 131.0, 129.2, 128.6, 128.2, 126.0, 117.6, 114.1, 112.1, 80.2, 68.2, 55.9, 55.4, 43.5, 42.2. HRMS-ESI (m/z): $[M + Na]^+$ calcd for C₂₄H₂₅NNaO₃ 398.1732; found, 398.1735.

(±)-(3*R*, 5*R*)-3-(4-nitrobenzyl)-2-(4-nitrophenyl) -5-(thiophen-2-yl)isoxazolidine (27). Reaction of 52 mg (0.307 mmol) of 3-(thiophen-2-yl) -*o*-homoallylhydroxylamine (**6**) with 1-bromo-4-nitrobenzene (127.2 mg, 0.63 mmol) and NaOt-Bu (70.7 mg, 0.74 mmol) following General procedures afforded 102.2 mg (81 %) of the title compound as a yellow solid. $R_f = 0.23$ (20% EtOAc : Petroleum ether); ¹H (300 MHz, CDCl₃) δ 8.18-8.21 (d, $J = 9$ Hz, 2H, *p*-NO₂-Ph), 8.04-8.07 (d, $J = 9$ Hz, 2H, *p*-NO₂-Ph), 7.51-7.54 (d, $J = 9$ Hz, 2H, *p*-NO₂-Ph), 7.39-7.41 (m, 1H, thienyl), 7.15-7.17 (m, 1H, thienyl), 7.04-7.07 (m, 1H, thienyl), 6.76-6.79 (d, $J = 9$ Hz, 2H, *p*-NO₂-Ph), 5.29-5.35 (m, 1H, OCH), 4.28-4.29 (m, 1H, NCH), 3.31-3.38 (dd, $J = 13.5, 9$ Hz, 1H, ArCH₂), 3.10-3.16 (dd, $J = 13.5, 4.8$ Hz, 1H, ArCH₂), 2.96-3.00 (m, 1H, CH₂), 2.28-2.37 (m, 1H, CH₂). ¹³C (75 MHz, CDCl₃) δ 155.7, 147.0, 145.7, 141.5, 139.3, 130.5, 127.1, 127.0, 126.6, 125.5, 123.9, 112.4, 76.7, 67.7, 43.5, 41.8. HRMS-ESI (m/z): $[M + Na]^+$ calcd for C₂₀H₁₇N₃NaO₅S 434.0781; found, 434.0806.

(±)-(3*R*, 5*R*)-3-(4-*tert*-butoxycarbonylbenzyl)-2-(4-*tert*-butoxycarbonylphenyl) -5-(thiophen-2-yl)isoxazolidine (28). Reaction of 52 mg (0.307 mmol) of 3-(thiophen-2-yl) -*o*-homoallylhydroxylamine (**6**) with *tert*-butyl 4-bromobenzoate (161.7 mg, 0.63 mmol) and NaOt-Bu (70.7 mg, 0.74 mmol) following General procedures afforded 115.2 mg (72 %) of the title compound as a white solid. $R_f = 0.28$ (15% EtOAc : Petroleum ether); ¹H (300 MHz, CDCl₃) δ 7.94-7.97 (m, 2H, *p*-CO₂*t*Bu-Ph), 7.83-7.86 (m, 2H, *p*-CO₂*t*Bu-Ph), 7.37-7.40 (m, 3H, Ar), 7.13-7.14 (m, 1H, thienyl), 7.02-7.05 (m, 1H, thienyl), 6.79-6.82 (m, 2H, *p*-CO₂*t*Bu-Ph), 5.28-5.34 (m, 1H, OCH), 4.18-4.19 (m, 1H, NCH), 3.28-3.36 (dd, $J = 13.5, 8.6$ Hz, 1H, ArCH₂), 2.97-3.03 (dd, $J = 13.5, 5.5$ Hz, 1H, ArCH₂), 2.80-2.84 (m, 1H, CH₂), 2.21-2.29 (m, 1H, CH₂), 1.59 (s, 9H, *t*-Bu), 1.56 (s, 9H, *t*-Bu). ¹³C (75 MHz, CDCl₃) δ 165.7, 165.5,

154.5, 143.4, 140.5, 130.8, 130.5, 129.8, 129.3, 126.9, 126.6, 126.2, 125.0, 112.8, 80.9, 80.4, 76.1, 68.7, 43.5, 42.4, 28.21, 28.19. HRMS-ESI (m/z): $[M + Na]^+$ calcd for $C_{30}H_{35}NNaO_5S$ 544.2128; found, 544.2160.

(±)-(3*R*, 5*R*)-3-(4-nitrobenzyl)-2-(4-nitrophenyl) -5-*p*-tolylisoxazolidine (29).

Reaction of 54.3 mg (0.307 mmol) of 3-*p*-tolyl-*o*-homoallylhydroxylamine (**7**) with 1-bromo-4-nitrobenzene (127.2 mg, 0.63 mmol) and NaOt-Bu (70.7 mg, 0.74 mmol) following General procedures afforded 105.5 mg (82 %) of the title compound as a yellow solid. R_f = 0.21 (20% EtOAc : Petroleum ether); 1H (300 MHz, $CDCl_3$) δ 8.19-8.22 (d, J = 9 Hz, 2H, *p*-NO₂-Ph), 8.04-8.07 (d, J = 9 Hz, 2H, *p*-NO₂-Ph), 7.51-7.54 (d, J = 9 Hz, 2H, *p*-NO₂-Ph), 7.21-7.31 (m, 4H, *p*-Me-Ph), 6.74-6.77 (d, J = 9 Hz, 2H, *p*-NO₂-Ph), 4.95-5.00 (m, 1H, OCH), 4.24-4.25 (m, 1H, NCH), 3.28-3.36 (dd, J = 13.5, 9 Hz, 1H, ArCH₂), 3.09-3.15 (dd, J = 13.5, 4.8 Hz, 1H, ArCH₂), 2.92-2.95 (m, 1H, CH₂), 2.39 (s, 3H, Me), 2.21-2.26 (m, 1H, CH₂). ^{13}C (75 MHz, $CDCl_3$) δ 156.1, 147.0, 145.8, 141.4, 138.9, 133.7, 130.5, 129.5, 126.6, 126.1, 125.5, 123.9, 112.3, 81.0, 67.7, 43.6, 42.2, 21.2. HRMS-ESI (m/z): $[M + K]^+$ calcd for $C_{23}H_{21}KN_3O_5$ 458.1113; found, 458.1110.

(±)-(3*R*, 5*R*)-3-(4-nitrobenzyl)-2-(4-nitrophenyl)-5-(4-chlorophenyl)isoxazolidine (30).

Reaction of 60.5 mg (0.307 mmol) of 3-(4-chlorophenyl)-*o*-homoallyl hydroxylamine (**8**) with 1-bromo-4-nitrobenzene (127.2 mg, 0.63 mmol) and NaOt-Bu (70.7 mg, 0.74 mmol) following General procedures afforded 113.5 mg (84 %) of the title compound as a yellow solid. R_f = 0.22 (20% EtOAc : Petroleum ether); 1H (300 MHz, $CDCl_3$) δ 8.17-8.20 (d, J = 9 Hz, 2H, *p*-NO₂-Ph), 8.01-8.04 (d, J = 9 Hz, 2H, *p*-NO₂-Ph), 7.50-7.53 (d, J = 9 Hz, 2H, *p*-NO₂-Ph), 7.32-7.40 (m, 4H, *p*-Cl-Ph), 6.73-6.76 (d, J = 9 Hz, 2H, *p*-NO₂-Ph), 4.97-5.03 (m, 1H, OCH), 4.24-4.25 (m, 1H, NCH), 3.25-3.33 (dd, J = 13.5, 9 Hz, 1H, ArCH₂), 3.08-3.14 (dd, J = 13.5, 4.8 Hz, 1H, ArCH₂), 2.95-2.99 (m, 1H, CH₂), 2.15-2.21 (m, 1H, CH₂). ^{13}C (75 MHz, $CDCl_3$) δ 155.9, 147.0, 145.6, 141.5, 135.5, 134.6, 130.5, 129.0, 127.8, 125.5, 123.8, 112.3, 80.2, 67.7, 43.7, 42.1. HRMS-ESI (m/z): $[M + Na]^+$ calcd for $C_{22}H_{18}N_3NaO_5$ 462.0827; found, 462.0813.

(±)-(3*R*, 5*R*)-3-(4-nitrobenzyl)-2-(4-nitrophenyl)-5-(4-methoxyphenyl)isoxazolidine (31). Reaction of 59.3 mg (0.307 mmol) of 3-(4-methoxyphenyl)-*o*-homoallyl hydroxylamine (**9**) with 1-bromo-4-nitrobenzene (127.2 mg, 0.63 mmol) and NaO*t*-Bu (70.7 mg, 0.74 mmol) following General procedures afforded 113.5 mg (85 %) of the title compound as a yellow solid. R_f = 0.24 (20% EtOAc : Petroleum ether); ^1H (300 MHz, CDCl_3) δ 8.19-8.22 (d, J = 9 Hz, 2H, *p*-NO₂-Ph), 8.03-8.06 (d, J = 9 Hz, 2H, *p*-NO₂-Ph), 7.51-7.54 (d, J = 9 Hz, 2H, *p*-NO₂-Ph), 7.32-7.35 (d, J = 9 Hz, 2H, *p*-MeO-Ph), 6.93-6.96 (d, J = 9 Hz, 2H, *p*-MeO-Ph), 6.73-6.76 (d, J = 9 Hz, 2H, *p*-NO₂-Ph), 4.92-4.98 (m, 1H, OCH), 4.23-4.26 (m, 1H, NCH), 3.84 (s, 3H, OMe), 3.28-3.36 (dd, J = 13.5, 9 Hz, 1H, ArCH₂), 3.11-3.16 (dd, J = 13.5, 4.8 Hz, 1H, ArCH₂), 2.89 (m, 1H, CH₂), 2.21-2.23 (m, 1H, CH₂). ^{13}C (75 MHz, CDCl_3) δ 160.1, 156.1, 147.0, 145.8, 141.4, 130.5, 128.4, 128.1, 125.5, 123.9, 114.2, 112.2, 80.9, 67.7, 55.4, 43.5, 42.2. HRMS-ESI (m/z): $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{23}\text{H}_{21}\text{N}_3\text{NaO}_6$ 458.1323; found, 458.1319.

(±)-(3*R*, 5*R*)-3-(4-nitrobenzyl)-2-(4-nitrophenyl)-5-styrylisoxazolidine (32). Reaction of 58.0 mg (0.307 mmol) of 3-styryl-*o*-homoallylhydroxylamine (**10**) with 1-bromo-4-nitrobenzene (127.2 mg, 0.63 mmol) and NaO*t*-Bu (70.7 mg, 0.74 mmol) following General procedures afforded 108.5 mg (82 %) of the title compound as a yellow solid. R_f = 0.22 (20% EtOAc : Petroleum ether); ^1H (300 MHz, CDCl_3) δ 8.19-8.22 (d, J = 9 Hz, 2H, *p*-NO₂-Ph), 8.03-8.06 (d, J = 9 Hz, 2H, *p*-NO₂-Ph), 7.50-7.53 (d, J = 9 Hz, 2H, *p*-NO₂-Ph), 7.26-7.45 (m, 5H, Ph), 6.73-6.78 (m, 3H, *p*-NO₂-Ph and =CH), 6.19-6.27 (m, 1H, =CH), 4.67-4.69 (m, 1H, OCH), 4.19-4.22 (m, 1H, NCH), 3.23-3.31 (dd, J = 13.5, 9 Hz, 1H, ArCH₂), 3.04-3.10 (dd, J = 13.5, 4.8 Hz, 1H, ArCH₂), 2.74-2.78 (m, 1H, CH₂), 2.04-2.09 (m, 1H, CH₂). ^{13}C (75 MHz, CDCl_3) δ 156.0, 147.0, 145.9, 141.5, 135.6, 135.4, 130.4, 128.7, 128.6, 126.7, 125.9, 124.6, 123.9, 112.5, 80.3, 67.7, 42.0, 41.8. HRMS-ESI (m/z): $[\text{M} + \text{K}]^+$ calcd for $\text{C}_{24}\text{H}_{21}\text{KN}_3\text{O}_5$ 470.1113; found, 470.1092.

(±)-(3*R*, 5*R*)-3-(4-nitrobenzyl)-2-(4-nitrophenyl)-5-isopropylisoxazolidine (33). Reaction of 39.6 mg (0.307 mmol) of 3-isopropyl-*o*-homoallylhydroxylamine (**11**)

with 1-bromo-4-nitrobenzene (127.2 mg, 0.63 mmol) and NaOt-Bu (70.7 mg, 0.74 mmol) following General procedures afforded 86.6 mg (76 %) of the title compound as a yellow solid. R_f = 0.27 (20% EtOAc : Petroleum ether); ^1H (300 MHz, CDCl_3) δ 8.19-8.22 (d, J = 9 Hz, 2H, $p\text{-NO}_2\text{-Ph}$), 8.04-8.07 (d, J = 9 Hz, 2H, $p\text{-NO}_2\text{-Ph}$), 7.51-7.54 (d, J = 9 Hz, 2H, $p\text{-NO}_2\text{-Ph}$), 6.74-6.77 (d, J = 9 Hz, 2H, $p\text{-NO}_2\text{-Ph}$), 4.52-4.58 (m, 1H, OCH), 4.17-4.21 (m, 1H, NCH), 3.21-3.29 (dd, J = 13.5, 9 Hz, 1H, ArCH_2), 3.04-3.11 (dd, J = 13.5, 4.8 Hz, 1H, ArCH_2), 2.52-2.58 (m, 1H, CH_2), 2.16-2.22 (m, 1H, CH_2), 1.90 (m, 1H, CH), 0.96 (d, J = 6.6 Hz, 3H, CH_3), 0.90 (d, J = 6.6 Hz, 3H, CH_3). ^{13}C (75 MHz, CDCl_3) δ 156.0, 147.0, 145.7, 143.1, 128.8, 126.5, 123.9, 112.3, 86.6, 67.9, 43.7, 40.2, 28.1, 18.2. HRMS-ESI (m/z): $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{19}\text{H}_{21}\text{N}_3\text{NaO}_5$ 394.1379; found, 394.1377

The relative *syn*-stereochemistry for the isoxazolidines was confirmed by HPLC and NOESY-measurements. NOE's were observed between H^a and both H^b and H^c .

Reference:

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Current Data Parameters
NAME 1369-pjs-254
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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RG 512
DM 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

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F2 - Processing parameters
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LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
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CY 8.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -150.06 ppm
F2 0.52500 ppm/cm
PPMCM 157.56825 Hz/cm

Current Data Parameters
NAME 1369-pjs-254
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PROCNO 1

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DS 4
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DM 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00020000 sec

===== CHANNEL f1 =====
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SF01 75.4775598 MHz

===== CHANNEL f2 =====
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NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SF02 300.1312005 MHz

F2 - Processing parameters
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SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
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CY 7.00 cm
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F2P -0.005 ppm
F2 -0.38 Hz
PPMCM 9.00050 ppm/cm
HZCM 679.24744 Hz/cm

Compound 9

Current Data Parameters
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 PROCNO 1

F2 - Acquisition Parameters
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 SOLVENT CDCl3
 NS 8
 DS 0
 SMH 5995.204 Hz
 FIDRES 0.182959 Hz
 AQ 2.7329011 sec
 RG 128
 DW 83.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

***** CHANNEL f1 *****
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F2 - Processing parameters
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 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
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 F2P -0.500 ppm
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Compound 9

Current Data Parameters
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 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060602
 Time 18.27
 INSTRUM av300
 PROBHD 5 mm GNP 1H/13
 PULPROG zgpg
 TD 65536
 SOLVENT CDCl3
 NS 64
 DS 4
 SMH 22675.736 Hz
 FIDRES 0.346004 Hz
 AQ 1.4451188 sec
 RG 8192
 DW 22.050 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 9.50 usec
 PL1 -6.00 dB
 SFO1 75.4775598 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -2.00 dB
 PL12 17.70 dB
 PL13 17.71 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4682871 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 9.00 cm
 F1P 200.000 ppm
 F1 15093.66 Hz
 F2P -0.005 ppm
 F2 -0.38 Hz
 PPMCH 10.00025 ppm/cm
 HZCM 754.70172 Hz/cm

Current Data Parameters
NAME 1369-p1s-288
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060605
Time 9.58
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 256
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320882 MHz

F2 - Processing parameters
SI 32768
SF 300.1300124 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

10 NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCH 0.52500 ppm/cm
HZCM 157.56825 Hz/cm

Current Data Parameters
NAME 1369-p1s-288
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060605
Time 11.35
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 240
DS 4
SWH 22675.736 Hz
FIDRES 0.346504 Hz
AQ 1.445189 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4775598 MHz

***** CHANNEL f2 *****
CPOPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677556 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

10 NMR plot parameters
CX 20.00 cm
CY 8.00 cm
F1P 180.005 ppm
F1 13584.57 Hz
F2P -0.005 ppm
F2 -0.38 Hz
PPMCH 9.00050 ppm/cm
HZCM 679.24756 Hz/cm

Compound 21

Current Data Parameters
NAME 1369-pjs-247
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060412
Time 17.31
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl₃
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 512
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SF01 300.1320884 MHz

F2 - Processing parameters
SI 32768
SF 300.1300124 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCH 0.52500 ppm/cm
HZCM 157.56825 Hz/cm

Compound 21

Current Data Parameters
NAME 1369-pjs-247
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060412
Time 18.22
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl₃
NS 2818
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SF01 75.4775598 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SF02 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677551 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 9.00 cm
F1P 180.005 ppm
F1 13584.57 Hz
F2P -0.050 ppm
F2 -3.77 Hz
PPMCH 9.00275 ppm/cm
HZCM 679.41736 Hz/cm

Current Data Parameters
 NAME 1369-pis-240
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060404
 Time 15.25
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SMH 5995.204 Hz
 FIDRES 0.182959 Hz
 AQ 2.7329011 sec
 RG 512
 DW 83.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 3.00 usec
 PL1 -2.00 dB
 SF01 300.1320884 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300124 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 10.00 cm
 F1P 10.000 ppm
 F1 3001.30 Hz
 F2P -0.500 ppm
 F2 -150.06 Hz
 PPMCM 0.52500 ppm/cm
 HZCM 157.56825 Hz/cm

Current Data Parameters
 NAME 1369-pis-240
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060404
 Time 18.37
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg
 TD 65536
 SOLVENT CDCl3
 NS 3626
 DS 4
 SMH 22675.736 Hz
 FIDRES 0.346004 Hz
 AQ 1.4451188 sec
 RG 8192
 DW 22.050 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 5.50 usec
 PL1 -6.00 dB
 SF01 75.4775598 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -2.00 dB
 PL12 17.70 dB
 PL13 17.71 dB
 SF02 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677546 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 8.00 cm
 F1P 180.005 ppm
 F1 13584.57 Hz
 F2P -0.005 ppm
 F2 -0.38 Hz
 PPMCM 9.00050 ppm/cm
 HZCM 679.24756 Hz/cm

Compound 12

Current Data Parameters
NAME 1369-pjs-225
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060314
Time 17.37
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl₃
NS 32
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 256
OW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320882 MHz

F2 - Processing parameters
SI 32768
SF 300.1300124 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCH 0.52500 ppm/cm
HZCM 157.56825 Hz/cm

Compound 12

Current Data Parameters
NAME 1369-pjs-225
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060404
Time 12.22
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl₃
NS 1980
DS 0
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
OW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4775598 MHz

***** CHANNEL f2 *****
CPOPRG2 waitz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677532 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 6.00 cm
F1P 180.005 ppm
F1 12584.57 Hz
F2P -0.38 Hz
F2 9.00050 ppm/cm
PPMCH 9.00050 ppm/cm
HZCM 679.24744 Hz/cm

Compound 22

Current Data Parameters
NAME 1369-pis-251
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060419
Time 17.15
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 17
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 512
DM 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320884 MHz

F2 - Processing parameters
SI 32768
SF 300.1300017 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 15.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -190.06 Hz/cm
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm

Compound 22

Current Data Parameters
NAME 1369-pis-251
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060420
Time 18.26
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 4342
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DM 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4775598 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677522 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 6.00 cm
F1P 180.005 ppm
F1 13584.57 Hz
F2P -0.005 ppm
F2 -0.38 Hz
PPMCM 9.00050 ppm/cm
HZCM 679.24744 Hz/cm

Current Data Parameters
NAME 1369-pjs-273
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060517
Time 12.11
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 256
DM 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320864 MHz

F2 - Processing parameters
SI 32768
SF 300.1300121 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 40.00 cm
FIP 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPHCH 0.52500 ppm/cm
HZCH 157.56825 Hz/cm

Current Data Parameters
NAME 1369-pjs-273
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060517
Time 12.15
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 2138
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DM 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4775598 MHz

***** CHANNEL f2 *****
CPOPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677533 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
FIP 200.005 ppm
F1 15093.93 Hz
F2P -0.005 ppm
F2 -0.38 Hz
PPHCH 10.00050 ppm/cm
HZCH 754.71527 Hz/cm

Current Data Parameters
NAME 1369-pjs-290
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060605
Time 10.21
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SMH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 256
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320882 MHz

F2 - Processing parameters
SI 32768
SF 300.1300124 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm

Current Data Parameters
NAME 1369-pjs-275
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060519
Time 18.50
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 280
DS 4
SMH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8152
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4775598 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677559 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
F1P 200.005 ppm
F1 15093.93 Hz
F2P -0.005 ppm
F2 -0.38 Hz
PPMCM 10.00050 ppm/cm
HZCM 754.71533 Hz/cm

Current Data Parameters
 NAME 1369-pjs-297
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060613
 Time 10.28
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TO 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SMH 5995.204 Hz
 FIDRES 0.182959 Hz
 AQ 2.7329011 sec
 RG 64
 DM 83.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 3.00 usec
 PL1 -2.00 dB
 SF01 300.1320884 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300125 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 12.00 cm
 F1P 10.000 ppm
 F1 3001.30 Hz
 F2P -0.500 ppm
 F2 -150.06 Hz
 PPMCM 0.52500 ppm/cm
 HZCM 157.56825 Hz/cm

Current Data Parameters
 NAME 1369-pjs-297
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060613
 Time 14.37
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg
 TO 65536
 SOLVENT CDCl3
 NS 272
 DS 4
 SMH 22675.736 Hz
 FIDRES 0.346004 Hz
 AQ 1.4451188 sec
 RG 8132
 DM 22.050 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 5.50 usec
 PL1 -6.00 dB
 SF01 75.4775598 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -2.00 dB
 PL12 17.70 dB
 PL13 17.71 dB
 SF02 300.1312095 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677542 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 6.00 cm
 F1P 200.005 ppm
 F1 15093.93 Hz
 F2P -0.005 ppm
 F2 -0.38 Hz
 PPMCM 10.00050 ppm/cm
 HZCM 754.71527 Hz/cm

Current Data Parameters
 NAME 1369-pis-299
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060613
 Time 18.46
 INSTRUM av300
 PROBHD 5 mm GNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 5995.204 Hz
 FIDRES 0.182959 Hz
 AQ 2.7329011 sec
 RG 128
 DM 83.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 3.00 usec
 PL1 -2.00 dB
 SF01 300.1320882 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300124 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 10.00 cm
 F1P 10.000 ppm
 F1 3001.30 Hz
 F2P -0.500 ppm
 F2 -150.06 Hz
 PPMCM 0.52500 ppm/cm
 HZCM 157.56825 Hz/cm

Current Data Parameters
 NAME 1369-pis-299
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060614
 Time 11.49
 INSTRUM av300
 PROBHD 5 mm GNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 136
 DS 4
 SWH 22675.736 Hz
 FIDRES 0.346004 Hz
 AQ 1.4451189 sec
 RG 8192
 DM 22.050 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 5.50 usec
 PL1 -6.00 dB
 SF01 75.4775598 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -2.00 dB
 PL12 17.70 dB
 PL13 17.71 dB
 SF02 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677556 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 4.00 cm
 F1P 200.000 ppm
 F1 15093.55 Hz
 F2P -0.505 ppm
 F2 -0.38 Hz
 PPMCM 10.00025 ppm/cm
 HZCM 754.69647 Hz/cm

Current Data Parameters
NAME 1369-pjs-301
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060614
Time 16.04
INSTRUM av300
PROBHD 5 mm GNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 32
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 128
DM 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SF01 300.1320882 MHz

F2 - Processing parameters
SI 32768
SF 300.1300124 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 30.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm

Current Data Parameters
NAME 1369-pjs-301
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060615
Time 12.24
INSTRUM av300
PROBHD 5 mm GNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 2142
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DM 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00020000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SF01 75.4775598 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SF02 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677528 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
F1P 200.005 ppm
F1 15093.93 Hz
F2P -0.005 ppm
F2 -0.38 Hz
PPMCM 10.00050 ppm/cm
HZCM 754.71527 Hz/cm

Current Data Parameters
NAME 1369-pjs-302
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060615
Time 15.04
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 512
DM 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 3.00 usec
PL1 2.00 dB
SF01 300.1320882 MHz

F2 - Processing parameters
SI 32768
SF 300.1300124 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52900 ppm/cm
HZCM 157.56825 Hz/cm

Current Data Parameters
NAME 1369-pjs-302
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060615
Time 15.39
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 280
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DM 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00000000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SF01 75.4775598 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SF02 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677574 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
F1P 180.005 ppm
F1 13584.58 Hz
F2P 0.005 ppm
F2 0.38 Hz
PPMCM 9.00000 ppm/cm
HZCM 679.20990 Hz/cm

Current Data Parameters
NAME 1369-pis-306
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060623
Time 10.09
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 512
OW 83.400 usec
OC 6.00 usec
TE 300.0 K
D1 1.00000000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320882 MHz

F2 - Processing parameters
SI 32768
SF 300.1300124 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
FIP 10.000 ppm
F1 3001.30 Hz
F2 -0.500 ppm
F2P -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm

Current Data Parameters
NAME 1369-pis-306
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060623
Time 14.33
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 480
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
OW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4775598 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677543 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 6.00 cm
FIP 200.005 ppm
F1 15093.93 Hz
F2 -0.005 ppm
F2P -0.38 Hz
PPMCM 10.00050 ppm/cm
HZCM 754.71527 Hz/cm

Current Data Parameters
NAME 1369-pis-244
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060411
Time 9.51
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 128
DM 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320864 MHz

F2 - Processing parameters
SI 32768
SF 300.1300124 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 15.00 cm
FIP 10.000 ppm
F1 3001.30 Hz
F2 -150.06 Hz
PPHCH 0.52500 ppm/cm
HZCH 157.56825 Hz/cm

Current Data Parameters
NAME 1369-pis-245
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060412
Time 12.29
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 1678
DS 4
SWH 22675.736 Hz
FIDRES 0.346804 Hz
AQ 1.4451188 sec
RG 8192
DM 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00020000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4775598 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677560 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 7.00 cm
FIP 180.005 ppm
F1 13584.57 Hz
F2 -0.38 Hz
PPHCH 9.00050 ppm/cm
HZCH 679.24756 Hz/cm

Compound 23

Current Data Parameters
 NAME 1369-pjs-311
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060711
 Time 10.25
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT D2O
 NS 8
 DS 0
 SMH 5995.204 Hz
 FIDRES 0.182959 Hz
 AQ 2.7329011 sec
 RG 128
 DW 83.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 3.00 usec
 PL1 -2.00 dB
 SFO1 300.1320882 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300119 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 12.00 cm
 F1P 10.000 ppm
 F1 3001.30 Hz
 F2P -0.500 ppm
 F2 -150.06 Hz
 PPMCM 0.52500 ppm/cm
 HZCM 157.56825 Hz/cm

Compound 23

Current Data Parameters
 NAME 1369-pjs-311
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060712
 Time 14.27
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 128
 DS 4
 SMH 22675.736 Hz
 FIDRES 0.346004 Hz
 AQ 1.4451188 sec
 RG 8192
 DW 22.050 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 5.50 usec
 PL1 -6.00 dB
 SFO1 75.4775998 MHz

===== CHANNEL f2 =====
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -2.00 dB
 PL12 17.70 dB
 PL13 17.71 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677598 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 6.00 cm
 F1P 180.005 ppm
 F1 13584.57 Hz
 F2P -0.005 ppm
 F2 -0.38 Hz
 PPMCM 9.00050 ppm/cm
 HZCM 679.24756 Hz/cm

Current Data Parameters
NAME 1369-pjs-315
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060728
Time 16.19
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 1024
DM 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320882 MHz

F2 - Processing parameters
SI 32768
SF 300.1300124 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
FIP 10.000 ppm
F1 3001.30 Hz
F2 -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm

Current Data Parameters
NAME 1369-pjs-315
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060728
Time 18.37
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 300
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DM 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4775998 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677528 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 6.00 cm
FIP 180.005 ppm
F1 13584.57 Hz
F2 -0.500 ppm
F2 -37.73 Hz
PPMCM 9.02525 ppm/cm
HZCM 681.11536 Hz/cm

P06518-1 NOESY

Current Data Parameters

NAME 1369-pjs-290
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters

Date_ 20060607
Time 18.34
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG noesyph
TD 1024
SOLVENT CDCl₃
NS 32
DS 4
SWH 2055.921 Hz
FIDRES 2.007735 Hz
AQ 0.2498668 sec
RG 32
OW 243.200 usec
DE 6.00 usec
TE 300.2 K
d0 0.0000300 sec
d1 2.0000000 sec
d8 1.0000000 sec
IND 0.00048640 sec
MCREST 0.0000000 sec
MCWAK 1.0000000 sec
STICHT 256

***** CHANNEL f1 *****

NUC1 1H
P1 7.00 usec
PL1 -2.00 dB
SFO1 300.1315955 MHz

F1 - Acquisition parameters

NUC1 13C
TD 512
SFO1 300.1316 MHz
FIDRES 4.015471 Hz
SW 6.850 ppm
FMODE States-TPPI

F2 - Processing parameters

SI 1024
SF 300.1300124 MHz
WDW Q5INE
SSB 2
LB 0.00 Hz
GB 0
PC 1.00

F1 - Processing parameters

SI 1024
MC2 States-TPPI
SF 300.1300124 MHz
WDW Q5INE
SSB 2
LB 0.00 Hz
GB 0

2D NMR plot parameters

CH2 15.00 cm
CH1 15.00 cm
F2PL0 8.700 ppm
F2L0 2611.08 Hz
F2PH1 1.850 ppm
F2H1 555.16 Hz
F1PL0 8.700 ppm
F1L0 2611.08 Hz
F1PH1 1.850 ppm
F1H1 555.16 Hz
F2PPH0 0.45667 ppm/cm
F2H2CM 137.06140 Hz/cm
F1PPH0 0.45667 ppm/cm
F1H2CM 137.06140 Hz/cm

Shimadzu CLASS-VP V 6.13 SP1

External Standard

Report

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Method Name: 1369-pjs

Data Name:

User: ZHH

Acquired: 2006-4-21 11:26:08

Printed: 2006-4-21 11:48:12

Detector
A
(254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	5.997	1485	0.43	23813	0.51
2	6.457	346459	99.30	4621871	99.15
3	7.803	607	0.17	10579	0.23
4	8.237	230	0.07	3620	0.08
5	8.860	36	0.01	342	0.01
6	11.100	29	0.01	304	0.01
7	14.313	46	0.01	1097	0.02

Totals		348892	100.00	4661626	100.00
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