# Catalytic Asymmetric Synthesis of Secondary Nitriles via Stereoconvergent Negishi Arylations and Alkenylations of Racemic α-Bromonitriles

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## Supporting Information

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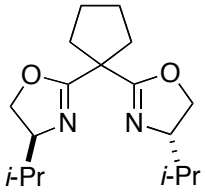
## I. General

The following reagents were purchased and used as received, unless otherwise noted: $NiCl_2$·glyme (Strem), THF (Aldrich; anhydrous), TMEDA (Aldrich; purified by distillation), and $Zn(OMe)_2$ (Aldrich; ground). The Grignard reagents were purchased (Aldrich) or prepared from aryl bromides and magnesium turnings (Strem). All reactions were carried out in oven-dried glassware under an inert atmosphere.

HPLC analyses were carried out on an Agilent 1100 series system with Daicel CHIRALPAK® columns or Daicel CHIRALCEL® columns (internal diameter 4.6 mm, column length 250 mm, particle size 5 μm or 3 μm). GC analyses were carried out on an Agilent 6890 series system with a DB-1 column (length 30 m, I.D. 0.25 mm) or an Agilent 6850 series system with a G-TA column (length 30 m, I.D. 0.25 mm) or a CP-Chirasil-Dex CB column (length 30 m, I.D. 0.25 mm). Supercritical fluid chromatography (SFC) analyses were carried out on a Berger SFC MiniGram system with Daicel CHIRALCEL® columns (internal diameter 4.6 mm, column length 250 mm, particle size 3 μm).

## II. Preparation of Materials




**(4*S*,4'*S*)-2,2'-(Cyclopentane-1,1-diyl)bis(4-isopropyl-4,5-dihydrooxazole).** Cyclopentane-1,1-dicarbonitrile was prepared from malononitrile and 1,4-dibromobutane according to a literature procedure.[1] A 500-mL round-bottom flask charged with cyclopentane-1,1-dicarbonitrile (2.85 g, 23.7 mmol) and zinc triflate (8.63 g, 23.7 mmol) was purged with argon, and anhydrous toluene (158 mL) was added. The mixture was stirred for 10 min, and then a solution of L-valinol (5.14 g, 49.8 mmol) in toluene (79 mL) was added. The mixture was heated at reflux for 48 h. Then, the mixture was allowed to cool to r.t., and the solution was washed with brine (3 × 60 mL) and saturated aqueous $NaHCO_3$ (3 × 60 mL). The organic layer was dried over $MgSO_4$ and concentrated. The residue was purified by column chromatography (2%→15% ethyl acetate and 1% $NEt_3$ in hexanes), which furnished a colorless oil (6.28 g, 91%).

$^1$H NMR (500 MHz, $CDCl_3$): δ 4.20 (dd, 2H, *J* = 7.8, 9.1 Hz), 4.01–3.94 (m, 4H), 2.37–2.31 (m, 2H), 2.18–2.13 (m, 2H), 1.83–1.67 (m, 6H), 0.91 (d, 6H, *J* = 6.8 Hz), 0.85 (d, 6H, *J* = 6.8 Hz).

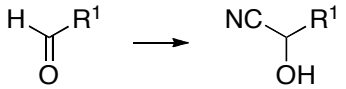
$^{13}$C NMR (126 MHz, $CDCl_3$): δ 168.2, 71.6, 70.1, 49.2, 35.5, 32.4, 25.0, 18.6, 17.6.

FT-IR (neat): 2958, 2873, 1661, 1468, 1386, 1350, 1301, 1273, 1238, 1158, 1116, 998, 962, 907, 893 $cm^{-1}$.

MS (EI) *m*/*z* ($M^+$): calcd for $C_{17}H_{28}N_2O_2$: 292, found: 292.

$[\alpha]^{25}_D = -68.0°$ (c = 1.00, $CHCl_3$).

**Synthesis of starting materials.** These procedures have not been optimized.

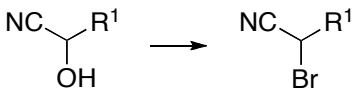



**Representative experimental procedure for the synthesis of cyanohydrins from aldehydes:** Trimethylsilyl cyanide (4.50 mL, 30.0 mmol) was added to a solution of the aldehyde (30.0 mmol) and $K_2CO_3$ (0.830 g, 6.00 mmol) in $Et_2O$ (60 mL) in a 250-mL round-bottom flask. The reaction mixture was stirred for 6 h at r.t., and then the reaction was quenched by the addition of saturated aqueous $NaHCO_3$ (30 mL). The reaction mixture was extracted with $Et_2O$ (2 × 20 mL), and the combined organic layer was concentrated.

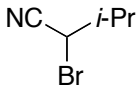
Next, an aqueous solution of HCl (1 M; 100 mL) was added to the residue, and the mixture was stirred for 2 h. Then, the reaction mixture was extracted with $Et_2O$ (3 × 50 mL), and the combined organic layer was rinsed with saturated aqueous $NaHCO_3$ (50 mL) and brine (50 mL), dried over

(1) Tsai, T.-Y.; Shia, K.-S.; Liu, H.-J. *Synlett* **2003**, 97–101.

$MgSO_4$, and concentrated. The residue was purified by column chromatography (10%→80% $Et_2O$/hexanes).




**Representative experimental procedure for the synthesis of secondary bromides from cyanohydrins:** Triphenylphosphine dibromide (15.2 g, 36.0 mmol) and then imidazole (2.45 g, 36.0 mmol) was added to a solution of the cyanohydrin (30.0 mmol) in dichloromethane (150 mL) at 0 °C. The solution was allowed to warm to r.t., and it was stirred for 6 h. Next, the reaction was quenched by the addition of saturated aqueous $NH_4Cl$ (100 mL). The aqueous layer was extracted with dichloromethane (2 × 50 mL), and the combined organic layer was rinsed with brine (50 mL), dried over $MgSO_4$, and concentrated.



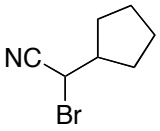

**2-Bromo-3-methylbutanenitrile.** The title compound was prepared from 2-hydroxy-3-methylbutanenitrile (2.39 g, 24.1 mmol). The product was purified by column chromatography (10% $Et_2O$/hexanes): 2.19 g (56%). Colorless oil.

$^1$H NMR (500 MHz, $CDCl_3$) δ 4.24 (d, 1H, $J$ = 5.1 Hz), 2.19 (doublet of septets, 1H, $J$ = 5.0, 6.7 Hz), 1.18 (d, 3H, $J$ = 6.7 Hz), 1.18 (d, 3H, $J$ = 6.7 Hz).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 116.6, 35.8, 34.0, 19.7, 19.2.

FT-IR (neat) 2972, 2936, 2878, 2242, 1466, 1392, 1373, 1319, 1271, 1187, 1120, 993, 966, 933, 911, 811, 695, 674 $cm^{-1}$.

MS (ESI) $m/z$ ($M^+$+H) calcd for $C_5H_9BrN$: 162.0, found: 162.0.



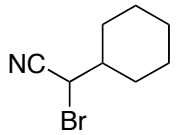

**2-Bromo-2-cyclopentylacetonitrile.** The title compound was prepared from 2-cyclopentyl-2-hydroxyacetonitrile (3.28 g, 26.2 mmol). The product was purified by column chromatography (2%→5% $Et_2O$/hexanes): 4.75 g (96%). Colorless oil.

$^1$H NMR (500 MHz, $CDCl_3$) δ 4.28 (d, 1H, $J$ = 6.9 Hz), 2.53–2.45 (m, 1H), 2.04–1.93 (m, 2H), 1.82–1.72 (m, 2H), 1.71–1.61 (m, 2H), 1.57–1.43 (m, 2H).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 117.1, 45.1, 32.7, 31.0, 30.5, 25.6.

FT-IR (neat) 2962, 2871, 2243, 1451, 1350, 1303, 1020, 1192, 771, 922, 690 $cm^{-1}$.

MS (EI) $m/z$ ($M^+$–HCN) calcd for $C_6H_9Br$: 160, found: 160.



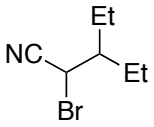

**2-Bromo-2-cyclohexylacetonitrile.** The title compound was prepared from 2-cyclohexyl-2-hydroxyacetonitrile (1.03 g, 7.43 mmol). The product was purified by column chromatography (1%→5% $Et_2O$/hexanes): 1.30 g (87%). Colorless oil.

$^1$H NMR (500 MHz, $CDCl_3$) δ 4.19 (d, 1H, *J* = 5.7 Hz), 2.04–1.99 (m, 1H), 1.96–1.91 (m, 1H), 1.87–1.77 (m, 3H), 1.72–1.66 (m, 1H), 1.34–1.22 (m, 3H), 1.22–1.13 (m, 2H).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 116.7, 42.8, 34.5, 30.5, 29.6, 25.6 (2C), 25.5.

FT-IR (neat) 2931, 2856, 2241, 1450, 1370, 1351, 1302, 1273, 1241, 1196, 1164, 1137, 970, 940, 916, 892, 855 cm$^{-1}$.

MS (ESI) *m*/*z* ($M^+$+Na) calcd for $C_8H_{12}BrNNa$: 224.0, found: 224.0.



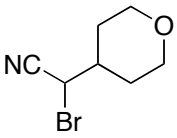

**2-Bromo-3-ethylpentanenitrile.** The title compound was prepared from 3-ethyl-2-hydroxypentanenitrile (1.93 g, 15.2 mmol). The product was purified by column chromatography (1%→5% $Et_2O$/hexanes): 2.39 g (83%). Colorless oil.

$^1$H NMR (500 MHz, $CDCl_3$) δ 4.43 (d, 1H, *J* = 4.1 Hz), 1.69–1.51 (m, 5H), 0.98 (t, 3H, *J* = 7.5 Hz), 0.96 (t, 3H, *J* = 7.2 Hz).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 117.2, 46.2, 33.1, 24.0, 23.4, 11.3, 11.2.

FT-IR (neat) 2968, 2937, 2879, 2242, 1462, 1385, 1358, 1316, 1264, 1176, 1118, 1092, 1014, 988, 945, 912, 827, 781, 755, 690, 666 cm$^{-1}$.

MS (ESI) *m*/*z* ($M^+$+H) calcd for $C_7H_{13}BrN$: 190.0, found: 190.0.



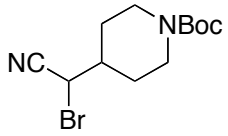

**2-Bromo-2-(tetrahydro-2*H*-pyran-4-yl)acetonitrile.** The title compound was prepared from 2-hydroxy-2-(tetrahydro-2*H*-pyran-4-yl)acetonitrile (0.85 g, 6.0 mmol). The product was purified by column chromatography (5%→100% $Et_2O$/hexanes): 0.71 g (58%). Light-yellow oil.

$^1$H NMR (500 MHz, $CDCl_3$) δ 4.18 (d, 1H, *J* = 6.4 Hz), 4.05 (ddd, 2H, *J* = 5.9, 5.9, 11.7 Hz), 3.39 (ddd, 1H, *J* = 2.2, 11.9, 11.9 Hz), 3.38 (ddd, 1H, *J* = 2.3, 12.0, 12.0 Hz), 2.06 (ddddd, 1H, *J* = 3.7, 3.7, 6.6, 11.8, 11.8 Hz), 1.92–1.87 (m, 1H), 1.87–1.82 (m, 1H), 1.61–1.47 (m, 2H).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 116.0, 67.1 (2C), 40.4, 33.2, 30.3, 29.7.

FT-IR (neat) 2947, 2849, 2763, 2242, 1468, 1446, 1388, 1371, 1275, 1238, 1172, 1133, 1114, 1090, 1014, 987, 947, 909, 874, 859, 816, 796 $cm^{-1}$.

MS (ESI) *m*/*z* ($M^+$+Na) calcd for $C_7H_{10}BrNNaO$: 226.0, found: 226.0.



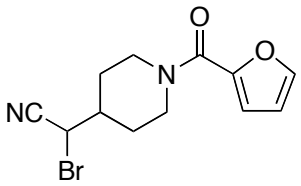

***tert*-Butyl 4-(bromo(cyano)methyl)piperidine-1-carboxylate.** The title compound was prepared from *tert*-butyl 4-(cyano(hydroxy)methyl)piperidine-1-carboxylate (3.77 g, 15.7 mmol). The product was purified by column chromatography (5%→100% $Et_2O$/hexanes): 3.53 g (74%). White solid.

$^1H$ NMR (500 MHz, $CDCl_3$) δ 4.22 (br s, 2H), 4.21 (d, 1H, *J* = 5.8 Hz), 2.70 (br s, 2H), 2.00–1.89 (m, 3H), 1.45 (s, 9H), 1.43–1.32 (m, 2H).

$^{13}C$ NMR (126 MHz, $CDCl_3$) δ 154.6, 116.0, 80.0, 43.3, 41.6, 33.1, 29.7, 29.1, 28.5.

FT-IR (neat) 2976, 2947, 2859, 2242, 1687, 1469, 1450, 1425, 1367, 1322, 1301, 1280, 1236, 1164, 1127, 1063, 1004, 974, 866, 770, 705 $cm^{-1}$.

MS (ESI) *m*/*z* ($M^+$–Boc+2H) calcd for $C_7H_{12}BrN_2$: 203.0, found: 203.0.



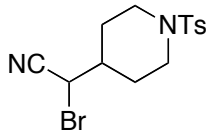

**2-Bromo-2-(1-(furan-2-carbonyl)piperidin-4-yl)acetonitrile.** Furan-2-yl(4-(hydroxymethyl)piperidin-1-yl)methanone was prepared from 4-piperidinemethanol and 2-furoyl chloride following a literature procedure. The title compound was prepared from 2-(1-(furan-2-carbonyl)piperidin-4-yl)-2-hydroxyacetonitrile (1.35 g, 5.76 mmol). The product was purified by column chromatography (10%→100% $Et_2O$/hexanes): 0.77 g (50%). White solid.

$^1H$ NMR (500 MHz, $CDCl_3$) δ 7.48 (dd, 1H, *J* = 0.8, 1.7 Hz), 7.01 (dd, 1H, *J* = 0.8, 3.4 Hz), 6.48 (dd, 1H, *J* = 1.8, 3.5 Hz), 4.70 (br s, 2H), 4.25 (d, 1H, *J* = 6.0 Hz), 2.93 (br s, 2H), 2.17–2.02 (m, 3H), 1.59–1.46 (m, 2H).

$^{13}C$ NMR (126 MHz, $CDCl_3$) δ 159.1, 147.9, 143.8, 116.6, 116.0, 111.4, 44.1, 41.5, 32.7, 29.8, 29.3.

FT-IR (neat) 3119, 2946, 2859, 2242, 1623, 1569, 1487, 1437, 1372, 1303, 1284, 1249, 1180, 1102, 1012, 977, 935, 886, 855, 756 $cm^{-1}$.

MS (ESI) *m*/*z* ($M^+$+H) calcd for $C_{12}H_{14}BrN_2O_2$: 297.0, found: 297.0.



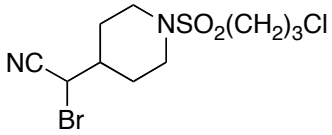

**2-Bromo-2-(1-tosylpiperidin-4-yl)acetonitrile.** (1-Tosylpiperidin-4-yl)methanol was prepared from 4-piperidinemethanol and *p*-toluenesulfonyl chloride following a literature procedure. The title compound was prepared from 2-hydroxy-2-(1-tosylpiperidin-4-yl)acetonitrile (4.63 g, 15.7 mmol). The product was purified by column chromatography (5%→100% ethyl acetate/hexanes): 3.37 g (60%). White solid.

$^1$H NMR (500 MHz, $CDCl_3$) δ 7.65–7.62 (m, 2H), 7.33 (d, 2H, *J* = 7.9 Hz), 4.18 (d, 1H, *J* = 6.4 Hz), 3.92–3.89 (m, 2H), 2.43 (s, 3H), 2.27 (dddd, 2H, *J* = 2.6, 3.2, 12.0, 12.0), 2.04–1.95 (m, 2H), 1.80–1.72 (m, 1H), 1.64–1.51 (m, 2H).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 144.0, 133.0, 129.9, 127.8, 115.9, 45.6 (2C), 40.6, 32.4, 29.0, 28.6, 21.7.

FT-IR (neat) 3031, 2961, 2926, 2854, 2245, 1598, 1493, 1469, 1448, 1354, 1330, 1306, 1253, 1164, 1113, 1094, 1071, 1049, 1011, 994, 932, 844, 813, 726, 706, 696, 652, 599 cm$^{-1}$.

MS (ESI) *m*/*z* ($M^+$+H) calcd for $C_{14}H_{18}BrN_2O_2S$: 357.0, found: 357.0.




**2-Bromo-2-(1-((3-chloropropyl)sulfonyl)piperidin-4-yl)acetonitrile.** (1-((3-Chloropropyl)sulfonyl)piperidin-4-yl)methanol was prepared from 4-piperidinemethanol and 3-chloropropanesulfonyl chloride following a literature procedure.[2] The title compound was prepared from 2-(1-((3-chloropropyl)sulfonyl)piperidin-4-yl)-2-hydroxyacetonitrile (4.77 g, 17.0 mmol). The product was purified by column chromatography (5%→100% ethyl acetate/hexanes): 3.20 g (55%). White solid.

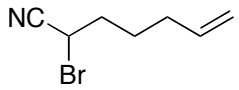
$^1$H NMR (500 MHz, $CDCl_3$) δ 4.25 (d, 1H, *J* = 6.0 Hz), 3.95–3.90 (m, 2H), 3.68 (dd, 2H, *J* = 6.1, 6.1 Hz), 3.09 (dd, 2H, *J* = 7.4, 7.4 Hz), 2.81 (dddd, 2H, *J* = 2.6, 3.5, 12.4, 12.4 Hz), 2.30–2.24 (m, 2H), 2.10–2.03 (m, 2H), 2.00–1.93 (m, 1H), 1.64–1.51 (m, 2H).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 115.9, 47.1, 45.3, 45.2, 43.0, 40.8, 32.5, 29.5, 29.1, 26.4.

FT-IR (neat) 2956, 2927, 2858, 2243, 1469, 1448, 1407, 1330, 1251, 1145, 1070, 1048, 993, 935, 798, 742, 696 cm$^{-1}$.

MS (ESI) *m*/*z* ($M^+$+H) calcd for $C_{10}H_{17}BrClN_2O_2S$: 345.0, found: 345.0.

(2) Wilsily, A.; Tramutola, F.; Owston, N. A.; Fu, G. C. *J. Am. Chem. Soc.* **2012**, *134*, 5794–5797.



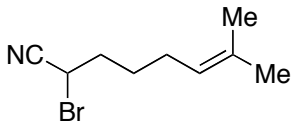

**2-Bromohept-6-enenitrile.** The title compound was prepared from 2-hydroxyhept-6-enenitrile (3.25 g, 26.0 mmol). The product was purified by column chromatography (5% $Et_2O$/hexanes): 4.10 g (84%). Colorless oil.

$^1$H NMR (500 MHz, $CDCl_3$) δ 5.78 (dddd, 1H, *J* = 6.7, 6.7, 10.3, 17.0 Hz), 5.08–5.02 (m, 2H), 4.31 (t, 1H, *J* = 7.0 Hz), 2.16–2.08 (m, 4H), 1.72–1.65 (m, 2H).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 137.0, 117.4, 116.0, 35.7, 32.4, 27.1, 26.0.

FT-IR (neat) 3079, 2935, 2865, 2244, 1641, 1458, 1418, 1290, 1220, 994, 917, 767, 698, 616 cm$^{-1}$.

MS (EI) *m*/*z* ($M^+$) calcd for $C_7H_{10}BrNNa$: 210.0, found: 210.0.



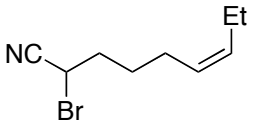

**2-Bromo-7-methyloct-6-enenitrile.** The title compound was prepared from 2-hydroxy-7-methyloct-6-enenitrile (1.81 g, 11.8 mmol). The product was purified by column chromatography (2%→15% $Et_2O$/hexanes): 2.53 g (88%). Colorless oil.

$^1$H NMR (500 MHz, $CDCl_3$) δ 5.08 (septet of triplets, 1H, *J* = 1.4, 7.2 Hz), 4.29 (t, 1H, *J* = 7.0 Hz), 2.11–2.04 (m, 4H), 1.70 (s, 3H), 1.65–1.58 (m, 5H).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 133.1, 122.9, 117.4, 36.0, 27.2, 27.1, 26.8, 25.8, 17.8.

FT-IR (neat) 2931, 2861, 2243, 1673, 1451, 1378, 1293, 1226, 1109, 1063, 985, 834, 771, 736, 696, 616 cm$^{-1}$.

MS (EI) *m*/*z* ($M^+$) calcd for $C_9H_{14}BrN$: 215, found: 215.




**(*Z*)-2-Bromonon-6-enenitrile.** The title compound was prepared from (*Z*)-2-hydroxynon-6-enenitrile (4.29 g, 28.0 mmol). The product was purified by column chromatography (5% $Et_2O$/hexanes): 5.71 g (94%). Colorless oil.

$^1$H NMR (500 MHz, $CDCl_3$) δ 5.45 (ddddd, 1H, *J* = 1.5, 1.5, 7.2, 7.2, 10.8 Hz), 5.29 (ddddd, 1H, *J* = 1.6, 1.6, 7.3, 7.3, 10.8 Hz), 4.30 (t, 1H, *J* = 7.0 Hz), 2.14–2.00 (m, 6H), 1.68–1.61 (m, 2H), 0.97 (t, 3H, *J* = 7.5 Hz).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 133.3, 127.1, 117.4, 35.9, 27.2, 26.9, 25.8, 20.6, 14.3.

FT-IR (neat) 3007, 2963, 2871, 2244, 1653, 1457, 1305, 1218, 1070, 691 cm$^{-1}$.

MS (ESI) *m*/*z* ($M^+$+Na) calcd for $C_9H_{14}BrNNa$: 238.0, found: 238.0.

## III. Negishi Cross-Coupling Reactions

**General procedure for Grignard reagent preparation:** A 25-mL two-neck round-bottom flask equipped with a reflux condenser and a stir bar was capped with a septum. Magnesium turnings (249 mg, 10.2 mmol) were added to the flask, and the flask was flame-dried under vacuum. The flask was filled with argon, and a solution of the aryl bromide (2.0 mmol) in THF (1.3 mL) was added dropwise over ~1 min. The reaction was initiated by gently heating the flask with a heat gun. Once the reaction had initiated, a solution of the aryl bromide (8.0 mmol) in THF (8.7 mL) was added dropwise over 15 min. The resulting mixture was stirred at reflux for 3 h, and then it was allowed to cool to r.t., transferred to a syringe, and filtered through an acrodisc into an oven-dried 20-mL vial sealed with a PTFE-lined septum cap under a positive pressure of argon. The Grignard reagent was titrated with $I_2$.[3]

**General procedure for asymmetric cross-coupling reactions with diarylzinc reagents prepared in situ (Tables 2, 3, and 4; no glovebox):** An oven-dried 8-mL vial equipped with a magnetic stir bar was capped with a PTFE-lined septum cap, cooled under vacuum, and filled with argon. $Zn(OMe)_2$ (124 mg, 0.972 mmol) was added to the vial, which was placed under vacuum. The vial was filled with argon, and this evacuation-refill cycle was repeated three times. THF (2.1 mL) was added to the vial, and then a solution of ArMgBr (1.0 M in THF; 1.92 mL). The mixture was stirred for 60 min at r.t. $NiCl_2$•glyme (17.6 mg, 0.080 mmol) and (*S,S*)-**L** (30.4 mg, 0.104 mmol) were added to an oven-dried 4-mL vial equipped with a magnetic stir bar. The vial was sealed with a PTFE-lined septum cap. The vial was placed under vacuum and then filled with argon; this cycle was repeated three times. Then, THF (0.80 mL) was added, and the mixture was stirred at r.t. for 10 min, at which time it had become homogenous. An oven-dried 20-mL vial equipped with a magnetic stir bar was charged with 2-bromo-2-cyclopentylacetonitrile (150 mg, 0.80 mmol) and TMEDA (24 μL, 0.16 mmol) and then capped with a PTFE-lined septum cap. Next, the vial was purged with argon for 10 min, and THF (3.2 mL) was added. An argon balloon was attached to the vial that contained the solution of the electrophile, which was cooled to –78 °C (any condensation around the septum cap on the 20-mL vial was removed), and then a 5-mL syringe containing the solution of diarylzinc and a 1-mL syringe containing the solution of $NiCl_2$•glyme and (*S,S*)-**L** were attached to the 20-mL vial containing the solution of the electrophile. The solution of the diarylzinc was injected, and the mixture was stirred for 10 min. Next, the solution of $NiCl_2$•glyme and (*S,S*)-**L** was added by syringe over 10 min. The argon-filled balloon was removed, and the septum cap was covered with grease. The reaction mixture was stirred at –78 °C for 48 h, and then the reaction was quenched by the addition of ethanol (0.8 mL). The mixture was allowed to warm to r.t., and then it was filtered through a pad of silica (eluted with $Et_2O$). The solution was concentrated, and the residue was purified by column chromatography.

A second run was conducted with (*R,R*)-**L**.

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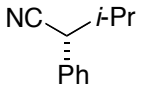
(3) Krasovskiy, A.; Knochel, P. *Synthesis* **2006**, 890–891.




**(*R*)-3-Methyl-2-phenylbutanenitrile (Table 2, entry 1).** 2-Bromo-3-methylbutanenitrile (97 mg, 0.60 mmol) was used. The product was purified by column chromatography (2% $Et_2O$/hexanes). Light-yellow oil. First run: 75 mg (79%, 92% ee). Second run: 72 mg (75%, 92% ee).

The ee was determined by GC analysis on a G-TA column (100 °C hold 5 min, then 100 °C→180 °C @ 5 °C/min, hold 10 min, 1.7 mL/min) with $t_r$ = 12.8 min (major), 13.8 min (minor).

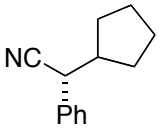
$^1$H NMR (500 MHz, $CDCl_3$) δ 7.40–7.36 (m, 2H), 7.34–7.29 (m, 3H), 3.66 (d, 1H, *J* = 6.3 Hz), 2.13 (apparent octet, 1H, *J* = 6.7 Hz), 1.06 (d, 3H, *J* = 6.7 Hz), 1.04 (d, 3H, *J* = 6.7 Hz).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 135.1, 128.9, 128.1, 128.0, 120.0, 45.3, 33.9, 20.9, 18.9.

FT-IR (neat) 3032, 2966, 2930, 2875, 2238, 1493, 1454, 1390, 1372, 1173, 1074, 1031, 918 cm$^{-1}$.

MS (EI) *m*/*z* ($M^+$) calcd for $C_{11}H_{13}N$: 159, found: 159.

$[\alpha]^{24}_D$ = +26.5° (c = 1.01, $CHCl_3$).




**(*R*)-2-Cyclopentyl-2-phenylacetonitrile (Table 2, entry 2).** 2-Bromo-2-cyclopentylacetonitrile (113 mg, 0.80 mmol) was used. The product was purified by column chromatography (1.5%→3% $Et_2O$/hexanes). White solid. First run: 147 mg (99%, 92% ee). Second run: 142 mg (96%, 93% ee).

The ee was determined by GC analysis on a G-TA column (100 °C hold 5 min, then 100 °C→180 °C @ 5 °C/min, hold 10 min, 1.7 mL/min) with $t_r$ = 20.0 min (major), 20.4 min (minor).

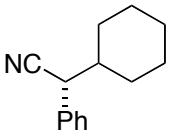
$^1$H NMR (500 MHz, $CDCl_3$) δ 7.39–7.35 (m, 2H), 7.33–7.30 (m, 3H), 3.71 (d, 1H, *J* = 7.7 Hz), 2.35–2.27 (m, 1H), 1.89–1.83 (m, 1H), 1.77–1.65 (m, 3H), 1.62–1.47 (m, 3H), 1.39–1.29 (m, 1H).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 136.0, 129.0, 128.0, 127.7, 120.7, 45.4, 42.6, 31.1, 30.3, 25.0, 24.9.

FT-IR (neat) 3033, 2955, 2868, 2233, 1647, 1495, 1456, 1361, 1302, 1146, 1078, 1030, 1003, 908, 755, 698 cm$^{-1}$.

MS (EI) *m*/*z* ($M^+$) calcd for $C_{13}H_{15}N$: 185, found: 185.

$[\alpha]^{25}_D$ = +35.7° (c = 1.00, $CHCl_3$).




**(*R*)-2-Cyclohexyl-2-phenylacetonitrile (Table 2, entry 3).** 2-Bromo-2-cyclohexylacetonitrile (121 mg, 0.60 mmol) was used. The product was purified by column chromatography (3% $Et_2O$/hexanes). White solid. First run: 106 mg (89%, 92% ee). Second run: 113 mg (95%, 93% ee).

The ee was determined by GC analysis on a G-TA column (130 °C hold 25 min, then 130 °C→180 °C @ 1 °C/min, hold 10 min, 1.5 mL/min) with $t_r$ = 39.0 min (major), 40.3 min (minor).

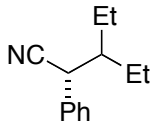
$^1$H NMR (500 MHz, $CDCl_3$) δ 7.39–7.35 (m, 2H), 7.33–7.27 (m, 3H), 3.63 (d, 1H, *J* = 6.7 Hz), 1.85–1.83 (m, 1H), 1.78–1.74 (m, 3H), 1.67–1.65 (m, 2H), 1.25–1.10 (m, 5H).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 134.8, 128.9, 128.1, 128.0, 120.3, 44.5, 42.9, 31.3, 29.7, 26.1, 26.0, 25.9.

FT-IR (neat) 2934, 2855, 2233, 1599, 1494, 1455, 1368, 1308, 1279, 1188, 1125, 1078, 1064, 1028, 982, 887, 753, 697 $cm^{-1}$.

MS (EI) *m*/*z* ($M^+$) calcd for $C_{14}H_{17}N$: 199, found: 199.

$[\alpha]^{24}_D$ = +27.9° (c = 1.01, $CHCl_3$).




**(*R*)-3-Ethyl-2-phenylpentanenitrile (Table 2, entry 4).** 2-Bromo-3-ethylpentanenitrile (114 mg, 0.60 mmol) was used. The product was purified by column chromatography (3% $Et_2O$/hexanes). Colorless oil. First run: 102 mg (91%, 92% ee). Second run: 105 mg (93%, 93% ee).

The ee was determined by GC analysis on a G-TA column (110 °C hold 20 min, then 110 °C→150 °C @ 1 °C/min, hold 10 min, 1.7 mL/min) with $t_r$ = 30.5 min (major), 31.9 min (minor).

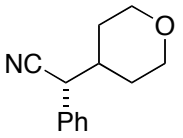
$^1$H NMR (500 MHz, $CDCl_3$) δ 7.40–7.36 (m, 2H), 7.33–7.30 (m, 3H), 3.92 (d, 1H, *J* = 6.0 Hz), 1.72–1.66 (m, 1H), 1.56–1.37 (m, 4H), 0.96 (t, 3H, *J* = 7.4 Hz), 0.87 (t, 3H, *J* = 7.4 Hz).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 135.2, 129.0, 128.1, 128.0, 120.2, 46.4, 40.8, 23.3, 22.6, 11.2, 11.1.

FT-IR (neat) 3065, 3032, 2965, 2935, 2878, 2238, 1602, 1494, 1455, 1384, 1315, 1228, 1157, 1077, 1031, 909, 821, 764, 747, 725, 699 $cm^{-1}$.

MS (EI) *m*/*z* ($M^+$) calcd for $C_{13}H_{17}N$: 187, found: 187.

$[\alpha]^{23}_D$ = +37.2° (c = 1.00, $CHCl_3$).




**(*R*)-2-Phenyl-2-(tetrahydro-2*H*-pyran-4-yl)acetonitrile (Table 2, entry 5).** 2-Bromo-2-(tetrahydro-2*H*-pyran-4-yl)acetonitrile (122 mg, 0.60 mmol) was used. The product was purified by column chromatography (40% $Et_2O$/hexanes). White solid. First run: 114 mg (94%, 92% ee). Second run: 114 mg (94%, 91% ee).

The ee was determined by HPLC analysis on a CHIRALCEL OD-H column (5% *i*-PrOH/hexanes, 1.0 mL/min) with $t_r$ = 16.8 min (minor), 19.1 min (major).

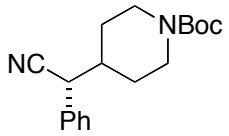
$^1$H NMR (500 MHz, $CDCl_3$) δ 7.42–7.36 (m, 2H), 7.36–7.32 (m, 1H), 7.32–7.27 (m, 2H), 4.05–3.99 (m, 1H), 3.98–3.94 (m, 1H), 3.61 (d, 1H, *J* = 7.6 Hz), 3.36–3.26 (m, 2H), 2.04–1.94 (m, 1H), 1.81–1.78 (m, 1H), 1.59–1.50 (m, 1H), 1.49–1.45 (m, 2H).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 133.8, 129.1, 128.4, 128.1, 119.6, 67.6, 67.5, 44.0, 40.4, 31.0, 30.2.

FT-IR (neat) 2969, 2932, 2854, 2233, 1494, 1455, 1393, 1366, 1303, 1278, 1263, 1244, 1215, 1139, 1116, 1092, 1068, 1018, 985, 912, 876, 823, 753, 697 $cm^{-1}$.

MS (EI) *m*/*z* ($M^+$) calcd for $C_{13}H_{15}NO$: 201, found: 201.

$[\alpha]^{24}_D$ = +27.9° (c = 1.01, $CHCl_3$).




**(*R*)-*tert*-Butyl 4-(cyano(phenyl)methyl)piperidine-1-carboxylate (Table 2, entry 6).** *tert*-Butyl 4-(bromo(cyano)methyl)piperidine-1-carboxylate (182 mg, 0.60 mmol) was used. The product was purified by column chromatography (30%→40% $Et_2O$/hexanes). White solid. First run: 172 mg (95%, 90% ee). Second run: 175 mg (97%, 90% ee).

The ee was determined by HPLC analysis on a CHIRALPAK AD-H column (3% *i*-PrOH/hexanes, 0.8 mL/min) with $t_r$ = 24.7 min (major), 27.1 min (minor).

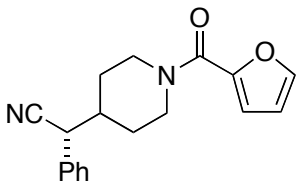
$^1$H NMR (500 MHz, $CDCl_3$) δ 7.40–7.37 (m, 2H), 7.36–7.32 (m, 1H), 7.29–7.28 (m, 2H), 4.15 (br s, 2H), 3.64 (d, 1H, *J* = 7.0 Hz), 2.62 (br s, 2H), 1.93–1.86 (m, 1H), 1.85–1.81 (m, 1H), 1.58–1.54 (m, 1H), 1.44 (s, 9H), 1.39–1.25 (m, 2H).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 154.7, 133.9, 129.2, 128.5, 128.1, 119.6, 79.8, 43.8, 43.6, 41.5, 30.2, 29.3, 28.6.

FT-IR (neat) 2976, 2937, 2856, 2239, 1690, 1494, 1454, 1424, 1366, 1318, 1279, 1248, 1169, 1125, 1081, 1058, 1031, 1004, 975, 952, 921, 868, 818, 758, 734, 702 $cm^{-1}$.

MS (ESI) *m*/*z* ($M^+$–Boc+2H) calcd for $C_{13}H_{17}N_2$: 201.1, found: 201.1.

$[\alpha]^{23}_D$ = +23.1° (c = 1.00, $CHCl_3$).




**(*R*)-2-(1-(Furan-2-carbonyl)piperidin-4-yl)-2-phenylacetonitrile (Table 2, entry 7).** 2-Bromo-2-(1-(furan-2-carbonyl)piperidin-4-yl)acetonitrile (178 mg, 0.60 mmol) was used. The product was purified by column chromatography (40%→50% ethyl acetate/hexanes). Light –yellow solid. First run: 166 mg (94%, 85% ee). Second run: 169 mg (96%, 85% ee).

The ee was determined by HPLC analysis on a CHIRALCEL OD-H column (50% *i*-PrOH/hexanes, 0.7 mL/min) with $t_r$ = 11.8 min (minor), 14.5 min (major).

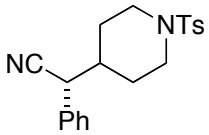
$^1$H NMR (500 MHz, $CDCl_3$) δ 7.46 (d, 1H, *J* = 0.9 Hz), 7.41–7.33 (m, 3H), 7.30–7.23 (m, 2H), 6.96 (dd, 1H, *J* = 0.6, 3.4 Hz), 6.46 (dd, 1H, *J* = 1.8, 3.4 Hz), 4.61 (br s, 2H), 3.66 (d, 1H, *J* = 7.3 Hz), 2.84 (br s, 2H), 2.09–2.01 (m, 1H), 1.99–1.94 (m, 1H), 1.70–1.65 (m, 1H), 1.52–1.37 (m, 2H).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 159.2, 148.0, 143.7, 133.6, 129.2, 128.5, 128.0, 119.5, 116.4, 111.4, 46.1, 43.5, 43.2, 41.6, 30.4, 29.8.

FT-IR (neat) 3117, 3032, 2923, 2857, 2238, 1625, 1569, 1488, 1437, 1372, 1319, 1283, 1222, 1173, 1098, 1057, 1012, 976, 938, 886, 757, 703 $cm^{-1}$.

MS (ESI) *m*/*z* ($M^+$+H) calcd for $C_{18}H_{19}N_2O_2$: 295.1, found: 295.1.

$[\alpha]^{25}_D$ = +19.7° (c = 1.00, $CHCl_3$).




**(*R*)-2-Phenyl-2-(1-tosylpiperidin-4-yl)acetonitrile (Table 2, entry 8).** 2-Bromo-2-(1-tosylpiperidin-4-yl)acetonitrile (244 mg, 0.60 mmol) was used. The product was purified by column chromatography (20%→25% ethyl acetate/hexanes). White solid. First run: 199 mg (94%, 91% ee). Second run: 202 mg (95%, 91% ee).

The ee was determined by HPLC analysis on a CHIRALPAK IB-3 column (20% *i*-PrOH/hexanes, 0.9 mL/min) with $t_r$ = 28.0 min (minor), 42.3 min (major).

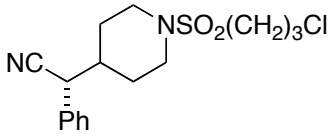
$^1$H NMR (500 MHz, $CDCl_3$) δ 7.62–7.60 (m, 2H), 7.38–7.32 (m, 3H), 7.32–7.29 (m, 2H), 7.23–7.21 (m, 2H), 3.88–3.84 (m, 1H), 3.82–3.78 (m, 1H), 3.57 (d, 1H, *J* = 7.6 Hz), 2.42 (s, 3H), 2.20 (ddd, 1H, *J* = 2.8, 12.0, 12.0 Hz), 2.15 (ddd, 1H, *J* = 2.8, 12.0, 12.0 Hz), 1.97–1.92 (m, 1H), 1.71–1.64 (m, 1H), 1.61–1.50 (m, 2H), 1.49–1.40 (m, 1H).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 143.8, 133.5, 133.0, 129.8, 129.2, 128.6, 128.0, 127.8, 119.4, 46.1, 46.0, 43.3, 40.7, 29.4, 28.9, 21.7.

FT-IR (neat) 3032, 2924, 2852, 2240, 1598, 1494, 1467, 1454, 1339, 1306, 1251, 1164, 1094, 1047, 932, 817, 761, 729, 702, 650 $cm^{-1}$.

MS (ESI) *m*/*z* ($M^+$+H) calcd for $C_{20}H_{23}N_2O_2S$: 355.1, found: 355.1.

$[\alpha]^{24}_D$ = +22.1° (c = 1.00, $CHCl_3$).




**(*R*)-2-(1-((3-Chloropropyl)sulfonyl)piperidin-4-yl)-2-phenylacetonitrile (Table 2, entry 9).** 2-Bromo-2-(1-((3-chloropropyl)sulfonyl)piperidin-4-yl)acetonitrile (206 mg, 0.60 mmol) was used. The product was purified by column chromatography (20%→35% ethyl acetate/hexanes). White solid. First run: 191 mg (93%, 89% ee). Second run: 192 mg (94%, 90% ee).

The ee was determined by SFC analysis on a CHIRALCEL OD-H column (15% MeOH/$CO_2$, 3.0 mL/min) with $t_r$ = 7.5 min (minor), 8.5 min (major).

$^1$H NMR (500 MHz, $CDCl_3$) δ 7.41–7.34 (m, 3H), 7.28 (d, 2H, *J* = 7.2 Hz), 3.87 (apparent d, 1H, *J* = 12.6 Hz), 3.82 (apparent d, 1H, *J* = 12.6 Hz), 3.67–3.65 (m, 3H), 3.05 (dd, 2H, *J* = 7.3, 7.3 Hz), 2.74

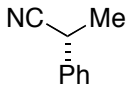
(ddd, 1H, *J* = 2.4, 12.3, 12.3 Hz), 2.69 (ddd, 1H, *J* = 2.4, 12.3, 12.3 Hz), 2.25 (ddd, 2H, *J* = 6.4, 6.4, 12.9 Hz), 1.99 (apparent d, 1H, *J* = 13.1 Hz), 1.92–1.85 (m, 1H), 1.66 (apparent d, 1H, *J* = 13.2 Hz), 1.53 (dddd, 1H, *J* = 4.3, 12.4, 12.4, 12.4 Hz), 1.44 (dddd, 1H, *J* = 4.3, 12.4, 12.4, 12.4 Hz).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 133.4, 129.3, 128.6, 128.0, 119.4, 46.9, 45.6 (2C), 43.3, 43.0, 40.8, 29.9, 29.3, 26.5.

FT-IR (neat) 3032, 2987, 2925, 2869, 2857, 2240, 1494, 1469, 1453, 1360, 1334, 1305, 1250, 1148, 1102, 1070, 1047, 1005, 993, 936, 914, 800, 761, 736, 703, 640, 623, 612 cm$^{-1}$.

MS (ESI) *m*/*z* ($M^+$+H) calcd for $C_{16}H_{22}ClN_2O_2S$: 341.1, found: 341.1.

$[\alpha]^{24}_D$ = +17.8° (c = 1.02, $CHCl_3$).




**(*R*)-2-Phenylpropanenitrile (Table 2, entry 10).** 2-Bromopropanenitrile (80 mg, 0.60 mmol; Adrich) was used. The product was purified by column chromatography (5% $Et_2O$/hexanes). Colorless oil. First run: 54 mg (69%, 81% ee). Second run: 51 mg (65%, 82% ee).

The ee was determined by GC analysis on a G-TA column (100 °C hold 5 min, then 100 °C→180 °C @ 5 °C/min, hold 10 min, 1.7 mL/min) with $t_r$ = 10.7 min (major), 11.9 min (minor).

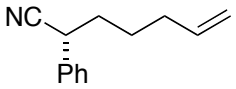
$^1$H NMR (500 MHz, $CDCl_3$) δ 7.41–7.31 (m, 5H), 3.91 (q, 1H, *J* = 7.3 Hz), 1.65 (d, 3H, *J* = 7.3 Hz).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 137.2, 129.3, 128.2, 126.8, 121.7, 31.4, 21.6.

FT-IR (neat) 3065, 3032, 2985, 2932, 2242, 1955, 1881, 1808, 1726, 1600, 1493, 1451, 1379, 1285, 1078, 1030, 988 cm$^{-1}$.

MS (EI) *m*/*z* ($M^+$) calcd for $C_9H_9N$: 131, found: 131.

$[\alpha]^{23}_D$ = +15.9° (c = 1.00, $CHCl_3$).




**(*R*)-2-Phenylhept-6-enenitrile (Table 2, entry 11).** 2-Bromohept-6-enenitrile (113 mg, 0.60 mmol) was used. The product was purified by column chromatography (2% $Et_2O$/hexanes). Colorless oil. First run: 104 mg (94%, 77% ee). Second run: 92 mg (83%, 78% ee).

The ee was determined by GC analysis on a G-TA column (100 °C hold 5 min, then 100 °C→180 °C @ 3 °C/min, hold 10 min, 1.7 mL/min) with $t_r$ = 23.0 min (major), 23.5 min (minor).

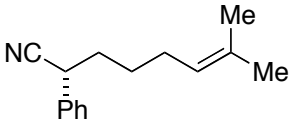
$^1$H NMR (500 MHz, $CDCl_3$) δ 7.40–7.36 (m, 2H), 7.34–7.31 (m, 3H), 5.76 (dddd, 1H, *J* = 6.8, 6.8, 10.2, 17.0 Hz), 5.04–4.97 (m, 2H), 3.79 (dd, 1H, *J* = 6.3, 8.6 Hz), 2.13–2.08 (m, 2H), 1.98–1.84 (m, 2H), 1.67–1.51 (m, 2H).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 137.7, 136.0, 129.2, 128.2, 127.4, 120.9, 115.6, 37.4, 35.3, 33.1, 26.2.

FT-IR (neat) 3066, 3032, 2978, 2929, 2863, 2240, 1954, 1811, 1641, 1601, 1494, 1455, 1416, 1344, 1079, 1031, 994, 914, 757, 699 cm$^{-1}$.

MS (EI) *m*/*z* ($M^+$) calcd for $C_{13}H_{15}N$: 185, found: 185.

$[\alpha]^{24}_D$ = +16.8° (c = 1.01, $CHCl_3$).




**(*R*)-7-Methyl-2-phenyloct-6-enenitrile (Table 2, entry 12).** 2-Bromo-7-methyloct-6-enenitrile (130 mg, 0.60 mmol) was used. The product was purified by column chromatography (2%→5% $Et_2O$/hexanes). Colorless oil. First run: 119 mg (93%, 76% ee). Second run: 120 mg (94%, 77% ee).

The ee was determined by GC analysis on a G-TA column (110 °C hold 5 min, then 110 °C→180 °C @ 1 °C/min, hold 10 min, 1.7 mL/min) with $t_r$ = 46.6 min (major), 47.7 min (minor).

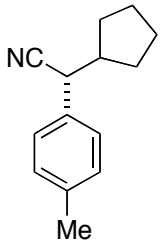
$^1$H NMR (500 MHz, $CDCl_3$) δ 7.39–7.37 (m, 2H), 7.33–7.31 (m, 3H), 5.06 (t, 1H, *J* = 7.1 Hz), 3.77 (dd, 1H, *J* = 6.3, 8.5 Hz), 2.02, (q, 2H, *J* = 7.2 Hz), 1.97–1.82 (m, 2H), 1.68 (s, 3H), 1.59 (s, 3H), 1.57–1.45 (m, 2H).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 136.2, 132.6, 129.2, 128.1, 127.4, 123.5, 121.0, 37.5, 35.5, 27.4, 27.3, 25.8, 17.9.

FT-IR (neat) 3032, 2928, 2861, 2240, 1602, 1495, 1454, 1377, 1110, 1080, 1031, 912, 833, 755, 699 $cm^{-1}$.

MS (EI) *m*/*z* ($M^+$) calcd for $C_{15}H_{19}N$: 213, found: 213.

$[\alpha]^{23}_D$ = +14.0° (c = 1.00, $CHCl_3$).




**(*R*)-2-Cyclopentyl-2-(*p*-tolyl)acetonitrile (Table 3, entry 1).** 2-Bromo-2-cyclopentylacetonitrile (150 mg, 0.80 mmol) and *p*-tolylmagnesium bromide (1.05 M in THF; Aldrich) were used. The product was purified by column chromatography (2%→3.5% $Et_2O$/hexanes). Colorless oil. First run: 149 mg (93%, 93% ee). Second run: 151 mg (95%, 94% ee).

The ee was determined by HPLC analysis on a CHIRALCEL OJ-H column (1% *i*-PrOH/hexanes, 1.0 mL/min) with $t_r$ = 10.4 min (major), 11.7 min (minor).

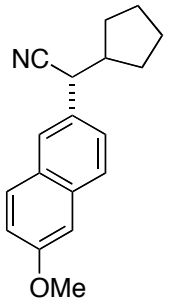
$^1$H NMR (500 MHz, $CDCl_3$) δ 7.21–7.16 (m, 4H), 3.67 (d, 1H, *J* = 7.8 Hz), 2.35 (s, 3H), 2.33–2.25 (m, 1H), 1.88–1.82 (m, 1H), 1.76–1.64 (m, 3H), 1.61–1.42 (m, 3H), 1.37–1.29 (m, 1H).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 137.8, 133.0, 129.7, 127.6, 120.9, 45.4, 42.2, 31.1, 30.4, 25.0 (2C), 21.2.

FT-IR (neat) 3026, 2957, 2870, 2239, 1904, 1799, 1653, 1616, 1515, 1452, 1417, 1380, 1351, 1309, 1215, 1186, 1113, 1041, 1022, 813, 770, 719 $cm^{-1}$.

MS (EI) *m*/*z* ($M^+$) calcd for $C_{14}H_{17}N$: 199, found: 199.

$[\alpha]^{24}_D$ = +29.6° (c = 0.99, $CHCl_3$).




**(*R*)-2-Cyclopentyl-2-(6-methoxynaphthalen-2-yl)acetonitrile (Table 3, entry 2).** 2-Bromo-2-cyclopentylacetonitrile (150 mg, 0.80 mmol) and (6-methoxynaphthalen-2-yl)magnesium bromide (1.22 M in THF) were used. The product was purified by column chromatography (5%→10% $Et_2O$/hexanes). Light-yellow solid. First run: 203 mg (96%, 94% ee). Second run: 184 mg (87%, 95% ee).

The ee was determined by HPLC analysis on a CHIRALCEL OJ-H column (10% *i*-PrOH/hexanes, 1.0 mL/min) with $t_r$ = 16.5 min (minor), 24.4 min (major).

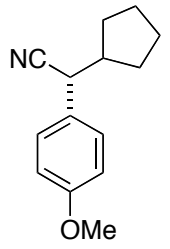
$^1$H NMR (500 MHz, $CDCl_3$) δ 7.75–7.71 (m, 3H), 7.37 (dd, 1H, *J* = 1.8, 8.5 Hz), 7.18 (dd, 1H, *J* = 2.5, 8.9 Hz), 7.13 (d, 1H, *J* = 2.5 Hz), 3.93 (s, 3H), 3.84 (d, 1H, *J* = 7.8 Hz), 2.44–2.36 (m, 1H), 1.90–1.84 (m, 1H), 1.78–1.67 (m, 3H), 1.63–1.51 (m, 3H), 1.43–1.35 (m, 1H).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 158.2, 134.1, 131.0, 129.4, 128.8, 127.7, 126.6, 125.8, 120.8, 119.6, 105.7, 55.5, 45.3, 42.6, 31.1, 30.4, 25.1, 25.0.

FT-IR (neat) 2957, 2869, 2237, 1635, 1607, 1507, 1485, 1457, 1419, 1393, 1348, 1266, 1230, 1213, 1174, 1121, 1031, 890, 852, 811, 673 $cm^{-1}$.

MS (EI) *m*/*z* ($M^+$) calcd for $C_{18}H_{19}NO$: 265, found: 265.

$[\alpha]^{25}_D$ = +26.4° (c = 1.00, $CHCl_3$).




**(*R*)-2-Cyclopentyl-2-(4-methoxyphenyl)acetonitrile** (**Table 3, entry 3**). 2-Bromo-2-cyclopentylacetonitrile (150 mg, 0.80 mmol) and 4-methoxyphenylmagnesium bromide (0.42 M in THF; Aldrich) were used. The product was purified by column chromatography (5%→10% $Et_2O$/hexanes). Light-yellow solid. First run: 135 mg (78%, 94% ee). Second run: 144 mg (84%, 95% ee).

The ee was determined by HPLC analysis on a CHIRALPAK AS-H column (10% *i*-PrOH/hexanes, 1.0 mL/min) with $t_r$ = 12.4 min (major), 16.1 min (minor).

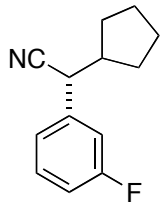
$^1$H NMR (500 MHz, $CDCl_3$) δ 7.23 (d, 2H, *J* = 8.8 Hz), 6.89 (d, 2H, *J* = 8.5 Hz), 3.81 (s, 3H), 3.65 (d, 1H, *J* = 7.9 Hz), 2.32–2.24 (m, 1H), 1.89–1.83 (m, 1H), 1.75–1.64 (m, 3H), 1.62–1.45 (m, 3H), 1.35–1.29 (m, 1H).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 159.3, 128.8, 128.0, 121.0, 114.4, 55.5, 45.5, 41.8, 31.0, 30.4, 25.0 (2C).

FT-IR (neat) 2959, 2868, 2838, 2234, 1613, 1514, 1465, 1442, 1424, 1348, 1303, 1252, 1180, 1107, 1035, 824 $cm^{-1}$.

MS (EI) *m*/*z* ($M^+$) calcd for $C_{14}H_{17}NO$: 215, found: 215.

$[\alpha]^{25}_D$ = +25.0° (c = 1.00, $CHCl_3$).




**(*R*)-2-Cyclopentyl-2-(3-fluorophenyl)acetonitrile (Table 3, entry 4).** 2-Bromo-2-cyclopentylacetonitrile (150 mg, 0.80 mmol) and (3-fluorophenyl)magnesium bromide (0.86 M in THF; Aldrich) were used. The reaction was run at –60 °C. The product was purified by column chromatography (1.6%→3% $Et_2O$/hexanes). Colorless oil. First run: 164 mg (100%, 93% ee). Second run: 161 mg (99%, 93% ee).

The ee was determined by GC analysis on a G-TA column (100 °C hold 5 min, then 100 °C→180 °C @ 5 °C/min, hold 10 min, 1.7 mL/min) with $t_r$ = 19.7 min (major), 20.4 min (minor).

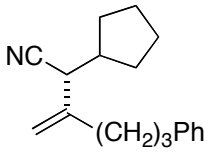
$^1$H NMR (500 MHz, $CDCl_3$) δ 7.34 (apparent ddd, 1H, *J* = 5.9, 7.9, 7.9 Hz), 7.12–7.10 (m, 1H), 7.06–7.03 (m, 1H), 7.01 (ddd, 1H, *J* = 0.9, 2.5, 3.4 Hz), 3.72 (d, 1H, *J* = 7.6 Hz), 2.34–2.26 (m, 1H), 1.88–1.82 (m, 1H), 1.77–1.67 (m, 3H), 1.63–1.45 (m, 3H), 1.39–1.31 (m, 1H).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 163.0 (d, *J* = 248 Hz), 138.4, 130.7 (d, *J* = 8 Hz), 123.4 (d, *J* = 3 Hz), 120.1, 115.2 (d, *J* = 21 Hz), 114.9 (d, *J* = 22 Hz), 45.3, 42.3, 31.1, 30.3, 25.0, 24.9.

FT-IR (neat) 3064, 2958, 2918, 2871, 2241, 1616, 1593, 1489, 1449, 1355, 1318, 1265, 1248, 1140, 1078, 871, 786, 761, 694 $cm^{-1}$.

MS (EI) *m*/*z* ($M^+$) calcd for $C_{13}H_{14}FN$: 203, found: 203.

$[\alpha]^{25}_D$ = +29.5° (c = 1.01, $CHCl_3$).




**(*R*)-2-Cyclopentyl-3-methylene-6-phenylhexanenitrile (Table 4, entry 1).** 2-Bromo-2-cyclopentylacetonitrile (150 mg, 0.80 mmol) and (5-phenylpent-1-en-2-yl)magnesium bromide (0.75 M in THF) were used. (4-Bromopent-4-en-1-yl)benzene was prepared from pent-4-yn-1-

ylbenzene following a literature procedure.[4] The reaction was run at –60 °C. The product was purified by column chromatography (3%→5% $Et_2O$/hexanes). Colorless oil. First run: 131 mg (65%, 80% ee). Second run: 128 mg (63%, 80% ee).

The ee was determined by HPLC analysis on a CHIRALCEL OJ-H column (1% *i*-PrOH/hexanes, 1.0 mL/min) with $t_r$ = 18.6 min (minor), 20.7 min (major).

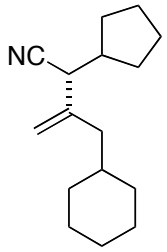
$^1$H NMR (500 MHz, $CDCl_3$) δ 7.31–7.28 (m, 2H), 7.21–7.18 (m, 3H), 5.15 (s, 1H), 5.02 (s, 1H), 3.13 (d, 1H, *J* = 7.5 Hz), 2.71–2.60 (m, 2H), 2.24–2.16 (m, 2H), 2.13–2.05 (m, 1H), 1.89–1.78 (m, 3H), 1.79–1.65 (m, 3H), 1.63–1.51 (m, 2H), 1.45–1.36 (m, 1H), 1.36–1.24 (m, 1H).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 142.9, 142.0, 128.5 (2C), 126.1, 120.3, 114.0, 43.7, 40.7, 35.5, 33.2, 31.2, 30.2, 29.4, 25.3, 25.1.

FT-IR (neat) 3085, 3062, 3027, 2948, 2868, 2237, 1647, 1603, 1496, 1453, 1353, 1080, 1030, 905, 750, 699 cm$^{-1}$.

MS (ESI) *m*/*z* ($M^+$+Na) calcd for $C_{18}H_{23}NNa$: 276.2, found: 276.2.

$[\alpha]^{24}_D$ = –2.9° (c = 1.00, $CHCl_3$).




**(*R*)-3-(Cyclohexylmethyl)-2-cyclopentylbut-3-enenitrile (Table 4, entry 2).** 2-Bromo-2-cyclopentylacetonitrile (150 mg, 0.80 mmol) and (3-cyclohexylprop-1-en-2-yl)magnesium bromide (0.72 M in THF) were used. (2-Bromoallyl)cyclohexane was prepared from prop-2-yn-1-ylcyclohexane following a literature procedure.[4] The reaction was run at –60 °C. The product was purified by column chromatography (2%→3% $Et_2O$/hexanes). Light-yellow oil. First run: 112 mg (61%, 85% ee). Second run: 106 mg (57%, 86% ee).

The ee was determined by GC analysis on a G-TA column (75 °C hold 1 min, then 75 °C→180 °C @ 2 °C/min, hold 15 min, 1.0 mL/min) with $t_r$ = 52.7 min (minor), 53.2 min (major).

$^1$H NMR (500 MHz, $CDCl_3$) δ 5.18 (s, 1H), 4.97 (d, 1H, *J* = 0.6 Hz), 3.13 (d, 1H, *J* = 7.1 Hz), 2.25–2.17 (m, 1H), 2.01–1.93 (m, 2H), 1.86–1.64 (m, 8H), 1.61–1.54 (m, 2H), 1.49–1.31 (m, 3H), 1.29–1.10 (m, 4H), 0.94–0.81 (m, 2H).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 141.4, 120.4, 114.9, 43.1, 42.2, 40.5, 35.7, 33.6, 33.0, 31.2, 30.0, 26.6, 26.4, 26.3, 25.4, 25.2.

FT-IR (neat) 3084, 2924, 2852, 2665, 2238, 1647, 1449, 1350, 1262, 1080, 905 cm$^{-1}$.

MS (EI) *m*/*z* ($M^+$) calcd for $C_{16}H_{25}N$: 231, found: 231.

$[\alpha]^{24}_D$ = –11.3° (c = 1.00, $CHCl_3$).

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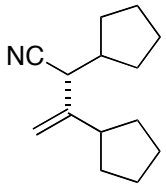
(4) Hara, S.; Dojo, H.; Takinami, S.; Suzuki, A. *Tetrahedron Lett.* **1983**, *24*, 731–734.




**(*R*)-2,3-Dicyclopentylbut-3-enenitrile (Table 4, entry 3).** 2-Bromo-2-cyclopentylacetonitrile (150.5 mg, 0.80 mmol) and (1-cyclopentylvinyl)magnesium bromide (0.73 M in THF) were used. (1-Bromovinyl)cyclopentane was prepared from ethynylcyclopentane following a literature procedure.[4] The reaction was run at –60 °C. The product was purified by column chromatography (3% $Et_2O$/hexanes). Colorless oil. First run: 125 mg (77%, 88% ee). Second run: 129 mg (79%, 90% ee).

The ee was determined by GC analysis on a CP-Chirasil-Dex CB column (120 °C hold 60 min, 1.0 mL/min) with $t_r$ = 44.0 min (major), 46.0 min (minor).

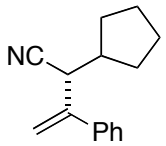
$^1$H NMR (500 MHz, $CDCl_3$) δ 5.13 (s, 1H), 5.06 (d, 1H, *J* = 1.3 Hz), 3.18 (d, 1H, *J* = 7.2 Hz), 2.43–2.37 (m, 1H), 2.30–2.22 (m, 1H), 1.94–1.82 (m, 3H), 1.81–1.67 (m, 5H), 1.64–1.54 (m, 4H), 1.49–1.31 (m, 4H).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 147.4, 120.6, 111.9, 44.3, 43.5, 41.0, 32.9, 32.6, 31.3, 30.1, 25.3, 25.2, 25.0, 24.9.

FT-IR (neat) 3091, 2956, 2869, 2237, 1645, 1473, 1452, 1351, 1306, 1162, 902 cm$^{-1}$.

MS (ESI) *m*/*z* ($M^+$+Na) calcd for $C_{14}H_{21}NNa$: 226.2, found: 226.2.

$[\alpha]^{24}_D$ = +7.2° (c = 1.00, $CHCl_3$).




**(*R*)-2-Cyclopentyl-3-phenylbut-3-enenitrile (Table 4, entry 4).** 2-Bromo-2-cyclopentylacetonitrile (150 mg, 0.80 mmol) and (1-phenylvinyl)magnesium bromide (0.80 M in THF) were used. The reaction was run at –60 °C. The product was purified by column chromatography (2%→3% $Et_2O$/hexanes). Colorless oil. First run: 157 mg (93%, 91% ee). Second run: 162 mg (96%, 91% ee).

The ee was determined by HPLC analysis on a CHIRALCEL OJ-H column (1% *i*-PrOH/hexanes, 1.0 mL/min) with $t_r$ = 9.1 min (major), 15.3 min (minor).

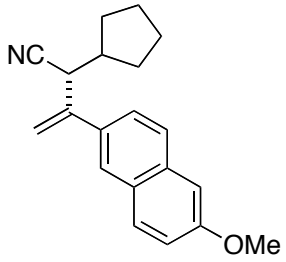
$^1$H NMR (500 MHz, $CDCl_3$) δ 7.39–7.31 (m, 5H), 5.55 (d, 1H, *J* = 1.2 Hz), 5.42 (s, 1H), 3.83 (dd, 1H, *J* = 1.1, 6.1 Hz), 2.12–2.04 (m, 1H), 1.76–1.64 (m, 4H), 1.54–1.36 (m, 4H).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 143.4, 139.1, 128.8, 128.4, 126.7, 120.0, 116.6, 42.5, 40.7, 31.2, 29.3, 25.5, 25.1.

FT-IR (neat) 3057, 2956, 2869, 2240, 1954, 1830, 1630, 1576, 1495, 1445, 1294, 1075, 1029, 910, 775, 700 cm$^{-1}$.

MS (EI) *m*/*z* ($M^+$) calcd for $C_{15}H_{17}N$: 211, found: 211.

$[\alpha]^{25}_D$ = –16.4° (c = 1.00, $CHCl_3$).




**(*R*)-2-Cyclopentyl-3-(6-methoxynaphthalen-2-yl)but-3-enenitrile (Table 4, entry 5).** 2-Bromo-2-cyclopentylacetonitrile (113 mg, 0.60 mmol) and (1-(6-methoxynaphthalen-2-yl)vinyl)magnesium bromide (0.51 M in THF) were used. The reaction was run at –60 °C. The product was purified by column chromatography (first purification: 5% $Et_2O$/hexanes, second purification: 50% toluene/hexanes). Yellow liquid. First run: 162 mg (93%, 92% ee). Second run (0.20 mmol): 53 mg (91%, 92% ee).

The ee was determined by HPLC on a CHIRALPAK IA column (1% *i*-PrOH/hexanes, 1.0 mL/min) with $t_r$ = 15.4 min (major), 20.0 min (minor).

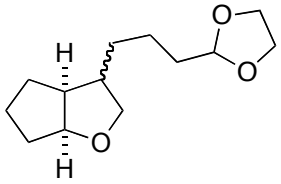
$^1$H NMR (500 MHz, $CDCl_3$) δ 7.75–7.72 (m, 3H), 7.45 (dd, 1H, *J* = 2.0, 8.5 Hz), 7.18 (dd, 1H, *J* = 2.5, 9.0 Hz), 7.13 (d, 1H, *J* = 2.5 Hz), 5.60 (d, 1H, *J* = 1.0 Hz), 5.53 (s, 1H), 3.95–3.93 (m, 4H), 2.17–2.09 (m, 1H), 1.78–1.65 (m, 4H), 1.52–1.40 (m, 4H).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 158.3, 143.4, 134.4, 134.2, 129.8, 128.8, 127.3, 125.4, 125.2, 120.2, 119.6, 116.4, 105.8, 55.5, 42.5, 41.0, 31.3, 29.4, 25.5, 25.1.

FT-IR (neat) 3058, 2956, 2869, 2239, 1630, 1603, 1502, 1484, 1463, 1453, 1411, 1392, 1336, 1270, 1208, 1165, 1127, 1032, 898, 854, 810, 758 $cm^{-1}$.

MS (EI) *m*/*z* ($M^+$) calcd for $C_{20}H_{21}NO$: 291, found: 291.

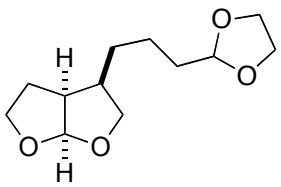
$[\alpha]^{26}_D$ = –23.9° (c = 1.00, $CHCl_3$).



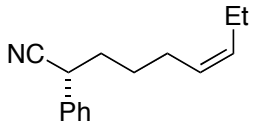

**3-(3-(1,3-Dioxolan-2-yl)propyl)hexahydro-2*H*-cyclopenta[*b*]furan (eq 3).** The title compound was prepared from *trans*-1-(allyloxy)-2-bromocyclopentane (123 mg, 0.60 mmol) and (2-(1,3-dioxolan-2-yl)ethyl)zinc bromide (0.96 mmol; ~0.75 M in DMA) following a procedure for nickel-catalyzed Negishi cross-couplings.[5] The product was purified by column chromatography on silica gel (20% ethyl acetate/hexanes) and then on C-18 silica gel (10%→100% acetonitrile/water).

(5) Zhou, J.; Fu, G. C. *J. Am. Chem. Soc.* **2003**, *125*, 14726–14727.

Light-yellow oil. First run: 103 mg (76%, endo:exo = 2.3:1). Second run: 111 mg (82%, endo:exo = 2.3:1). The spectral data matched previously reported data.[6]



**(3*R**,3a*S**,6a*R**)-3-(3-(1,3-dioxolan-2-yl)propyl)hexahydrofuro[2,3-*b*]furan (eq 3).** The title compound was prepared from *trans*-2-(allyloxy)-3-bromotetrahydrofuran (124 mg, 0.60 mmol) and (2-(1,3-dioxolan-2-yl)ethyl)zinc bromide (0.96 mmol; ~0.75 M in DMA) following a procedure for nickel-catalyzed Negishi cross-couplings.[5] The product was purified by column chromatography (40% ethyl acetate/hexanes). Light-yellow oil. First run: 118 mg (86%, endo:exo = 44:1). Second run: 120 mg (88%, endo:exo = 44:1). The spectral data matched previously reported data.[6]



**(*R*,*Z*)-2-Phenylnon-6-enenitrile (eq 4).** (*Z*)-2-Bromonon-6-enenitrile (130 mg, 0.60 mmol) was used. The product was purified by column chromatography (2%→3% $Et_2O$/hexanes). Colorless oil. First run: 122 mg (95%, 77% ee). Second run: 120 mg (94%, 77% ee).

The ee was determined by HPLC analysis on a CHIRALCEL OJ-H column (1% *i*-PrOH/hexanes, 1.0 mL/min) with $t_r$ = 9.1 min (major), 11.0 min (minor).

$^1$H NMR (500 MHz, $CDCl_3$) δ 7.40–7.36 (m, 2H), 7.34–7.31 (m, 3H), 5.40 (ddddd, 1H, *J* = 1.6, 1.6, 7.2, 7.2, 10.8 Hz), 5.27 (ddddd, 1H, *J* = 1.5, 1.5, 7.2, 7.2, 10.8 Hz), 3.78 (dd, 1H, *J* = 6.3, 8.5 Hz), 2.08 (q, 2H, *J* = 7.3 Hz), 2.02 (quintet, 2H, *J* = 7.5 Hz), 1.98–1.84 (m, 2H), 1.62–1.47 (m, 2H), 0.95 (t, 3H, *J* = 7.5 Hz).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 136.1, 132.9, 129.2, 128.2, 127.8, 127.4, 121.0, 37.4, 35.5, 27.1, 26.4, 20.7, 14.4.

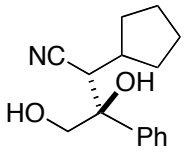
FT-IR (neat) 3066, 3007, 2931, 2863, 2240, 1653, 1602, 1495, 1455, 1405, 1373, 1304, 1070, 1030, 969, 912, 756, 698 $cm^{-1}$.

MS (EI) *m*/*z* ($M^+$) calcd for $C_{13}H_{19}N$: 213, found: 213.

$[\alpha]^{24}_D$ = +12.6° (c = 1.00, $CHCl_3$).

(6) Phapale, V. B.; Bunuel, E.; García-Iglesias, M.; Cárdenas, D. J. *Angew. Chem., Int. Ed.* **2007**, *46*, 8790–8795.

## IV. Functionalization of the Cross-Coupling Product




**(2*S*,3*S*)-2-Cyclopentyl-3,4-dihydroxy-3-phenylbutanenitrile (eq 2).** The title compound was prepared via a modification of a literature procedure.[7] (*R*)-2-Cyclopentyl-3-phenylbut-3-enenitrile (80 mg, 0.38 mmol; Table 4, entry 4; from a reaction using (*S*,*S*)-**L**), $K_3Fe(CN)_6$ (374 mg, 1.14 mmol), $K_2CO_3$ (157 mg, 1.14 mmol), 1,4-diazabicyclo[2.2.2]octane (21 mg, 0.19 mmol), water (1.89 mL), and *t*-BuOH (1.31 mL) were added to a 20-mL vial equipped with a magnetic stir bar. The vial was sealed with a PTFE-lined septum cap, and the mixture was stirred at r.t. for 10 min. Then, the solution was cooled to 0 °C, and $OsO_4$ (0.58 mL; 2.5 wt% solution in *t*-BuOH; Aldrich) was added to the vial. The reaction mixture was stirred at 0 °C for 72 h, and then the reaction was quenched by the addition of saturated aqueous $Na_2SO_3$ (5 mL). The solution was stirred for 1 h, and then the reaction mixture was extracted with ethyl acetate (3 × 10 mL). The combined organic layers were dried over $Na_2SO_4$ and concentrated. The product was purified by column chromatography (20%→25% ethyl acetate/hexanes). Brown oil. First run: 75 mg (80% yield, 13:1 dr). Second run (0.43 mmol; from a reaction using (*R*,*R*)-**L**): 79 mg (75% yield, 12:1 dr).

The dr was determined by HPLC analysis on a CHIRALCEL OD-H column (10% *i*-PrOH/hexanes, 1.0 mL/min) with $t_r$ = 9.3, 12.6 min (major), 17.9, 23.7 min (minor). The stereochemistry of the major isomer was assigned on the basis of an X-ray crystal structure of the cyclic-carbonate derivative.

$^1$H NMR (500 MHz, $CDCl_3$) δ 7.60–7.57 (m, 2H), 7.43–7.39 (m, 2H), 7.36–7.33 (m, 1H), 4.12 (dd, 1H, *J* = 11.1, 7.0 Hz), 4.04 (dd, 1H, *J* = 11.1, 4.0 Hz), 3.19 (d, 1H, *J* = 4.8 Hz), 3.12 (s, 1H), 2.10–2.02 (m, 1H), 1.86–1.80 (m, 2H), 1.65–1.51 (m, 2H), 1.45–1.31 (m, 3H), 1.30–1.23 (m, 1H), 1.13–1.05 (m, 1H).

$^{13}$C NMR (126 MHz, $CDCl_3$) δ 140.0, 128.8, 128.4, 125.9, 119.6, 76.7, 68.4, 45.6, 37.0, 33.2, 30.1, 25.2, 24.8.

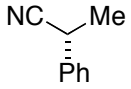
FT-IR (neat) 3439, 2955, 2870, 2242, 1496, 1449, 1395, 1289, 1184, 1135, 1069, 959, 909, 771, 703 $cm^{-1}$.

MS (EI) *m*/*z* ($M^+$+H) calcd for $C_{15}H_{20}NO_2$: 246.1, found: 246.1.

$[\alpha]^{24}_D$ = 37.4° (c = 0.96, $CHCl_3$).

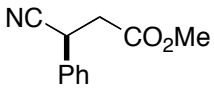
(7) Petrova, K. V.; Mohr , J. T.; Stoltz, B. M. *Org. Lett.* **2009**, *11*, 293–295.

## V. Assignment of Absolute Stereochemistry




**(*R*)-2-Phenylpropanenitrile (from a reaction using (*S,S*)-L).** 2-Phenylpropanenitrile was prepared from 2-bromopropanenitrile and $Ph_2Zn$ according to the general procedure.

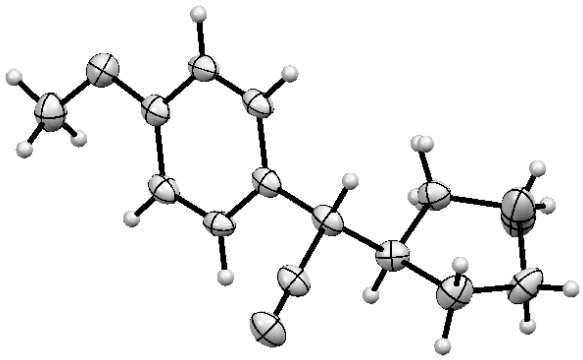
To determine the absolute stereochemistry, the specific rotation of the product was compared with the literature: $[\alpha]^{25}_D = +22.6°$ (c = 1.00, $CHCl_3$; 90% ee); lit.[8] $[\alpha]^{RT}_D = +18.5°$ (c = 1.2, $CHCl_3$; ≥95% ee; *R* enantiomer). Therefore, the absolute configuration of the cross-coupling product is assigned as *R*.




**(*S*)-Methyl 3-cyano-3-phenylpropanoate (from a reaction using (*R,R*)-L).** Methyl 3-cyano-3-phenylpropanoate was prepared from methyl 3-bromo-3-cyanopropanoate and $Ph_2Zn$ according to the general procedure.

To determine the absolute stereochemistry, the specific rotation of the product was compared with the literature: $[\alpha]^{25}_D = -16.0°$ (c = 1.02, MeOH; 88% ee); lit.[9] $[\alpha]^{29}_D = -15.3°$ (c = 1.15, MeOH; 94% ee). Therefore, the absolute configuration of the cross-coupling product is assigned as *S*.

**Product from entry 4 of Table 3 (run with (*R,R*)-L). (*S*)-2-Cyclopentyl-2-(4-methoxyphenyl)acetonitrile.** A crystal suitable for X-ray crystallography was grown by vapor diffusion with dichloromethane and pentane.



(8) Enders, D.; Plant, A.; Backhaus, D.; Reinhold, U. *Tetrahedron* **1995**, *51*, 10699–10714.

(9) Fryszkowska, A.; Fisher, K.; Gardiner, J. M.; Stephens, G. M. *Org. Biomol. Chem.* **2010**, *8*, 533–535.

Reference for the Hooft/Spek method: Hooft, R. W. W.; Straver, L. H.; Spek, A. L. *J. Appl. Cryst.* **2007**, *41*, 96–103. Absolute configuration: The Flack test is inconclusive because this is a light-atom structure. However the method by Spek and Hooft, which is based on Bayesian statistics, results in the following probabilities (see also file X11176_t4.lis): The probability P2(true) of the model to be correct assuming that the structure is either right or wrong is 1.000. The probability P3(true) of the model to be correct assuming that the structure is either right or wrong or a 50:50 racemic twin is 1.000. The probability P3(rac-twin) of the model to be a 50:50 racemic twin is 0.1E-14. The probability P3(false) of the model to be wrong is 0.4E-95. There are two independent molecules in the asymmetric unit, and two of the atoms in the cyclohexane group in both are disordered with appropriate restraints. For the second molecule the anisotropic displacement parameters of one of the carbons was constrained to be equivalent to the major component.

Table 1. Crystal data and structure refinement for X11176_t5.

| | | |
|---|---|---|
| Identification code | x11176_t5 | |
| Empirical formula | C14 H17 N O | |
| Formula weight | 215.29 | |
| Temperature | 100(2) K | |
| Wavelength | 1.54178 Å | |
| Crystal system | Monoclinic | |
| Space group | P2(1) | |
| Unit cell dimensions | a = 5.6768(2) Å | α= 90°. |
| | b = 9.5536(3) Å | β= 92.523(2)°. |
| | c = 21.6728(7) Å | γ = 90°. |
| Volume | 1174.26(7) $Å^3$ | |
| Z | 4 | |
| Density (calculated) | 1.218 $Mg/m^3$ | |
| Absorption coefficient | 0.595 $mm^{-1}$ | |
| F(000) | 464 | |
| Crystal size | 0.25 x 0.20 x 0.15 $mm^3$ | |
| Theta range for data collection | 2.04 to 70.23°. | |
| Index ranges | -6<=h<=6, -11<=k<=11, -26<=l<=26 | |
| Reflections collected | 4343 | |
| Independent reflections | 4349 [R(int) = 0.0395] | |
| Completeness to theta = 70.23° | 98.5 % | |
| Absorption correction | Semi-empirical from equivalents | |
| Max. and min. transmission | 0.9160 and 0.8655 | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 4349 / 276 / 324 | |
| Goodness-of-fit on $F^2$ | 1.053 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0326, wR2 = 0.0841 | |
| R indices (all data) | R1 = 0.0326, wR2 = 0.0842 | |
| Absolute structure parameter | 0.1(3) | |
| Largest diff. peak and hole | 0.188 and -0.131 $e.Å^{-3}$ | |

Table 2. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($Å^2$x $10^3$) for X11176_t5. U(eq) is defined as one third of the trace of the orthogonalized $U^{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| O(1) | 560(3) | 7685(2) | 7064(1) | 39(1) |
| C(1) | 335(4) | 7459(2) | 6444(1) | 29(1) |
| C(2) | -1575(4) | 7894(2) | 6066(1) | 30(1) |
| C(3) | -1634(3) | 7582(2) | 5445(1) | 27(1) |
| C(4) | 169(3) | 6828(2) | 5181(1) | 26(1) |
| C(8) | 16(3) | 6448(2) | 4500(1) | 28(1) |
| C(9) | -2195(3) | 5676(2) | 4359(1) | 30(1) |
| N(1) | -3973(3) | 5142(2) | 4254(1) | 41(1) |
| C(11) | 128(3) | 7724(2) | 4060(1) | 29(1) |
| C(12) | -12(4) | 7273(2) | 3370(1) | 38(1) |
| C(13) | 2458(7) | 7515(5) | 3145(1) | 39(1) |
| C(14) | 3199(9) | 8845(5) | 3500(2) | 38(1) |
| C(13A) | 1792(15) | 8262(11) | 3060(3) | 42(2) |
| C(14A) | 3776(16) | 8333(12) | 3523(4) | 42(2) |
| C(15) | 2483(4) | 8523(2) | 4148(1) | 32(1) |
| C(5) | 2074(3) | 6414(2) | 5563(1) | 28(1) |
| C(6) | 2165(3) | 6723(2) | 6182(1) | 30(1) |
| C(7) | -1410(4) | 8310(2) | 7347(1) | 41(1) |
| O(2) | 5483(3) | 782(2) | -1866(1) | 38(1) |
| C(21) | 5262(4) | 955(2) | -1248(1) | 30(1) |
| C(22) | 3345(3) | 495(2) | -922(1) | 29(1) |
| C(23) | 3308(3) | 751(2) | -293(1) | 28(1) |
| C(24) | 5113(3) | 1475(2) | 22(1) | 27(1) |
| C(28) | 5070(3) | 1744(2) | 709(1) | 30(1) |
| C(29) | 2855(3) | 2478(2) | 856(1) | 30(1) |
| N(2) | 1111(3) | 2998(2) | 971(1) | 39(1) |
| C(31) | 5303(4) | 406(2) | 1111(1) | 31(1) |
| C(32) | 5310(4) | 713(2) | 1808(1) | 40(1) |
| C(33) | 6727(6) | -446(3) | 2101(1) | 40(1) |
| C(34) | 8690(16) | -650(8) | 1662(2) | 61(2) |
| C(33A) | 7840(20) | 314(15) | 2046(4) | 49(3) |
| C(34A) | 8510(60) | -890(30) | 1651(7) | 61(2) |
| C(35) | 7639(3) | -378(2) | 1014(1) | 32(1) |
| C(25) | 7020(3) | 1919(2) | -313(1) | 30(1) |
| C(26) | 7097(3) | 1662(2) | -934(1) | 32(1) |
| C(27) | 3583(4) | 110(2) | -2202(1) | 40(1) |

Table 3. Bond lengths [Å] and angles [°] for X11176_t5.

| | |
|---|---|
| O(1)-C(1) | 1.362(2) |
| O(1)-C(7) | 1.429(3) |
| C(1)-C(2) | 1.392(3) |
| C(1)-C(6) | 1.395(3) |
| C(2)-C(3) | 1.378(2) |
| C(2)-H(2) | 0.9500 |
| C(3)-C(4) | 1.395(3) |
| C(3)-H(3) | 0.9500 |
| C(4)-C(5) | 1.390(2) |
| C(4)-C(8) | 1.519(2) |
| C(8)-C(9) | 1.475(2) |
| C(8)-C(11) | 1.551(2) |
| C(8)-H(8) | 1.0000 |
| C(9)-N(1) | 1.145(3) |
| C(11)-C(15) | 1.544(3) |
| C(11)-C(12) | 1.554(2) |
| C(11)-H(11) | 1.0000 |
| C(12)-C(13) | 1.522(4) |
| C(12)-C(13A) | 1.567(7) |
| C(12)-H(12A) | 0.9900 |
| C(12)-H(12B) | 0.9900 |
| C(12)-H(12C) | 0.9900 |
| C(12)-H(12D) | 0.9900 |
| C(13)-C(14) | 1.535(5) |
| C(13)-H(13A) | 0.9900 |
| C(13)-H(13B) | 0.9900 |
| C(14)-C(15) | 1.512(4) |
| C(14)-H(14A) | 0.9900 |
| C(14)-H(14B) | 0.9900 |
| C(13A)-C(14A) | 1.478(11) |
| C(13A)-H(13C) | 0.9900 |
| C(13A)-H(13D) | 0.9900 |
| C(14A)-C(15) | 1.579(9) |
| C(14A)-H(14C) | 0.9900 |
| C(14A)-H(14D) | 0.9900 |
| C(15)-H(15A) | 0.9900 |
| C(15)-H(15B) | 0.9900 |
| C(15)-H(15C) | 0.9900 |
| C(15)-H(15D) | 0.9900 |
| C(5)-C(6) | 1.374(2) |
| C(5)-H(5) | 0.9500 |
| C(6)-H(6) | 0.9500 |
| C(7)-H(7A) | 0.9800 |
| C(7)-H(7B) | 0.9800 |
| C(7)-H(7C) | 0.9800 |
| O(2)-C(21) | 1.361(2) |
| O(2)-C(27) | 1.427(3) |
| C(21)-C(26) | 1.394(3) |
| C(21)-C(22) | 1.394(3) |
| C(22)-C(23) | 1.384(2) |
| C(22)-H(22) | 0.9500 |
| C(23)-C(24) | 1.390(2) |
| C(23)-H(23) | 0.9500 |
| C(24)-C(25) | 1.396(3) |
| C(24)-C(28) | 1.512(2) |

| | |
|---|---|
| C(28)-C(29) | 1.486(2) |
| C(28)-C(31) | 1.549(2) |
| C(28)-H(28) | 1.0000 |
| C(29)-N(2) | 1.145(3) |
| C(31)-C(32) | 1.538(2) |
| C(31)-C(35) | 1.545(2) |
| C(31)-H(31) | 1.0000 |
| C(32)-C(33) | 1.493(3) |
| C(32)-C(33A) | 1.550(9) |
| C(32)-H(32A) | 0.9900 |
| C(32)-H(32B) | 0.9900 |
| C(32)-H(32C) | 0.9900 |
| C(32)-H(32D) | 0.9900 |
| C(33)-C(34) | 1.509(8) |
| C(33)-H(33A) | 0.9900 |
| C(33)-H(33B) | 0.9900 |
| C(34)-C(35) | 1.523(5) |
| C(34)-H(34A) | 0.9900 |
| C(34)-H(34B) | 0.9900 |
| C(33A)-C(34A) | 1.497(16) |
| C(33A)-H(33C) | 0.9900 |
| C(33A)-H(33D) | 0.9900 |
| C(34A)-C(35) | 1.526(14) |
| C(34A)-H(34C) | 0.9900 |
| C(34A)-H(34D) | 0.9900 |
| C(35)-H(35A) | 0.9900 |
| C(35)-H(35B) | 0.9900 |
| C(35)-H(35C) | 0.9900 |
| C(35)-H(35D) | 0.9900 |
| C(25)-C(26) | 1.371(3) |
| C(25)-H(25) | 0.9500 |
| C(26)-H(26) | 0.9500 |
| C(27)-H(27A) | 0.9800 |
| C(27)-H(27B) | 0.9800 |
| C(27)-H(27C) | 0.9800 |

| | |
|---|---|
| C(1)-O(1)-C(7) | 116.65(16) |
| O(1)-C(1)-C(2) | 124.69(17) |
| O(1)-C(1)-C(6) | 116.22(17) |
| C(2)-C(1)-C(6) | 119.09(16) |
| C(3)-C(2)-C(1) | 119.63(17) |
| C(3)-C(2)-H(2) | 120.2 |
| C(1)-C(2)-H(2) | 120.2 |
| C(2)-C(3)-C(4) | 121.72(17) |
| C(2)-C(3)-H(3) | 119.1 |
| C(4)-C(3)-H(3) | 119.1 |
| C(5)-C(4)-C(3) | 117.90(16) |
| C(5)-C(4)-C(8) | 121.38(16) |
| C(3)-C(4)-C(8) | 120.71(16) |
| C(9)-C(8)-C(4) | 109.38(15) |
| C(9)-C(8)-C(11) | 108.86(14) |
| C(4)-C(8)-C(11) | 114.07(14) |
| C(9)-C(8)-H(8) | 108.1 |
| C(4)-C(8)-H(8) | 108.1 |
| C(11)-C(8)-H(8) | 108.1 |
| N(1)-C(9)-C(8) | 176.43(19) |
| C(15)-C(11)-C(8) | 111.79(14) |

| | |
|---|---|
| C(15)-C(11)-C(12) | 105.32(15) |
| C(8)-C(11)-C(12) | 111.84(14) |
| C(15)-C(11)-H(11) | 109.3 |
| C(8)-C(11)-H(11) | 109.3 |
| C(12)-C(11)-H(11) | 109.3 |
| C(13)-C(12)-C(11) | 104.93(18) |
| C(13)-C(12)-C(13A) | 30.9(3) |
| C(11)-C(12)-C(13A) | 103.8(3) |
| C(13)-C(12)-H(12A) | 110.8 |
| C(11)-C(12)-H(12A) | 110.8 |
| C(13A)-C(12)-H(12A) | 135.5 |
| C(13)-C(12)-H(12B) | 110.8 |
| C(11)-C(12)-H(12B) | 110.8 |
| C(13A)-C(12)-H(12B) | 83.0 |
| H(12A)-C(12)-H(12B) | 108.8 |
| C(13)-C(12)-H(12C) | 134.3 |
| C(11)-C(12)-H(12C) | 111.0 |
| C(13A)-C(12)-H(12C) | 111.0 |
| H(12A)-C(12)-H(12C) | 82.0 |
| H(12B)-C(12)-H(12C) | 29.6 |
| C(13)-C(12)-H(12D) | 82.3 |
| C(11)-C(12)-H(12D) | 111.0 |
| C(13A)-C(12)-H(12D) | 111.0 |
| H(12A)-C(12)-H(12D) | 30.0 |
| H(12B)-C(12)-H(12D) | 130.6 |
| H(12C)-C(12)-H(12D) | 109.0 |
| C(12)-C(13)-C(14) | 101.6(3) |
| C(12)-C(13)-H(13A) | 111.5 |
| C(14)-C(13)-H(13A) | 111.5 |
| C(12)-C(13)-H(13B) | 111.5 |
| C(14)-C(13)-H(13B) | 111.5 |
| H(13A)-C(13)-H(13B) | 109.3 |
| C(15)-C(14)-C(13) | 102.7(3) |
| C(15)-C(14)-H(14A) | 111.2 |
| C(13)-C(14)-H(14A) | 111.2 |
| C(15)-C(14)-H(14B) | 111.2 |
| C(13)-C(14)-H(14B) | 111.2 |
| H(14A)-C(14)-H(14B) | 109.1 |
| C(14A)-C(13A)-C(12) | 103.2(6) |
| C(14A)-C(13A)-H(13C) | 111.1 |
| C(12)-C(13A)-H(13C) | 111.1 |
| C(14A)-C(13A)-H(13D) | 111.1 |
| C(12)-C(13A)-H(13D) | 111.1 |
| H(13C)-C(13A)-H(13D) | 109.1 |
| C(13A)-C(14A)-C(15) | 102.7(6) |
| C(13A)-C(14A)-H(14C) | 111.2 |
| C(15)-C(14A)-H(14C) | 111.2 |
| C(13A)-C(14A)-H(14D) | 111.2 |
| C(15)-C(14A)-H(14D) | 111.2 |
| H(14C)-C(14A)-H(14D) | 109.1 |
| C(14)-C(15)-C(11) | 104.6(2) |
| C(14)-C(15)-C(14A) | 21.9(3) |
| C(11)-C(15)-C(14A) | 105.6(4) |
| C(14)-C(15)-H(15A) | 110.8 |
| C(11)-C(15)-H(15A) | 110.8 |
| C(14A)-C(15)-H(15A) | 127.9 |
| C(14)-C(15)-H(15B) | 110.8 |

| | |
|---|---|
| C(11)-C(15)-H(15B) | 110.8 |
| C(14A)-C(15)-H(15B) | 90.7 |
| H(15A)-C(15)-H(15B) | 108.9 |
| C(14)-C(15)-H(15C) | 128.9 |
| C(11)-C(15)-H(15C) | 110.6 |
| C(14A)-C(15)-H(15C) | 110.6 |
| H(15A)-C(15)-H(15C) | 90.0 |
| H(15B)-C(15)-H(15C) | 21.4 |
| C(14)-C(15)-H(15D) | 91.3 |
| C(11)-C(15)-H(15D) | 110.6 |
| C(14A)-C(15)-H(15D) | 110.6 |
| H(15A)-C(15)-H(15D) | 20.9 |
| H(15B)-C(15)-H(15D) | 125.3 |
| H(15C)-C(15)-H(15D) | 108.8 |
| C(6)-C(5)-C(4) | 121.05(17) |
| C(6)-C(5)-H(5) | 119.5 |
| C(4)-C(5)-H(5) | 119.5 |
| C(5)-C(6)-C(1) | 120.59(17) |
| C(5)-C(6)-H(6) | 119.7 |
| C(1)-C(6)-H(6) | 119.7 |
| O(1)-C(7)-H(7A) | 109.5 |
| O(1)-C(7)-H(7B) | 109.5 |
| H(7A)-C(7)-H(7B) | 109.5 |
| O(1)-C(7)-H(7C) | 109.5 |
| H(7A)-C(7)-H(7C) | 109.5 |
| H(7B)-C(7)-H(7C) | 109.5 |
| C(21)-O(2)-C(27) | 117.16(15) |
| O(2)-C(21)-C(26) | 116.10(17) |
| O(2)-C(21)-C(22) | 124.65(17) |
| C(26)-C(21)-C(22) | 119.24(17) |
| C(23)-C(22)-C(21) | 119.25(17) |
| C(23)-C(22)-H(22) | 120.4 |
| C(21)-C(22)-H(22) | 120.4 |
| C(22)-C(23)-C(24) | 121.93(16) |
| C(22)-C(23)-H(23) | 119.0 |
| C(24)-C(23)-H(23) | 119.0 |
| C(23)-C(24)-C(25) | 117.87(17) |
| C(23)-C(24)-C(28) | 121.68(17) |
| C(25)-C(24)-C(28) | 120.43(17) |
| C(29)-C(28)-C(24) | 109.97(15) |
| C(29)-C(28)-C(31) | 108.73(15) |
| C(24)-C(28)-C(31) | 114.12(14) |
| C(29)-C(28)-H(28) | 107.9 |
| C(24)-C(28)-H(28) | 107.9 |
| C(31)-C(28)-H(28) | 107.9 |
| N(2)-C(29)-C(28) | 177.56(19) |
| C(32)-C(31)-C(35) | 105.09(15) |
| C(32)-C(31)-C(28) | 113.05(15) |
| C(35)-C(31)-C(28) | 112.10(14) |
| C(32)-C(31)-H(31) | 108.8 |
| C(35)-C(31)-H(31) | 108.8 |
| C(28)-C(31)-H(31) | 108.8 |
| C(33)-C(32)-C(31) | 104.72(16) |
| C(33)-C(32)-C(33A) | 37.2(5) |
| C(31)-C(32)-C(33A) | 104.0(4) |
| C(33)-C(32)-H(32A) | 110.8 |
| C(31)-C(32)-H(32A) | 110.8 |

| | |
|---|---|
| C(33A)-C(32)-H(32A) | 76.9 |
| C(33)-C(32)-H(32B) | 110.8 |
| C(31)-C(32)-H(32B) | 110.8 |
| C(33A)-C(32)-H(32B) | 139.0 |
| H(32A)-C(32)-H(32B) | 108.9 |
| C(33)-C(32)-H(32C) | 76.5 |
| C(31)-C(32)-H(32C) | 111.0 |
| C(33A)-C(32)-H(32C) | 111.0 |
| H(32A)-C(32)-H(32C) | 133.7 |
| H(32B)-C(32)-H(32C) | 36.0 |
| C(33)-C(32)-H(32D) | 138.2 |
| C(31)-C(32)-H(32D) | 111.0 |
| C(33A)-C(32)-H(32D) | 111.0 |
| H(32A)-C(32)-H(32D) | 35.7 |
| H(32B)-C(32)-H(32D) | 76.2 |
| H(32C)-C(32)-H(32D) | 109.0 |
| C(32)-C(33)-C(34) | 103.1(3) |
| C(32)-C(33)-H(33A) | 111.1 |
| C(34)-C(33)-H(33A) | 111.1 |
| C(32)-C(33)-H(33B) | 111.1 |
| C(34)-C(33)-H(33B) | 111.1 |
| H(33A)-C(33)-H(33B) | 109.1 |
| C(33)-C(34)-C(35) | 106.8(4) |
| C(33)-C(34)-H(34A) | 110.4 |
| C(35)-C(34)-H(34A) | 110.4 |
| C(33)-C(34)-H(34B) | 110.4 |
| C(35)-C(34)-H(34B) | 110.4 |
| H(34A)-C(34)-H(34B) | 108.6 |
| C(34A)-C(33A)-C(32) | 104.8(12) |
| C(34A)-C(33A)-H(33C) | 110.8 |
| C(32)-C(33A)-H(33C) | 110.8 |
| C(34A)-C(33A)-H(33D) | 110.8 |
| C(32)-C(33A)-H(33D) | 110.8 |
| H(33C)-C(33A)-H(33D) | 108.9 |
| C(33A)-C(34A)-C(35) | 100.8(10) |
| C(33A)-C(34A)-H(34C) | 111.6 |
| C(35)-C(34A)-H(34C) | 111.6 |
| C(33A)-C(34A)-H(34D) | 111.6 |
| C(35)-C(34A)-H(34D) | 111.6 |
| H(34C)-C(34A)-H(34D) | 109.4 |
| C(34)-C(35)-C(34A) | 9.6(18) |
| C(34)-C(35)-C(31) | 105.2(3) |
| C(34A)-C(35)-C(31) | 106.3(9) |
| C(34)-C(35)-H(35A) | 110.7 |
| C(34A)-C(35)-H(35A) | 101.8 |
| C(31)-C(35)-H(35A) | 110.7 |
| C(34)-C(35)-H(35B) | 110.7 |
| C(34A)-C(35)-H(35B) | 118.2 |
| C(31)-C(35)-H(35B) | 110.7 |
| H(35A)-C(35)-H(35B) | 108.8 |
| C(34)-C(35)-H(35C) | 119.1 |
| C(34A)-C(35)-H(35C) | 110.5 |
| C(31)-C(35)-H(35C) | 110.5 |
| H(35A)-C(35)-H(35C) | 9.5 |
| H(35B)-C(35)-H(35C) | 100.7 |
| C(34)-C(35)-H(35D) | 102.6 |
| C(34A)-C(35)-H(35D) | 110.5 |

| | |
|---|---|
| C(31)-C(35)-H(35D) | 110.5 |
| H(35A)-C(35)-H(35D) | 116.4 |
| H(35B)-C(35)-H(35D) | 9.0 |
| H(35C)-C(35)-H(35D) | 108.7 |
| C(26)-C(25)-C(24) | 120.97(17) |
| C(26)-C(25)-H(25) | 119.5 |
| C(24)-C(25)-H(25) | 119.5 |
| C(25)-C(26)-C(21) | 120.73(17) |
| C(25)-C(26)-H(26) | 119.6 |
| C(21)-C(26)-H(26) | 119.6 |
| O(2)-C(27)-H(27A) | 109.5 |
| O(2)-C(27)-H(27B) | 109.5 |
| H(27A)-C(27)-H(27B) | 109.5 |
| O(2)-C(27)-H(27C) | 109.5 |
| H(27A)-C(27)-H(27C) | 109.5 |
| H(27B)-C(27)-H(27C) | 109.5 |

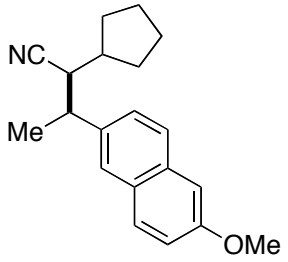
Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for X11176_t5. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| O(1) | 39(1) | 45(1) | 34(1) | 0(1) | 1(1) | 1(1) |
| C(1) | 29(1) | 24(1) | 34(1) | 3(1) | 4(1) | -3(1) |
| C(2) | 26(1) | 24(1) | 39(1) | 2(1) | 7(1) | 0(1) |
| C(3) | 25(1) | 21(1) | 36(1) | 4(1) | -1(1) | -1(1) |
| C(4) | 25(1) | 18(1) | 36(1) | 3(1) | 4(1) | -2(1) |
| C(8) | 24(1) | 20(1) | 39(1) | -2(1) | 1(1) | 0(1) |
| C(9) | 31(1) | 22(1) | 36(1) | -3(1) | -1(1) | 1(1) |
| N(1) | 33(1) | 30(1) | 59(1) | -7(1) | -3(1) | -2(1) |
| C(11) | 34(1) | 24(1) | 31(1) | -1(1) | 0(1) | 4(1) |
| C(12) | 40(1) | 42(1) | 32(1) | -5(1) | -2(1) | -1(1) |
| C(13) | 44(2) | 37(2) | 36(1) | -4(1) | 5(1) | 5(2) |
| C(14) | 42(2) | 34(2) | 39(2) | 0(2) | 7(2) | 0(2) |
| C(13A) | 54(4) | 41(4) | 31(2) | 1(3) | 5(2) | -1(4) |
| C(14A) | 46(4) | 45(5) | 35(3) | 8(4) | 9(3) | -6(4) |
| C(15) | 37(1) | 26(1) | 34(1) | -4(1) | 3(1) | -4(1) |
| C(5) | 24(1) | 19(1) | 41(1) | 2(1) | 3(1) | 0(1) |
| C(6) | 25(1) | 25(1) | 40(1) | 6(1) | -4(1) | -2(1) |
| C(7) | 43(1) | 45(1) | 35(1) | -2(1) | 8(1) | -6(1) |
| O(2) | 38(1) | 41(1) | 34(1) | 4(1) | 1(1) | 0(1) |
| C(21) | 28(1) | 22(1) | 39(1) | 4(1) | 0(1) | 3(1) |
| C(22) | 24(1) | 23(1) | 40(1) | 1(1) | -2(1) | -2(1) |
| C(23) | 22(1) | 22(1) | 40(1) | 4(1) | 5(1) | -1(1) |
| C(24) | 23(1) | 18(1) | 40(1) | 1(1) | 0(1) | 2(1) |
| C(28) | 25(1) | 23(1) | 40(1) | -3(1) | 0(1) | 2(1) |
| C(29) | 28(1) | 26(1) | 38(1) | -7(1) | -2(1) | -2(1) |
| N(2) | 32(1) | 35(1) | 49(1) | -10(1) | 0(1) | 7(1) |
| C(31) | 32(1) | 26(1) | 34(1) | -3(1) | 5(1) | 0(1) |
| C(32) | 49(1) | 37(1) | 36(1) | -3(1) | 8(1) | 7(1) |
| C(33) | 58(2) | 31(1) | 31(1) | 1(1) | 12(1) | 5(1) |
| C(34) | 54(2) | 89(3) | 41(1) | 8(1) | 7(1) | 31(2) |
| C(33A) | 45(5) | 62(6) | 41(4) | 5(4) | 4(3) | 7(5) |
| C(34A) | 54(2) | 89(3) | 41(1) | 8(1) | 7(1) | 31(2) |
| C(35) | 31(1) | 29(1) | 36(1) | 0(1) | 6(1) | 4(1) |
| C(25) | 22(1) | 24(1) | 43(1) | 2(1) | -2(1) | -3(1) |
| C(26) | 22(1) | 30(1) | 44(1) | 10(1) | 3(1) | 0(1) |
| C(27) | 45(1) | 42(1) | 34(1) | -3(1) | -2(1) | 4(1) |

Table 5. Hydrogen coordinates ( x $10^4$) and isotropic displacement parameters ($Å^2$x $10^3$) for X11176_t5.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(2) | -2830 | 8402 | 6236 | 35 |
| H(3) | -2936 | 7889 | 5190 | 33 |
| H(8) | 1365 | 5813 | 4415 | 33 |
| H(11) | -1207 | 8373 | 4139 | 35 |
| H(12A) | -459 | 6274 | 3329 | 46 |
| H(12B) | -1183 | 7846 | 3131 | 46 |
| H(12C) | -1623 | 7407 | 3186 | 46 |
| H(12D) | 443 | 6279 | 3325 | 46 |
| H(13A) | 2431 | 7668 | 2693 | 47 |
| H(13B) | 3517 | 6721 | 3253 | 47 |
| H(14A) | 4919 | 9004 | 3487 | 46 |
| H(14B) | 2356 | 9677 | 3331 | 46 |
| H(13C) | 2304 | 7863 | 2666 | 50 |
| H(13D) | 1103 | 9200 | 2980 | 50 |
| H(14C) | 4819 | 9139 | 3445 | 50 |
| H(14D) | 4720 | 7461 | 3527 | 50 |
| H(15A) | 2268 | 9396 | 4386 | 39 |
| H(15B) | 3687 | 7938 | 4369 | 39 |
| H(15C) | 3445 | 8129 | 4499 | 39 |
| H(15D) | 2194 | 9527 | 4230 | 39 |
| H(5) | 3333 | 5909 | 5393 | 34 |
| H(6) | 3486 | 6434 | 6435 | 37 |
| H(7A) | -1648 | 9261 | 7187 | 61 |
| H(7B) | -1105 | 8348 | 7795 | 61 |
| H(7C) | -2827 | 7750 | 7253 | 61 |
| H(22) | 2078 | 12 | -1128 | 35 |
| H(23) | 2012 | 423 | -71 | 34 |
| H(28) | 6422 | 2375 | 827 | 35 |
| H(31) | 3956 | -233 | 1000 | 37 |
| H(32A) | 6045 | 1632 | 1903 | 49 |
| H(32B) | 3684 | 713 | 1956 | 49 |
| H(32C) | 4116 | 138 | 2010 | 49 |
| H(32D) | 4985 | 1714 | 1885 | 49 |
| H(33A) | 5772 | -1307 | 2134 | 47 |
| H(33B) | 7358 | -176 | 2517 | 47 |
| H(34A) | 9313 | -1617 | 1694 | 73 |
| H(34B) | 9995 | 11 | 1760 | 73 |
| H(33C) | 8933 | 1109 | 1999 | 59 |
| H(33D) | 7851 | 38 | 2486 | 59 |
| H(34C) | 7703 | -1766 | 1768 | 73 |
| H(34D) | 10238 | -1048 | 1670 | 73 |
| H(35A) | 7340 | -1269 | 792 | 38 |
| H(35B) | 8716 | 203 | 774 | 38 |
| H(35C) | 7372 | -1177 | 729 | 38 |
| H(35D) | 8810 | 259 | 838 | 38 |
| H(25) | 8284 | 2407 | -107 | 35 |
| H(26) | 8415 | 1969 | -1153 | 38 |
| H(27A) | 3419 | -851 | -2052 | 61 |
| H(27B) | 3907 | 96 | -2642 | 61 |
| H(27C) | 2118 | 626 | -2142 | 61 |




**(2*S*,3*S*)-2-Cyclopentyl-3-(6-methoxynaphthalen-2-yl)butanenitrile.** (*S*)-2-Cyclopentyl-3-(6-methoxynaphthalen-2-yl)but-3-enenitrile (32 mg, 0.11 mmol; Table 4, entry 5; from a reaction using (*R*,*R*)-**L**) and Pd/C (3.2 mg; 10 wt%; Aldrich) were added to a 4-mL vial equipped with a magnetic stir bar. The vial was sealed with a PTFE-lined septum cap, and it was placed under vacuum. The vial was filled with hydrogen, and this evacuation-refill cycle was repeated three times. EtOH (1.1 mL) was added to the vial, and the mixture was stirred overnight under hydrogen. Next, the mixture was filtered through a pad of celite (eluted with $Et_2O$), and the solution was concentrated. The major diastereomer (3:1 dr) was isolated by preparative HPLC on a Daicel CHIRALPAK IC column (250 mm x 250 mm, 5 μm; 1% *i*-PrOH/hexanes, 20 mL/min) with $t_r$ = 26.4 min (minor), 29.3 min (major). White solid. 22 mg (68%, 91% ee).

The ee was determined by HPLC analysis on a CHIRALPAK AD-H column (3% *i*-PrOH/hexanes, 1.0 mL/min) with $t_r$ = 24.1 min (minor), 27.8 min (major).

$^1$H NMR (500 MHz, $CDCl_3$) δ 7.73–7.68 (m, 3H), 7.46 (dd, 1H, *J* = 1.9, 8.4 Hz), 7.16 (dd, 1H, *J* = 2.6, 8.7 Hz), 7.13 (d, 1H, *J* = 2.5 Hz), 3.92 (s, 3H), 3.12 (pentet, 1H, *J* = 7.0 Hz), 2.76 (dd, 1H, *J* = 6.3 Hz, 7.9 Hz), 1.94–1.84 (m, 2H), 1.83–1.77 (m, 1H), 1.72–1.62 (m, 2H), 1.56–1.44 (m, 2H), 1.52 (d, 3H, *J* = 7.2 Hz), 1.40–1.29 (m, 2H).

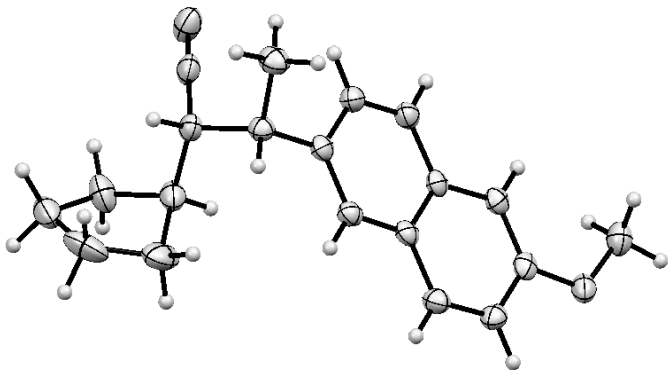
$^{13}$C NMR (126 MHz, $CDCl_3$) δ 157.7, 137.4, 133.9, 129.5, 129.0, 127.3, 126.5, 126.4, 120.9, 119.1, 105.7, 55.5, 45.2, 40.4, 39.8, 31.4, 30.8, 25.3, 25.1, 20.7.

FT-IR (neat) 2961, 2933, 2869, 2235, 1631, 1606, 1506, 1484, 1463, 1382, 1266, 1241, 1220, 1197, 1184, 1164, 1029, 891, 858, 818 $cm^{-1}$.

MS (EI) *m*/*z* ($M^+$) calcd for $C_{20}H_{23}NO$: 293, found: 293.

$[\alpha]^{24}_D$ = –16.0° (c = 0.98, $CHCl_3$).

A crystal suitable for X-ray crystallography was grown by vapor diffusion with $Et_2O$ and pentane.



Stereochemistry at C1: S; stereochemistry at C2: S

Eight independent molecules, refined using residues. Two molecules (number seven and eight) show disorder in the five-membered ring. Pseudo-merohedral twin. Twin-law 0 0 1 0 -1 0 1 0 0. Twin ratio: 0.3500(7). Flack-x has high standard uncertainty; Hooft test gives more reliable results. See Platon output.

Table 1. Crystal data and structure refinement for X12022.

| | | |
|---|---|---|
| Identification code | x12022 | |
| Empirical formula | C20 H23 N O | |
| Formula weight | 293.39 | |
| Temperature | 100(2) K | |
| Wavelength | 1.54178 Å | |
| Crystal system | Monoclinic | |
| Space group | P2(1) | |
| Unit cell dimensions | a = 14.4823(5) Å | α= 90°. |
| | b = 32.2596(10) Å | β= 104.760(2)°. |
| | c = 14.5199(5) Å | γ = 90°. |
| Volume | 6559.7(4) $Å^3$ | |
| Z | 16 | |
| Density (calculated) | 1.188 $Mg/m^3$ | |
| Absorption coefficient | 0.559 $mm^{-1}$ | |
| F(000) | 2528 | |
| Crystal size | 0.30 x 0.11 x 0.08 $mm^3$ | |
| Theta range for data collection | 1.37 to 68.22°. | |
| Index ranges | -17<=h<=15, -38<=k<=38, -17<=l<=17 | |
| Reflections collected | 196268 | |
| Independent reflections | 23690 [R(int) = 0.0369] | |
| Completeness to theta = 68.22° | 99.5 % | |
| Absorption correction | Semi-empirical from equivalents | |
| Max. and min. transmission | 0.9567 and 0.8503 | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 23690 / 1932 / 1676 | |
| Goodness-of-fit on $F^2$ | 1.065 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0405, wR2 = 0.1032 | |
| R indices (all data) | R1 = 0.0421, wR2 = 0.1066 | |
| Absolute structure parameter | 0.06(13) | |
| Largest diff. peak and hole | 0.300 and -0.159 $e.Å^{-3}$ | |

Table 2. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($Å^2$x $10^3$) for X12022. U(eq) is defined as one third of the trace of the orthogonalized $U^{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| O11 | 4117(1) | -79(1) | 3269(1) | 33(1) |
| N11 | 4239(2) | 2770(1) | 3836(2) | 39(1) |
| C21 | 3141(2) | 2128(1) | 5186(2) | 24(1) |
| C31 | 3901(2) | 2261(1) | 6076(2) | 29(1) |
| C41 | 3631(2) | 2659(1) | 4144(2) | 29(1) |
| C11 | 2831(2) | 2506(1) | 4508(2) | 25(1) |
| C111 | 1971(2) | 2421(1) | 3653(2) | 28(1) |
| C121 | 1079(2) | 2278(1) | 3934(2) | 36(1) |
| C131 | 238(2) | 2402(1) | 3107(2) | 48(1) |
| C141 | 647(2) | 2674(1) | 2442(2) | 42(1) |
| C151 | 1620(2) | 2809(1) | 3057(2) | 35(1) |
| C211 | 3445(2) | 1753(1) | 4707(2) | 23(1) |
| C221 | 2862(2) | 1412(1) | 4473(2) | 24(1) |
| C231 | 3162(2) | 1046(1) | 4091(2) | 25(1) |
| C241 | 2565(2) | 691(1) | 3866(2) | 28(1) |
| C251 | 2896(2) | 335(1) | 3580(2) | 31(1) |
| C261 | 3860(2) | 308(1) | 3499(2) | 27(1) |
| C271 | 4432(2) | 649(1) | 3652(2) | 27(1) |
| C281 | 4098(2) | 1027(1) | 3956(2) | 24(1) |
| C291 | 4678(2) | 1382(1) | 4167(2) | 26(1) |
| C301 | 4370(2) | 1734(1) | 4529(2) | 26(1) |
| C311 | 5085(2) | -140(1) | 3252(2) | 39(1) |
| O12 | 8399(1) | 4941(1) | 4274(1) | 38(1) |
| N12 | 8122(2) | 7771(1) | 3561(2) | 31(1) |
| C22 | 9384(2) | 7146(1) | 2336(2) | 28(1) |
| C32 | 8683(2) | 7283(1) | 1409(2) | 32(1) |
| C42 | 8772(2) | 7662(1) | 3326(2) | 27(1) |
| C12 | 9630(2) | 7514(1) | 3045(2) | 28(1) |
| C112 | 10429(2) | 7422(1) | 3942(2) | 29(1) |
| C122 | 11389(2) | 7322(1) | 3726(2) | 38(1) |
| C132 | 12121(2) | 7372(1) | 4673(2) | 44(1) |
| C142 | 11678(2) | 7673(1) | 5257(2) | 40(1) |
| C152 | 10690(2) | 7790(1) | 4627(2) | 36(1) |
| C212 | 9050(2) | 6763(1) | 2763(2) | 26(1) |
| C222 | 9651(2) | 6435(1) | 3071(2) | 27(1) |
| C232 | 9347(2) | 6065(1) | 3442(2) | 26(1) |
| C242 | 9968(2) | 5724(1) | 3750(2) | 30(1) |
| C252 | 9634(2) | 5367(1) | 4038(2) | 34(1) |
| C262 | 8653(2) | 5326(1) | 4026(2) | 30(1) |
| C272 | 8043(2) | 5653(1) | 3773(2) | 30(1) |
| C282 | 8374(2) | 6031(1) | 3471(2) | 26(1) |
| C292 | 7772(2) | 6376(1) | 3181(2) | 27(1) |
| C302 | 8085(2) | 6726(1) | 2837(2) | 28(1) |
| C312 | 7417(2) | 4870(1) | 4225(2) | 40(1) |
| O13 | 648(1) | 4934(1) | 1887(1) | 36(1) |
| N13 | 779(2) | 7775(1) | 1196(2) | 40(1) |
| C23 | 2056(2) | 7105(1) | 101(2) | 24(1) |
| C33 | 1412(2) | 7275(1) | -829(2) | 31(1) |
| C43 | 1460(2) | 7639(1) | 1039(2) | 30(1) |
| C13 | 2333(2) | 7456(1) | 859(2) | 24(1) |
| C113 | 3005(2) | 7319(1) | 1810(2) | 29(1) |
| C123 | 3964(2) | 7165(1) | 1696(2) | 41(1) |

| | | | | |
|---|---|---|---|---|
| C133 | 4520(2) | 7558(1) | 1627(2) | 46(1) |
| C143 | 4189(2) | 7873(1) | 2262(2) | 39(1) |
| C153 | 3308(2) | 7684(1) | 2506(2) | 37(1) |
| C213 | 1639(2) | 6729(1) | 464(2) | 24(1) |
| C223 | 2174(2) | 6383(1) | 747(2) | 27(1) |
| C233 | 1798(2) | 6018(1) | 1071(2) | 25(1) |
| C243 | 2345(2) | 5656(1) | 1367(2) | 29(1) |
| C253 | 1951(2) | 5311(1) | 1645(2) | 32(1) |
| C263 | 964(2) | 5306(1) | 1626(2) | 29(1) |
| C273 | 410(2) | 5651(1) | 1369(2) | 29(1) |
| C283 | 812(2) | 6016(1) | 1086(2) | 26(1) |
| C293 | 269(2) | 6376(1) | 799(2) | 29(1) |
| C303 | 659(2) | 6722(1) | 498(2) | 27(1) |
| C313 | -347(2) | 4900(1) | 1820(2) | 42(1) |
| O14 | 5487(1) | 6351(1) | 1998(1) | 32(1) |
| N14 | 5042(2) | 3488(1) | 2051(2) | 42(1) |
| C24 | 3910(2) | 4129(1) | 3349(2) | 26(1) |
| C34 | 2984(2) | 3954(1) | 2711(2) | 36(1) |
| C44 | 4892(2) | 3617(1) | 2728(2) | 33(1) |
| C14 | 4702(2) | 3789(1) | 3602(2) | 28(1) |
| C114 | 5650(2) | 3936(1) | 4276(2) | 35(1) |
| C124 | 5543(2) | 4042(1) | 5266(2) | 52(1) |
| C134 | 5598(2) | 3626(1) | 5763(2) | 57(1) |
| C144 | 6240(2) | 3353(1) | 5341(2) | 47(1) |
| C154 | 6413(2) | 3596(1) | 4515(2) | 45(1) |
| C214 | 4239(2) | 4518(1) | 2931(2) | 26(1) |
| C224 | 4505(2) | 4867(1) | 3475(2) | 26(1) |
| C234 | 4792(2) | 5234(1) | 3106(2) | 26(1) |
| C244 | 5053(2) | 5598(1) | 3670(2) | 30(1) |
| C254 | 5284(2) | 5954(1) | 3275(2) | 29(1) |
| C264 | 5274(2) | 5971(1) | 2302(2) | 27(1) |
| C274 | 5068(2) | 5625(1) | 1742(2) | 27(1) |
| C284 | 4821(2) | 5249(1) | 2142(2) | 25(1) |
| C294 | 4563(2) | 4884(1) | 1586(2) | 28(1) |
| C304 | 4279(2) | 4536(1) | 1969(2) | 29(1) |
| C314 | 5449(2) | 6392(1) | 1016(2) | 36(1) |
| O15 | 8020(1) | 6326(1) | -607(1) | 35(1) |
| N15 | 7392(2) | 3495(1) | -562(1) | 32(1) |
| C25 | 6164(2) | 4143(1) | 659(2) | 26(1) |
| C35 | 5227(2) | 3997(1) | -11(2) | 32(1) |
| C45 | 7154(2) | 3614(1) | 80(2) | 27(1) |
| C15 | 6872(2) | 3774(1) | 918(2) | 26(1) |
| C115 | 7782(2) | 3869(1) | 1699(2) | 29(1) |
| C125 | 7572(2) | 4004(1) | 2640(2) | 41(1) |
| C135 | 8492(2) | 3918(1) | 3382(2) | 54(1) |
| C145 | 8997(2) | 3576(1) | 2999(2) | 62(1) |
| C155 | 8438(2) | 3491(1) | 1987(2) | 39(1) |
| C215 | 6581(2) | 4520(1) | 283(2) | 25(1) |
| C225 | 6840(2) | 4870(1) | 832(2) | 27(1) |
| C235 | 7187(2) | 5228(1) | 482(2) | 26(1) |
| C245 | 7435(2) | 5594(1) | 1033(2) | 30(1) |
| C255 | 7714(2) | 5945(1) | 650(2) | 31(1) |
| C265 | 7765(2) | 5951(1) | -302(2) | 29(1) |
| C275 | 7570(2) | 5601(1) | -857(2) | 26(1) |
| C285 | 7273(2) | 5233(1) | -463(2) | 25(1) |
| C295 | 7021(2) | 4870(1) | -1024(2) | 26(1) |
| C305 | 6688(2) | 4527(1) | -656(2) | 26(1) |

| | | | | |
|---|---|---|---|---|
| C315 | 7967(2) | 6364(1) | -1592(2) | 38(1) |
| O16 | -584(1) | 11364(1) | 2997(1) | 39(1) |
| N16 | -222(2) | 8558(1) | 2784(2) | 41(1) |
| C26 | 1306(2) | 9170(1) | 1840(2) | 26(1) |
| C36 | 2173(2) | 8999(1) | 2580(2) | 36(1) |
| C46 | 140(2) | 8671(1) | 2219(2) | 32(1) |
| C16 | 562(2) | 8821(1) | 1463(2) | 27(1) |
| C116 | -247(2) | 8952(1) | 601(2) | 34(1) |
| C126 | 141(2) | 9072(1) | -253(2) | 43(1) |
| C136 | -602(2) | 8917(1) | -1114(2) | 52(1) |
| C146 | -898(3) | 8509(1) | -758(2) | 61(1) |
| C156 | -981(2) | 8604(1) | 218(2) | 40(1) |
| C216 | 876(2) | 9548(1) | 2200(2) | 26(1) |
| C226 | 630(2) | 9897(1) | 1637(2) | 26(1) |
| C236 | 264(2) | 10257(1) | 1963(2) | 26(1) |
| C246 | 12(2) | 10619(1) | 1389(2) | 30(1) |
| C256 | -274(2) | 10971(1) | 1754(2) | 33(1) |
| C266 | -340(2) | 10983(1) | 2707(2) | 32(1) |
| C276 | -156(2) | 10638(1) | 3271(2) | 29(1) |
| C286 | 147(2) | 10266(1) | 2899(2) | 26(1) |
| C296 | 370(2) | 9904(1) | 3460(2) | 28(1) |
| C306 | 720(2) | 9558(1) | 3115(2) | 28(1) |
| C316 | -547(2) | 11405(1) | 3976(2) | 44(1) |
| O17 | 6944(1) | 11381(1) | 5395(1) | 34(1) |
| N17 | 7070(2) | 8562(1) | 5122(2) | 41(1) |
| C27 | 8557(2) | 9156(1) | 4110(2) | 25(1) |
| C37 | 9406(2) | 8957(1) | 4819(2) | 36(1) |
| C47 | 7375(2) | 8677(1) | 4528(2) | 31(1) |
| C17 | 7754(2) | 8833(1) | 3742(2) | 24(1) |
| C117 | 6927(2) | 8996(1) | 2932(2) | 33(1) |
| C127 | 7264(4) | 9005(2) | 1977(4) | 36(1) |
| C137 | 7091(4) | 8566(2) | 1579(3) | 32(1) |
| C147 | 6326(4) | 8380(2) | 1989(4) | 27(1) |
| C157 | 6049(3) | 8719(2) | 2613(3) | 26(1) |
| C12A7 | 7109(11) | 9135(4) | 2057(8) | 40(2) |
| C13A7 | 7151(9) | 8739(5) | 1540(9) | 41(2) |
| C14A7 | 6565(12) | 8420(4) | 1855(11) | 39(2) |
| C15A7 | 6251(9) | 8605(4) | 2698(9) | 36(2) |
| C217 | 8206(2) | 9544(1) | 4511(2) | 24(1) |
| C227 | 7966(2) | 9895(1) | 3959(2) | 25(1) |
| C237 | 7658(2) | 10262(1) | 4316(2) | 24(1) |
| C247 | 7419(2) | 10623(1) | 3752(2) | 29(1) |
| C257 | 7173(2) | 10980(1) | 4134(2) | 30(1) |
| C267 | 7150(2) | 10999(1) | 5095(2) | 28(1) |
| C277 | 7330(2) | 10652(1) | 5659(2) | 27(1) |
| C287 | 7592(2) | 10274(1) | 5272(2) | 24(1) |
| C297 | 7815(2) | 9910(1) | 5821(2) | 27(1) |
| C307 | 8110(2) | 9560(1) | 5454(2) | 28(1) |
| C317 | 6952(2) | 11421(1) | 6372(2) | 39(1) |
| O18 | 6802(1) | -82(1) | 645(1) | 31(1) |
| N18 | 6507(2) | 2740(1) | 1046(2) | 44(1) |
| C28 | 5428(2) | 2100(1) | 2395(2) | 28(1) |
| C38 | 6140(2) | 2273(1) | 3280(2) | 39(1) |
| C48 | 5888(2) | 2618(1) | 1327(2) | 31(1) |
| C18 | 5078(2) | 2449(1) | 1644(2) | 26(1) |
| C118 | 4288(2) | 2316(1) | 762(2) | 37(1) |
| C128 | 3421(5) | 2123(2) | 903(6) | 40(2) |

| | | | | |
|---|---|---|---|---|
| C138 | 2846(5) | 2478(3) | 1107(5) | 43(2) |
| C148 | 3092(6) | 2850(2) | 556(7) | 34(1) |
| C158 | 3904(8) | 2708(3) | 162(7) | 37(2) |
| C12A8 | 3339(7) | 2241(3) | 1132(8) | 46(2) |
| C13A8 | 2886(6) | 2660(4) | 1100(7) | 41(2) |
| C14A8 | 3225(10) | 2909(4) | 361(10) | 42(2) |
| C15A8 | 3917(9) | 2639(4) | 18(9) | 39(2) |
| C218 | 5828(2) | 1726(1) | 1996(2) | 25(1) |
| C228 | 5304(2) | 1370(1) | 1781(2) | 26(1) |
| C238 | 5672(2) | 1008(1) | 1446(2) | 25(1) |
| C248 | 5135(2) | 638(1) | 1228(2) | 30(1) |
| C258 | 5528(2) | 288(1) | 956(2) | 34(1) |
| C268 | 6494(2) | 293(1) | 891(2) | 27(1) |
| C278 | 7020(2) | 648(1) | 1061(2) | 27(1) |
| C288 | 6623(2) | 1013(1) | 1342(2) | 25(1) |
| C298 | 7154(2) | 1383(1) | 1562(2) | 27(1) |
| C308 | 6778(2) | 1728(1) | 1880(2) | 27(1) |
| C318 | 7787(2) | -116(1) | 655(2) | 38(1) |

Table 3. Bond lengths [Å] and angles [°] for X12022.

| | |
|---|---|
| O11-C261 | 1.369(3) |
| O11-C311 | 1.422(3) |
| N11-C41 | 1.142(3) |
| C21-C211 | 1.516(3) |
| C21-C31 | 1.530(3) |
| C21-C11 | 1.559(3) |
| C21-H21 | 1.0000 |
| C31-H3A1 | 0.9800 |
| C31-H3B1 | 0.9800 |
| C31-H3C1 | 0.9800 |
| C41-C11 | 1.475(3) |
| C11-C111 | 1.543(3) |
| C11-H11 | 1.0000 |
| C111-C121 | 1.522(3) |
| C111-C151 | 1.531(3) |
| C111-H111 | 1.0000 |
| C121-C131 | 1.529(3) |
| C121-H12A1 | 0.9900 |
| C121-H12B1 | 0.9900 |
| C131-C141 | 1.531(4) |
| C131-H13A1 | 0.9900 |
| C131-H13B1 | 0.9900 |
| C141-C151 | 1.526(3) |
| C141-H14A1 | 0.9900 |
| C141-H14B1 | 0.9900 |
| C151-H15A1 | 0.9900 |
| C151-H15B1 | 0.9900 |
| C211-C221 | 1.376(3) |
| C211-C301 | 1.430(3) |
| C221-C231 | 1.420(3) |
| C221-H221 | 0.9500 |
| C231-C281 | 1.418(3) |
| C231-C241 | 1.421(3) |
| C241-C251 | 1.351(3) |
| C241-H241 | 0.9500 |
| C251-C261 | 1.434(3) |
| C251-H251 | 0.9500 |
| C261-C271 | 1.360(3) |
| C271-C281 | 1.422(3) |
| C271-H271 | 0.9500 |
| C281-C291 | 1.408(3) |
| C291-C301 | 1.371(3) |
| C291-H291 | 0.9500 |
| C301-H301 | 0.9500 |
| C311-H31A1 | 0.9800 |
| C311-H31B1 | 0.9800 |
| C311-H31C1 | 0.9800 |
| O12-C262 | 1.368(3) |
| O12-C312 | 1.424(3) |
| N12-C42 | 1.135(3) |
| C22-C212 | 1.515(3) |
| C22-C32 | 1.531(3) |
| C22-C12 | 1.553(3) |
| C22-H22 | 1.0000 |
| C32-H3A2 | 0.9800 |

| | |
|---|---|
| C32-H3B2 | 0.9800 |
| C32-H3C2 | 0.9800 |
| C42-C12 | 1.482(3) |
| C12-C112 | 1.535(3) |
| C12-H12 | 1.0000 |
| C112-C152 | 1.533(3) |
| C112-C122 | 1.536(3) |
| C112-H112 | 1.0000 |
| C122-C132 | 1.517(3) |
| C122-H12A2 | 0.9900 |
| C122-H12B2 | 0.9900 |
| C132-C142 | 1.535(4) |
| C132-H13A2 | 0.9900 |
| C132-H13B2 | 0.9900 |
| C142-C152 | 1.536(3) |
| C142-H14A2 | 0.9900 |
| C142-H14B2 | 0.9900 |
| C152-H15A2 | 0.9900 |
| C152-H15B2 | 0.9900 |
| C212-C222 | 1.372(3) |
| C212-C302 | 1.433(3) |
| C222-C232 | 1.423(3) |
| C222-H222 | 0.9500 |
| C232-C242 | 1.419(3) |
| C232-C282 | 1.424(3) |
| C242-C252 | 1.357(3) |
| C242-H242 | 0.9500 |
| C252-C262 | 1.423(4) |
| C252-H252 | 0.9500 |
| C262-C272 | 1.364(3) |
| C272-C282 | 1.420(3) |
| C272-H272 | 0.9500 |
| C282-C292 | 1.410(3) |
| C292-C302 | 1.360(3) |
| C292-H292 | 0.9500 |
| C302-H302 | 0.9500 |
| C312-H31A2 | 0.9800 |
| C312-H31B2 | 0.9800 |
| C312-H31C2 | 0.9800 |
| O13-C263 | 1.373(3) |
| O13-C313 | 1.424(3) |
| N13-C43 | 1.153(3) |
| C23-C213 | 1.509(3) |
| C23-C33 | 1.533(3) |
| C23-C13 | 1.559(3) |
| C23-H23 | 1.0000 |
| C33-H3A3 | 0.9800 |
| C33-H3B3 | 0.9800 |
| C33-H3C3 | 0.9800 |
| C43-C13 | 1.478(3) |
| C13-C113 | 1.539(3) |
| C13-H13 | 1.0000 |
| C113-C123 | 1.523(3) |
| C113-C153 | 1.542(3) |
| C113-H113 | 1.0000 |
| C123-C133 | 1.518(4) |
| C123-H12A3 | 0.9900 |

| | |
|---|---|
| C123-H12B3 | 0.9900 |
| C133-C143 | 1.531(4) |
| C133-H13A3 | 0.9900 |
| C133-H13B3 | 0.9900 |
| C143-C153 | 1.535(4) |
| C143-H14A3 | 0.9900 |
| C143-H14B3 | 0.9900 |
| C153-H15A3 | 0.9900 |
| C153-H15B3 | 0.9900 |
| C213-C223 | 1.361(3) |
| C213-C303 | 1.433(3) |
| C223-C233 | 1.426(3) |
| C223-H223 | 0.9500 |
| C233-C243 | 1.416(3) |
| C233-C283 | 1.434(3) |
| C243-C253 | 1.359(3) |
| C243-H243 | 0.9500 |
| C253-C263 | 1.422(4) |
| C253-H253 | 0.9500 |
| C263-C273 | 1.368(4) |
| C273-C283 | 1.419(3) |
| C273-H273 | 0.9500 |
| C283-C293 | 1.406(3) |
| C293-C303 | 1.370(3) |
| C293-H293 | 0.9500 |
| C303-H303 | 0.9500 |
| C313-H31A3 | 0.9800 |
| C313-H31B3 | 0.9800 |
| C313-H31C3 | 0.9800 |
| O14-C264 | 1.364(3) |
| O14-C314 | 1.418(3) |
| N14-C44 | 1.139(3) |
| C24-C214 | 1.521(3) |
| C24-C34 | 1.530(3) |
| C24-C14 | 1.560(3) |
| C24-H24 | 1.0000 |
| C34-H3A4 | 0.9800 |
| C34-H3B4 | 0.9800 |
| C34-H3C4 | 0.9800 |
| C44-C14 | 1.473(3) |
| C14-C114 | 1.544(3) |
| C14-H14 | 1.0000 |
| C114-C124 | 1.522(4) |
| C114-C154 | 1.534(3) |
| C114-H114 | 1.0000 |
| C124-C134 | 1.514(5) |
| C124-H12A4 | 0.9900 |
| C124-H12B4 | 0.9900 |
| C134-C144 | 1.517(4) |
| C134-H13A4 | 0.9900 |
| C134-H13B4 | 0.9900 |
| C144-C154 | 1.506(4) |
| C144-H14A4 | 0.9900 |
| C144-H14B4 | 0.9900 |
| C154-H15A4 | 0.9900 |
| C154-H15B4 | 0.9900 |
| C214-C224 | 1.375(3) |

| | |
|---|---|
| C214-C304 | 1.415(3) |
| C224-C234 | 1.406(3) |
| C224-H224 | 0.9500 |
| C234-C284 | 1.412(3) |
| C234-C244 | 1.425(3) |
| C244-C254 | 1.362(4) |
| C244-H244 | 0.9500 |
| C254-C264 | 1.411(4) |
| C254-H254 | 0.9500 |
| C264-C274 | 1.368(3) |
| C274-C284 | 1.430(3) |
| C274-H274 | 0.9500 |
| C284-C294 | 1.422(3) |
| C294-C304 | 1.361(3) |
| C294-H294 | 0.9500 |
| C304-H304 | 0.9500 |
| C314-H31A4 | 0.9800 |
| C314-H31B4 | 0.9800 |
| C314-H31C4 | 0.9800 |
| O15-C265 | 1.373(3) |
| O15-C315 | 1.417(3) |
| N15-C45 | 1.138(3) |
| C25-C215 | 1.521(3) |
| C25-C35 | 1.529(3) |
| C25-C15 | 1.551(3) |
| C25-H25 | 1.0000 |
| C35-H3A5 | 0.9800 |
| C35-H3B5 | 0.9800 |
| C35-H3C5 | 0.9800 |
| C45-C15 | 1.473(3) |
| C15-C115 | 1.534(3) |
| C15-H15 | 1.0000 |
| C115-C155 | 1.536(3) |
| C115-C125 | 1.536(3) |
| C115-H115 | 1.0000 |
| C125-C135 | 1.511(3) |
| C125-H12A5 | 0.9900 |
| C125-H12B5 | 0.9900 |
| C135-C145 | 1.505(4) |
| C135-H13A5 | 0.9900 |
| C135-H13B5 | 0.9900 |
| C145-C155 | 1.511(4) |
| C145-H14A5 | 0.9900 |
| C145-H14B5 | 0.9900 |
| C155-H15A5 | 0.9900 |
| C155-H15B5 | 0.9900 |
| C215-C225 | 1.377(3) |
| C215-C305 | 1.411(3) |
| C225-C235 | 1.408(3) |
| C225-H225 | 0.9500 |
| C235-C285 | 1.408(3) |
| C235-C245 | 1.418(3) |
| C245-C255 | 1.367(4) |
| C245-H245 | 0.9500 |
| C255-C265 | 1.404(4) |
| C255-H255 | 0.9500 |
| C265-C275 | 1.372(3) |

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|---|---|
| C275-C285 | 1.430(3) |
| C275-H275 | 0.9500 |
| C285-C295 | 1.422(3) |
| C295-C305 | 1.368(3) |
| C295-H295 | 0.9500 |
| C305-H305 | 0.9500 |
| C315-H31A5 | 0.9800 |
| C315-H31B5 | 0.9800 |
| C315-H31C5 | 0.9800 |
| O16-C266 | 1.374(3) |
| O16-C316 | 1.415(4) |
| N16-C46 | 1.138(3) |
| C26-C216 | 1.521(3) |
| C26-C36 | 1.532(3) |
| C26-C16 | 1.558(3) |
| C26-H26 | 1.0000 |
| C36-H3A6 | 0.9800 |
| C36-H3B6 | 0.9800 |
| C36-H3C6 | 0.9800 |
| C46-C16 | 1.469(3) |
| C16-C116 | 1.540(3) |
| C16-H16 | 1.0000 |
| C116-C126 | 1.534(3) |
| C116-C156 | 1.549(3) |
| C116-H116 | 1.0000 |
| C126-C136 | 1.512(3) |
| C126-H12A6 | 0.9900 |
| C126-H12B6 | 0.9900 |
| C136-C146 | 1.514(4) |
| C136-H13A6 | 0.9900 |
| C136-H13B6 | 0.9900 |
| C146-C156 | 1.486(4) |
| C146-H14A6 | 0.9900 |
| C146-H14B6 | 0.9900 |
| C156-H15A6 | 0.9900 |
| C156-H15B6 | 0.9900 |
| C216-C226 | 1.385(3) |
| C216-C306 | 1.403(3) |
| C226-C236 | 1.407(3) |
| C226-H226 | 0.9500 |
| C236-C286 | 1.412(3) |
| C236-C246 | 1.426(3) |
| C246-C256 | 1.362(4) |
| C246-H246 | 0.9500 |
| C256-C266 | 1.412(4) |
| C256-H256 | 0.9500 |
| C266-C276 | 1.366(4) |
| C276-C286 | 1.431(3) |
| C276-H276 | 0.9500 |
| C286-C296 | 1.414(3) |
| C296-C306 | 1.373(3) |
| C296-H296 | 0.9500 |
| C306-H306 | 0.9500 |
| C316-H31A6 | 0.9800 |
| C316-H31B6 | 0.9800 |
| C316-H31C6 | 0.9800 |
| O17-C267 | 1.364(3) |

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|---|---|
| O17-C317 | 1.422(3) |
| N17-C47 | 1.128(3) |
| C27-C217 | 1.522(3) |
| C27-C37 | 1.529(3) |
| C27-C17 | 1.551(3) |
| C27-H27 | 1.0000 |
| C37-H3A7 | 0.9800 |
| C37-H3B7 | 0.9800 |
| C37-H3C7 | 0.9800 |
| C47-C17 | 1.475(3) |
| C17-C117 | 1.542(3) |
| C17-H17 | 1.0000 |
| C117-C12A7 | 1.432(10) |
| C117-C157 | 1.528(4) |
| C117-C15A7 | 1.579(10) |
| C117-C127 | 1.582(6) |
| C117-H11A7 | 1.0000 |
| C117-H11B7 | 1.0000 |
| C127-C137 | 1.524(5) |
| C127-H12A7 | 0.9900 |
| C127-H12B7 | 0.9900 |
| C137-C147 | 1.510(5) |
| C137-H13A7 | 0.9900 |
| C137-H13B7 | 0.9900 |
| C147-C157 | 1.538(5) |
| C147-H14A7 | 0.9900 |
| C147-H14B7 | 0.9900 |
| C157-H15A7 | 0.9900 |
| C157-H15B7 | 0.9900 |
| C12A7-C13A7 | 1.492(10) |
| C12A7-H12C7 | 0.9900 |
| C12A7-H12D7 | 0.9900 |
| C13A7-C14A7 | 1.478(11) |
| C13A7-H13C7 | 0.9900 |
| C13A7-H13D7 | 0.9900 |
| C14A7-C15A7 | 1.532(11) |
| C14A7-H14C7 | 0.9900 |
| C14A7-H14D7 | 0.9900 |
| C15A7-H15C7 | 0.9900 |
| C15A7-H15D7 | 0.9900 |
| C217-C227 | 1.379(3) |
| C217-C307 | 1.411(3) |
| C227-C237 | 1.412(3) |
| C227-H227 | 0.9500 |
| C237-C247 | 1.415(3) |
| C237-C287 | 1.416(3) |
| C247-C257 | 1.363(3) |
| C247-H247 | 0.9500 |
| C257-C267 | 1.406(4) |
| C257-H257 | 0.9500 |
| C267-C277 | 1.372(3) |
| C277-C287 | 1.432(3) |
| C277-H277 | 0.9500 |
| C287-C297 | 1.410(3) |
| C297-C307 | 1.364(3) |
| C297-H297 | 0.9500 |
| C307-H307 | 0.9500 |

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|---|---|
| C317-H31A7 | 0.9800 |
| C317-H31B7 | 0.9800 |
| C317-H31C7 | 0.9800 |
| O18-C268 | 1.366(3) |
| O18-C318 | 1.428(3) |
| N18-C48 | 1.144(3) |
| C28-C218 | 1.517(3) |
| C28-C38 | 1.533(3) |
| C28-C18 | 1.559(3) |
| C28-H28 | 1.0000 |
| C38-H3A8 | 0.9800 |
| C38-H3B8 | 0.9800 |
| C38-H3C8 | 0.9800 |
| C48-C18 | 1.472(3) |
| C18-C118 | 1.545(3) |
| C18-H18 | 1.0000 |
| C118-C128 | 1.462(7) |
| C118-C15A8 | 1.500(9) |
| C118-C158 | 1.554(7) |
| C118-C12A8 | 1.615(9) |
| C118-H11A8 | 1.0000 |
| C118-H11B8 | 1.0000 |
| C128-C138 | 1.491(7) |
| C128-H12A8 | 0.9900 |
| C128-H12B8 | 0.9900 |
| C138-C148 | 1.532(6) |
| C138-H13A8 | 0.9900 |
| C138-H13B8 | 0.9900 |
| C148-C158 | 1.504(8) |
| C148-H14A8 | 0.9900 |
| C148-H14B8 | 0.9900 |
| C158-H15A8 | 0.9900 |
| C158-H15B8 | 0.9900 |
| C12A8-C13A8 | 1.499(9) |
| C12A8-H12C8 | 0.9900 |
| C12A8-H12D8 | 0.9900 |
| C13A8-C14A8 | 1.519(9) |
| C13A8-H13C8 | 0.9900 |
| C13A8-H13D8 | 0.9900 |
| C14A8-C15A8 | 1.504(10) |
| C14A8-H14C8 | 0.9900 |
| C14A8-H14D8 | 0.9900 |
| C15A8-H15C8 | 0.9900 |
| C15A8-H15D8 | 0.9900 |
| C218-C228 | 1.368(3) |
| C218-C308 | 1.427(3) |
| C228-C238 | 1.419(3) |
| C228-H228 | 0.9500 |
| C238-C248 | 1.417(3) |
| C238-C288 | 1.423(3) |
| C248-C258 | 1.366(4) |
| C248-H248 | 0.9500 |
| C258-C268 | 1.426(4) |
| C258-H258 | 0.9500 |
| C268-C278 | 1.363(3) |
| C278-C288 | 1.417(3) |
| C278-H278 | 0.9500 |

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|---|---|
| C288-C298 | 1.411(3) |
| C298-C308 | 1.371(3) |
| C298-H298 | 0.9500 |
| C308-H308 | 0.9500 |
| C318-H31A8 | 0.9800 |
| C318-H31B8 | 0.9800 |
| C318-H31C8 | 0.9800 |

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|---|---|
| C261-O11-C311 | 117.48(19) |
| C211-C21-C31 | 112.36(18) |
| C211-C21-C11 | 113.87(17) |
| C31-C21-C11 | 110.12(17) |
| C211-C21-H21 | 106.7 |
| C31-C21-H21 | 106.7 |
| C11-C21-H21 | 106.7 |
| C21-C31-H3A1 | 109.5 |
| C21-C31-H3B1 | 109.5 |
| H3A1-C31-H3B1 | 109.5 |
| C21-C31-H3C1 | 109.5 |
| H3A1-C31-H3C1 | 109.5 |
| H3B1-C31-H3C1 | 109.5 |
| N11-C41-C11 | 177.8(3) |
| C41-C11-C111 | 108.50(19) |
| C41-C11-C21 | 111.21(17) |
| C111-C11-C21 | 114.37(17) |
| C41-C11-H11 | 107.5 |
| C111-C11-H11 | 107.5 |
| C21-C11-H11 | 107.5 |
| C121-C111-C151 | 102.53(19) |
| C121-C111-C11 | 113.87(19) |
| C151-C111-C11 | 113.17(18) |
| C121-C111-H111 | 109.0 |
| C151-C111-H111 | 109.0 |
| C11-C111-H111 | 109.0 |
| C111-C121-C131 | 105.7(2) |
| C111-C121-H12A1 | 110.6 |
| C131-C121-H12A1 | 110.6 |
| C111-C121-H12B1 | 110.6 |
| C131-C121-H12B1 | 110.6 |
| H12A1-C121-H12B1 | 108.7 |
| C121-C131-C141 | 106.7(2) |
| C121-C131-H13A1 | 110.4 |
| C141-C131-H13A1 | 110.4 |
| C121-C131-H13B1 | 110.4 |
| C141-C131-H13B1 | 110.4 |
| H13A1-C131-H13B1 | 108.6 |
| C151-C141-C131 | 103.98(19) |
| C151-C141-H14A1 | 111.0 |
| C131-C141-H14A1 | 111.0 |
| C151-C141-H14B1 | 111.0 |
| C131-C141-H14B1 | 111.0 |
| H14A1-C141-H14B1 | 109.0 |
| C141-C151-C111 | 102.87(18) |
| C141-C151-H15A1 | 111.2 |
| C111-C151-H15A1 | 111.2 |
| C141-C151-H15B1 | 111.2 |
| C111-C151-H15B1 | 111.2 |

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| H15A1-C151-H15B1 | 109.1 |
| C221-C211-C301 | 117.7(2) |
| C221-C211-C21 | 121.3(2) |
| C301-C211-C21 | 120.89(19) |
| C211-C221-C231 | 122.1(2) |
| C211-C221-H221 | 118.9 |
| C231-C221-H221 | 118.9 |
| C281-C231-C221 | 119.2(2) |
| C281-C231-C241 | 118.7(2) |
| C221-C231-C241 | 122.0(2) |
| C251-C241-C231 | 120.8(2) |
| C251-C241-H241 | 119.6 |
| C231-C241-H241 | 119.6 |
| C241-C251-C261 | 120.4(2) |
| C241-C251-H251 | 119.8 |
| C261-C251-H251 | 119.8 |
| C271-C261-O11 | 125.9(2) |
| C271-C261-C251 | 120.2(2) |
| O11-C261-C251 | 113.9(2) |
| C261-C271-C281 | 120.1(2) |
| C261-C271-H271 | 119.9 |
| C281-C271-H271 | 119.9 |
| C291-C281-C231 | 118.2(2) |
| C291-C281-C271 | 122.2(2) |
| C231-C281-C271 | 119.5(2) |
| C301-C291-C281 | 121.5(2) |
| C301-C291-H291 | 119.2 |
| C281-C291-H291 | 119.2 |
| C291-C301-C211 | 121.1(2) |
| C291-C301-H301 | 119.5 |
| C211-C301-H301 | 119.5 |
| O11-C311-H31A1 | 109.5 |
| O11-C311-H31B1 | 109.5 |
| H31A1-C311-H31B1 | 109.5 |
| O11-C311-H31C1 | 109.5 |
| H31A1-C311-H31C1 | 109.5 |
| H31B1-C311-H31C1 | 109.5 |
| C262-O12-C312 | 117.6(2) |
| C212-C22-C32 | 112.66(19) |
| C212-C22-C12 | 113.34(18) |
| C32-C22-C12 | 110.51(18) |
| C212-C22-H22 | 106.6 |
| C32-C22-H22 | 106.6 |
| C12-C22-H22 | 106.6 |
| C22-C32-H3A2 | 109.5 |
| C22-C32-H3B2 | 109.5 |
| H3A2-C32-H3B2 | 109.5 |
| C22-C32-H3C2 | 109.5 |
| H3A2-C32-H3C2 | 109.5 |
| H3B2-C32-H3C2 | 109.5 |
| N12-C42-C12 | 178.4(2) |
| C42-C12-C112 | 109.42(19) |
| C42-C12-C22 | 111.21(18) |
| C112-C12-C22 | 114.26(18) |
| C42-C12-H12 | 107.2 |
| C112-C12-H12 | 107.2 |
| C22-C12-H12 | 107.2 |

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| C152-C112-C12 | 114.13(18) |
| C152-C112-C122 | 102.19(19) |
| C12-C112-C122 | 113.2(2) |
| C152-C112-H112 | 109.0 |
| C12-C112-H112 | 109.0 |
| C122-C112-H112 | 109.0 |
| C132-C122-C112 | 104.6(2) |
| C132-C122-H12A2 | 110.8 |
| C112-C122-H12A2 | 110.8 |
| C132-C122-H12B2 | 110.8 |
| C112-C122-H12B2 | 110.8 |
| H12A2-C122-H12B2 | 108.9 |
| C122-C132-C142 | 105.9(2) |
| C122-C132-H13A2 | 110.5 |
| C142-C132-H13A2 | 110.5 |
| C122-C132-H13B2 | 110.5 |
| C142-C132-H13B2 | 110.5 |
| H13A2-C132-H13B2 | 108.7 |
| C132-C142-C152 | 106.53(19) |
| C132-C142-H14A2 | 110.4 |
| C152-C142-H14A2 | 110.4 |
| C132-C142-H14B2 | 110.4 |
| C152-C142-H14B2 | 110.4 |
| H14A2-C142-H14B2 | 108.6 |
| C112-C152-C142 | 103.51(19) |
| C112-C152-H15A2 | 111.1 |
| C142-C152-H15A2 | 111.1 |
| C112-C152-H15B2 | 111.1 |
| C142-C152-H15B2 | 111.1 |
| H15A2-C152-H15B2 | 109.0 |
| C222-C212-C302 | 117.5(2) |
| C222-C212-C22 | 121.3(2) |
| C302-C212-C22 | 121.2(2) |
| C212-C222-C232 | 122.5(2) |
| C212-C222-H222 | 118.8 |
| C232-C222-H222 | 118.8 |
| C242-C232-C222 | 122.4(2) |
| C242-C232-C282 | 118.8(2) |
| C222-C232-C282 | 118.8(2) |
| C252-C242-C232 | 120.6(2) |
| C252-C242-H242 | 119.7 |
| C232-C242-H242 | 119.7 |
| C242-C252-C262 | 120.6(2) |
| C242-C252-H252 | 119.7 |
| C262-C252-H252 | 119.7 |
| C272-C262-O12 | 125.0(2) |
| C272-C262-C252 | 120.4(2) |
| O12-C262-C252 | 114.6(2) |
| C262-C272-C282 | 120.1(2) |
| C262-C272-H272 | 120.0 |
| C282-C272-H272 | 120.0 |
| C292-C282-C272 | 122.6(2) |
| C292-C282-C232 | 118.1(2) |
| C272-C282-C232 | 119.4(2) |
| C302-C292-C282 | 121.8(2) |
| C302-C292-H292 | 119.1 |
| C282-C292-H292 | 119.1 |

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|---|---|
| C292-C302-C212 | 121.2(2) |
| C292-C302-H302 | 119.4 |
| C212-C302-H302 | 119.4 |
| O12-C312-H31A2 | 109.5 |
| O12-C312-H31B2 | 109.5 |
| H31A2-C312-H31B2 | 109.5 |
| O12-C312-H31C2 | 109.5 |
| H31A2-C312-H31C2 | 109.5 |
| H31B2-C312-H31C2 | 109.5 |
| C263-O13-C313 | 116.8(2) |
| C213-C23-C33 | 112.84(18) |
| C213-C23-C13 | 113.02(18) |
| C33-C23-C13 | 110.54(18) |
| C213-C23-H23 | 106.7 |
| C33-C23-H23 | 106.7 |
| C13-C23-H23 | 106.7 |
| C23-C33-H3A3 | 109.5 |
| C23-C33-H3B3 | 109.5 |
| H3A3-C33-H3B3 | 109.5 |
| C23-C33-H3C3 | 109.5 |
| H3A3-C33-H3C3 | 109.5 |
| H3B3-C33-H3C3 | 109.5 |
| N13-C43-C13 | 178.4(3) |
| C43-C13-C113 | 109.27(19) |
| C43-C13-C23 | 109.67(18) |
| C113-C13-C23 | 114.49(18) |
| C43-C13-H13 | 107.7 |
| C113-C13-H13 | 107.7 |
| C23-C13-H13 | 107.7 |
| C123-C113-C13 | 112.47(19) |
| C123-C113-C153 | 101.79(19) |
| C13-C113-C153 | 112.13(19) |
| C123-C113-H113 | 110.1 |
| C13-C113-H113 | 110.1 |
| C153-C113-H113 | 110.1 |
| C133-C123-C113 | 104.5(2) |
| C133-C123-H12A3 | 110.9 |
| C113-C123-H12A3 | 110.9 |
| C133-C123-H12B3 | 110.9 |
| C113-C123-H12B3 | 110.9 |
| H12A3-C123-H12B3 | 108.9 |
| C123-C133-C143 | 105.2(2) |
| C123-C133-H13A3 | 110.7 |
| C143-C133-H13A3 | 110.7 |
| C123-C133-H13B3 | 110.7 |
| C143-C133-H13B3 | 110.7 |
| H13A3-C133-H13B3 | 108.8 |
| C133-C143-C153 | 106.28(19) |
| C133-C143-H14A3 | 110.5 |
| C153-C143-H14A3 | 110.5 |
| C133-C143-H14B3 | 110.5 |
| C153-C143-H14B3 | 110.5 |
| H14A3-C143-H14B3 | 108.7 |
| C143-C153-C113 | 105.6(2) |
| C143-C153-H15A3 | 110.6 |
| C113-C153-H15A3 | 110.6 |
| C143-C153-H15B3 | 110.6 |

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|---|---|
| C113-C153-H15B3 | 110.6 |
| H15A3-C153-H15B3 | 108.8 |
| C223-C213-C303 | 118.0(2) |
| C223-C213-C23 | 121.1(2) |
| C303-C213-C23 | 120.8(2) |
| C213-C223-C233 | 122.6(2) |
| C213-C223-H223 | 118.7 |
| C233-C223-H223 | 118.7 |
| C243-C233-C223 | 123.3(2) |
| C243-C233-C283 | 118.3(2) |
| C223-C233-C283 | 118.4(2) |
| C253-C243-C233 | 121.4(2) |
| C253-C243-H243 | 119.3 |
| C233-C243-H243 | 119.3 |
| C243-C253-C263 | 120.0(2) |
| C243-C253-H253 | 120.0 |
| C263-C253-H253 | 120.0 |
| C273-C263-O13 | 125.1(2) |
| C273-C263-C253 | 120.8(2) |
| O13-C263-C253 | 114.1(2) |
| C263-C273-C283 | 119.9(2) |
| C263-C273-H273 | 120.0 |
| C283-C273-H273 | 120.0 |
| C293-C283-C273 | 122.1(2) |
| C293-C283-C233 | 118.4(2) |
| C273-C283-C233 | 119.5(2) |
| C303-C293-C283 | 121.5(2) |
| C303-C293-H293 | 119.3 |
| C283-C293-H293 | 119.3 |
| C293-C303-C213 | 121.1(2) |
| C293-C303-H303 | 119.5 |
| C213-C303-H303 | 119.5 |
| O13-C313-H31A3 | 109.5 |
| O13-C313-H31B3 | 109.5 |
| H31A3-C313-H31B3 | 109.5 |
| O13-C313-H31C3 | 109.5 |
| H31A3-C313-H31C3 | 109.5 |
| H31B3-C313-H31C3 | 109.5 |
| C264-O14-C314 | 117.06(19) |
| C214-C24-C34 | 112.15(19) |
| C214-C24-C14 | 112.73(18) |
| C34-C24-C14 | 111.26(19) |
| C214-C24-H24 | 106.8 |
| C34-C24-H24 | 106.8 |
| C14-C24-H24 | 106.8 |
| C24-C34-H3A4 | 109.5 |
| C24-C34-H3B4 | 109.5 |
| H3A4-C34-H3B4 | 109.5 |
| C24-C34-H3C4 | 109.5 |
| H3A4-C34-H3C4 | 109.5 |
| H3B4-C34-H3C4 | 109.5 |
| N14-C44-C14 | 179.2(3) |
| C44-C14-C114 | 109.3(2) |
| C44-C14-C24 | 110.46(19) |
| C114-C14-C24 | 114.55(19) |
| C44-C14-H14 | 107.4 |
| C114-C14-H14 | 107.4 |

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|---|---|
| C24-C14-H14 | 107.4 |
| C124-C114-C154 | 100.8(2) |
| C124-C114-C14 | 112.3(2) |
| C154-C114-C14 | 113.4(2) |
| C124-C114-H114 | 110.0 |
| C154-C114-H114 | 110.0 |
| C14-C114-H114 | 110.0 |
| C134-C124-C114 | 104.3(2) |
| C134-C124-H12A4 | 110.9 |
| C114-C124-H12A4 | 110.9 |
| C134-C124-H12B4 | 110.9 |
| C114-C124-H12B4 | 110.9 |
| H12A4-C124-H12B4 | 108.9 |
| C124-C134-C144 | 106.5(2) |
| C124-C134-H13A4 | 110.4 |
| C144-C134-H13A4 | 110.4 |
| C124-C134-H13B4 | 110.4 |
| C144-C134-H13B4 | 110.4 |
| H13A4-C134-H13B4 | 108.6 |
| C154-C144-C134 | 105.3(2) |
| C154-C144-H14A4 | 110.7 |
| C134-C144-H14A4 | 110.7 |
| C154-C144-H14B4 | 110.7 |
| C134-C144-H14B4 | 110.7 |
| H14A4-C144-H14B4 | 108.8 |
| C144-C154-C114 | 107.2(2) |
| C144-C154-H15A4 | 110.3 |
| C114-C154-H15A4 | 110.3 |
| C144-C154-H15B4 | 110.3 |
| C114-C154-H15B4 | 110.3 |
| H15A4-C154-H15B4 | 108.5 |
| C224-C214-C304 | 117.2(2) |
| C224-C214-C24 | 121.2(2) |
| C304-C214-C24 | 121.6(2) |
| C214-C224-C234 | 122.6(2) |
| C214-C224-H224 | 118.7 |
| C234-C224-H224 | 118.7 |
| C224-C234-C284 | 119.4(2) |
| C224-C234-C244 | 122.4(2) |
| C284-C234-C244 | 118.1(2) |
| C254-C244-C234 | 120.7(2) |
| C254-C244-H244 | 119.7 |
| C234-C244-H244 | 119.7 |
| C244-C254-C264 | 120.9(2) |
| C244-C254-H254 | 119.6 |
| C264-C254-H254 | 119.6 |
| O14-C264-C274 | 125.0(2) |
| O14-C264-C254 | 114.5(2) |
| C274-C264-C254 | 120.5(2) |
| C264-C274-C284 | 119.4(2) |
| C264-C274-H274 | 120.3 |
| C284-C274-H274 | 120.3 |
| C234-C284-C294 | 117.8(2) |
| C234-C284-C274 | 120.3(2) |
| C294-C284-C274 | 121.9(2) |
| C304-C294-C284 | 120.9(2) |
| C304-C294-H294 | 119.6 |

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|---|---|
| C284-C294-H294 | 119.6 |
| C294-C304-C214 | 122.0(2) |
| C294-C304-H304 | 119.0 |
| C214-C304-H304 | 119.0 |
| O14-C314-H31A4 | 109.5 |
| O14-C314-H31B4 | 109.5 |
| H31A4-C314-H31B4 | 109.5 |
| O14-C314-H31C4 | 109.5 |
| H31A4-C314-H31C4 | 109.5 |
| H31B4-C314-H31C4 | 109.5 |
| C265-O15-C315 | 116.8(2) |
| C215-C25-C35 | 112.72(19) |
| C215-C25-C15 | 113.70(18) |
| C35-C25-C15 | 110.12(18) |
| C215-C25-H25 | 106.6 |
| C35-C25-H25 | 106.6 |
| C15-C25-H25 | 106.6 |
| C25-C35-H3A5 | 109.5 |
| C25-C35-H3B5 | 109.5 |
| H3A5-C35-H3B5 | 109.5 |
| C25-C35-H3C5 | 109.5 |
| H3A5-C35-H3C5 | 109.5 |
| H3B5-C35-H3C5 | 109.5 |
| N15-C45-C15 | 178.3(3) |
| C45-C15-C115 | 108.28(19) |
| C45-C15-C25 | 111.97(18) |
| C115-C15-C25 | 114.69(18) |
| C45-C15-H15 | 107.2 |
| C115-C15-H15 | 107.2 |
| C25-C15-H15 | 107.2 |
| C15-C115-C155 | 113.55(19) |
| C15-C115-C125 | 112.70(19) |
| C155-C115-C125 | 103.35(19) |
| C15-C115-H115 | 109.0 |
| C155-C115-H115 | 109.0 |
| C125-C115-H115 | 109.0 |
| C135-C125-C115 | 104.1(2) |
| C135-C125-H12A5 | 110.9 |
| C115-C125-H12A5 | 110.9 |
| C135-C125-H12B5 | 110.9 |
| C115-C125-H12B5 | 110.9 |
| H12A5-C125-H12B5 | 108.9 |
| C145-C135-C125 | 107.1(2) |
| C145-C135-H13A5 | 110.3 |
| C125-C135-H13A5 | 110.3 |
| C145-C135-H13B5 | 110.3 |
| C125-C135-H13B5 | 110.3 |
| H13A5-C135-H13B5 | 108.6 |
| C135-C145-C155 | 107.6(2) |
| C135-C145-H14A5 | 110.2 |
| C155-C145-H14A5 | 110.2 |
| C135-C145-H14B5 | 110.2 |
| C155-C145-H14B5 | 110.2 |
| H14A5-C145-H14B5 | 108.5 |
| C145-C155-C115 | 105.2(2) |
| C145-C155-H15A5 | 110.7 |
| C115-C155-H15A5 | 110.7 |

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| C145-C155-H15B5 | 110.7 |
| C115-C155-H15B5 | 110.7 |
| H15A5-C155-H15B5 | 108.8 |
| C225-C215-C305 | 117.8(2) |
| C225-C215-C25 | 121.4(2) |
| C305-C215-C25 | 120.7(2) |
| C215-C225-C235 | 122.3(2) |
| C215-C225-H225 | 118.9 |
| C235-C225-H225 | 118.9 |
| C225-C235-C285 | 119.3(2) |
| C225-C235-C245 | 122.8(2) |
| C285-C235-C245 | 117.9(2) |
| C255-C245-C235 | 121.1(2) |
| C255-C245-H245 | 119.4 |
| C235-C245-H245 | 119.4 |
| C245-C255-C265 | 120.5(2) |
| C245-C255-H255 | 119.8 |
| C265-C255-H255 | 119.8 |
| C275-C265-O15 | 124.6(2) |
| C275-C265-C255 | 120.8(2) |
| O15-C265-C255 | 114.6(2) |
| C265-C275-C285 | 119.0(2) |
| C265-C275-H275 | 120.5 |
| C285-C275-H275 | 120.5 |
| C235-C285-C295 | 118.4(2) |
| C235-C285-C275 | 120.6(2) |
| C295-C285-C275 | 121.0(2) |
| C305-C295-C285 | 120.5(2) |
| C305-C295-H295 | 119.7 |
| C285-C295-H295 | 119.7 |
| C295-C305-C215 | 121.8(2) |
| C295-C305-H305 | 119.1 |
| C215-C305-H305 | 119.1 |
| O15-C315-H31A5 | 109.5 |
| O15-C315-H31B5 | 109.5 |
| H31A5-C315-H31B5 | 109.5 |
| O15-C315-H31C5 | 109.5 |
| H31A5-C315-H31C5 | 109.5 |
| H31B5-C315-H31C5 | 109.5 |
| C266-O16-C316 | 116.4(2) |
| C216-C26-C36 | 112.36(19) |
| C216-C26-C16 | 113.09(18) |
| C36-C26-C16 | 110.88(19) |
| C216-C26-H26 | 106.7 |
| C36-C26-H26 | 106.7 |
| C16-C26-H26 | 106.7 |
| C26-C36-H3A6 | 109.5 |
| C26-C36-H3B6 | 109.5 |
| H3A6-C36-H3B6 | 109.5 |
| C26-C36-H3C6 | 109.5 |
| H3A6-C36-H3C6 | 109.5 |
| H3B6-C36-H3C6 | 109.5 |
| N16-C46-C16 | 177.3(3) |
| C46-C16-C116 | 108.8(2) |
| C46-C16-C26 | 111.01(19) |
| C116-C16-C26 | 113.73(18) |
| C46-C16-H16 | 107.7 |

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| C116-C16-H16 | 107.7 |
| C26-C16-H16 | 107.7 |
| C126-C116-C16 | 111.5(2) |
| C126-C116-C156 | 104.6(2) |
| C16-C116-C156 | 113.8(2) |
| C126-C116-H116 | 108.9 |
| C16-C116-H116 | 108.9 |
| C156-C116-H116 | 108.9 |
| C136-C126-C116 | 104.8(2) |
| C136-C126-H12A6 | 110.8 |
| C116-C126-H12A6 | 110.8 |
| C136-C126-H12B6 | 110.8 |
| C116-C126-H12B6 | 110.8 |
| H12A6-C126-H12B6 | 108.9 |
| C126-C136-C146 | 102.0(2) |
| C126-C136-H13A6 | 111.4 |
| C146-C136-H13A6 | 111.4 |
| C126-C136-H13B6 | 111.4 |
| C146-C136-H13B6 | 111.4 |
| H13A6-C136-H13B6 | 109.2 |
| C156-C146-C136 | 104.2(2) |
| C156-C146-H14A6 | 110.9 |
| C136-C146-H14A6 | 110.9 |
| C156-C146-H14B6 | 110.9 |
| C136-C146-H14B6 | 110.9 |
| H14A6-C146-H14B6 | 108.9 |
| C146-C156-C116 | 106.1(2) |
| C146-C156-H15A6 | 110.5 |
| C116-C156-H15A6 | 110.5 |
| C146-C156-H15B6 | 110.5 |
| C116-C156-H15B6 | 110.5 |
| H15A6-C156-H15B6 | 108.7 |
| C226-C216-C306 | 117.5(2) |
| C226-C216-C26 | 121.0(2) |
| C306-C216-C26 | 121.5(2) |
| C216-C226-C236 | 122.3(2) |
| C216-C226-H226 | 118.8 |
| C236-C226-H226 | 118.8 |
| C226-C236-C286 | 119.2(2) |
| C226-C236-C246 | 122.7(2) |
| C286-C236-C246 | 118.0(2) |
| C256-C246-C236 | 120.9(2) |
| C256-C246-H246 | 119.5 |
| C236-C246-H246 | 119.5 |
| C246-C256-C266 | 120.4(2) |
| C246-C256-H256 | 119.8 |
| C266-C256-H256 | 119.8 |
| C276-C266-O16 | 124.8(2) |
| C276-C266-C256 | 120.9(2) |
| O16-C266-C256 | 114.3(2) |
| C266-C276-C286 | 119.2(2) |
| C266-C276-H276 | 120.4 |
| C286-C276-H276 | 120.4 |
| C236-C286-C296 | 118.2(2) |
| C236-C286-C276 | 120.3(2) |
| C296-C286-C276 | 121.5(2) |
| C306-C296-C286 | 120.9(2) |

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| C306-C296-H296 | 119.6 |
| C286-C296-H296 | 119.6 |
| C296-C306-C216 | 121.8(2) |
| C296-C306-H306 | 119.1 |
| C216-C306-H306 | 119.1 |
| O16-C316-H31A6 | 109.5 |
| O16-C316-H31B6 | 109.5 |
| H31A6-C316-H31B6 | 109.5 |
| O16-C316-H31C6 | 109.5 |
| H31A6-C316-H31C6 | 109.5 |
| H31B6-C316-H31C6 | 109.5 |
| C267-O17-C317 | 117.1(2) |
| C217-C27-C37 | 112.13(18) |
| C217-C27-C17 | 112.93(17) |
| C37-C27-C17 | 110.77(18) |
| C217-C27-H27 | 106.9 |
| C37-C27-H27 | 106.9 |
| C17-C27-H27 | 106.9 |
| C27-C37-H3A7 | 109.5 |
| C27-C37-H3B7 | 109.5 |
| H3A7-C37-H3B7 | 109.5 |
| C27-C37-H3C7 | 109.5 |
| H3A7-C37-H3C7 | 109.5 |
| H3B7-C37-H3C7 | 109.5 |
| N17-C47-C17 | 178.7(3) |
| C47-C17-C117 | 109.7(2) |
| C47-C17-C27 | 110.89(18) |
| C117-C17-C27 | 113.72(17) |
| C47-C17-H17 | 107.4 |
| C117-C17-H17 | 107.4 |
| C27-C17-H17 | 107.4 |
| C12A7-C117-C157 | 104.0(6) |
| C12A7-C117-C17 | 120.0(6) |
| C157-C117-C17 | 117.1(3) |
| C12A7-C117-C15A7 | 107.6(7) |
| C157-C117-C15A7 | 17.1(4) |
| C17-C117-C15A7 | 102.1(5) |
| C12A7-C117-C127 | 18.4(5) |
| C157-C117-C127 | 99.5(3) |
| C17-C117-C127 | 108.7(3) |
| C15A7-C117-C127 | 97.9(5) |
| C12A7-C117-H11A7 | 92.2 |
| C157-C117-H11A7 | 110.3 |
| C17-C117-H11A7 | 110.3 |
| C15A7-C117-H11A7 | 126.0 |
| C127-C117-H11A7 | 110.3 |
| C12A7-C117-H11B7 | 108.8 |
| C157-C117-H11B7 | 95.0 |
| C17-C117-H11B7 | 108.8 |
| C15A7-C117-H11B7 | 108.8 |
| C127-C117-H11B7 | 127.2 |
| H11A7-C117-H11B7 | 19.9 |
| C137-C127-C117 | 105.0(3) |
| C137-C127-H12A7 | 110.8 |
| C117-C127-H12A7 | 110.8 |
| C137-C127-H12B7 | 110.8 |
| C117-C127-H12B7 | 110.8 |

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| H12A7-C127-H12B7 | 108.8 |
| C147-C137-C127 | 106.7(3) |
| C147-C137-H13A7 | 110.4 |
| C127-C137-H13A7 | 110.4 |
| C147-C137-H13B7 | 110.4 |
| C127-C137-H13B7 | 110.4 |
| H13A7-C137-H13B7 | 108.6 |
| C137-C147-C157 | 106.0(3) |
| C137-C147-H14A7 | 110.5 |
| C157-C147-H14A7 | 110.5 |
| C137-C147-H14B7 | 110.5 |
| C157-C147-H14B7 | 110.5 |
| H14A7-C147-H14B7 | 108.7 |
| C117-C157-C147 | 105.9(3) |
| C117-C157-H15A7 | 110.6 |
| C147-C157-H15A7 | 110.6 |
| C117-C157-H15B7 | 110.6 |
| C147-C157-H15B7 | 110.6 |
| H15A7-C157-H15B7 | 108.7 |
| C117-C12A7-C13A7 | 102.7(7) |
| C117-C12A7-H12C7 | 111.2 |
| C13A7-C12A7-H12C7 | 111.2 |
| C117-C12A7-H12D7 | 111.2 |
| C13A7-C12A7-H12D7 | 111.2 |
| H12C7-C12A7-H12D7 | 109.1 |
| C14A7-C13A7-C12A7 | 109.8(9) |
| C14A7-C13A7-H13C7 | 109.7 |
| C12A7-C13A7-H13C7 | 109.7 |
| C14A7-C13A7-H13D7 | 109.7 |
| C12A7-C13A7-H13D7 | 109.7 |
| H13C7-C13A7-H13D7 | 108.2 |
| C13A7-C14A7-C15A7 | 106.6(9) |
| C13A7-C14A7-H14C7 | 110.4 |
| C15A7-C14A7-H14C7 | 110.4 |
| C13A7-C14A7-H14D7 | 110.4 |
| C15A7-C14A7-H14D7 | 110.4 |
| H14C7-C14A7-H14D7 | 108.6 |
| C14A7-C15A7-C117 | 101.0(7) |
| C14A7-C15A7-H15C7 | 111.6 |
| C117-C15A7-H15C7 | 111.6 |
| C14A7-C15A7-H15D7 | 111.6 |
| C117-C15A7-H15D7 | 111.6 |
| H15C7-C15A7-H15D7 | 109.4 |
| C227-C217-C307 | 117.6(2) |
| C227-C217-C27 | 120.7(2) |
| C307-C217-C27 | 121.69(19) |
| C217-C227-C237 | 122.1(2) |
| C217-C227-H227 | 118.9 |
| C237-C227-H227 | 118.9 |
| C227-C237-C247 | 122.1(2) |
| C227-C237-C287 | 119.3(2) |
| C247-C237-C287 | 118.6(2) |
| C257-C247-C237 | 120.6(2) |
| C257-C247-H247 | 119.7 |
| C237-C247-H247 | 119.7 |
| C247-C257-C267 | 121.1(2) |
| C247-C257-H257 | 119.5 |

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| C267-C257-H257 | 119.5 |
| O17-C267-C277 | 124.8(2) |
| O17-C267-C257 | 114.8(2) |
| C277-C267-C257 | 120.5(2) |
| C267-C277-C287 | 119.3(2) |
| C267-C277-H277 | 120.3 |
| C287-C277-H277 | 120.3 |
| C297-C287-C237 | 118.0(2) |
| C297-C287-C277 | 122.2(2) |
| C237-C287-C277 | 119.8(2) |
| C307-C297-C287 | 121.2(2) |
| C307-C297-H297 | 119.4 |
| C287-C297-H297 | 119.4 |
| C297-C307-C217 | 121.8(2) |
| C297-C307-H307 | 119.1 |
| C217-C307-H307 | 119.1 |
| O17-C317-H31A7 | 109.5 |
| O17-C317-H31B7 | 109.5 |
| H31A7-C317-H31B7 | 109.5 |
| O17-C317-H31C7 | 109.5 |
| H31A7-C317-H31C7 | 109.5 |
| H31B7-C317-H31C7 | 109.5 |
| C268-O18-C318 | 117.36(19) |
| C218-C28-C38 | 111.7(2) |
| C218-C28-C18 | 113.26(18) |
| C38-C28-C18 | 110.71(19) |
| C218-C28-H28 | 106.9 |
| C38-C28-H28 | 106.9 |
| C18-C28-H28 | 106.9 |
| C28-C38-H3A8 | 109.5 |
| C28-C38-H3B8 | 109.5 |
| H3A8-C38-H3B8 | 109.5 |
| C28-C38-H3C8 | 109.5 |
| H3A8-C38-H3C8 | 109.5 |
| H3B8-C38-H3C8 | 109.5 |
| N18-C48-C18 | 177.1(3) |
| C48-C18-C118 | 109.0(2) |
| C48-C18-C28 | 110.10(18) |
| C118-C18-C28 | 114.90(18) |
| C48-C18-H18 | 107.5 |
| C118-C18-H18 | 107.5 |
| C28-C18-H18 | 107.5 |
| C128-C118-C15A8 | 103.6(6) |
| C128-C118-C18 | 118.9(4) |
| C15A8-C118-C18 | 117.3(6) |
| C128-C118-C158 | 103.2(5) |
| C15A8-C118-C158 | 11.4(8) |
| C18-C118-C158 | 108.8(5) |
| C128-C118-C12A8 | 19.4(4) |
| C15A8-C118-C12A8 | 99.5(6) |
| C18-C118-C12A8 | 106.2(5) |
| C158-C118-C12A8 | 95.5(5) |
| C128-C118-H11A8 | 108.5 |
| C15A8-C118-H11A8 | 97.9 |
| C18-C118-H11A8 | 108.5 |
| C158-C118-H11A8 | 108.5 |
| C12A8-C118-H11A8 | 127.8 |

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|---|---|
| C128-C118-H11B8 | 92.1 |
| C15A8-C118-H11B8 | 111.0 |
| C18-C118-H11B8 | 111.0 |
| C158-C118-H11B8 | 122.3 |
| C12A8-C118-H11B8 | 111.0 |
| H11A8-C118-H11B8 | 18.6 |
| C118-C128-C138 | 104.0(4) |
| C118-C128-H12A8 | 111.0 |
| C138-C128-H12A8 | 111.0 |
| C118-C128-H12B8 | 111.0 |
| C138-C128-H12B8 | 111.0 |
| H12A8-C128-H12B8 | 109.0 |
| C128-C138-C148 | 106.4(5) |
| C128-C138-H13A8 | 110.5 |
| C148-C138-H13A8 | 110.5 |
| C128-C138-H13B8 | 110.5 |
| C148-C138-H13B8 | 110.5 |
| H13A8-C138-H13B8 | 108.6 |
| C158-C148-C138 | 105.6(5) |
| C158-C148-H14A8 | 110.6 |
| C138-C148-H14A8 | 110.6 |
| C158-C148-H14B8 | 110.6 |
| C138-C148-H14B8 | 110.6 |
| H14A8-C148-H14B8 | 108.8 |
| C148-C158-C118 | 103.7(5) |
| C148-C158-H15A8 | 111.0 |
| C118-C158-H15A8 | 111.0 |
| C148-C158-H15B8 | 111.0 |
| C118-C158-H15B8 | 111.0 |
| H15A8-C158-H15B8 | 109.0 |
| C13A8-C12A8-C118 | 104.9(6) |
| C13A8-C12A8-H12C8 | 110.8 |
| C118-C12A8-H12C8 | 110.8 |
| C13A8-C12A8-H12D8 | 110.8 |
| C118-C12A8-H12D8 | 110.8 |
| H12C8-C12A8-H12D8 | 108.8 |
| C12A8-C13A8-C14A8 | 106.3(7) |
| C12A8-C13A8-H13C8 | 110.5 |
| C14A8-C13A8-H13C8 | 110.5 |
| C12A8-C13A8-H13D8 | 110.5 |
| C14A8-C13A8-H13D8 | 110.5 |
| H13C8-C13A8-H13D8 | 108.7 |
| C15A8-C14A8-C13A8 | 106.9(8) |
| C15A8-C14A8-H14C8 | 110.3 |
| C13A8-C14A8-H14C8 | 110.3 |
| C15A8-C14A8-H14D8 | 110.3 |
| C13A8-C14A8-H14D8 | 110.3 |
| H14C8-C14A8-H14D8 | 108.6 |
| C118-C15A8-C14A8 | 108.1(8) |
| C118-C15A8-H15C8 | 110.1 |
| C14A8-C15A8-H15C8 | 110.1 |
| C118-C15A8-H15D8 | 110.1 |
| C14A8-C15A8-H15D8 | 110.1 |
| H15C8-C15A8-H15D8 | 108.4 |
| C228-C218-C308 | 118.2(2) |
| C228-C218-C28 | 120.8(2) |
| C308-C218-C28 | 120.9(2) |

| | |
|---|---|
| C218-C228-C238 | 122.3(2) |
| C218-C228-H228 | 118.9 |
| C238-C228-H228 | 118.9 |
| C248-C238-C228 | 122.5(2) |
| C248-C238-C288 | 118.5(2) |
| C228-C238-C288 | 119.0(2) |
| C258-C248-C238 | 121.0(2) |
| C258-C248-H248 | 119.5 |
| C238-C248-H248 | 119.5 |
| C248-C258-C268 | 119.9(2) |
| C248-C258-H258 | 120.0 |
| C268-C258-H258 | 120.0 |
| C278-C268-O18 | 125.8(2) |
| C278-C268-C258 | 120.7(2) |
| O18-C268-C258 | 113.6(2) |
| C268-C278-C288 | 120.1(2) |
| C268-C278-H278 | 120.0 |
| C288-C278-H278 | 120.0 |
| C298-C288-C278 | 122.1(2) |
| C298-C288-C238 | 118.2(2) |
| C278-C288-C238 | 119.7(2) |
| C308-C298-C288 | 121.5(2) |
| C308-C298-H298 | 119.2 |
| C288-C298-H298 | 119.2 |
| C298-C308-C218 | 120.9(2) |
| C298-C308-H308 | 119.6 |
| C218-C308-H308 | 119.6 |
| O18-C318-H31A8 | 109.5 |
| O18-C318-H31B8 | 109.5 |
| H31A8-C318-H31B8 | 109.5 |
| O18-C318-H31C8 | 109.5 |
| H31A8-C318-H31C8 | 109.5 |
| H31B8-C318-H31C8 | 109.5 |

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for X12022. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| O11 | 36(1) | 26(1) | 33(1) | -4(1) | 5(1) | 4(1) |
| N11 | 38(1) | 26(1) | 58(1) | 6(1) | 18(1) | 1(1) |
| C21 | 22(1) | 24(1) | 22(1) | 0(1) | -1(1) | -2(1) |
| C31 | 25(1) | 32(1) | 26(1) | -2(1) | -4(1) | 1(1) |
| C41 | 31(1) | 18(1) | 34(1) | 2(1) | 3(1) | 4(1) |
| C11 | 22(1) | 21(1) | 28(1) | 1(1) | -1(1) | 1(1) |
| C111 | 29(1) | 23(1) | 25(1) | -1(1) | -6(1) | -1(1) |
| C121 | 27(1) | 40(1) | 38(1) | 6(1) | 0(1) | -2(1) |
| C131 | 25(1) | 52(2) | 55(2) | 9(1) | -11(1) | -5(1) |
| C141 | 30(1) | 42(1) | 42(1) | 5(1) | -12(1) | 6(1) |
| C151 | 36(1) | 31(1) | 32(1) | 8(1) | 0(1) | 3(1) |
| C211 | 22(1) | 22(1) | 22(1) | 4(1) | 0(1) | 0(1) |
| C221 | 19(1) | 26(1) | 24(1) | 3(1) | 0(1) | 0(1) |
| C231 | 22(1) | 28(1) | 22(1) | 5(1) | 0(1) | -2(1) |
| C241 | 24(1) | 28(1) | 30(1) | 0(1) | 4(1) | -2(1) |
| C251 | 32(1) | 31(1) | 27(1) | -3(1) | 3(1) | -5(1) |
| C261 | 35(1) | 24(1) | 20(1) | -1(1) | -1(1) | 3(1) |
| C271 | 25(1) | 32(1) | 22(1) | 2(1) | 2(1) | 3(1) |
| C281 | 24(1) | 25(1) | 20(1) | 4(1) | -1(1) | 1(1) |
| C291 | 18(1) | 28(1) | 29(1) | 5(1) | 2(1) | 1(1) |
| C301 | 21(1) | 24(1) | 32(1) | 4(1) | 3(1) | -4(1) |
| C311 | 49(2) | 28(1) | 42(2) | 2(1) | 16(1) | 8(1) |
| O12 | 46(1) | 30(1) | 35(1) | 4(1) | 4(1) | -7(1) |
| N12 | 30(1) | 26(1) | 38(1) | 0(1) | 9(1) | 3(1) |
| C22 | 24(1) | 27(1) | 32(1) | -2(1) | 7(1) | 1(1) |
| C32 | 30(1) | 34(1) | 31(1) | 3(1) | 4(1) | 2(1) |
| C42 | 32(1) | 20(1) | 27(1) | 2(1) | 2(1) | -2(1) |
| C12 | 24(1) | 22(1) | 36(1) | 0(1) | 4(1) | 0(1) |
| C112 | 27(1) | 26(1) | 32(1) | -2(1) | 1(1) | 0(1) |
| C122 | 25(1) | 44(1) | 41(1) | -4(1) | 1(1) | 3(1) |
| C132 | 27(1) | 56(2) | 42(1) | 1(1) | -5(1) | -1(1) |
| C142 | 31(1) | 48(1) | 35(1) | -4(1) | -2(1) | -5(1) |
| C152 | 35(1) | 28(1) | 40(1) | -6(1) | -3(1) | 0(1) |
| C212 | 24(1) | 24(1) | 28(1) | -8(1) | 6(1) | -3(1) |
| C222 | 20(1) | 30(1) | 28(1) | -4(1) | 2(1) | -5(1) |
| C232 | 22(1) | 31(1) | 24(1) | -6(1) | 2(1) | 2(1) |
| C242 | 24(1) | 32(1) | 32(1) | -1(1) | 1(1) | 2(1) |
| C252 | 33(1) | 33(1) | 32(1) | 1(1) | 0(1) | 4(1) |
| C262 | 38(1) | 29(1) | 22(1) | 0(1) | 2(1) | -3(1) |
| C272 | 29(1) | 34(1) | 25(1) | -4(1) | 4(1) | -4(1) |
| C282 | 24(1) | 29(1) | 22(1) | -5(1) | 1(1) | -2(1) |
| C292 | 18(1) | 31(1) | 30(1) | -7(1) | 2(1) | -1(1) |
| C302 | 22(1) | 27(1) | 33(1) | -5(1) | 2(1) | 2(1) |
| C312 | 51(2) | 32(1) | 39(1) | -6(1) | 15(1) | -11(1) |
| O13 | 46(1) | 28(1) | 32(1) | 1(1) | 5(1) | -8(1) |
| N13 | 35(1) | 29(1) | 60(1) | -6(1) | 17(1) | -2(1) |
| C23 | 19(1) | 25(1) | 25(1) | -1(1) | 0(1) | 1(1) |
| C33 | 28(1) | 34(1) | 27(1) | 4(1) | 1(1) | 2(1) |
| C43 | 28(1) | 22(1) | 38(1) | -1(1) | 6(1) | -4(1) |
| C13 | 24(1) | 21(1) | 27(1) | -1(1) | 3(1) | -2(1) |
| C113 | 34(1) | 25(1) | 24(1) | 1(1) | 1(1) | -7(1) |
| C123 | 32(1) | 44(1) | 36(1) | -7(1) | -11(1) | 7(1) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C133 | 24(1) | 76(2) | 33(1) | 10(1) | 0(1) | 1(1) |
| C143 | 31(1) | 33(1) | 42(1) | 7(1) | -10(1) | -6(1) |
| C153 | 44(1) | 38(1) | 28(1) | -8(1) | 4(1) | -12(1) |
| C213 | 20(1) | 25(1) | 23(1) | -6(1) | -1(1) | -2(1) |
| C223 | 21(1) | 32(1) | 23(1) | -4(1) | -1(1) | -2(1) |
| C233 | 26(1) | 26(1) | 22(1) | -2(1) | 4(1) | 1(1) |
| C243 | 25(1) | 32(1) | 26(1) | -2(1) | 0(1) | 3(1) |
| C253 | 40(1) | 27(1) | 26(1) | 2(1) | 2(1) | 0(1) |
| C263 | 37(1) | 27(1) | 21(1) | -3(1) | 4(1) | -8(1) |
| C273 | 29(1) | 30(1) | 29(1) | -5(1) | 7(1) | -5(1) |
| C283 | 24(1) | 28(1) | 25(1) | -5(1) | 4(1) | -1(1) |
| C293 | 20(1) | 30(1) | 35(1) | -5(1) | 3(1) | 1(1) |
| C303 | 21(1) | 25(1) | 31(1) | -4(1) | 1(1) | 3(1) |
| C313 | 50(2) | 38(1) | 41(2) | -5(1) | 16(1) | -18(1) |
| O14 | 29(1) | 27(1) | 37(1) | 6(1) | 0(1) | -2(1) |
| N14 | 56(1) | 30(1) | 45(1) | 1(1) | 22(1) | 6(1) |
| C24 | 24(1) | 27(1) | 27(1) | -2(1) | 4(1) | -2(1) |
| C34 | 31(1) | 41(1) | 34(1) | -1(1) | 3(1) | -6(1) |
| C44 | 40(1) | 22(1) | 36(1) | 5(1) | 9(1) | 4(1) |
| C14 | 33(1) | 23(1) | 28(1) | 2(1) | 8(1) | 0(1) |
| C114 | 31(1) | 29(1) | 40(1) | 6(1) | 0(1) | -2(1) |
| C124 | 41(2) | 57(2) | 47(2) | -16(1) | -12(1) | 8(1) |
| C134 | 50(2) | 90(2) | 25(1) | -2(1) | -2(1) | -12(2) |
| C144 | 48(2) | 40(1) | 42(2) | 11(1) | -7(1) | -6(1) |
| C154 | 40(1) | 48(1) | 43(1) | 16(1) | 4(1) | 9(1) |
| C214 | 22(1) | 27(1) | 27(1) | 1(1) | 2(1) | 4(1) |
| C224 | 24(1) | 29(1) | 24(1) | -1(1) | 3(1) | 4(1) |
| C234 | 20(1) | 29(1) | 25(1) | 0(1) | 0(1) | 4(1) |
| C244 | 29(1) | 32(1) | 26(1) | -2(1) | 3(1) | -2(1) |
| C254 | 25(1) | 26(1) | 34(1) | -2(1) | 2(1) | -2(1) |
| C264 | 16(1) | 26(1) | 35(1) | 3(1) | 2(1) | 2(1) |
| C274 | 21(1) | 29(1) | 28(1) | 2(1) | 4(1) | 4(1) |
| C284 | 19(1) | 26(1) | 27(1) | 2(1) | 2(1) | 5(1) |
| C294 | 30(1) | 29(1) | 25(1) | 1(1) | 5(1) | 6(1) |
| C304 | 30(1) | 26(1) | 29(1) | -2(1) | 2(1) | 2(1) |
| C314 | 37(1) | 30(1) | 42(1) | 8(1) | 11(1) | 2(1) |
| O15 | 30(1) | 27(1) | 43(1) | 3(1) | 3(1) | -3(1) |
| N15 | 38(1) | 27(1) | 31(1) | -4(1) | 9(1) | 1(1) |
| C25 | 22(1) | 26(1) | 26(1) | -3(1) | 1(1) | 1(1) |
| C35 | 25(1) | 36(1) | 32(1) | 0(1) | 2(1) | -2(1) |
| C45 | 26(1) | 21(1) | 30(1) | 1(1) | -2(1) | -2(1) |
| C15 | 25(1) | 23(1) | 27(1) | -3(1) | 4(1) | -4(1) |
| C115 | 26(1) | 26(1) | 30(1) | -1(1) | 0(1) | 1(1) |
| C125 | 41(1) | 48(2) | 28(1) | -6(1) | -2(1) | 5(1) |
| C135 | 40(2) | 80(2) | 34(1) | -6(1) | -5(1) | 10(1) |
| C145 | 66(2) | 66(2) | 41(2) | 3(1) | -10(1) | 24(2) |
| C155 | 36(1) | 34(1) | 38(1) | 1(1) | -5(1) | 8(1) |
| C215 | 18(1) | 27(1) | 26(1) | -1(1) | 0(1) | 4(1) |
| C225 | 22(1) | 30(1) | 25(1) | -2(1) | 0(1) | 2(1) |
| C235 | 19(1) | 28(1) | 26(1) | -2(1) | -3(1) | 4(1) |
| C245 | 27(1) | 34(1) | 28(1) | -6(1) | 3(1) | -4(1) |
| C255 | 24(1) | 28(1) | 39(1) | -10(1) | 3(1) | -3(1) |
| C265 | 16(1) | 25(1) | 43(1) | 1(1) | 2(1) | 3(1) |
| C275 | 22(1) | 26(1) | 31(1) | 2(1) | 4(1) | 3(1) |
| C285 | 15(1) | 26(1) | 30(1) | 1(1) | 1(1) | 4(1) |
| C295 | 28(1) | 25(1) | 24(1) | 0(1) | 2(1) | 5(1) |
| C305 | 26(1) | 22(1) | 27(1) | -1(1) | -1(1) | 4(1) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C315 | 37(1) | 27(1) | 52(2) | 4(1) | 17(1) | 3(1) |
| O16 | 35(1) | 27(1) | 51(1) | -8(1) | 5(1) | 5(1) |
| N16 | 55(1) | 29(1) | 42(1) | -4(1) | 20(1) | -10(1) |
| C26 | 25(1) | 24(1) | 29(1) | 2(1) | 6(1) | -1(1) |
| C36 | 29(1) | 37(1) | 37(1) | -2(1) | 2(1) | 4(1) |
| C46 | 39(1) | 23(1) | 33(1) | -5(1) | 10(1) | -4(1) |
| C16 | 26(1) | 24(1) | 30(1) | -1(1) | 8(1) | -2(1) |
| C116 | 32(1) | 33(1) | 33(1) | -5(1) | 4(1) | -6(1) |
| C126 | 40(1) | 54(2) | 30(1) | 5(1) | -2(1) | -9(1) |
| C136 | 53(2) | 63(2) | 30(1) | 2(1) | -8(1) | -10(1) |
| C146 | 71(2) | 58(2) | 47(2) | -16(1) | 2(2) | -18(2) |
| C156 | 36(1) | 44(1) | 40(1) | -8(1) | 7(1) | -16(1) |
| C216 | 19(1) | 26(1) | 29(1) | -2(1) | 0(1) | -6(1) |
| C226 | 22(1) | 28(1) | 26(1) | 1(1) | 3(1) | -4(1) |
| C236 | 20(1) | 26(1) | 29(1) | 0(1) | -2(1) | -4(1) |
| C246 | 26(1) | 30(1) | 32(1) | 3(1) | 4(1) | 0(1) |
| C256 | 25(1) | 28(1) | 41(1) | 7(1) | 2(1) | 3(1) |
| C266 | 19(1) | 25(1) | 49(2) | -4(1) | 3(1) | -2(1) |
| C276 | 22(1) | 30(1) | 32(1) | -4(1) | 3(1) | -3(1) |
| C286 | 17(1) | 26(1) | 32(1) | -4(1) | 1(1) | -6(1) |
| C296 | 28(1) | 29(1) | 26(1) | -2(1) | 4(1) | -8(1) |
| C306 | 27(1) | 22(1) | 30(1) | 0(1) | 0(1) | -6(1) |
| C316 | 41(2) | 31(1) | 62(2) | -14(1) | 17(1) | -4(1) |
| O17 | 33(1) | 26(1) | 40(1) | -5(1) | 4(1) | 2(1) |
| N17 | 61(2) | 29(1) | 36(1) | -3(1) | 20(1) | -10(1) |
| C27 | 22(1) | 28(1) | 23(1) | -1(1) | 2(1) | 2(1) |
| C37 | 29(1) | 45(1) | 29(1) | -3(1) | -3(1) | 10(1) |
| C47 | 37(1) | 21(1) | 35(1) | -6(1) | 8(1) | -5(1) |
| C17 | 27(1) | 20(1) | 25(1) | -2(1) | 4(1) | 2(1) |
| C117 | 25(1) | 26(1) | 38(1) | -8(1) | -7(1) | 5(1) |
| C127 | 33(2) | 30(3) | 34(2) | 15(2) | -11(2) | -5(2) |
| C137 | 32(2) | 39(3) | 22(2) | -2(2) | 1(1) | -3(2) |
| C147 | 26(2) | 24(2) | 27(2) | -3(1) | -1(2) | -2(1) |
| C157 | 17(2) | 33(2) | 24(2) | -3(2) | -2(1) | 0(2) |
| C12A7 | 37(4) | 26(4) | 40(3) | 14(3) | -20(3) | -6(4) |
| C13A7 | 35(4) | 44(5) | 39(3) | 0(4) | 3(3) | -6(4) |
| C14A7 | 35(5) | 36(4) | 40(4) | -9(3) | -3(3) | -5(3) |
| C15A7 | 28(4) | 34(4) | 41(4) | 5(3) | 0(3) | 2(3) |
| C217 | 17(1) | 25(1) | 28(1) | -3(1) | 0(1) | -6(1) |
| C227 | 22(1) | 31(1) | 20(1) | 1(1) | 1(1) | -4(1) |
| C237 | 17(1) | 26(1) | 24(1) | -2(1) | -3(1) | -2(1) |
| C247 | 28(1) | 32(1) | 25(1) | 2(1) | 2(1) | 2(1) |
| C257 | 29(1) | 27(1) | 32(1) | 5(1) | 2(1) | 4(1) |
| C267 | 18(1) | 26(1) | 38(1) | -4(1) | 3(1) | -3(1) |
| C277 | 23(1) | 28(1) | 29(1) | -4(1) | 5(1) | -4(1) |
| C287 | 18(1) | 26(1) | 26(1) | -1(1) | 1(1) | -6(1) |
| C297 | 27(1) | 31(1) | 21(1) | -2(1) | 2(1) | -6(1) |
| C307 | 30(1) | 24(1) | 27(1) | 0(1) | 1(1) | -5(1) |
| C317 | 40(1) | 30(1) | 49(2) | -12(1) | 13(1) | -5(1) |
| O18 | 33(1) | 26(1) | 32(1) | 1(1) | 2(1) | 3(1) |
| N18 | 40(1) | 30(1) | 67(2) | 12(1) | 22(1) | 4(1) |
| C28 | 25(1) | 27(1) | 33(1) | 5(1) | 8(1) | 0(1) |
| C38 | 38(1) | 41(1) | 36(1) | -3(1) | 8(1) | 3(1) |
| C48 | 31(1) | 24(1) | 39(1) | 4(1) | 10(1) | 4(1) |
| C18 | 24(1) | 21(1) | 33(1) | -1(1) | 9(1) | -1(1) |
| C118 | 35(1) | 28(1) | 40(1) | 0(1) | -4(1) | 5(1) |
| C128 | 33(2) | 31(3) | 45(3) | 9(2) | -13(2) | -5(2) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C138 | 33(2) | 47(4) | 50(3) | 14(3) | 10(2) | -3(3) |
| C148 | 23(3) | 33(3) | 39(3) | 3(2) | -3(2) | 0(2) |
| C158 | 36(3) | 38(3) | 34(3) | 8(2) | 3(2) | 1(2) |
| C12A8 | 36(3) | 37(4) | 57(4) | 6(3) | -4(3) | -11(3) |
| C13A8 | 23(3) | 53(4) | 47(3) | -2(3) | 8(2) | -1(3) |
| C14A8 | 33(4) | 43(3) | 47(4) | 7(3) | 3(3) | 3(3) |
| C15A8 | 22(3) | 57(4) | 36(4) | 12(3) | 2(2) | 12(3) |
| C218 | 24(1) | 24(1) | 25(1) | 7(1) | 1(1) | 4(1) |
| C228 | 19(1) | 32(1) | 27(1) | 7(1) | 3(1) | 3(1) |
| C238 | 24(1) | 27(1) | 21(1) | 6(1) | 1(1) | -2(1) |
| C248 | 24(1) | 34(1) | 31(1) | 3(1) | 3(1) | -4(1) |
| C258 | 33(1) | 31(1) | 33(1) | -2(1) | 1(1) | -7(1) |
| C268 | 29(1) | 25(1) | 24(1) | 1(1) | 1(1) | 4(1) |
| C278 | 23(1) | 31(1) | 26(1) | 8(1) | 3(1) | 3(1) |
| C288 | 24(1) | 26(1) | 20(1) | 7(1) | 0(1) | 1(1) |
| C298 | 20(1) | 27(1) | 33(1) | 6(1) | 6(1) | 0(1) |
| C308 | 22(1) | 24(1) | 31(1) | 7(1) | 3(1) | -2(1) |
| C318 | 43(2) | 32(1) | 41(1) | 5(1) | 15(1) | 9(1) |

Table 5. Hydrogen coordinates ( x $10^4$) and isotropic displacement parameters ($Å^2$x $10^3$) for X12022.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H21 | 2567 | 2043 | 5404 | 29 |
| H3A1 | 4094 | 2021 | 6496 | 44 |
| H3B1 | 3639 | 2476 | 6414 | 44 |
| H3C1 | 4458 | 2371 | 5890 | 44 |
| H11 | 2645 | 2735 | 4890 | 30 |
| H111 | 2154 | 2206 | 3237 | 34 |
| H12A1 | 1032 | 2415 | 4531 | 44 |
| H12B1 | 1091 | 1974 | 4030 | 44 |
| H13A1 | -242 | 2558 | 3346 | 57 |
| H13B1 | -71 | 2153 | 2766 | 57 |
| H14A1 | 716 | 2515 | 1881 | 50 |
| H14B1 | 233 | 2917 | 2222 | 50 |
| H15A1 | 1559 | 3047 | 3467 | 41 |
| H15B1 | 2058 | 2884 | 2660 | 41 |
| H221 | 2238 | 1422 | 4571 | 29 |
| H241 | 1925 | 705 | 3918 | 34 |
| H251 | 2488 | 101 | 3432 | 37 |
| H271 | 5056 | 635 | 3556 | 32 |
| H291 | 5296 | 1378 | 4056 | 31 |
| H301 | 4779 | 1969 | 4663 | 32 |
| H31A1 | 5234 | 35 | 2758 | 58 |
| H31B1 | 5184 | -432 | 3113 | 58 |
| H31C1 | 5505 | -67 | 3873 | 58 |
| H22 | 9990 | 7068 | 2169 | 33 |
| H3A2 | 8515 | 7044 | 981 | 48 |
| H3B2 | 8981 | 7497 | 1103 | 48 |
| H3C2 | 8105 | 7394 | 1550 | 48 |
| H12 | 9854 | 7747 | 2704 | 33 |
| H112 | 10234 | 7183 | 4288 | 35 |
| H12A2 | 11392 | 7035 | 3485 | 46 |
| H12B2 | 11520 | 7515 | 3246 | 46 |
| H13A2 | 12725 | 7485 | 4578 | 53 |
| H13B2 | 12257 | 7101 | 5003 | 53 |
| H14A2 | 12082 | 7923 | 5426 | 48 |
| H14B2 | 11615 | 7539 | 5852 | 48 |
| H15A2 | 10220 | 7824 | 5012 | 44 |
| H15B2 | 10719 | 8051 | 4276 | 44 |
| H222 | 10296 | 6455 | 3036 | 32 |
| H242 | 10623 | 5746 | 3755 | 37 |
| H252 | 10059 | 5142 | 4249 | 41 |
| H272 | 7397 | 5628 | 3799 | 36 |
| H292 | 7130 | 6362 | 3226 | 32 |
| H302 | 7656 | 6951 | 2643 | 34 |
| H31A2 | 7039 | 4906 | 3566 | 60 |
| H31B2 | 7202 | 5069 | 4637 | 60 |
| H31C2 | 7334 | 4588 | 4437 | 60 |
| H23 | 2661 | 7013 | -48 | 29 |
| H3A3 | 1265 | 7053 | -1305 | 46 |
| H3B3 | 1741 | 7501 | -1065 | 46 |
| H3C3 | 817 | 7380 | -711 | 46 |
| H13 | 2665 | 7678 | 586 | 29 |

| | | | | |
|---|---|---|---|---|
| H113 | 2692 | 7098 | 2110 | 35 |
| H12A3 | 4296 | 6997 | 2253 | 49 |
| H12B3 | 3880 | 6995 | 1113 | 49 |
| H13A3 | 4377 | 7657 | 960 | 55 |
| H13B3 | 5216 | 7508 | 1856 | 55 |
| H14A3 | 4023 | 8140 | 1922 | 47 |
| H14B3 | 4699 | 7924 | 2850 | 47 |
| H15A3 | 2788 | 7891 | 2419 | 45 |
| H15B3 | 3467 | 7585 | 3173 | 45 |
| H223 | 2825 | 6385 | 729 | 32 |
| H243 | 3002 | 5655 | 1371 | 35 |
| H253 | 2334 | 5073 | 1852 | 39 |
| H273 | -243 | 5647 | 1381 | 35 |
| H293 | -383 | 6380 | 814 | 35 |
| H303 | 273 | 6961 | 310 | 32 |
| H31A3 | -533 | 5111 | 2224 | 64 |
| H31B3 | -487 | 4624 | 2032 | 64 |
| H31C3 | -708 | 4942 | 1157 | 64 |
| H24 | 3766 | 4213 | 3960 | 32 |
| H3A4 | 2496 | 4172 | 2578 | 54 |
| H3B4 | 2763 | 3723 | 3035 | 54 |
| H3C4 | 3101 | 3857 | 2111 | 54 |
| H14 | 4451 | 3559 | 3929 | 34 |
| H114 | 5902 | 4181 | 3996 | 42 |
| H12A4 | 6065 | 4227 | 5605 | 63 |
| H12B4 | 4923 | 4178 | 5228 | 63 |
| H13A4 | 4954 | 3502 | 5653 | 68 |
| H13B4 | 5869 | 3659 | 6457 | 68 |
| H14A4 | 6849 | 3297 | 5818 | 56 |
| H14B4 | 5923 | 3086 | 5122 | 56 |
| H15A4 | 6365 | 3412 | 3959 | 54 |
| H15B4 | 7059 | 3721 | 4689 | 54 |
| H224 | 4495 | 4860 | 4127 | 32 |
| H244 | 5067 | 5592 | 4327 | 36 |
| H254 | 5453 | 6194 | 3660 | 35 |
| H274 | 5088 | 5635 | 1093 | 32 |
| H294 | 4589 | 4883 | 939 | 34 |
| H304 | 4103 | 4298 | 1577 | 35 |
| H31A4 | 5909 | 6201 | 851 | 54 |
| H31B4 | 5608 | 6677 | 884 | 54 |
| H31C4 | 4805 | 6325 | 635 | 54 |
| H25 | 6010 | 4231 | 1263 | 31 |
| H3A5 | 4780 | 4230 | -158 | 48 |
| H3B5 | 4950 | 3776 | 296 | 48 |
| H3C5 | 5350 | 3892 | -602 | 48 |
| H15 | 6531 | 3546 | 1159 | 31 |
| H115 | 8145 | 4094 | 1473 | 35 |
| H12A5 | 7407 | 4302 | 2625 | 49 |
| H12B5 | 7039 | 3841 | 2768 | 49 |
| H13A5 | 8894 | 4170 | 3502 | 65 |
| H13B5 | 8356 | 3831 | 3988 | 65 |
| H14A5 | 9028 | 3323 | 3392 | 74 |
| H14B5 | 9657 | 3662 | 3012 | 74 |
| H15A5 | 8057 | 3234 | 1954 | 46 |
| H15B5 | 8872 | 3462 | 1564 | 46 |
| H225 | 6782 | 4868 | 1469 | 32 |
| H245 | 7407 | 5594 | 1680 | 37 |

| | | | | |
|---|---|---|---|---|
| H255 | 7874 | 6186 | 1032 | 37 |
| H275 | 7630 | 5604 | -1493 | 32 |
| H295 | 7085 | 4864 | -1659 | 31 |
| H305 | 6524 | 4287 | -1043 | 32 |
| H31A5 | 8392 | 6160 | -1771 | 56 |
| H31B5 | 8163 | 6644 | -1725 | 56 |
| H31C5 | 7310 | 6314 | -1961 | 56 |
| H26 | 1544 | 9265 | 1285 | 31 |
| H3A6 | 2623 | 9225 | 2819 | 53 |
| H3B6 | 2487 | 8787 | 2286 | 53 |
| H3C6 | 1962 | 8877 | 3111 | 53 |
| H16 | 908 | 8584 | 1259 | 32 |
| H116 | -588 | 9196 | 784 | 40 |
| H12A6 | 218 | 9376 | -283 | 52 |
| H12B6 | 766 | 8938 | -209 | 52 |
| H13A6 | -326 | 8875 | -1664 | 62 |
| H13B6 | -1148 | 9111 | -1297 | 62 |
| H14A6 | -1517 | 8414 | -1168 | 73 |
| H14B6 | -411 | 8293 | -744 | 73 |
| H15A6 | -836 | 8355 | 629 | 48 |
| H15B6 | -1636 | 8699 | 202 | 48 |
| H226 | 711 | 9894 | 1008 | 32 |
| H246 | 43 | 10614 | 743 | 36 |
| H256 | -430 | 11211 | 1364 | 39 |
| H276 | -229 | 10646 | 3902 | 35 |
| H296 | 276 | 9900 | 4084 | 34 |
| H306 | 861 | 9318 | 3507 | 33 |
| H31A6 | -1005 | 11214 | 4142 | 66 |
| H31B6 | -708 | 11690 | 4107 | 66 |
| H31C6 | 98 | 11339 | 4357 | 66 |
| H27 | 8789 | 9245 | 3548 | 30 |
| H3A7 | 9908 | 9165 | 5036 | 54 |
| H3B7 | 9655 | 8728 | 4509 | 54 |
| H3C7 | 9199 | 8851 | 5367 | 54 |
| H17 | 8045 | 8593 | 3484 | 29 |
| H11A7 | 6735 | 9280 | 3087 | 39 |
| H11B7 | 6588 | 9221 | 3188 | 39 |
| H12A7 | 6889 | 9209 | 1524 | 43 |
| H12B7 | 7949 | 9078 | 2108 | 43 |
| H13A7 | 6877 | 8574 | 875 | 39 |
| H13B7 | 7685 | 8401 | 1768 | 39 |
| H14A7 | 6573 | 8132 | 2376 | 32 |
| H14B7 | 5766 | 8297 | 1473 | 32 |
| H15A7 | 5496 | 8880 | 2244 | 31 |
| H15B7 | 5879 | 8594 | 3172 | 31 |
| H12C7 | 7721 | 9288 | 2174 | 48 |
| H12D7 | 6587 | 9315 | 1698 | 48 |
| H13C7 | 6910 | 8784 | 845 | 49 |
| H13D7 | 7821 | 8643 | 1668 | 49 |
| H14C7 | 6944 | 8165 | 2052 | 47 |
| H14D7 | 6001 | 8349 | 1331 | 47 |
| H15C7 | 6364 | 8411 | 3242 | 43 |
| H15D7 | 5569 | 8687 | 2515 | 43 |
| H227 | 8009 | 9888 | 3317 | 30 |
| H247 | 7431 | 10617 | 3101 | 35 |
| H257 | 7014 | 11219 | 3745 | 37 |
| H277 | 7281 | 10662 | 6299 | 32 |

| | | | | |
|---|---|---|---|---|
| H297 | 7759 | 9909 | 6459 | 32 |
| H307 | 8256 | 9320 | 5843 | 34 |
| H31A7 | 6471 | 11236 | 6518 | 59 |
| H31B7 | 6805 | 11708 | 6503 | 59 |
| H31C7 | 7584 | 11347 | 6770 | 59 |
| H28 | 4858 | 2003 | 2604 | 34 |
| H3A8 | 6362 | 2049 | 3738 | 58 |
| H3B8 | 5827 | 2487 | 3573 | 58 |
| H3C8 | 6687 | 2395 | 3095 | 58 |
| H18 | 4814 | 2678 | 1965 | 31 |
| H11A8 | 4575 | 2126 | 367 | 44 |
| H11B8 | 4473 | 2058 | 471 | 44 |
| H12A8 | 3574 | 1926 | 1444 | 49 |
| H12B8 | 3074 | 1973 | 323 | 49 |
| H13A8 | 3011 | 2538 | 1799 | 52 |
| H13B8 | 2156 | 2415 | 895 | 52 |
| H14A8 | 2535 | 2930 | 34 | 40 |
| H14B8 | 3287 | 3090 | 984 | 40 |
| H15A8 | 4404 | 2924 | 244 | 44 |
| H15B8 | 3677 | 2638 | -523 | 44 |
| H12C8 | 2902 | 2044 | 711 | 55 |
| H12D8 | 3506 | 2130 | 1789 | 55 |
| H13C8 | 3084 | 2796 | 1731 | 49 |
| H13D8 | 2181 | 2636 | 917 | 49 |
| H14C8 | 2678 | 2984 | -176 | 51 |
| H14D8 | 3543 | 3167 | 647 | 51 |
| H15C8 | 3591 | 2506 | -593 | 47 |
| H15D8 | 4451 | 2809 | -86 | 47 |
| H228 | 4673 | 1364 | 1858 | 32 |
| H248 | 4491 | 632 | 1271 | 36 |
| H258 | 5158 | 42 | 810 | 40 |
| H278 | 7655 | 650 | 990 | 33 |
| H298 | 7787 | 1393 | 1488 | 33 |
| H308 | 7154 | 1972 | 2025 | 32 |
| H31A8 | 7939 | 83 | 207 | 57 |
| H31B8 | 7918 | -398 | 467 | 57 |
| H31C8 | 8182 | -59 | 1298 | 57 |

JC7061 1H CDCl3

exp1 s2pul

| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Jan 7 2012 | dfrq | 125.844 |
| solvent | CDCl3 | dn | C13 |
| file | /data/export/~ | dpwr | 30 |
| | home/gfu/FUjcho/ca~ | dof | 0 |
| | sper/JC7061_1H_CDC~ | dm | nnn |
| | l3.fid | dmm | c |
| ACQUISITION | | dmf | 200 |
| sfrq | 500.431 | dseq | |
| tn | H1 | dres | 1.0 |
| at | 4.999 | homo | n |
| np | 120102 | PROCESSING | |
| sw | 12012.0 | wtfile | |
| fb | not used | proc | ft |
| bs | 8 | fn | 262144 |
| tpwr | 60 | math | f |
| pw | 8.0 | | |
| d1 | 0.100 | werr | |
| tof | 3003.2 | wexp | |
| nt | 16 | wbs | |
| ct | 16 | wnt | |
| alock | n | | |
| gain | not used | | |
| FLAGS | | | |
| il | n | | |
| in | n | | |
| dp | y | | |
| hs | nn | | |
| DISPLAY | | | |
| sp | 45.1 | | |
| wp | 4954.0 | | |
| vs | 38 | | |
| sc | 0 | | |
| wc | 250 | | |
| hzmm | 19.82 | | |
| is | 33.57 | | |
| rfl | 4148.0 | | |
| rfp | 3633.1 | | |
| th | 7 | | |
| ins | 1.000 | | |
| ai cdc ph | | | |

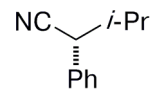



Table 2, entry 1

JC6205 1H CDCl3

exp1 s2pul

| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Sep 24 2011 | dfrq | 125.844 |
| solvent | CDCl3 | dn | C13 |
| file | /data/export/~ | dpwr | 30 |
| | home/gfu/FUjcho/ca~ | dof | 0 |
| | sper/JC6205_1H_CDC~ | dm | nnn |
| | 13.fid | dmm | c |
| ACQUISITION | | dmf | 200 |
| sfrq | 500.431 | dseq | |
| tn | H1 | dres | 1.0 |
| at | 4.999 | homo | n |
| np | 120102 | PROCESSING | |
| sw | 12012.0 | wtfile | |
| fb | not used | proc | ft |
| bs | 8 | fn | 262144 |
| tpwr | 60 | math | f |
| pw | 8.0 | | |
| d1 | 0.100 | werr | |
| tof | 3003.2 | wexp | |
| nt | 16 | wbs | |
| ct | 16 | wnt | |
| alock | n | | |
| gain | not used | | |
| FLAGS | | | |
| il | n | | |
| in | n | | |
| dp | y | | |
| hs | nn | | |
| DISPLAY | | | |
| sp | 42.0 | | |
| wp | 4961.5 | | |
| vs | 46 | | |
| sc | 0 | | |
| wc | 250 | | |
| hzmm | 19.85 | | |
| is | 33.57 | | |
| rfl | 4149.1 | | |
| rfp | 3633.1 | | |
| th | 7 | | |
| ins | 1.000 | | |
| ai cdc ph | | | |

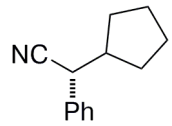
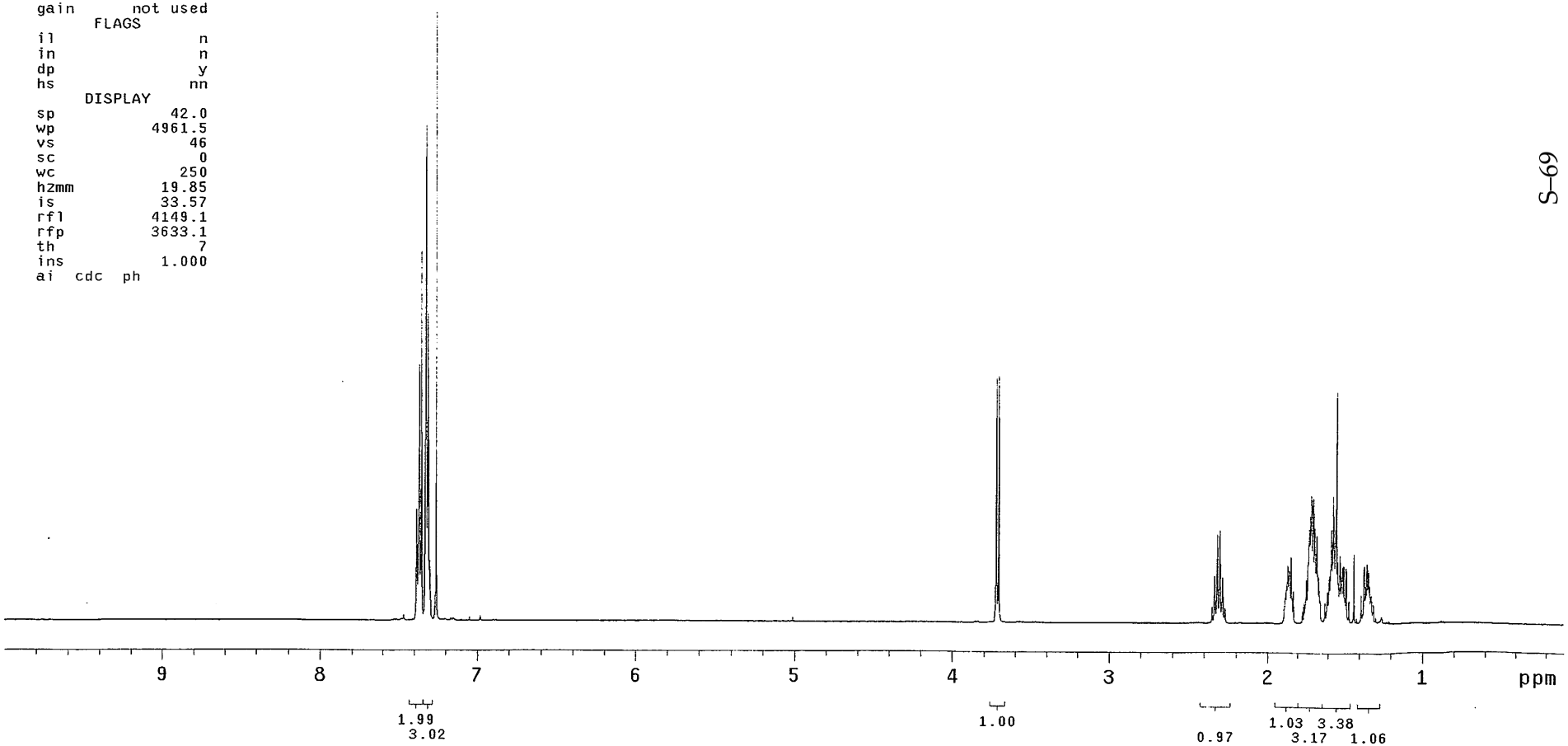



Table 2, entry 2



JC7189B 1H CDCl3

exp2 s2pul

| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Feb 22 2012 | dfrq | 125.672 |
| solvent | CDCl3 | dn | C13 |
| file | /data/export/~ | dpwr | 30 |
| | home/gfu/FUjcho/bu~ | dof | 0 |
| | llwinkle/JC7189B_1~ | dm | nnn |
| | H_CDCl3.fid | dmm | w |
| ACQUISITION | | dmf | 10000 |
| sfrq | 499.746 | dseq | |
| tn | H1 | dres | 1.0 |
| at | 3.001 | homo | n |
| np | 63050 | PROCESSING | |
| sw | 10504.2 | wtfile | |
| fb | not used | proc | ft |
| bs | 8 | fn | 262144 |
| tpwr | 56 | math | f |
| pw | 8.6 | | |
| d1 | 2.000 | werr | |
| tof | 1519.5 | wexp | |
| nt | 16 | wbs | |
| ct | 16 | wnt | wft |
| alock | n | | |
| gain | not used | | |
| FLAGS | | | |
| il | n | | |
| in | n | | |
| dp | y | | |
| hs | nn | | |
| DISPLAY | | | |
| sp | 40.8 | | |
| wp | 4958.9 | | |
| vs | 93 | | |
| sc | 0 | | |
| wc | 250 | | |
| hzmm | 19.84 | | |
| is | 33.57 | | |
| rfl | 4866.4 | | |
| rfp | 3628.1 | | |
| th | 7 | | |
| ins | 1.000 | | |
| ai cdc ph | | | |

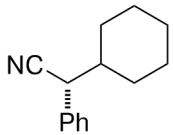
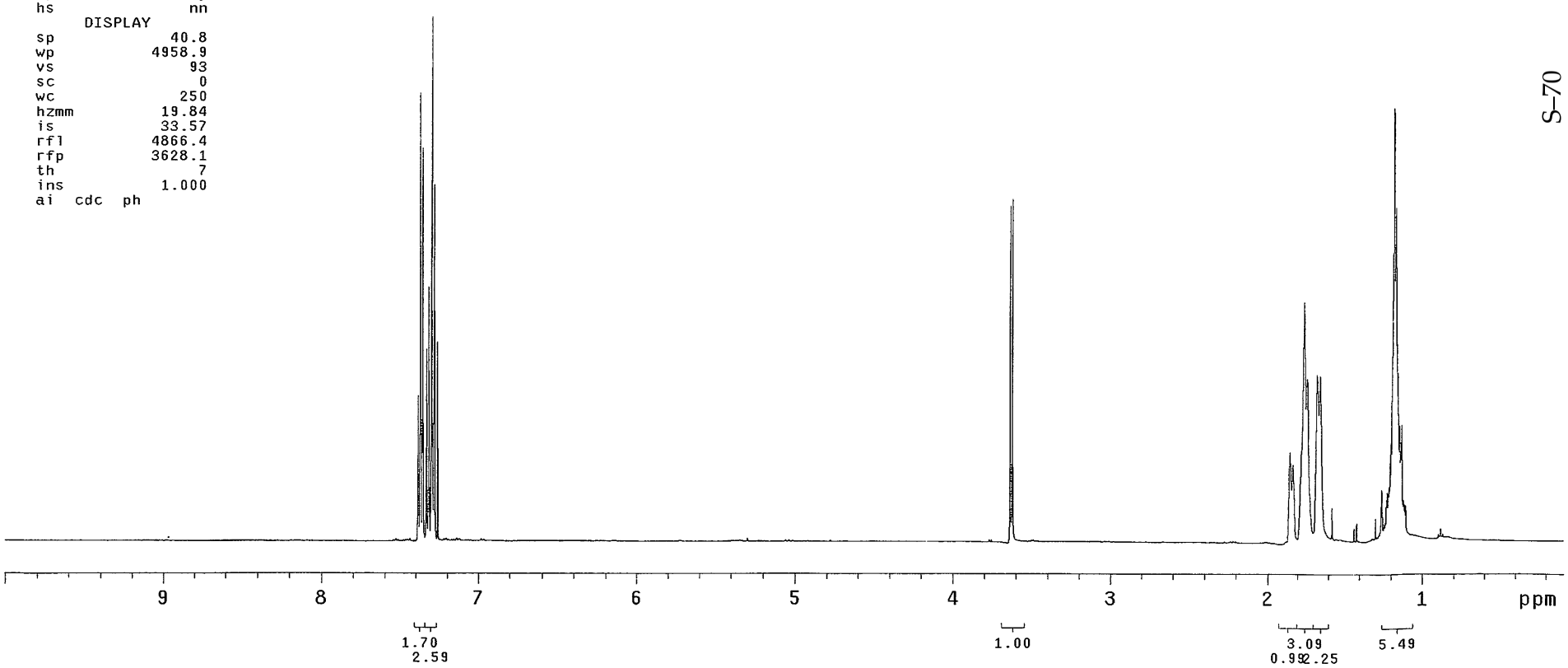



Table 2, entry 3



JC7243A 1H CDCl3

exp1 s2pul

| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Mar 15 2012 | dfrq | 125.672 |
| solvent | CDCl3 | dn | C13 |
| file | /data/export/~ | dpwr | 30 |
| | home/gfu/FUjcho/bu~ | dof | 0 |
| | llwinkle/JC7243A_1~ | dm | nnn |
| | H_CDCl3.fid | dmm | w |
| ACQUISITION | | dmf | 10000 |
| sfrq | 499.746 | dseq | |
| tn | H1 | dres | 1.0 |
| at | 3.001 | homo | n |
| np | 63050 | temp | 22.0 |
| sw | 10504.2 | PROCESSING | |
| fb | not used | wtfile | |
| bs | 8 | proc | ft |
| tpwr | 56 | fn | 262144 |
| pw | 8.6 | math | f |
| d1 | 2.000 | | |
| tof | 1519.5 | werr | |
| nt | 16 | wexp | |
| ct | 16 | wbs | |
| alock | n | wnt | wft |
| gain | not used | | |
| FLAGS | | | |
| il | n | | |
| in | n | | |
| dp | y | | |
| hs | nn | | |
| DISPLAY | | | |
| sp | 39.9 | | |
| wp | 4960.9 | | |
| vs | 57 | | |
| sc | 0 | | |
| wc | 250 | | |
| hzmm | 19.84 | | |
| is | 33.57 | | |
| rfl | 4865.4 | | |
| rfp | 3628.1 | | |
| th | 7 | | |
| ins | 1.000 | | |
| ai cdc ph | | | |

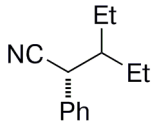



Table 2, entry 4

JC7211B 1H CDCl3

exp1 s2pul

| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Feb 28 2012 | dfrq | 125.844 |
| solvent | CDCl3 | dn | C13 |
| file | /data/export/~ | dpwr | 30 |
| | home/gfu/FUjcho/ca~ | dof | 0 |
| | sper/JC7211B_1H_CD~ | dm | nnn |
| | C13.fid | dmm | c |
| ACQUISITION | | dmf | 200 |
| sfrq | 500.431 | dseq | |
| tn | H1 | dres | 1.0 |
| at | 4.999 | homo | n |
| np | 120102 | PROCESSING | |
| sw | 12012.0 | wtfile | |
| fb | not used | proc | ft |
| bs | 8 | fn | 262144 |
| tpwr | 60 | math | f |
| pw | 8.0 | | |
| d1 | 0.100 | werr | |
| tof | 3003.2 | wexp | |
| nt | 16 | wbs | |
| ct | 16 | wnt | |
| alock | n | | |
| gain | not used | | |
| FLAGS | | | |
| il | n | | |
| in | n | | |
| dp | y | | |
| hs | nn | | |
| DISPLAY | | | |
| sp | 51.4 | | |
| wp | 4947.8 | | |
| vs | 50 | | |
| sc | 0 | | |
| wc | 250 | | |
| hzmm | 19.79 | | |
| is | 176.67 | | |
| rfl | 4148.0 | | |
| rfp | 3633.1 | | |
| th | 7 | | |
| ins | 1.000 | | |
| ai cdc ph | | | |

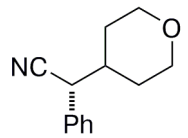
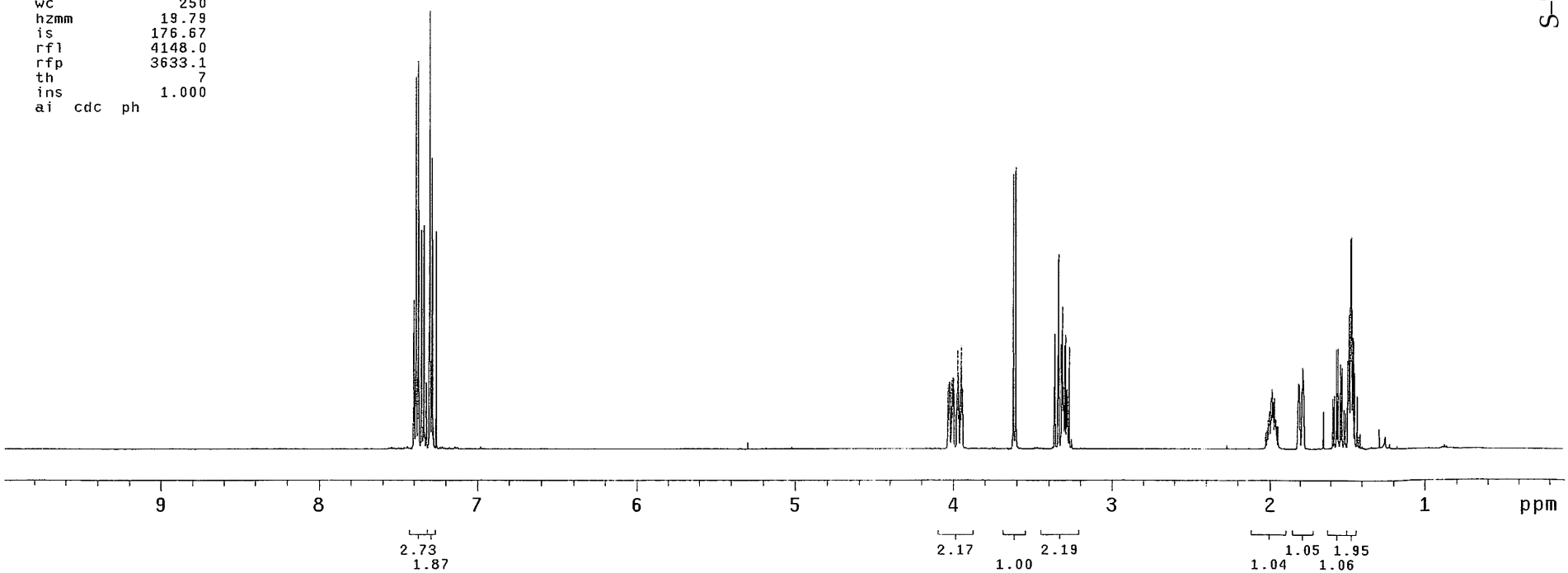



Table 2, entry 5



JC7229A 1H CDCl3

exp1 s2pul

| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Mar 11 2012 | dfrq | 125.844 |
| solvent | CDCl3 | dn | C13 |
| file | /data/export/~ | dpwr | 30 |
| | home/gfu/FUjcho/ca~ | dof | 0 |
| | sper/JC7229A_1H_CD~ | dm | nnn |
| | C13.fid | dmm | c |
| ACQUISITION | | dmf | 200 |
| sfrq | 500.431 | dseq | |
| tn | H1 | dres | 1.0 |
| at | 4.999 | homo | n |
| np | 120102 | PROCESSING | |
| sw | 12012.0 | wtfile | |
| fb | not used | proc | ft |
| bs | 8 | fn | 262144 |
| tpwr | 60 | math | f |
| pw | 8.0 | | |
| d1 | 0.100 | werr | |
| tof | 3003.2 | wexp | |
| nt | 16 | wbs | |
| ct | 16 | wnt | |
| alock | n | | |
| gain | not used | | |
| FLAGS | | | |
| il | n | | |
| in | n | | |
| dp | y | | |
| hs | nn | | |
| DISPLAY | | | |
| sp | 47.5 | | |
| wp | 4956.6 | | |
| vs | 92 | | |
| sc | 0 | | |
| wc | 250 | | |
| hzmm | 19.83 | | |
| is | 33.57 | | |
| rfl | 4148.6 | | |
| rfp | 3633.1 | | |
| th | 7 | | |
| ins | 1.000 | | |
| ai cdc ph | | | |

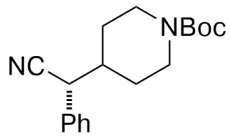



Table 2, entry 6

JC7257A 1H CDCl3

exp2 s2pul

| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Mar 27 2012 | dfrq | 125.672 |
| solvent | CDCl3 | dn | C13 |
| file | exp | dpwr | 30 |
| ACQUISITION | | dof | 0 |
| sfrq | 499.746 | dm | nnn |
| tn | H1 | dmm | w |
| at | 3.001 | dmf | 10000 |
| np | 63050 | dseq | |
| sw | 10504.2 | dres | 1.0 |
| fb | not used | homo | n |
| bs | 8 | DEC2 | |
| tpwr | 56 | dfrq2 | 0 |
| pw | 8.6 | dn2 | |
| d1 | 2.000 | dpwr2 | 1 |
| tof | 1519.5 | dof2 | 0 |
| nt | 16 | dm2 | n |
| ct | 16 | dmm2 | c |
| alock | n | dmf2 | 200 |
| gain | not used | dseq2 | |
| FLAGS | | dres2 | 1.0 |
| il | n | homo2 | n |
| in | n | DEC3 | |
| dp | y | dfrq3 | 0 |
| hs | nn | dn3 | |
| DISPLAY | | dpwr3 | 1 |
| sp | 48.4 | dof3 | 0 |
| wp | 4952.3 | dm3 | n |
| vs | 83 | dmm3 | c |
| sc | 0 | dmf3 | 200 |
| wc | 250 | dseq3 | |
| hzmm | 19.81 | dres3 | 1.0 |
| is | 33.57 | homo3 | n |
| rfl | 4865.7 | PROCESSING | |
| rfp | 3628.1 | wtfile | |
| th | 7 | proc | ft |
| ins | 1.000 | fn | 262144 |
| ai cdc ph | | math | f |
| | | werr | |
| | | wexp | |
| | | wbs | |
| | | wnt | wft |

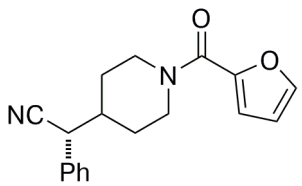
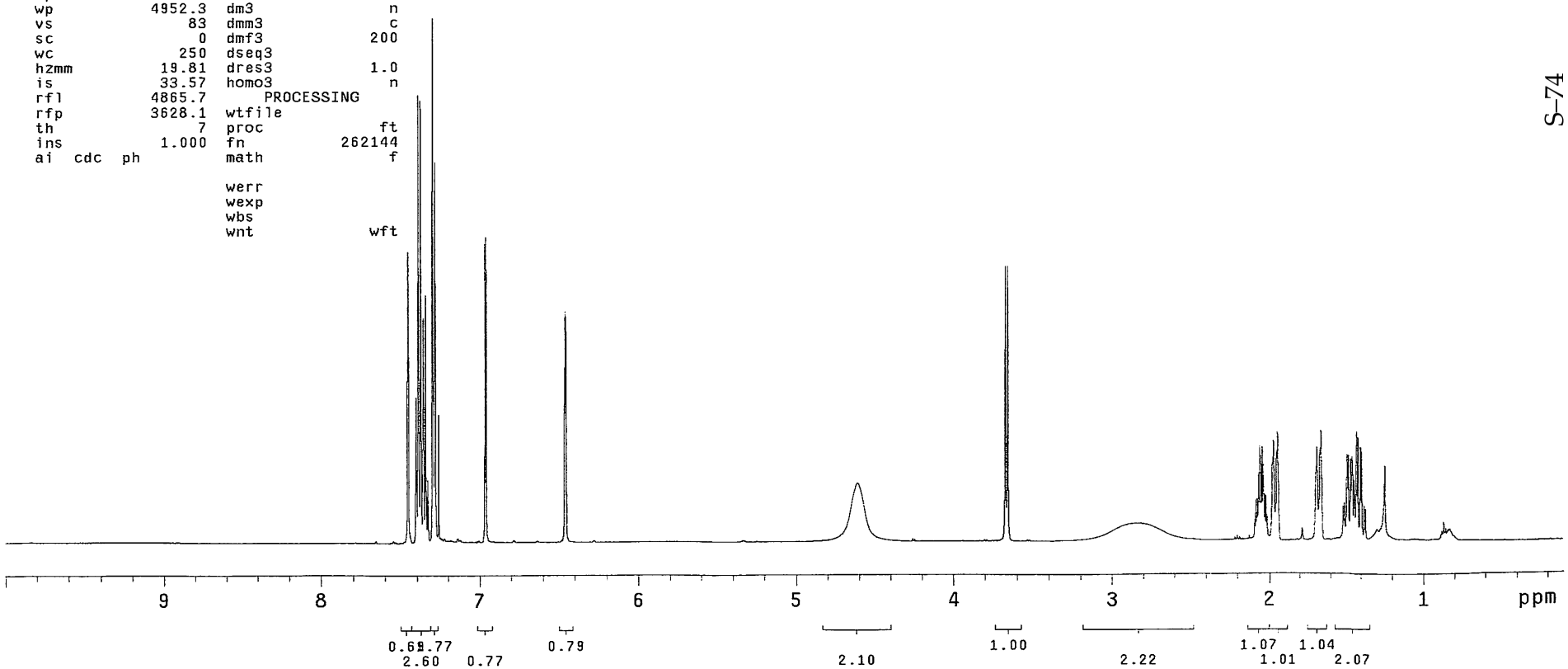



Table 2, entry 7



JC7255A 1H CDCl3

exp1 s2pul

| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Mar 23 2012 | dfrq | 125.844 |
| solvent | CDCl3 | dn | C13 |
| file | exp | dpwr | 30 |
| ACQUISITION | | dof | 0 |
| sfrq | 500.431 | dm | nnn |
| tn | H1 | dmm | c |
| at | 4.999 | dmf | 200 |
| np | 120102 | dseq | |
| sw | 12012.0 | dres | 1.0 |
| fb | not used | homo | n |
| bs | 8 | PROCESSING | |
| tpwr | 60 | wtfile | |
| pw | 8.0 | proc | ft |
| d1 | 0.100 | fn | 262144 |
| tof | 3003.2 | math | f |
| nt | 16 | | |
| ct | 16 | werr | |
| alock | n | wexp | |
| gain | not used | wbs | |
| FLAGS | | wnt | |
| il | n | | |
| in | n | | |
| dp | y | | |
| hs | nn | | |
| DISPLAY | | | |
| sp | 61.2 | | |
| wp | 4947.8 | | |
| vs | 42 | | |
| sc | 0 | | |
| wc | 250 | | |
| hzmm | 19.79 | | |
| is | 33.57 | | |
| rfl | 4148.2 | | |
| rfp | 3633.1 | | |
| th | 7 | | |
| ins | 1.000 | | |
| ai cdc ph | | | |

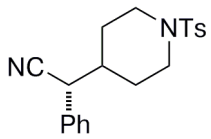



Table 2, entry 8

JC7231B 1H CDCl3

exp1 s2pul

| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Mar 15 2012 | dfrq | 125.844 |
| solvent | CDCl3 | dn | C13 |
| file | /data/export/~ | dpwr | 30 |
| home/gfu/FUjcho/ca~ | | dof | 0 |
| sper/JC7231B_1H_CD~ | | dm | nnn |
| | Cl3.fid | dmm | c |
| ACQUISITION | | dmf | 200 |
| sfrq | 500.431 | dseq | |
| tn | H1 | dres | 1.0 |
| at | 4.999 | homo | n |
| np | 120102 | PROCESSING | |
| sw | 12012.0 | wtfile | |
| fb | not used | proc | ft |
| bs | 8 | fn | 262144 |
| tpwr | 60 | math | f |
| pw | 8.0 | | |
| d1 | 0.100 | werr | |
| tof | 3003.2 | wexp | |
| nt | 8 | wbs | |
| ct | 8 | wnt | |
| alock | n | | |
| gain | not used | | |
| FLAGS | | | |
| il | n | | |
| in | n | | |
| dp | y | | |
| hs | nn | | |
| DISPLAY | | | |
| sp | 35.2 | | |
| wp | 4969.1 | | |
| vs | 76 | | |
| sc | 0 | | |
| wc | 250 | | |
| hzmm | 19.88 | | |
| is | 33.57 | | |
| rfl | 4148.4 | | |
| rfp | 3633.1 | | |
| th | 7 | | |
| ins | 2.000 | | |
| ai cdc ph | | | |

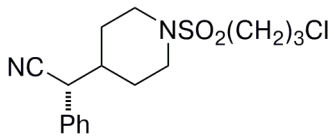
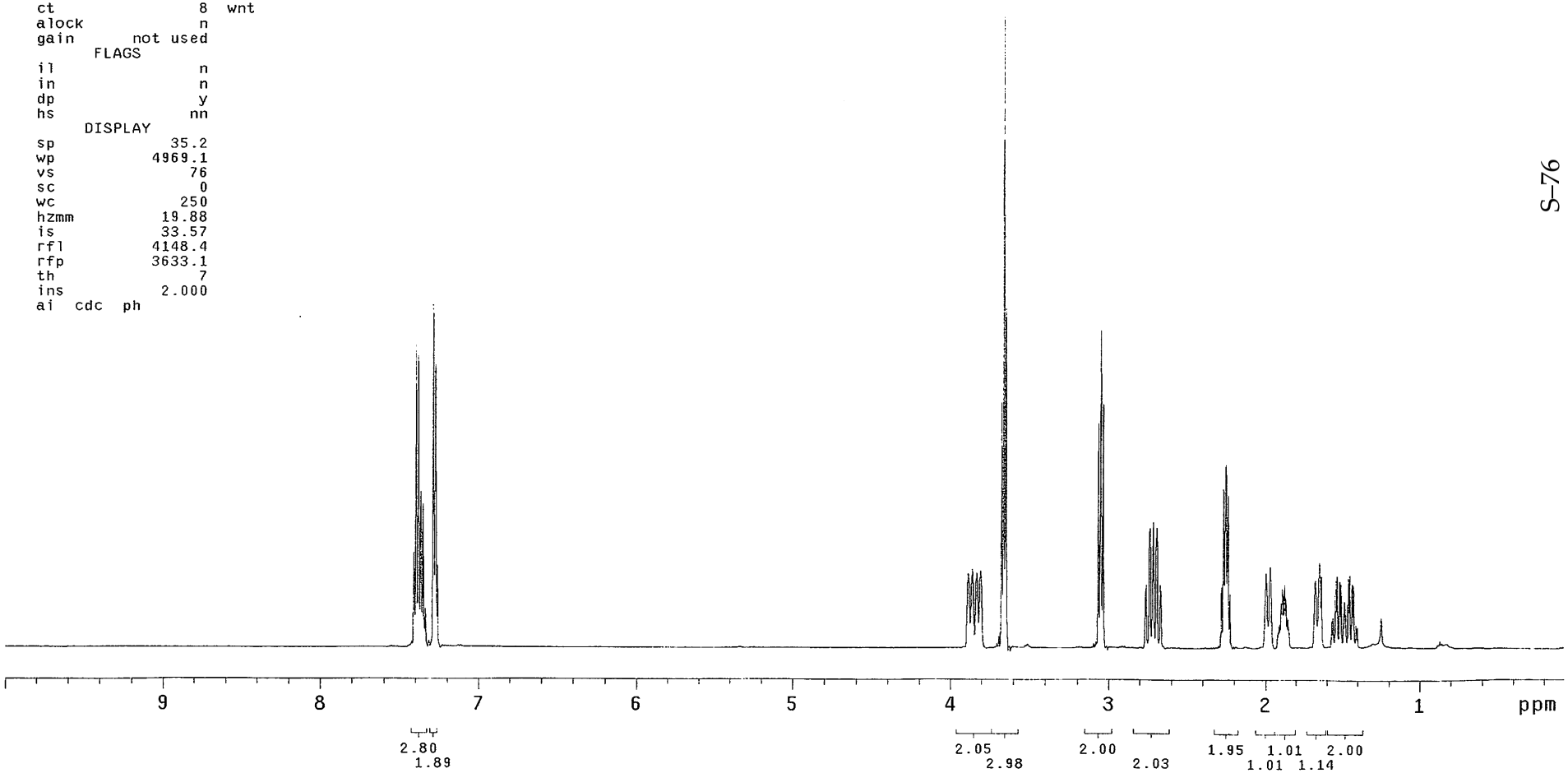



Table 2, entry 9



JC7183B 1H CDCl3

exp1 s2pul

| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Feb 20 2012 | dfrq | 125.844 |
| solvent | CDCl3 | dn | C13 |
| file | /data/export/~ | dpwr | 30 |
| home/gfu/FUjcho/ca~ | | dof | 0 |
| sper/JC7183B_1H_CD~ | | dm | nnn |
| | Cl3.fid | dmm | c |
| ACQUISITION | | dmf | 200 |
| sfrq | 500.431 | dseq | |
| tn | H1 | dres | 1.0 |
| at | 4.999 | homo | n |
| np | 120102 | PROCESSING | |
| sw | 12012.0 | wtfile | |
| fb | not used | proc | ft |
| bs | 8 | fn | 262144 |
| tpwr | 60 | math | f |
| pw | 8.0 | | |
| d1 | 0.100 | werr | |
| tof | 3003.2 | wexp | |
| nt | 16 | wbs | |
| ct | 16 | wnt | |
| alock | n | | |
| gain | not used | | |
| FLAGS | | | |
| il | n | | |
| in | n | | |
| dp | y | | |
| hs | nn | | |
| DISPLAY | | | |
| sp | 37.1 | | |
| wp | 4961.5 | | |
| vs | 14 | | |
| sc | 0 | | |
| wc | 250 | | |
| hzmm | 19.85 | | |
| is | 33.57 | | |
| rfl | 515.5 | | |
| rfp | 0 | | |
| th | 7 | | |
| ins | 1.000 | | |

ai cdc ph

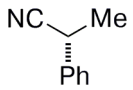



Table 2, entry 10

JC7197B 1H CDCl3

exp1 s2pul

| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Feb 23 2012 | dfrq | 125.844 |
| solvent | CDCl3 | dn | C13 |
| file | /data/export/~ | dpwr | 30 |
| | home/gfu/FUjcho/ca~ | dof | 0 |
| | sper/JC7197B_1H_CD~ | dm | nnn |
| | C13.fid | dmm | c |
| ACQUISITION | | dmf | 200 |
| sfrq | 500.431 | dseq | |
| tn | H1 | dres | 1.0 |
| at | 4.999 | homo | n |
| np | 120102 | PROCESSING | |
| sw | 12012.0 | wtfile | |
| fb | not used | proc | ft |
| bs | 8 | fn | 262144 |
| tpwr | 60 | math | f |
| pw | 8.0 | | |
| d1 | 0.100 | werr | |
| tof | 3003.2 | wexp | |
| nt | 16 | wbs | |
| ct | 16 | wnt | |
| alock | n | | |
| gain | not used | | |
| FLAGS | | | |
| il | n | | |
| in | n | | |
| dp | y | | |
| hs | nn | | |
| DISPLAY | | | |
| sp | 40.7 | | |
| wp | 4967.7 | | |
| vs | 64 | | |
| sc | 0 | | |
| wc | 250 | | |
| hzmm | 19.87 | | |
| is | 33.57 | | |
| rfl | 4148.8 | | |
| rfp | 3633.1 | | |
| th | 7 | | |
| ins | 1.000 | | |
| ai cdc ph | | | |

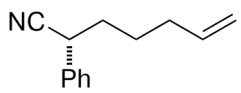
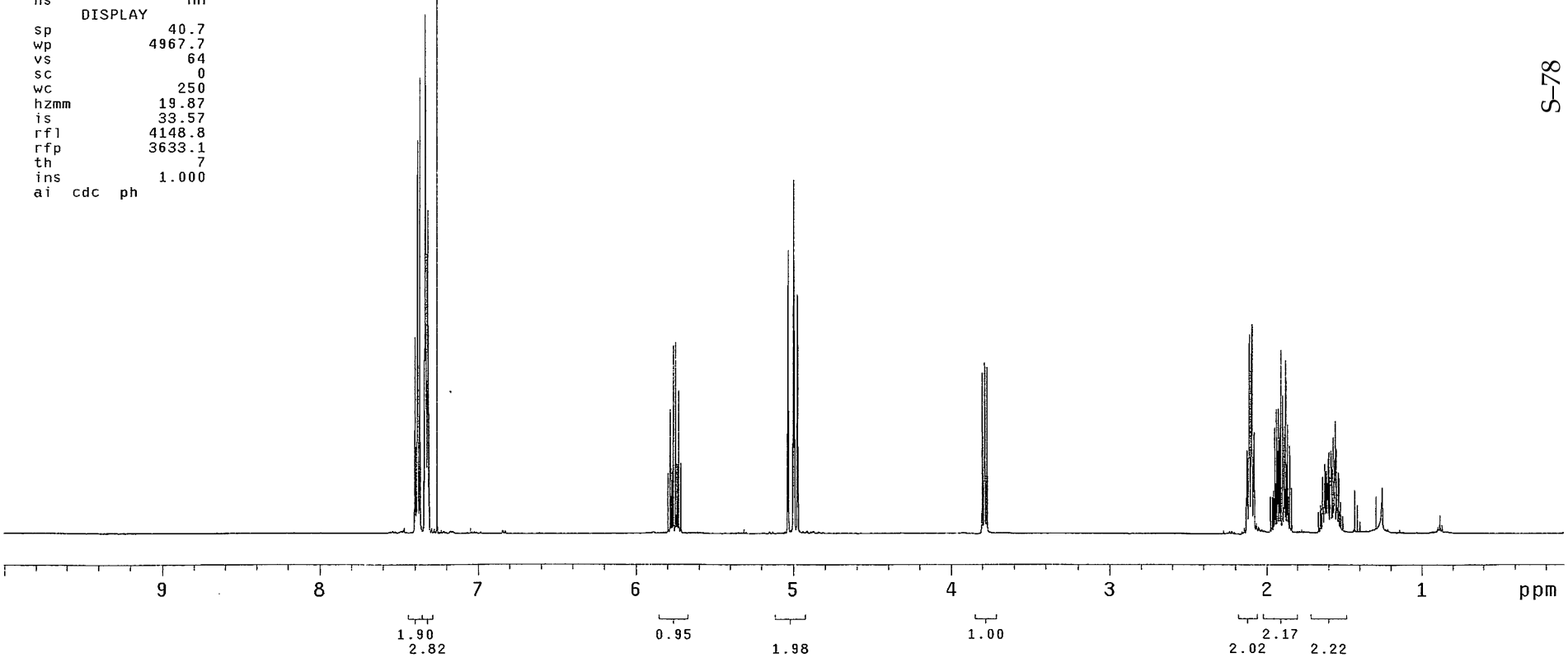



Table 2, entry 11



JC7203B_1H_CDCl3

expl s2pul

| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Feb 24 2012 | dfrq | 125.844 |
| solvent | CDCl3 | dn | C13 |
| file | /data/export/~ | dpwr | 30 |
| | home/gfu/FUjcho/ca~ | dof | 0 |
| | sper/JC7203B_1H_CD~ | dm | nnn |
| | C13.fid | dmm | c |
| ACQUISITION | | dmf | 200 |
| sfrq | 500.431 | dseq | |
| tn | H1 | dres | 1.0 |
| at | 4.999 | homo | n |
| np | 120102 | PROCESSING | |
| sw | 12012.0 | wtfile | |
| fb | not used | proc | ft |
| bs | 8 | fn | 262144 |
| tpwr | 60 | math | f |
| pw | 8.0 | | |
| d1 | 0.100 | werr | |
| tof | 3003.2 | wexp | |
| nt | 16 | wbs | |
| ct | 16 | wnt | |
| alock | n | | |
| gain | not used | | |
| FLAGS | | | |
| il | n | | |
| in | n | | |
| dp | y | | |
| hs | nn | | |
| DISPLAY | | | |
| sp | 50.8 | | |
| wp | 4957.7 | | |
| vs | 54 | | |
| sc | 0 | | |
| wc | 250 | | |
| hzmm | 19.83 | | |
| is | 33.57 | | |
| rfl | 4148.6 | | |
| rfp | 3633.1 | | |
| th | 7 | | |
| ins | 1.000 | | |
| ai cdc ph | | | |

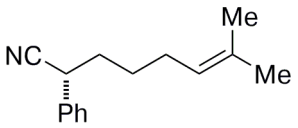



Table 2, entry 12

JC6221A 1H CDCl3

exp1 s2pul

| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Sep 30 2011 | dfrq | 125.844 |
| solvent | CDCl3 | dn | C13 |
| file | /data/export/~ | dpwr | 30 |
| | home/gfu/FUjcho/ca~ | dof | 0 |
| | sper/JC6221A_1H_CD~ | dm | nnn |
| | C13.fid | dmm | c |
| ACQUISITION | | dmf | 200 |
| sfrq | 500.431 | dseq | |
| tn | H1 | dres | 1.0 |
| at | 4.999 | homo | n |
| np | 120102 | PROCESSING | |
| sw | 12012.0 | wtfile | |
| fb | not used | proc | ft |
| bs | 8 | fn | 262144 |
| tpwr | 60 | math | f |
| pw | 8.0 | | |
| d1 | 0.100 | werr | |
| tof | 3003.2 | wexp | |
| nt | 16 | wbs | |
| ct | 16 | wnt | |
| alock | n | | |
| gain | not used | | |
| FLAGS | | | |
| il | n | | |
| in | n | | |
| dp | y | | |
| hs | nn | | |
| DISPLAY | | | |
| sp | 53.5 | | |
| wp | 4949.2 | | |
| vs | 31 | | |
| sc | 0 | | |
| wc | 250 | | |
| hzmm | 19.80 | | |
| is | 33.57 | | |
| rfl | 4149.7 | | |
| rfp | 3633.1 | | |
| th | 7 | | |
| ins | 1.000 | | |
| ai cdc ph | | | |

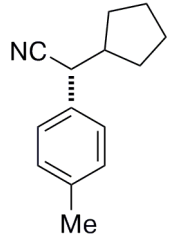



Table 3, entry 1

JC6223A_1H_CDCl3

exp2 s2pul

| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Oct 11 2011 | dfrq | 125.844 |
| solvent | CDCl3 | dn | C13 |
| file | /data/export/~ | dpwr | 30 |
| | home/gfu/FUjcho/ca~ | dof | 0 |
| | sper/JC6223A_1H_CD~ | dm | nnn |
| | Cl3.fid | dmm | c |
| ACQUISITION | | dmf | 200 |
| sfrq | 500.431 | dseq | |
| tn | H1 | dres | 1.0 |
| at | 4.999 | homo | n |
| np | 120102 | PROCESSING | |
| sw | 12012.0 | wtfile | |
| fb | not used | proc | ft |
| bs | 8 | fn | 262144 |
| tpwr | 60 | math | f |
| pw | 8.0 | | |
| d1 | 0.100 | werr | |
| tof | 3003.2 | wexp | |
| nt | 16 | wbs | |
| ct | 16 | wnt | |
| alock | n | | |
| gain | not used | | |
| FLAGS | | | |
| il | n | | |
| in | n | | |
| dp | y | | |
| hs | nn | | |
| DISPLAY | | | |
| sp | 0.8 | | |
| wp | 5004.9 | | |
| vs | 31 | | |
| sc | 0 | | |
| wc | 250 | | |
| hzmm | 20.02 | | |
| is | 33.57 | | |
| rfl | 4149.0 | | |
| rfp | 3633.1 | | |
| th | 7 | | |
| ins | 1.000 | | |
| ai cdc ph | | | |

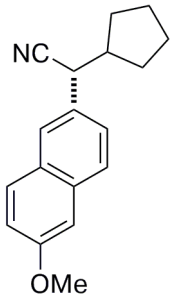



Table 3, entry 2

JC6217A 1H CDCl3

exp1 s2pul

| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Sep 30 2011 | dfrq | 125.844 |
| solvent | CDCl3 | dn | C13 |
| file | /data/export/~ | dpwr | 30 |
| | home/gfu/FUjcho/ca~ | dof | 0 |
| | sper/JC6217A_1H_CD~ | dm | nnn |
| | CT3.fid | dmm | c |
| ACQUISITION | | dmf | 200 |
| sfrq | 500.431 | dseq | |
| tn | H1 | dres | 1.0 |
| at | 4.999 | homo | n |
| np | 120102 | PROCESSING | |
| sw | 12012.0 | wtfile | |
| fb | not used | proc | ft |
| bs | 8 | fn | 262144 |
| tpwr | 60 | math | f |
| pw | 8.0 | | |
| d1 | 0.100 | werr | |
| tof | 3003.2 | wexp | |
| nt | 16 | wbs | |
| ct | 16 | wnt | |
| alock | n | | |
| gain | not used | | |
| FLAGS | | | |
| il | n | | |
| in | n | | |
| dp | y | | |
| hs | nn | | |
| DISPLAY | | | |
| sp | 42.9 | | |
| wp | 4954.0 | | |
| vs | 34 | | |
| sc | 0 | | |
| wc | 250 | | |
| hzmm | 19.82 | | |
| is | 33.57 | | |
| rfl | 4148.2 | | |
| rfp | 3633.1 | | |
| th | 7 | | |
| ins | 1.000 | | |
| ai cdc ph | | | |

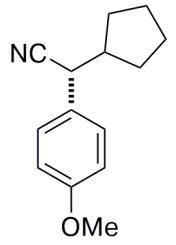



Table 3, entry 3

JC6239A 1H CDCl3

exp1 s2pul

| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Oct 26 2011 | dfrq | 125.844 |
| solvent | CDCl3 | dn | C13 |
| file | /data/export/~ | dpwr | 30 |
| home/gfu/FUjcho/ca~ | | dof | 0 |
| sper/JC6239A_1H_CD~ | | dm | nnn |
| | Cl3.fid | dmm | c |
| ACQUISITION | | dmf | 200 |
| sfrq | 500.431 | dseq | |
| tn | H1 | dres | 1.0 |
| at | 4.999 | homo | n |
| np | 120102 | PROCESSING | |
| sw | 12012.0 | wtfile | |
| fb | not used | proc | ft |
| bs | 8 | fn | 262144 |
| tpwr | 60 | math | f |
| pw | 8.0 | | |
| d1 | 0.100 | werr | |
| tof | 3003.2 | wexp | |
| nt | 16 | wbs | |
| ct | 16 | wnt | |
| alock | n | | |
| gain | not used | | |
| FLAGS | | | |
| il | n | | |
| in | n | | |
| dp | y | | |
| hs | nn | | |
| DISPLAY | | | |
| sp | 44.7 | | |
| wp | 4964.9 | | |
| vs | 64 | | |
| sc | 0 | | |
| wc | 250 | | |
| hzmm | 19.86 | | |
| is | 33.57 | | |
| rfl | 4148.6 | | |
| rfp | 3633.1 | | |
| th | 7 | | |
| ins | 1.000 | | |
| ai cdc ph | | | |

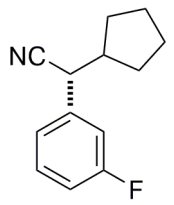
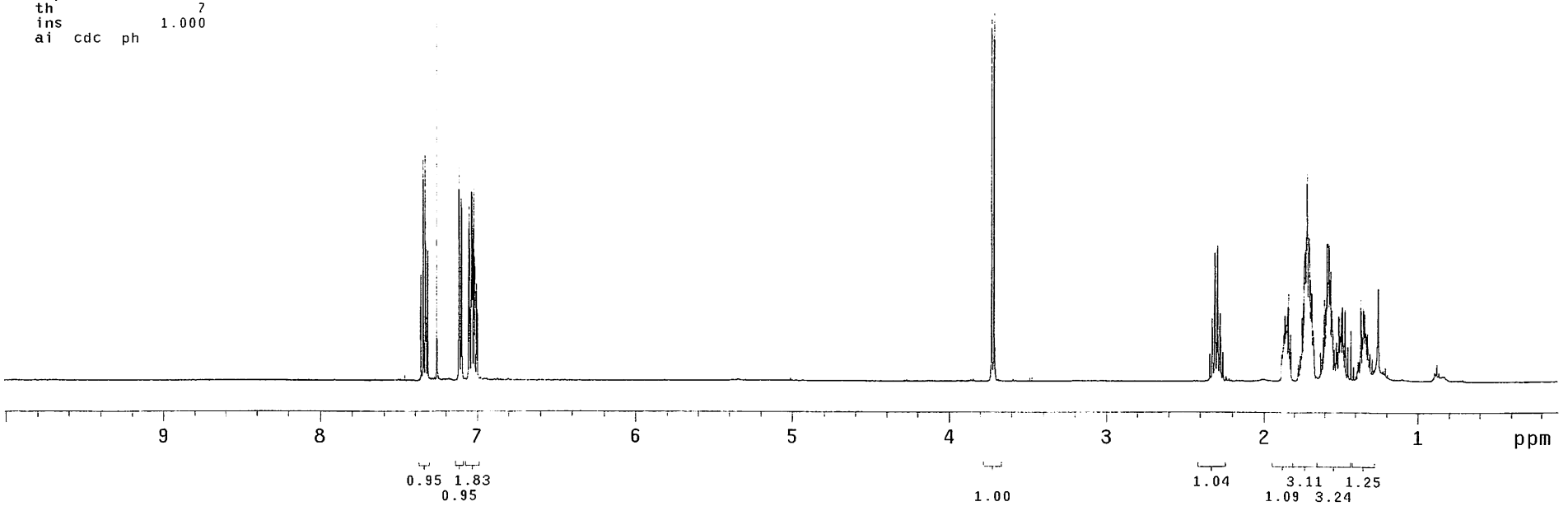



Table 3, entry 4



JC7005A 1H CDCl3

exp1 s2pul

| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Dec 2 2011 | dfrq | 125.844 |
| solvent | CDCl3 | dn | C13 |
| file | /data/export/~ | dpwr | 30 |
| home/gfu/FUjcho/ca~ | | dof | 0 |
| sper/JC7005A_1H_CD~ | | dm | nnn |
| | Cl3.fid | dmm | c |
| ACQUISITION | | dmf | 200 |
| sfrq | 500.431 | dseq | |
| tn | H1 | dres | 1.0 |
| at | 4.999 | homo | n |
| np | 120102 | PROCESSING | |
| sw | 12012.0 | wtfile | |
| fb | not used | proc | ft |
| bs | 8 | fn | 262144 |
| tpwr | 60 | math | f |
| pw | 8.0 | | |
| d1 | 0.100 | werr | |
| tof | 3003.2 | wexp | |
| nt | 16 | wbs | |
| ct | 16 | wnt | |
| alock | n | | |
| gain | not used | | |
| FLAGS | | | |
| il | n | | |
| in | n | | |
| dp | y | | |
| hs | nn | | |
| DISPLAY | | | |
| sp | 40.8 | | |
| wp | 4960.4 | | |
| vs | 82 | | |
| sc | 0 | | |
| wc | 250 | | |
| hzmm | 19.84 | | |
| is | 33.57 | | |
| rfl | 4148.9 | | |
| rfp | 3633.1 | | |
| th | 7 | | |
| ins | 1.000 | | |
| ai cdc ph | | | |

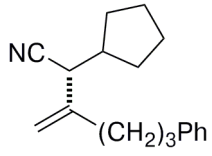
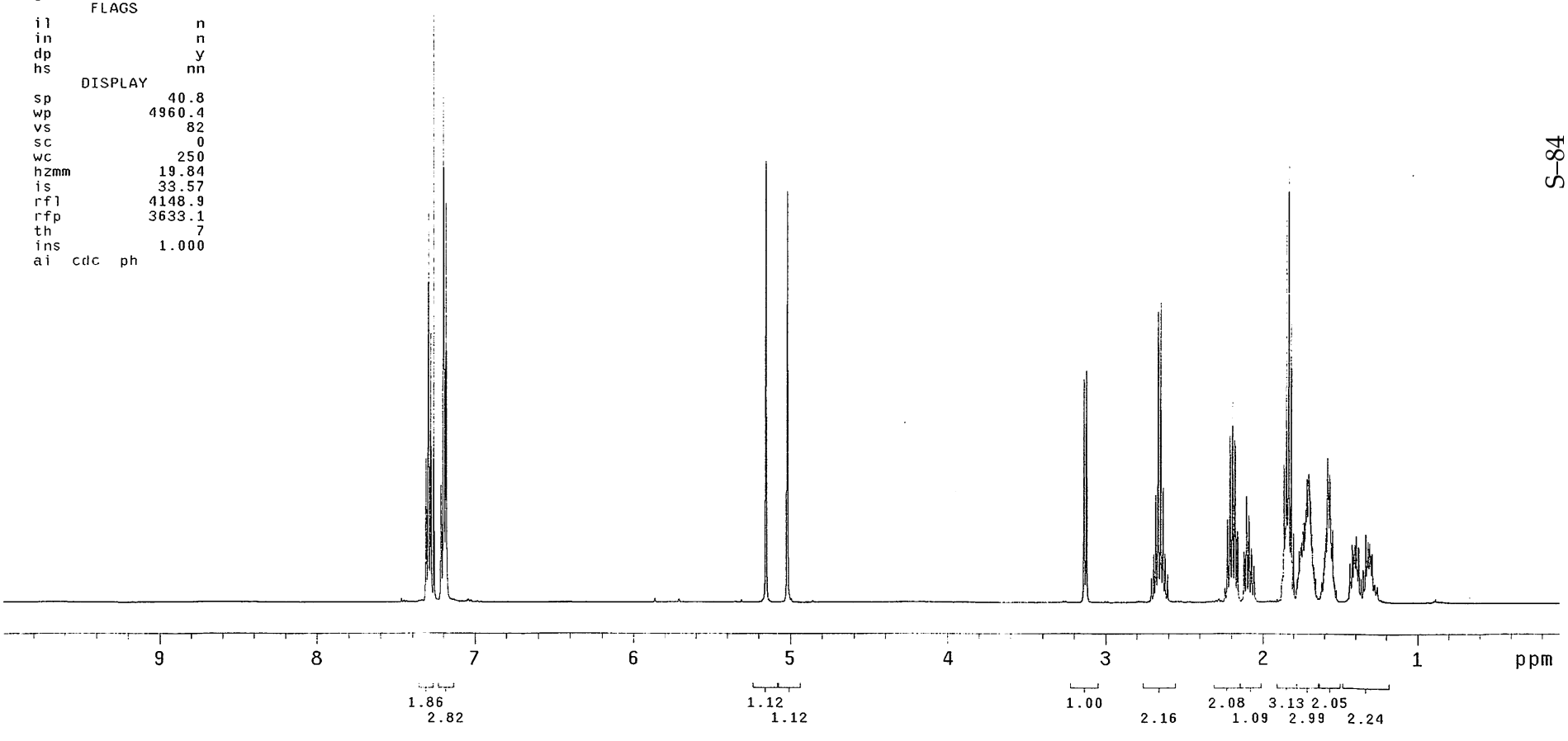



Table 4, entry 1



JC7011A 1H CDCl3

exp1 s2pul

| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Dec 6 2011 | dfrq | 125.844 |
| solvent | CDCl3 | dn | C13 |
| file | /data/export/~ | dpwr | 30 |
| home/gfu/FUjcho/ca~ | | dof | 0 |
| sper/JC7011A_1H_CD~ | | dm | nnn |
| | Cl3.fid | dmm | c |
| ACQUISITION | | dmf | 200 |
| sfrq | 500.431 | dseq | |
| tn | H1 | dres | 1.0 |
| at | 4.999 | homo | n |
| np | 120102 | PROCESSING | |
| sw | 12012.0 | wtfile | |
| fb | not used | proc | ft |
| bs | 8 | fn | 262144 |
| tpwr | 60 | math | f |
| pw | 8.0 | | |
| d1 | 0.100 | werr | |
| tof | 3003.2 | wexp | |
| nt | 16 | wbs | |
| ct | 16 | wnt | |
| alock | n | | |
| gain | not used | | |
| FLAGS | | | |
| il | n | | |
| in | n | | |
| dp | y | | |
| hs | nn | | |
| DISPLAY | | | |
| sp | 50.8 | | |
| wp | 4957.5 | | |
| vs | 122 | | |
| sc | 0 | | |
| wc | 250 | | |
| hzmm | 19.83 | | |
| is | 33.57 | | |
| rfl | 4148.6 | | |
| rfp | 3633.1 | | |
| th | 7 | | |
| ins | 1.000 | | |
| ai cdc ph | | | |

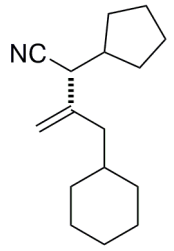
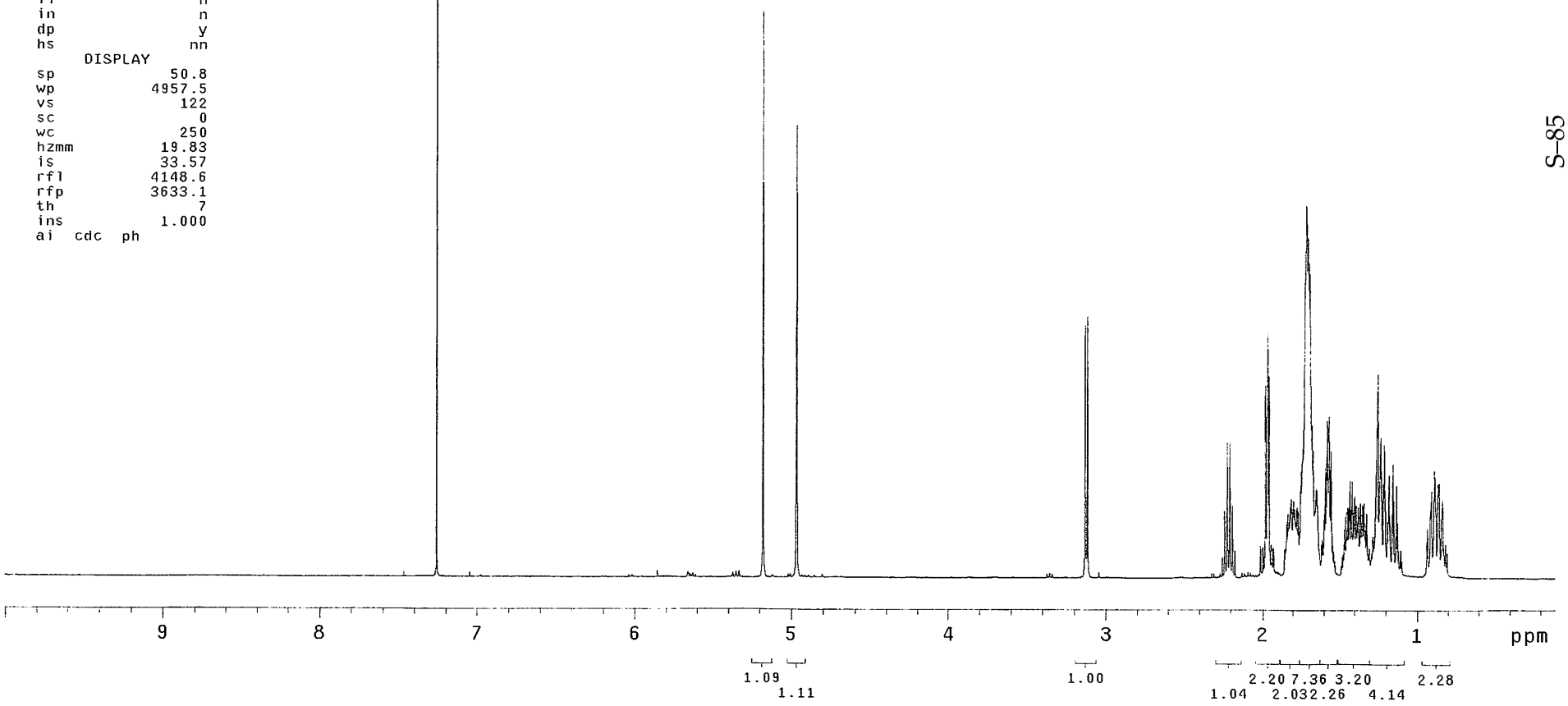



Table 4, entry 2



JC7009A 1H CDCl3

exp1 s2pul

| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Dec 4 2011 | dfrq | 125.844 |
| solvent | CDCl3 | dn | C13 |
| file | /data/export/~ | dpwr | 30 |
| home/gfu/FUjcho/ca~ | | dof | 0 |
| sper/JC7009A_1H_CD~ | | dm | nnn |
| | CT3.fid | dmm | c |
| ACQUISITION | | dmf | 200 |
| sfrq | 500.431 | dseq | |
| tn | H1 | dres | 1.0 |
| at | 4.999 | homo | n |
| np | 120102 | PROCESSING | |
| sw | 12012.0 | wtfile | |
| fb | not used | proc | ft |
| bs | 8 | fn | 262144 |
| tpwr | 60 | math | f |
| pw | 8.0 | | |
| d1 | 0.100 | werr | |
| tof | 3003.2 | wexp | |
| nt | 16 | wbs | |
| ct | 16 | wnt | |
| alock | n | | |
| gain | not used | | |
| FLAGS | | | |
| il | n | | |
| in | n | | |
| dp | y | | |
| hs | nn | | |
| DISPLAY | | | |
| sp | 59.7 | | |
| wp | 4939.5 | | |
| vs | 120 | | |
| sc | 0 | | |
| wc | 250 | | |
| hzmm | 19.76 | | |
| is | 33.57 | | |
| rfl | 4148.6 | | |
| rfp | 3633.1 | | |
| th | 7 | | |
| ins | 1.000 | | |
| ai cdc ph | | | |

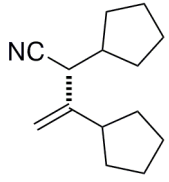
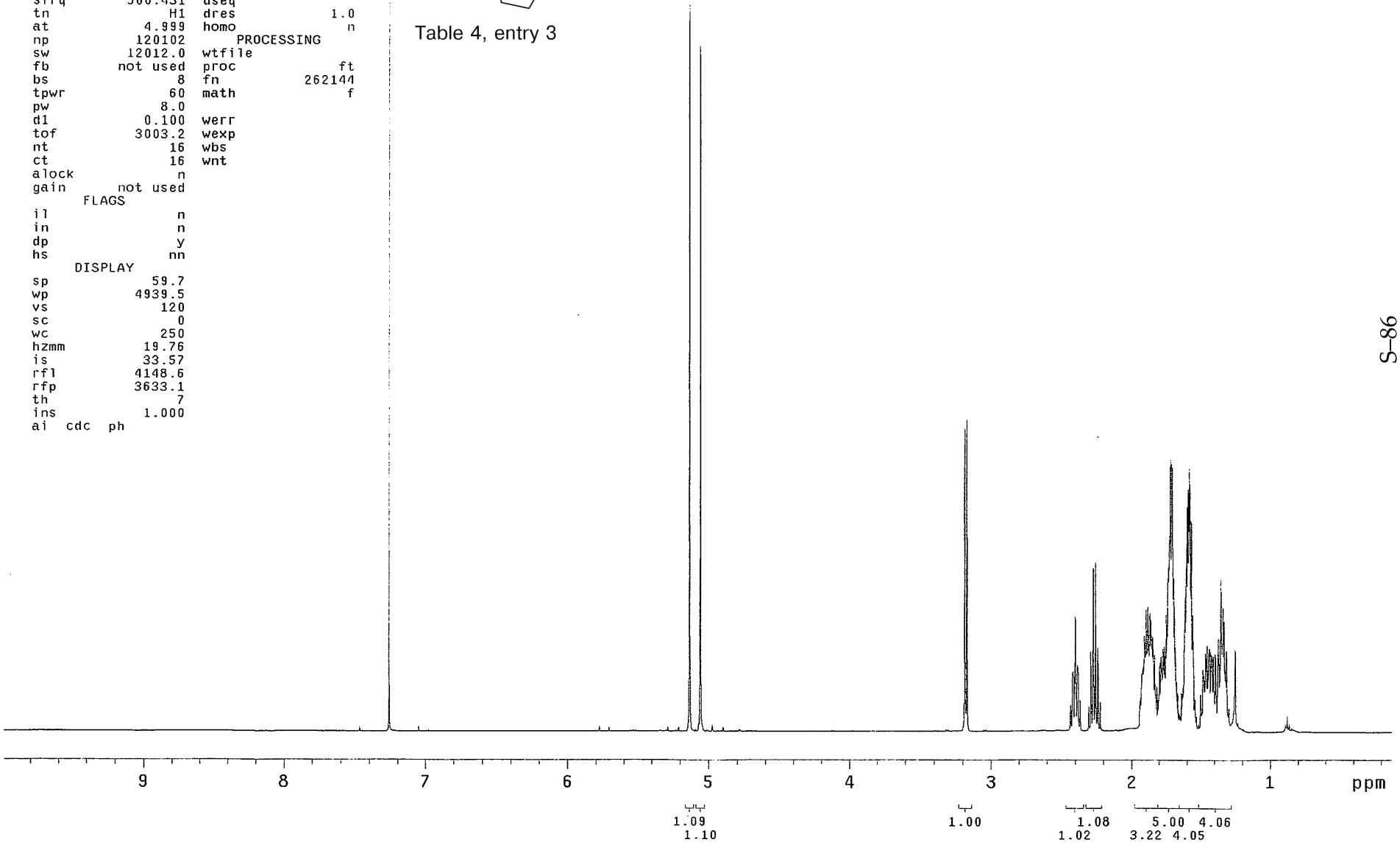



Table 4, entry 3



JC6275B 1H CDCl3

exp1 s2pul

| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Nov 19 2011 | dfrq | 125.844 |
| solvent | CDCl3 | dn | C13 |
| file | /data/export/~ | dpwr | 30 |
| home/gfu/FUjcho/ca~ | | dof | 0 |
| sper/JC6275B_1H_CD~ | | dm | nnn |
| | Cl3.fid | dmm | c |
| ACQUISITION | | dmf | 200 |
| sfrq | 500.431 | dseq | |
| tn | H1 | dres | 1.0 |
| at | 4.999 | homo | n |
| np | 120102 | PROCESSING | |
| sw | 12012.0 | wtfile | |
| fb | not used | proc | ft |
| bs | 8 | fn | 262144 |
| tpwr | 60 | math | f |
| pw | 8.0 | | |
| d1 | 0.100 | werr | |
| tof | 3003.2 | wexp | |
| nt | 16 | wbs | |
| ct | 16 | wnt | |
| alock | n | | |
| gain | not used | | |
| FLAGS | | | |
| il | n | | |
| in | n | | |
| dp | y | | |
| hs | nn | | |
| DISPLAY | | | |
| sp | -4.3 | | |
| wp | 5010.2 | | |
| vs | 33 | | |
| sc | 0 | | |
| wc | 250 | | |
| hzmm | 20.04 | | |
| is | 33.57 | | |
| rfl | 4147.8 | | |
| rfp | 3633.1 | | |
| th | 79 | | |
| ins | 1.000 | | |
| ai cdc ph | | | |

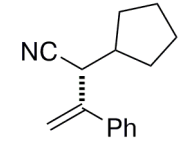
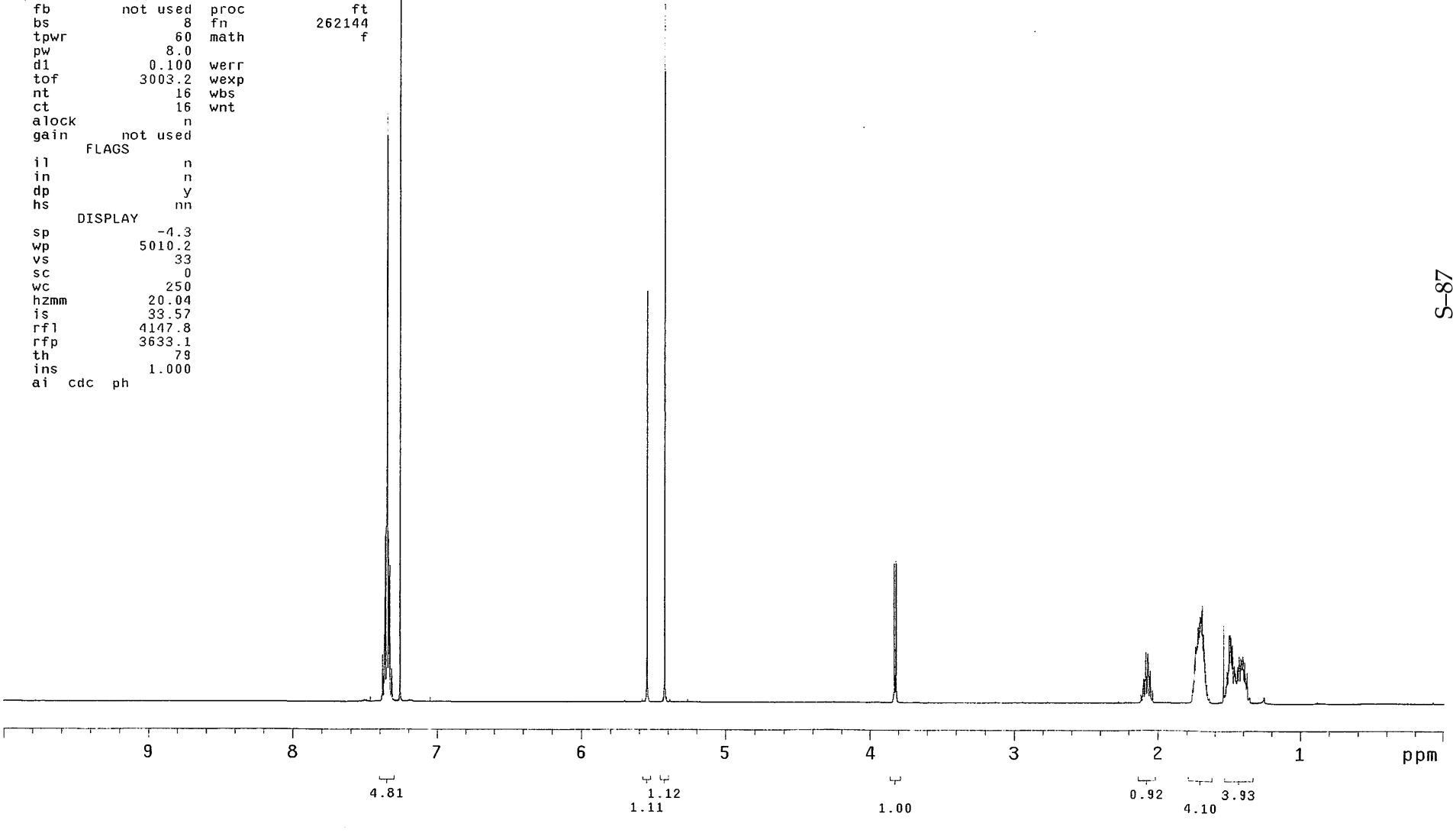



Table 4, entry 4



JC7099 1H CDCl3

exp2 s2pul

| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Jan 27 2012 | dfrq | 125.844 |
| solvent | CDCl3 | dn | C13 |
| file | /data/export/~ | dpwr | 30 |
| | home/gfu/FUjcho/ca~ | dof | 0 |
| | sper/JC7099_1H_CDC~ | dm | nnn |
| | 13.fid | dmm | c |
| ACQUISITION | | dmf | 200 |
| sfrq | 500.431 | dseq | |
| tn | H1 | dres | 1.0 |
| at | 4.999 | homo | n |
| np | 120102 | PROCESSING | |
| sw | 12012.0 | wtfile | |
| fb | not used | proc | ft |
| bs | 8 | fn | 262144 |
| tpwr | 60 | math | f |
| pw | 8.0 | | |
| d1 | 0.100 | werr | |
| tof | 3003.2 | wexp | |
| nt | 64 | wbs | |
| ct | 24 | wnt | |
| alock | n | | |
| gain | not used | | |
| FLAGS | | | |
| il | n | | |
| in | n | | |
| dp | y | | |
| hs | nn | | |
| DISPLAY | | | |
| sp | 51.1 | | |
| wp | 4957.7 | | |
| vs | 21 | | |
| sc | 0 | | |
| wc | 250 | | |
| hzmm | 19.83 | | |
| is | 33.57 | | |
| rfl | 4148.4 | | |
| rfp | 3633.1 | | |
| th | 7 | | |
| ins | 1.000 | | |
| ai cdc ph | | | |

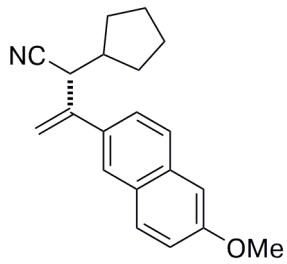
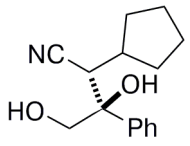



Table 4, entry 5

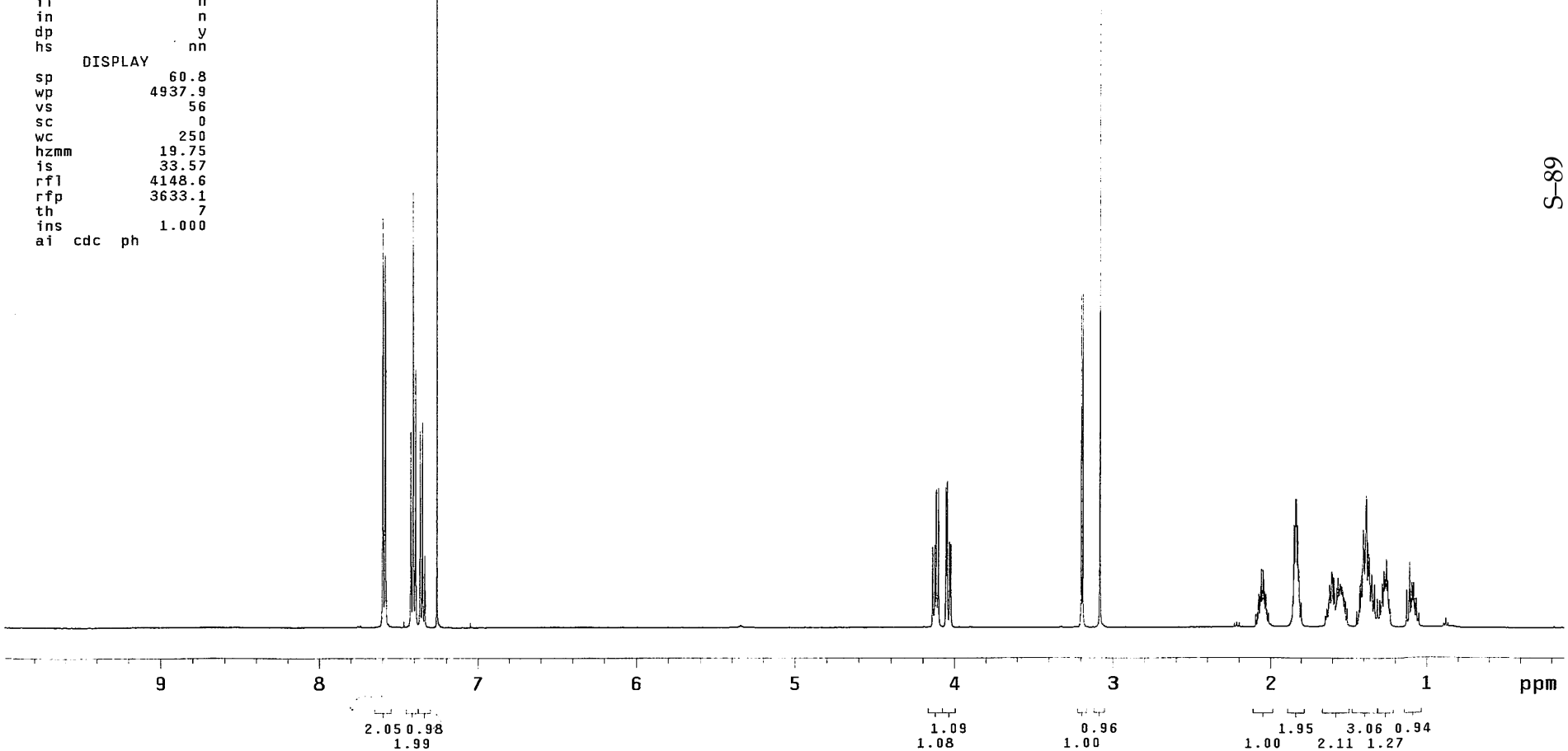
JC7279A 1H CDCl3

exp1 s2pul

| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Apr 7 2012 | dfrq | 125.844 |
| solvent | CDCl3 | dn | C13 |
| file | exp | dpwr | 30 |
| ACQUISITION | | dof | 0 |
| sfrq | 500.431 | dm | nnn |
| tn | H1 | dmm | c |
| at | 4.999 | dmf | 200 |
| np | 120102 | dseq | |
| sw | 12012.0 | dres | 1.0 |
| fb | not used | homo | n |
| bs | 8 | PROCESSING | |
| tpwr | 60 | wtfile | |
| pw | 8.0 | proc | ft |
| d1 | 0.100 | fn | 262144 |
| tof | 3003.2 | math | f |
| nt | 16 | | |
| ct | 16 | werr | |
| alock | n | wexp | |
| gain | not used | wbs | |
| FLAGS | | wnt | |
| il | n | | |
| in | n | | |
| dp | y | | |
| hs | nn | | |
| DISPLAY | | | |
| sp | 60.8 | | |
| wp | 4937.9 | | |
| vs | 56 | | |
| sc | 0 | | |
| wc | 250 | | |
| hzmm | 19.75 | | |
| is | 33.57 | | |
| rfl | 4148.6 | | |
| rfp | 3633.1 | | |
| th | 7 | | |
| ins | 1.000 | | |
| ai cdc ph | | | |



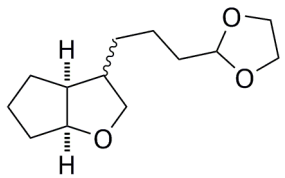

eq 2



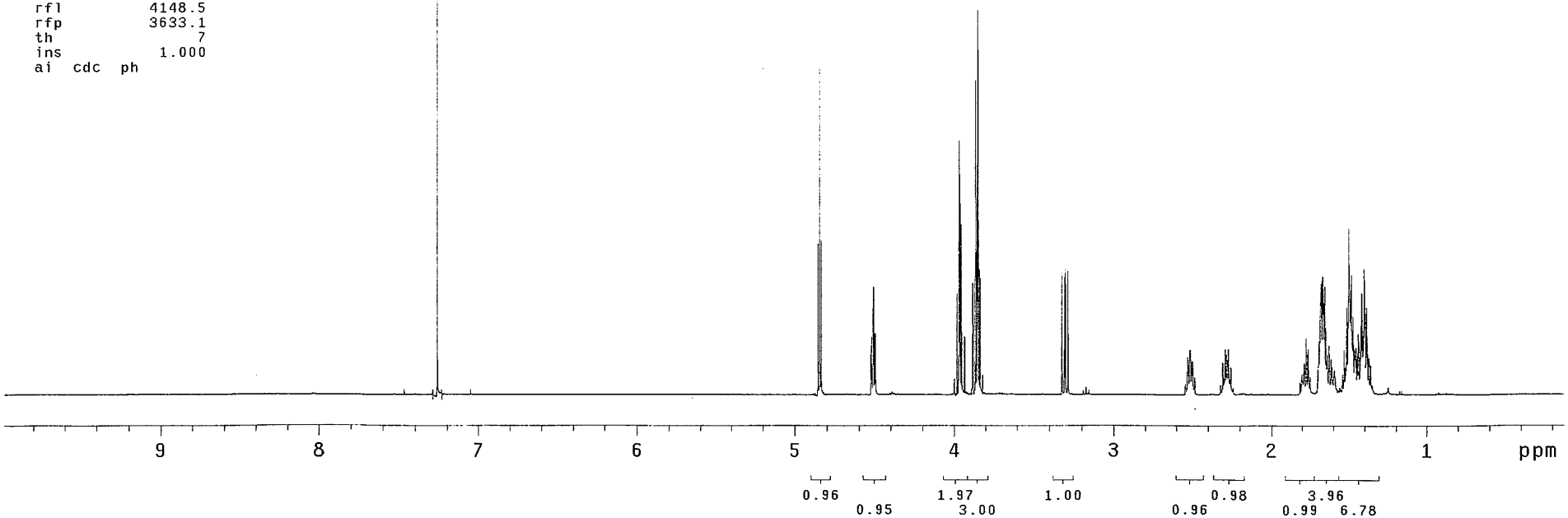
JC7151A Major Diastereomer 1H CDCl3

exp2 s2pul

| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Feb 6 2012 | dfrq | 125.844 |
| solvent | CDCl3 | dn | C13 |
| file | /data/export/~ | dpwr | 30 |
| home/gfu/FUjcho/ca~ | | dof | 0 |
| sper/JC7151A_Major~ | | dm | nnn |
| -Diastereomer_1H_C~ | | dmm | c |
| DCl3.fid | | dmf | 200 |
| ACQUISITION | | dseq | |
| sfrq | 500.431 | dres | 1.0 |
| tn | H1 | homo | n |
| at | 4.999 | PROCESSING | |
| np | 120102 | wtfile | |
| sw | 12012.0 | proc | ft |
| fb | not used | fn | 262144 |
| bs | 8 | math | f |
| tpwr | 60 | | |
| pw | 8.0 | werr | |
| d1 | 0.100 | wexp | |
| tof | 3003.2 | wbs | |
| nt | 16 | wnt | |
| ct | 16 | | |
| alock | n | | |
| gain | not used | | |
| FLAGS | | | |
| il | n | | |
| in | n | | |
| dp | y | | |
| hs | nn | | |
| DISPLAY | | | |
| sp | 60.9 | | |
| wp | 4937.9 | | |
| vs | 61 | | |
| sc | 0 | | |
| wc | 250 | | |
| hzmm | 19.75 | | |
| is | 33.57 | | |
| rfl | 4148.5 | | |
| rfp | 3633.1 | | |
| th | 7 | | |
| ins | 1.000 | | |
| ai cdc ph | | | |



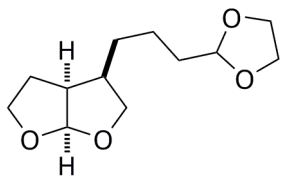

eq 3



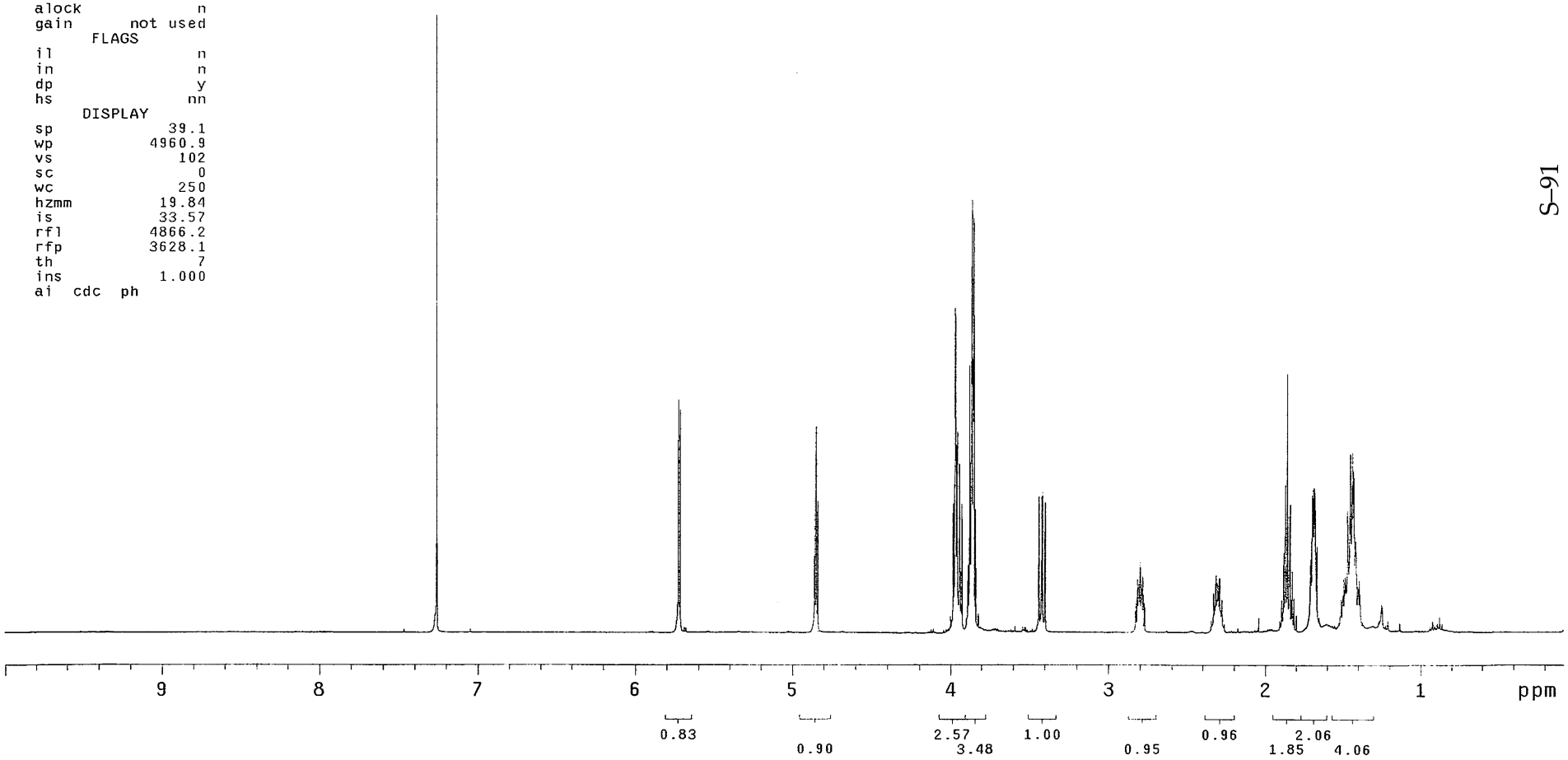
JC7153B Major 1H CDCl3

exp1 s2pul

| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Feb 4 2012 | dfrq | 125.672 |
| solvent | CDCl3 | dn | C13 |
| file | /data/export/~ | dpwr | 30 |
| | home/gfu/FUjcho/bu~ | dof | 0 |
| | llwinkle/JC7153B_M~ | dm | nnn |
| | ajor_1H_CDCl3.fid | dmm | w |
| ACQUISITION | | dmf | 10000 |
| sfrq | 499.746 | dseq | |
| tn | H1 | dres | 1.0 |
| at | 3.001 | homo | n |
| np | 63050 | PROCESSING | |
| sw | 10504.2 | wtfile | |
| fb | not used | proc | ft |
| bs | 8 | fn | 262144 |
| tpwr | 56 | math | f |
| pw | 8.6 | | |
| d1 | 2.000 | werr | |
| tof | 1519.5 | wexp | |
| nt | 16 | wbs | |
| ct | 16 | wnt | wft |
| alock | n | | |
| gain | not used | | |
| FLAGS | | | |
| il | n | | |
| in | n | | |
| dp | y | | |
| hs | nn | | |
| DISPLAY | | | |
| sp | 39.1 | | |
| wp | 4960.9 | | |
| vs | 102 | | |
| sc | 0 | | |
| wc | 250 | | |
| hzmm | 19.84 | | |
| is | 33.57 | | |
| rfl | 4866.2 | | |
| rfp | 3628.1 | | |
| th | 7 | | |
| ins | 1.000 | | |
| ai cdc ph | | | |



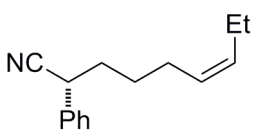

eq 3



JC7205A 1H CDCl3

exp2 s2pul

| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Feb 24 2012 | dfrq | 125.844 |
| solvent | CDCl3 | dn | C13 |
| file | /data/export/~ | dpwr | 30 |
| | home/gfu/FUjcho/ca~ | dof | 0 |
| | sper/JC7205A_1H_CD~ | dm | nnn |
| | C13.fid | dmm | c |
| ACQUISITION | | dmf | 200 |
| sfrq | 500.431 | dseq | |
| tn | H1 | dres | 1.0 |
| at | 4.999 | homo | n |
| np | 120102 | PROCESSING | |
| sw | 12012.0 | wtfile | |
| fb | not used | proc | ft |
| bs | 8 | fn | 262144 |
| tpwr | 60 | math | f |
| pw | 8.0 | | |
| d1 | 0.100 | werr | |
| tof | 3003.2 | wexp | |
| nt | 16 | wbs | |
| ct | 16 | wnt | |
| alock | n | | |
| gain | not used | | |
| FLAGS | | | |
| il | n | | |
| in | n | | |
| dp | y | | |
| hs | nn | | |
| DISPLAY | | | |
| sp | 50.9 | | |
| wp | 4947.8 | | |
| vs | 42 | | |
| sc | 0 | | |
| wc | 250 | | |
| hzmm | 19.79 | | |
| is | 1701.11 | | |
| rfl | 4148.6 | | |
| rfp | 3633.1 | | |
| th | 7 | | |
| ins | 1.000 | | |
| ai cdc ph | | | |




eq 4