

SUPPORTING INFORMATION

Carbocyclization by Radical Closure onto *O*-Trityl Oximes: Dramatic Effect of Diphenyl Diselenide

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Experimental procedures for:

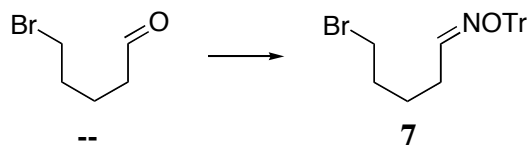
Experimental procedures for **7-11**, **12** (from **11**), **13-17**, **19-27-30**, **32-33**, **34** (from **22**), **35**, **37**, **39**, **41-45**, **47**, **49**, **51**, **53-54**, **56-58**, **60-62**, *trans*-2-[(2-bromocyclohexyl)oxy]acetaldehyde *O*-(phenylmethyl)oxime, *O*-benzyl-*N*-[(3 α ,7 α)octahydrobenzofuran-3-yl]hydroxylamine S2

References and footnotes

S29

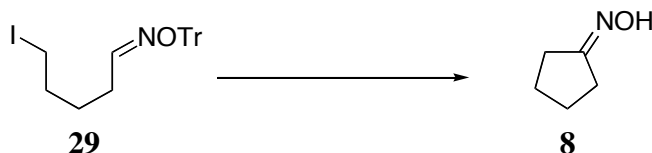
NMR spectra of **7**, **9-32**, **34-38**, **41-42**, **47**, **49**, **51**, **53-54**, **56-58**, **60-62**, *trans*-[(2-iodocyclohexyl)oxy]acetaldehyde, acetic acid (1-iodomethyl)pent-4-enyl ester, acetic acid 1-(iodomethyl)-4-oxobutyl ester, *trans*-2-[(2-bromocyclohexyl)oxy]acetaldehyde *O*-(phenylmethyl)oxime, *O*-benzyl-*N*-[3 α ,7 α]-octahydrobenzofuran-3-yl]hydroxylamine S30

5-Bromopentanal *O*-(Triphenylmethyl)oxime (**7**).



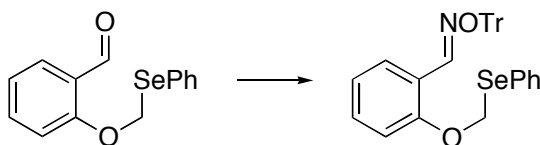
The general procedure for making *O*-trityl oximes was followed, using TrONH₂ (850 mg, 3.08 mmol) and 5-bromopentanal¹⁴ (505 mg, 3.08 mmol) in THF (10 mL). Flash chromatography of the residue over silica gel (1.7 x 35 cm), using 5% EtOAc-hexane, gave **7** (1.07 g, 82%) as a foam, which was a 1:1 mixture of *Z* and *E* isomers (¹³C NMR): FTIR (CH₂Cl₂ cast) 3056, 3022, 2935, 1596, 1491 cm⁻¹; ¹H NMR (CDCl₃, 500 MHz) δ 1.43-1.54 (m, 1 H), 1.60-1.71 (m, 2 H), 1.81-1.93 (m, 1 H), 2.11-2.15 (m, 1 H), 2.53-2.60 (m, 1 H), 3.22 (t, *J* = 6.7 Hz, 1 H), 3.38 (t, *J* = 6.7 Hz, 1 H), 6.71 (t, *J* = 6.0 Hz, 0.42 H), 7.49-7.34 (m, 15 H), 7.52 (t, *J* = 6.0 Hz, 0.5 H); ¹³C NMR (CDCl₃, 125.7 MHz) 24.7 (t), 24.8 (t), 25.5 (t), 28.5 (t), 31.4 (t), 32.3 (t), 33.2 (t), 33.4 (t), 90.3 (s), 90.4 (s), 126.9 (d), 127.0 (d), 127.4 (d), 127.5 (d), 128.8 (d), 129.1 (d), 144.4 (s), 144.5 (s), 150.1 (d), 151.2 (d); exact mass (electrospray) *m/z* calcd for C₂₄H₂₄⁷⁹BrNNaO 444.0938, found 444.0943.

Cyclopentanone Oxime (**8**).



General procedure B for radical cyclization was followed using **29** (353.3 mg, 0.7535 mmol), PhSeSePh (47 mg, 0.15 mmol) and Hünig's base (0.52 mL, 3.014 mmol) in THF (20 mL), Bu₃SnH (0.24 mL, 0.892 mmol) in THF (5 mL) and ABC (184 mg, 0.753 mmol) in THF (5 mL). After evaporation of the solvent, flash chromatography of the residue over silica gel (1.5 x 30 cm), using 30% EtOAc-hexane, gave cyclopentanone oxime (**8**)²⁵ (0.0689, 92%).

2-[(Phenylseleno)methoxy]benzaldehyde *O*-(Triphenylmethyl)oxime (**9**).



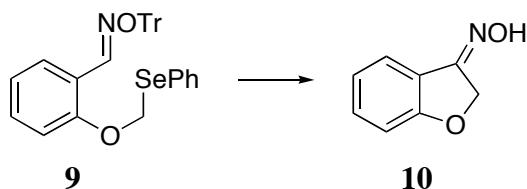
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9

The general procedure for making *O*-trityl oximes was followed, using TrONH₂ (193 mg, 0.702 mmol) and 2-[(phenylseleno)methoxy]benzaldehyde¹⁵ (204.6 mg, 0.7007 mmol) in THF (5 mL). Flash chromatography of the residue over silica gel (1.7 x 30 cm), using 7% EtOAc-hexane, gave **9** (362 mg, 95%) as a foam, which appeared to be a single isomer (¹H and ¹³C NMR): FTIR (CH₂Cl₂ cast) 3056, 3032, 1599, 1577, 1484, 1448 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 5.66 (s, 2 H), 6.82-6.91 (m, 2 H), 7.20-7.63 (m, 22 H), 8.52 (s, 1 H); ¹³C NMR (CDCl₃, 100.6 MHz) δ 68.7 (t), 91.1 (s), 114.2 (d), 122.3 (d), 123.2 (s), 127.1 (d), 127.5 (d), 127.9 (d), 129.4 (d), 129.5 (s), 130.5 (d), 133.8 (d), 144.4 (d), 144.6 (s), 154.8 (s); exact mass (electrospray) *m/z* calcd for C₃₃H₂₇NNaO₂⁸⁰Se 572.1104, found 572.1100.

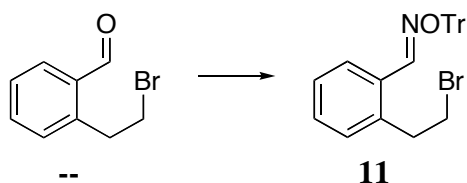
The oxime geometry was not determined.

3(2*H*)-Benzofuranone Oxime (**10**).²⁶



General procedure A for radical cyclization was followed, using **9** (238 mg, 0.434 mmol) in THF (30 mL), Bu₃SnH (510.0 mg, 1.737 mmol) in THF (10 mL), ABC (11 mg, 0.043 mmol) in THF (10 mL), and *i*-Pr₂NEt (230 mg, 1.74 mmol). Flash chromatography of the residue over silica gel (1.7 x 20 cm), using 10% EtOAc-hexane, gave **10** (59.2 mg, 91%) as a crystalline solid, which was a single isomer of unestablished geometry: mp 158-161 °C; FTIR 3131, 3046, 2841, 1666, 1605, 1591, 1481 cm⁻¹; ¹H NMR (CD₂Cl₂, 400 MHz) δ 5.18 (s, 2 H), 6.86-7.11 (m, 2 H), 7.32-7.4 (m, 1 H), 7.58-7.60 (m, 1 H), 7.98 (s, 1 H); ¹³C NMR (CDCl₃, 125.7 MHz) δ 70.6 (t), 111.4 (d), 119.6 (s), 121.5 (d), 121.9 (d), 132.7 (d), 158.3 (s), 165.5 (s); exact mass *m/z* calcd for C₈H₇NO₂ 149.0476, found 149.0477.

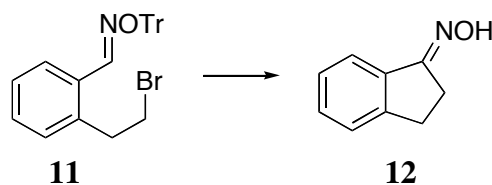
2-(2-Bromoethyl)benzaldehyde *O*-(Triphenylmethyl)oxime (**11**).



The general procedure for making *O*-trityl oximes was followed, using TrONH₂ (1.094 g, 3.978 mmol) and 2-(2-bromoethyl)benzaldehyde¹⁶ (1.06 g, 4.97 mmol) in THF (15 mL). Flash chromatography of the residue over silica gel (4 x 32 cm), using 5% EtOAc-hexane, gave **11** (1.75 g, 94%) as a foam, which was a single isomer: FTIR (CH₂Cl₂ cast) 3057, 3022, 1957, 1597, 1490 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz) δ 3.01-3.18 (m, 4 H), 7.11-7.45 (m, 19 H), 8.41 (s, 1 H); ¹³C NMR (CDCl₃, 125.7 MHz) δ 32.9 (t), 37.5 (t), 91.3 (s), 127.1 (d), 127.2 (d), 127.5 (d), 127.8 (d), 129.1 (d), 129.2 (d), 130.4 (s), 130.5 (d), 131.5 (d), 137.4 (s), 144.3 (s), 149.1 (d); exact mass (electrospray) *m/z* calcd for C₂₈H₂₄⁷⁹BrNNaO 492.0938 found 492.0928.

The oxime geometry was not determined.

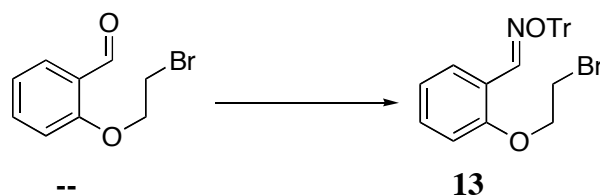
2,3-Dihydro-1*H*-inden-1-one Oxime (**12**) (from **11**).^{22,23}



General procedure A for radical cyclization was followed, using **11** (211 mg, 0.449 mmol) in THF (30 mL), Bu₃SnH (524 mg, 1.81 mmol) in THF (8 mL), ABC (11 mg, 0.045 mmol) in THF (8 mL), and *i*-Pr₂NEt (232 mg, 1.81 mmol). Flash chromatography of the residue over silica gel (1.7 x 18 cm), using 10% EtOAc-hexane, gave **12** (57.3 mg, 87%) as a crystalline solid, which was a single isomer: mp 148-150 °C (Lit.²² mp 153-154 °C); FTIR (CH₂Cl₂ cast) 3064, 2861, 1654, 1598, 1479, 1460 cm⁻¹; ¹H NMR (CDCl₃, 500 MHz) δ 2.92-3.01 (m, 2 H), 3.05-3.18 (m, 2 H), 7.20-7.41 (m, 3 H), 7.64-7.67 (m, 1 H), 9.15 (s, 1 H); ¹³C NMR (CDCl₃, 125.7 MHz) δ 26.0 (t), 28.6 (t), 121.5 (d), 125.5 (d), 126.9 (d), 130.3 (d), 135.8 (s), 148.3 (s), 163.9 (s); exact mass *m/z* calcd for C₉H₉NO 147.0684, found 147.0682.

The ¹³C NMR spectrum (DMSO-*d*₆) of the so-called *E*-isomer has been reported;²³ but the spectrum of our material, also in DMSO-*d*₆, is different from the reported spectrum. Our sample had: ¹³C NMR (DMSO-*d*₆, 125.7 MHz) δ 25.4 (t), 27.8 (t), 120.4 (d), 125.6 (d), 126.7 (d), 129.5 (d), 136.4 (s), 147.5 (s), 160.8 (s). A sample crystallized from a mixture of *i*-PrOH and hexane gave material suitable for X-ray analysis, which established the *E*-geometry.

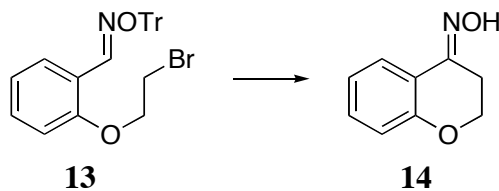
2-(2-Bromoethoxy)benzaldehyde *O*-(Triphenylmethyl)oxime (**13**).



The general procedure for making *O*-trityl oximes was followed, using TrONH₂ (180 mg, 0.654 mmol) and 2-(2-bromoethoxy)benzaldehyde¹⁷ (150 mg, 0.654 mmol) in THF (10 mL). Flash chromatography of the residue over silica gel (1.7 x 30 cm), using 5% EtOAc-hexane, gave **13** (289 mg, 92%) as a foam, which appeared to be a single isomer (¹H and ¹³C NMR): FTIR (CH₂Cl₂ cast) 3056, 3033, 1599, 1488, 1448, 1421 cm⁻¹; ¹H NMR (CDCl₃, 200 MHz) δ 3.66 (t, *J* = 9.0 Hz, 2 H), 4.31 (t, *J* = 9.0 Hz, 2 H), 6.81-7.59 (m, 19 H), 8.65 (s, 1 H); ¹³C NMR (CDCl₃, 50.3 MHz) δ 28.9 (t), 68.3 (t), 91.1 (s), 112.3 (d), 121.5 (d), 121.9 (s), 127.1 (d), 127.3 (d), 127.5 (d), 127.9 (d), 129.3 (d), 130.7 (d), 144.3 (d), 144.5 (s), 155.9 (s); exact mass (electrospray) *m/z* calcd for C₂₈H₂₄⁷⁹BrNNaO 508.0888, found 508.08828.

The oxime geometry was not determined.

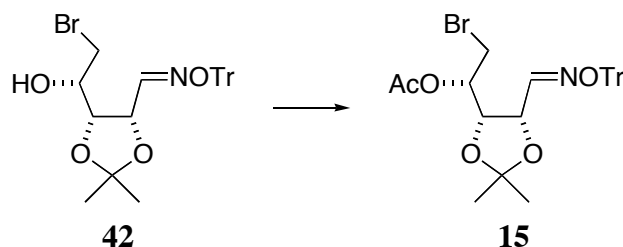
2,3-Dihydro-4*H*-1-benzopyran-4-one Oxime (**14**).²⁷



General procedure A for radical cyclization was followed, using **13** (151.7 mg, 0.3121 mmol) in THF (20 mL), Bu₃SnH (340.0 mg, 1.248 mmol) in THF (5 mL), ABC (8 mg, 0.03 mmol) in THF (5 mL), and *i*-Pr₂NEt (200 mg, 1.56 mmol). Flash chromatography of the residue over silica gel (1.7 x 18 cm) gave **14** (46.1 mg, 92%) as a crystalline solid: mp 139-141 °C (lit.²⁷ 138 °C); FTIR (CH₂Cl₂ cast) 3263, 2988, 2922, 1958, 1647 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 2.99 (t, *J* = 7.0 Hz, 2 H), 4.22 (t, *J* = 7.0 Hz, 2 H), 6.84-6.88 (m, 2 H), 7.21-7.26 (m, 1 H), 7.67-7.81 (m, 1 H), 8.85 (br s, 1 H); ¹³C NMR (CDCl₃, 125.7 MHz) δ 23.6 (t), 64.9 (t), 117.7 (d), 118.2 (s), 121.4 (d), 123.9 (d), 131.1 (d), 150.0 (s), 156.6 (s); exact mass *m/z* calcd for C₉H₉NO₂ 163.0633, found 163.0632.

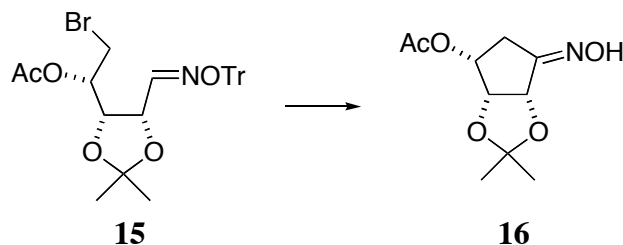
Although the compound is known,²⁷ its geometry was not reported, and we did not establish the geometry. In a separate experiment triphenylmethane (78 mg, 56%) was isolated.

4-*O*-Acetyl-5-bromo-5-deoxy-2,3-*O*-(1-methylethylidene)-D-ribose *O*-(Triphenylmethyl)oxime (15**).**



Ac₂O (0.1 mL, 1 mmol) was added to a stirred solution of **42** (265.4 mg, 0.5214 mmol) in pyridine (1.0 mL), and stirring was continued overnight. Water (10 mL) was added and the solution was extracted with Et₂O (2 x 10 mL). The combined organic extracts were washed with saturated aqueous NaHCO₃ (3 x 10 mL), water (2 x 10 mL) and brine (2 x 10 mL), dried (MgSO₄), and evaporated. Flash chromatography of the residue over silica gel (1.7 x 32 cm), using 5% EtOAc-hexane, gave **15** (270 mg, 96%) as a foam, which was a 4.3:1 mixture of isomers (¹H NMR): FTIR (CH₂Cl₂ cast) 3057, 3034, 2987, 1749, 1491 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 1.31 and 1.40 (two singlets, 3 H in all), 1.43 and 1.49 (two singlets, 3 H in all), 1.59 and 1.78 (two singlets, 3 H in all), 3.44-3.58 (two overlapping m, 2 H), 4.31-4.39 (m, 0.84 H), 4.61-4.72 (m, 1 H), 4.88-4.99 (m, 1 H), 5.46-5.51 (m, 0.22 H), 6.83 (d, *J* = 5.0 Hz, 0.69 H), 7.17-7.35 (m, 15 H), 7.42 (d, *J* = 8.0 Hz, 0.86 H); ¹³C NMR (CDCl₃, 125.7 MHz) δ 20.4 (q), 20.5 (q), 25.3 (q), 25.4 (q), 27.7 (q), 32.1 (t), 32.3 (t), 69.1 (d), 69.7 (d), 71.1 (d), 74.7 (d), 76.02 (d), 76.06 (d), 91.1 (s), 91.9 (s), 109.8 (s), 110.0 (s), 127.1 (d), 127.3 (d), 127.53 (d), 127.56 (d), 127.6 (d), 127.8 (d), 129.14 (d), 129.18 (d), 129.19 (d), 129.2 (d), 143.7 (s), 143.9 (s), 146.5 (d), 146.9 (d), 169.5 (s), 169.7 (s); exact mass (electrospray) *m/z* calcd for C₂₉H₃₀⁷⁹BrNNaO₅ 574.1205, found 574.1202.

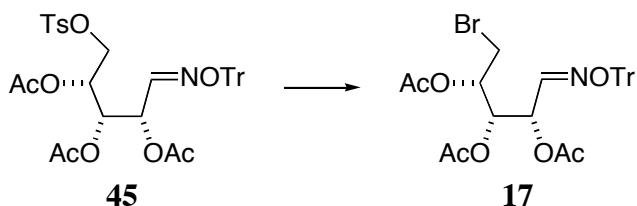
[4*R*-(3α,4β,6α)]-Tetrahydro-2,2-dimethyl-6-hydroximino-4*H*-cyclopenta-1,3-dioxol-4-yl Acetate (16**).**



General procedure A for radical cyclization was followed, using **15** (250.0 mg, 0.4537 mmol) in THF (30 mL), Bu₃SnH (0.49 mL, 1.814 mmol) in THF (10 mL), ABC (11 mg, 0.067 mmol) in THF (10 mL), and *i*-Pr₂NEt (237.4 mg, 1.814 mmol). As some starting material was present after the arbitrary reflux period (TLC control), further portions of Bu₃SnH (0.07 mL, 0.24 mmol), and ABC (8 mg, 0.03 mmol), each in THF (4 mL), were added slowly (4 h) as before, and refluxing was continued for 2 h after the addition. Flash chromatography of the residue over silica gel (1.7 x 20 cm), using 40% EtOAc-hexane, gave **16** (96.0 mg, 93%) as a crystalline solid, which was a single isomer: mp 151-154 °C; FTIR 3358, 2991, 2980, 2945, 1736 cm⁻¹; ¹H NMR (CDCl₃, 200 MHz) δ 1.33 (s, 3 H), 1.51 (s, 3 H), 2.15 (s, 3 H), 2.51-2.74 (m, 1 H), 3.11-3.32 (m, 1 H), 4.73-4.88 (m, 2 H), 7.62 (s, 1 H), δ 4.89-5.12 (m, 1 H); ¹³C NMR (CD₂Cl₂, 50.3 MHz) δ 22.5 (q), 26.7 (q), 28.2 (q), 30.4 (t), 72.0 (d), 79.5 (d), 80.3 (d), 114.7 (s), 159.6 (s), 172.2 (s); exact mass (electrospray) *m/z* calcd for C₁₀H₁₅NNaO₅ 252.0847, found 252.0848.

The oxime geometry was not established.

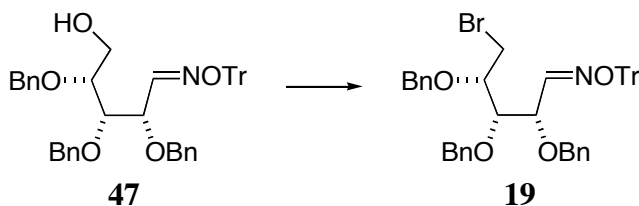
2,3,4-Tris-*O*-Acetyl-5-Bromo-5-deoxy-D-ribose *O*-(Triphenylmethyl)oxime (17).



DMF (10 mL) was added to a stirred mixture of LiBr (dried at 100 °C, 140 mg, 1.55 mmol) and **45** (266 mg, 0.387 mmol). The solution was stirred for 8 h, Et₂O (20 mL) was added, and the mixture was washed with water (4 x 10 mL). The ether extract was washed with water (2 x 10 mL), dried (MgSO₄) and evaporated. Flash chromatography of the residue over silica gel (1.7 x 30 cm), using 15% EtOAc-hexane, gave **17** (198 mg, 86%) as a foam. The material, which was used with only partial characterization (¹H NMR), appeared to be a single isomer: ¹H NMR (CDCl₃, 300 MHz) δ 1.98 and 2.01 (two singlets, 9 H), 3.24-3.58 (m, 2 H), 5.01-5.16 (m, 1 H), 5.34-5.46 (m, 1 H), 5.48-5.51 (m, 1 H), 7.01-7.49 (m, 15 H), 7.52 (d, *J* = 7 Hz, 1 H).

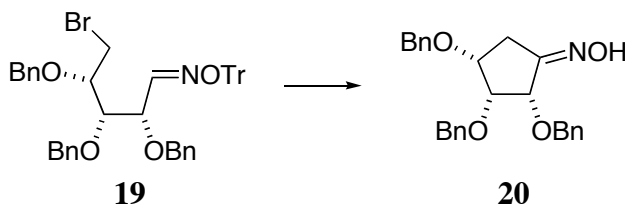
The oxime geometry was not determined.

5-Bromo-5-deoxy-2,3,4-tris-*O*-(phenylmethyl)-D-ribose *O*-(Triphenylmethyl)oxime (19).



Ph₃P (90 mg, 0.34 mmol) was added to a stirred and cooled (ice-water) solution of **47** (115 mg, 0.169 mmol) in pyridine (5 mL), and then CBr₄ (70 mg, 0.2 mmol) was added in one portion. The mixture was heated at 65 °C for 2 h, cooled, and diluted with MeOH (5 mL). Evaporation of the solvent, and flash chromatography of the residue over silica gel (1.7 x 20 cm), using 10% EtOAc-hexane, gave **19** (109 mg, 86%) as a foam, which was a 6:1 mixture of isomers (¹H NMR): FTIR (CH₂Cl₂ cast) 3060, 3030, 2866, 1958, 1597 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 3.34-3.43 (m, 1 H), 3.53-3.74 (m, 2 H), 3.79-4.08 (m, 2 H), 4.19-5.30 (m including several AB q, 6 H), 6.88 (d, *J* = 7 Hz, 0.13 H), 7.22-7.46 (m, 30 H), 7.68 (d, *J* = 7 Hz, 0.77 H); ¹³C NMR (CDCl₃, 100.6 MHz) δ 33.6 (t), 33.7 (t), 70.7 (t), 71.71 (d), 71.73 (t), 72.2 (t), 72.5 (t), 73.4 (t), 74.7 (t), 76.5 (d), 77.1 (d), 77.2 (d), 79.8 (d), 80.8 (d), 90.8 (s), 127.1 (d), 127.2 (d), 127.5 (d), 127.6 (d), 127.64 (d), 127.7 (d), 127.75 (d), 127.81 (d), 127.85 (d), 127.9 (d), 128.11 (d), 128.17 (d), 128.2 (d), 128.3 (d), 128.31 (d), 128.35 (d), 128.4 (d), 129.1 (d), 129.2 (d), 129.3 (d), 137.5 (s), 137.6 (s), 138.1 (s), 144.1 (s), 144.3 (s), 149.3 (d); exact mass (electrospray) *m/z* calcd for C₄₅H₄₂⁷⁹BrNNaO₄ 762.2194, found 762.2183.

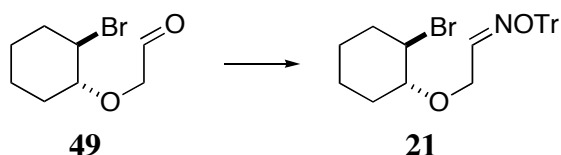
[2*S*-(2α,3α,4α)]-2,3,4-Tris(phenylmethoxy)cyclopentanone Oxime (20).



General procedure A for radical cyclization was followed, using **19** (260 mg, 0.351 mmol) in THF (25 mL), Bu₃SnH (0.21 mL, 0.702 mmol) in THF (5 mL), ABC (5 mg, 0.02 mmol) in THF (5 mL), and *i*-Pr₂NEt (90 mg, 0.71 mmol). As a considerable amount of starting material was present after the arbitrary reflux period (TLC control), further portions of Bu₃SnH (0.21 mL, 0.702 mmol) and ABC (5.9 mg, 0.024 mmol), each in THF (5 mL), were added slowly (10 h) as before. As a considerable amount of starting material was still present after the arbitrary reflux period (TLC control), further portions of Bu₃SnH (0.21 mg, 0.702 mmol) and ABC (5 mg, 0.02 mmol), each in THF (5 mL), were added slowly (10 h) as before. Flash

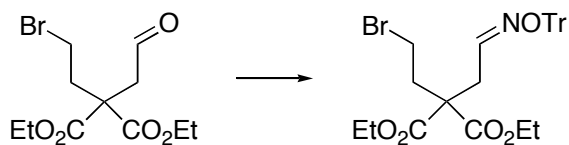
chromatography of the residue over silica gel (1.7 x 20 cm), using 30% EtOAc-hexane, gave **20** (132 mg, 91%) as a crystalline solid, which was a mixture of *Z* and *E* isomers (^1H NMR): mp 145-147 °C; FTIR (CH_2Cl_2 cast) 3228, 3087, 3062, 2869, 1495 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 2.48-2.92 [m, including a doublet of doublets at δ 2.54 (dd, $J = 17.0$, 6.3 Hz), 2 H in all], 3.76-3.97 [m, including a triplet at δ 3.8 ($J = 4.6$ Hz), 2 H in all], 4.16 (d, $J = 4$ Hz, 1 H), 4.53-4.84 (m, 6 H), 7.03-7.61 (m, 15 H), 7.78-8.87 (br s, 1 H); ^{13}C NMR (CDCl_3 , 100.6 MHz) δ 30.5 (t), 33.7 (t), 71.41 (t), 71.47 (t, shows only in expanded spectrum), 71.5 (t), 71.6 (t), 72.5 (t), 72.7 (t), 73.4 (d), 75.2 (d), 75.3 (d), 77.0 (d), 78.4 (d), 127.5 (d), 127.53 (d), 127.59 (d), 127.6 (d), 127.71 (d), 127.75 (d), 127.86 (d), 127.89 (d), 128.14 (d), 128.16 (d), 128.22 (d), 128.28 (d), 128.3 (d), 137.8 (s), 138.1 (s), 138.18 (s), 138.2 (s), 138.21 (s), 138.4 (s), 158.6 (s), 160.1 (s); exact mass m/z calcd for $\text{C}_{26}\text{H}_{27}\text{NO}_4$ 417.1940, found 417.1943.

***Trans*-2-[2-Bromocyclohexyl]oxy]acetaldehyde *O*-(Triphenylmethyl)oxime (**21**).**



The general procedure for making *O*-trityl oximes was followed, using TrONH_2 (651 mg, 2.37 mmol) and **49** (523 mg, 2.36 mmol) in THF (8 mL). Flash chromatography of the residue over silica gel (1.7 x 35 cm), using 5% EtOAc-hexane, gave **21** (1.06 g, 94%) as a foam, which was a 1:1 mixture of *Z* and *E* isomers (^1H NMR): FTIR (CH_2Cl_2 cast) 3056, 3033, 2860, 1597, 1491 cm^{-1} ; ^1H NMR (CD_2Cl_2 , 300 MHz) δ 1.10-1.43 (m, 3 H), 1.58-2.03 (m, 4 H), 2.11-2.41 (m, 1 H), 3.11-3.23 (m, 0.6 H), 3.34-3.43 (m, 0.46 H), 3.81-4.20 (m, 2 H), 4.64 (apparent t, $J = 4$ Hz, 1 H), 6.98 (t, $J = 5$ Hz, 0.5 H), 7.14-7.40 (m, 15 H), 7.69 (t, $J = 5$ Hz, 0.5 H); ^{13}C NMR (CDCl_3 , 125.7 MHz) δ 23.3 (t), 25.49 (t), 25.5 (t), 25.51 (t), 31.0 (t), 35.6 (t), 35.7 (t), 55.1 (d), 55.3 (d), 64.6 (t), 66.2 (t), 80.7 (d), 82.5 (d), 90.7 (s), 91.0 (s), 127.0 (d), 127.2 (d), 127.4 (d), 127.5 (d), 128.8 (d), 129.1 (d), 129.2 (d), 144.1 (s), 144.2 (s), 147.8 (d), 151.1 (d); exact mass (electrospray) m/z calcd for $\text{C}_{27}\text{H}_{28}^{79}\text{BrNNaO}_2$ 500.1195, found 500.1199.

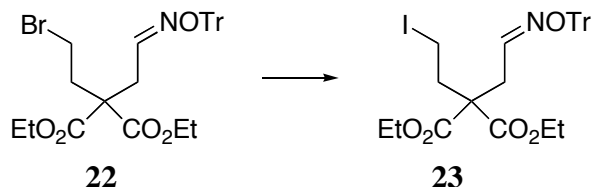
Diethyl (2-Bromoethyl)[2-[(triphenylmethoxy)imino]ethyl]propanedioate (22**).**



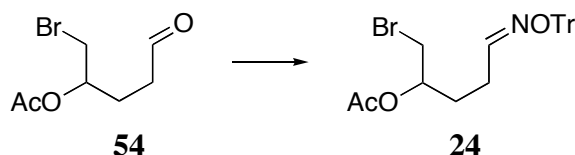
51**22**

The general procedure for making *O*-trityl oximes was followed, using TrONH₂ (260 mg, 0.943 mmol) and **51** (290 mg, 0.943 mmol) in THF (10 mL). Flash chromatography of the residue over silica gel (1.7 x 30 cm), using 10% EtOAc-hexane, gave **22** (512 mg, 97%) as a foam, which was a mixture of *Z* and *E* isomers (¹H NMR): FTIR (CH₂Cl₂ cast) 3533, 3087, 2980, 2936, 1958, 1731, 1597 cm⁻¹; ¹H NMR (CDCl₃, 500 MHz) δ 1.17 (t, *J* = 7.0 Hz) and 1.22 (t, *J* = 7.0 Hz) (6 H in all), 2.24 (t, *J* = 8.0 Hz, 1.3 H), 2.44 (t, *J* = 8.0 Hz, 0.7 H), 2.73-2.80 (m, 1.3 H), 3.11-3.18 (m, 2 H), 3.30-3.38 (m, 0.72 H), 4.04-4.23 (m, 4 H), 6.71 (dt, *J* = 5.5, 0.8 Hz, 0.36 H), 7.19-7.32 (m, 15 H), 7.50 (dt, *J* = 6.0, 0.6 Hz, 0.8 H); ¹³C NMR (CDCl₃, 125.7 MHz) δ 14.0 (q), 14.1 (q), 26.7 (t), 26.9 (t), 30.1 (t), 33.2 (t), 35.9 (t), 37.2 (t), 55.9 (s or t), 56.2 (s or t), 61.9 (s or t), 62.1 (s or t), 90.7 (s), 91.1 (s), 127.10 (d), 127.13 (d), 127.5 (d), 127.8 (d), 128.9 (d), 129.0 (d), 144.1 (s), 144.2 (s), 145.9 (d), 146.0 (d), 169.5 (s), 169.6 (s); exact mass (electrospray) *m/z* calcd for C₃₀H₃₂⁷⁹BrNNaO₅ 588.1356, found 588.1361.

Diethyl (2-Iodoethyl)[2-[(triphenylmethoxy)imino]ethyl]propanedioate (23**).**

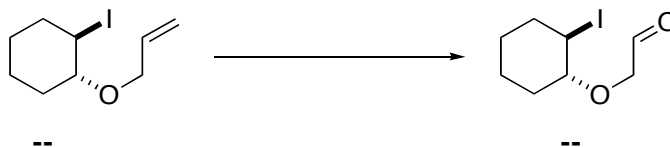


Bromide **22** (365 mg, 0.645 mmol) was added to a stirred solution of NaI (200 mg, 1.29 mmol) in dry acetone (10 mL), and the mixture was refluxed for 24 h and cooled. Saturated aqueous Na₂S₂O₃ was added to the mixture which was then extracted with Et₂O (2 x 10 mL). The combined organic extracts were washed with brine (1 x 10 mL), dried (MgSO₄), and evaporated. Flash chromatography of the residue over silica gel (1.7 x 30 cm), using 8% EtOAc-hexane, gave **23** (342 mg, 83%) as a foam, which was a 3.8:1 mixture of isomers (¹H NMR): FTIR (CH₂Cl₂ cast) 3532, 3057, 2979, 1958, 1730, 1597 cm⁻¹; ¹H NMR (CDCl₃, 500 MHz) δ 1.20 (t, *J* = 7.1 Hz), 1.24 (t, *J* = 7.1 Hz) (6 H in all), 2.31-2.98 (m, 5 H), 3.03-3.18 (m, 1 H), 4.01-4.23 (m, 4 H), 6.71 (t, *J* = 5 Hz, 0.18 H), 7.21-7.35 (m, 15 H), 7.49 (t, *J* = 5 Hz, 0.70 H); ¹³C NMR (CDCl₃, 125.7 MHz) δ -3.0 (t), -2.8 (t), 13.95 (q), 13.98 (q), 29.7 (s or t), 32.9 (s or t), 37.5 (s or t), 38.7 (s or t), 57.3 (s or t), 57.6 (s or t), 61.8 (s or t), 61.9 (s or t), 90.7 (s), 91.1 (s), 127.1 (d), 127.20 (d), 127.58 (d), 127.64 (d), 129.0 (d), 129.1 (d), 144.2 (s), 146.0 (d), 146.1 (d), 146.9 (s), 169.5 (s), 169.6 (s); exact mass (electrospray) *m/z* calcd for C₃₀H₃₂INNaO₅ 636.1222, found 636.1228.

(1-Bromomethyl)-4-[(Triphenylmethoxy)imino]butyl Acetate (24).

The general procedure for making *O*-trityl oximes was followed, using TrONH₂ (335 mg, 1.22 mmol) and **54** (272 mg, 1.22 mmol) in THF (10 mL). Flash chromatography of the residue over silica gel (1.7 x 32 cm), using 10% EtOAc-hexane, gave **24** (571 mg, 98%) as a foam, which was a 1:1 mixture of *Z* and *E* isomers (¹H NMR): FTIR (CH₂Cl₂ cast) 3057, 3023, 2928, 1958, 1741, 1596 cm⁻¹; ¹H NMR (CD₂Cl₂, 300 MHz) δ 1.71-1.85 (m, 1 H), 1.89-2.09 (m including two s, 4 H), 2.11-2.31 (m, 1 H), 2.44-2.72 (m, 1 H), 3.23-3.34 (m, 1 H), 3.42-3.59 (m, 1 H), 4.78-5.10 (m, 1 H), 6.78 (t, *J* = 6 Hz, 0.5 H), 7.11-7.42 (m, 15 H), 7.60 (t, *J* = 6 Hz, 0.5 H); ¹³C NMR (CDCl₃, 100.6 MHz) δ 20.8 (q), 20.9 (q), 22.2 (t), 25.4 (t), 28.82 (t), 28.83 (t), 33.3 (t), 33.8 (t), 71.3 (d), 71.9 (d), 90.4 (s), 90.6 (s), 126.96 (d), 127.1 (d), 127.4 (d), 127.9 (d), 128.9 (d), 129.1 (d), 144.3 (s), 144.4 (s), 149.3 (d), 150.1 (d), 170.1 (s); exact mass (electrospray) *m/z* calcd for C₂₆H₂₆⁷⁹BrNNaO₃ 502.0993, found 502.0993.

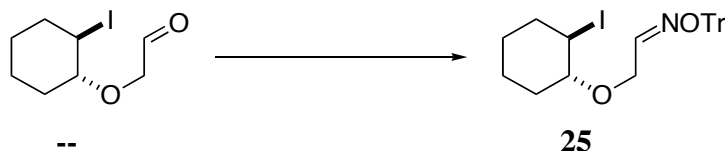
***Trans*-[(2-Iodocyclohexyl)oxy]acetaldehyde *O*-(Triphenylmethyl)oxime (25). (a)**
***Trans*-[(2-Iodocyclohexyl)oxy]acetaldehyde.**



O₃ was bubbled through a stirred and cooled (-78 °C) solution of alkene *trans*-1-(allyloxy)-2-iodocyclohexane^{28,29} (504 mg, 1.90 mmol) in CH₂Cl₂ (10 mL) for 1 h and the mixture was then flushed with O₂ for 0.5 h at -78 °C. Ph₃P (1.49 g, 5.68 mmol) was tipped in, the cold bath was removed and the mixture was stirred overnight. Evaporation of the solvent and flash chromatography of the residue over silica gel (3 x 20 cm), using 30% EtOAc-hexane, gave *trans*-[(2-iodocyclohexyl)oxy]acetaldehyde (280 mg, 59%) as a colorless oil: FTIR (CH₂Cl₂ cast) 2934, 2857, 1735, 1446 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) 1.21-1.42 (m, 3 H), 1.49-1.59 (m, 1 H), 1.78-1.87 (m, 1 H), 1.91-2.03 (m, 1 H), 2.09-2.20 (m, 1 H), 2.38-2.48 (m, 1 H), 3.39 (ddd, *J* = 9.2, 9.2, 4.4 Hz, 1 H), 4.07 (ddd, *J* = 13.1, 8.9, 4.3 Hz, 1 H), 4.10 (s, 2 H), 9.79 (t, *J* =

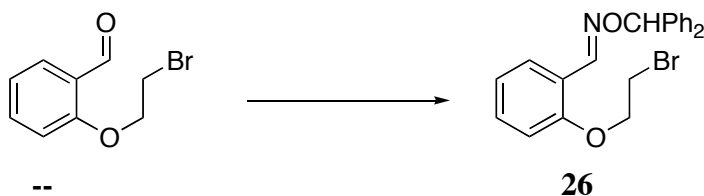
1.1 Hz, 1 H); ^{13}C NMR (CDCl_3 , 100.6 MHz) δ 23.4 (t), 26.9 (t), 31.1 (t), 34.4 (d), 37.8 (t), 75.0 (t), 83.9 (d), 201.0 (d); exact mass m/z calcd for $\text{C}_6\text{H}_{10}\text{IO}$ ($\text{M} - \text{C}_2\text{H}_3\text{O}$) 224.97765, found 224.97771.

(b) *Trans*-[(2-Iodocyclohexyl)oxy]acetaldehyde *O*-(Triphenylmethyl)oxime (25**).**



TrONH_2 (103 mg, 0.373 mmol) was added in one portion to a stirred solution of *trans*-[(2-iodocyclohexyl)oxy]acetaldehyde (100 mg, 0.373 mmol) in dry CH_2Cl_2 (5 mL) and the mixture was refluxed overnight. Evaporation of the solvent and flash chromatography of the residue over silica gel (3 x 20 cm), using 20% EtOAc-hexane, gave **25** (200 mg, 100%) as an oil, which was a mixture (ca 1:1) of *Z* and *E* isomers (^1H NMR): FTIR (CH_2Cl_2 cast) 3086, 3056, 3022, 2935, 2858, 1954, 1811, 1491, 1447, 699; ^1H NMR (CDCl_3 , 400 MHz) δ 1.05-2.24 (m, 7 H), 2.29-2.48 (m, 1 H), 3.15 (ddd, $J = 8.9, 8.9, 4.5$ Hz, 1 H), 3.39 (ddd, $J = 8.9, 8.9, 4.3$ Hz, 1 H), 3.86-4.21 (m, 2 H), 4.52-4.68 (m, 1 H), 7.05 (t, $J = 3.6$ Hz, 0.5 H), 7.17-7.40 (m, 15 H), 7.75 (t, $J = 5.9$ Hz, 0.5 H); ^{13}C NMR (CDCl_3 , 100.6 MHz) δ 23.5 (t), 27.06 (t), 27.12 (t), 31.0 (t), 31.1 (t), 34.7 (d), 35.1 (d), 37.87 (t), 37.89 (t), 64.2 (t), 65.8 (t), 81.3 (d), 83.2 (d), 90.7 (s), 90.9 (s), 127.0 (d), 127.2 (d), 127.5 (d), 127.6 (d), 128.9 (d), 129.1 (d), 144.2 (s), 146.8 (s), 147.9 (d), 151.1 (d); exact mass (electrospray) m/z calcd for $\text{C}_{27}\text{H}_{28}\text{INO}_2$ 548.10570, found 548.10578.

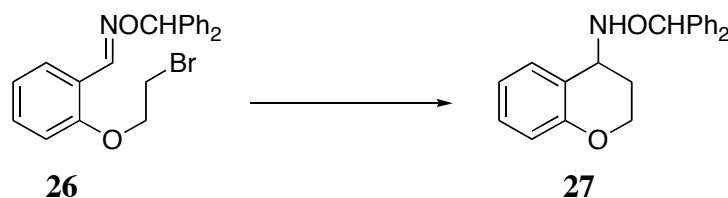
2-(2-Bromoethoxy)benzaldehyde *O*-(Diphenylmethyl)oxime (26**).**



$\text{Ph}_2\text{CHONH}_2^{30}$ (0.0868g, 0.436 mmol) in THF (6 mL) was added dropwise to a stirred solution of 2-(2-bromoethoxy)benzaldehyde¹⁷ (100.0 mg, 0.436 mmol) in THF (2 mL), and the mixture was heated at 65 °C for 4 h. Evaporation of the solvent and flash chromatography of the residue over silica gel (2 x 30 cm), using 10% EtOAc-hexane, gave **26** (176 mg, 98%) as an oil which was a single isomer (^1H NMR): FTIR (CH_2Cl_2 cast) 3061, 3029, 1601, 1451, 1249 cm^{-1} ;

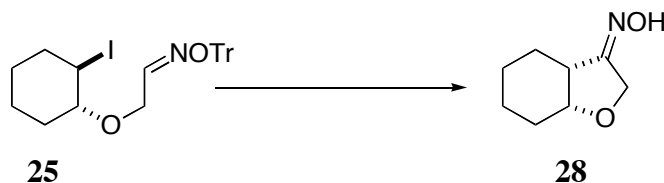
^1H NMR (CDCl_3 , 400 MHz) δ 3.64 (t, J = 6.2 Hz, 2 H), 4.32 (t, J = 6.2 Hz, 2 H), 6.36 (s, 1 H), 6.80-6.98 (m, 2 H), 7.22-7.44 (m, 11 H), 7.70-7.77 (m, 1 H), 8.67 (s, 1 H); ^{13}C NMR (CDCl_3 , 100.6 MHz) δ 28.9 (t), 68.2 (t), 86.8 (d), 112.3 (d), 121.2 (s), 121.4 (d), 126.7 (d), 127.47 (d), 127.5 (d), 128.3 (d), 131.0 (d), 141.2 (s), 145.1 (d), 155.9 (s); exact mass m/z calcd for $\text{C}_{22}\text{H}_{20}^{79}\text{BrNO}_2$ 409.06775, found 409.06671.

***O*-(Diphenylmethyl)-*N*-(chroman-4-yl)hydroxylamine (**27**).**



General procedure A for radical cyclization was followed, using **26** (67.6 mg, 0.165 mmol) in THF (20 mL), Bu_3SnH (0.18 mL, 0.669 mmol) in THF (2 mL), ABC (0.0041 mg, 0.0017 mmol) in THF (2 mL), and $i\text{-Pr}_2\text{NEt}$ (0.144 mL, 0.83 mmol). Evaporation of the solvent and flash chromatography of the residue over silica gel (2 x 30 cm), using 5% EtOAc-hexane, gave hydroxylamine **27** (13.2 mg, 49%) as an oil: FTIR (CH_2Cl_2 cast) 3257, 3061, 3029, 2883, 1951, 1904, 1808, 1608, 1584, 1489, 1454 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 1.95-2.07 (m, 1 H), 2.22 (dddd, J = 14.2, 3.0, 3.0, 3.0 Hz, 1 H), 4.12-4.29 (m, 3 H), 5.30-6.0 (br signal containing s at δ 5.73, 2 H in all), 6.74-6.90 (m, 2 H), 7.10-7.20 (m, 2 H), 7.22-7.42 (m, 10 H); ^{13}C NMR (CDCl_3 , 100.6 MHz) δ 25.7 (t), 53.7 (d), 62.0 (t), 87.4 (d), 117.0 (d), 119.8 (s), 120.1 (d), 127.2 (d), 127.4 (d), 127.60 (d), 127.65 (D), 128.3 (d), 128.4 (d), 129.3(d), 130.3 (d), 141.3 (s), 141.4 (s), 155.7 (s); exact mass (electrospray) m/z calcd for $\text{C}_{22}\text{H}_{22}\text{NO}_2$ ($M + H$) 332.16451, found 332.16431.

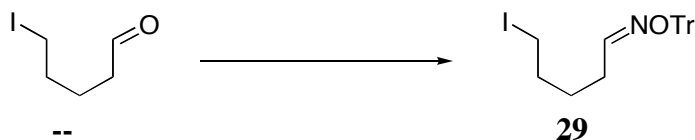
***Cis*-Hexahydrobenzofuran-3(2*H*)-one oxime (**28**).**



General procedure B for radical cyclization was followed, using **25** (180 mg, 0.343 mmol), PhSeSePh (24.0 mg, 0.077 mmol) and Hünig's base (0.26 mL, 1.49 mmol) in THF (20 mL), Bu_3SnH (0.165 mL, 0.613 mmol) in THF (5 mL) and ABC (92.0 mg, 0.377 mmol) in THF

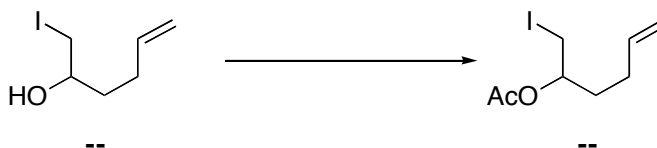
(5 mL). After evaporation of the solvent, flash chromatography of the residue over silica gel (2 x 20 cm), using 20% EtOAc-hexane, gave **28** (4.9 mg, 91%) as an oil which was a mixture (ca 1:2.4) of geometric isomers (^1H NMR): FTIR (CH_2Cl_2 , cast) 2934, 2857 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 1.10-2.11 (m, 8 H), 2.64 (ddd, $J = 10.3, 5.7, 5.7$ Hz, 0.7 H), 3.04 (ddd, $J = 10.9, 5.1, 5.1$ Hz, 0.3 H), 3.91 (ddd, $J = 3.7, 3.7, 3.7$ Hz, 0.3 H), 4.03 (ddd, $J = 4.5, 4.5, 4.5$ Hz, 0.7 H), 4.15-4.66 (two overlapping m, 2 H), 8.38 (s, 0.3 H), 8.54 (s, 0.7 H); ^{13}C NMR (CDCl_3 , 100.6 MHz) δ 20.0 (t), 20.5 (t), 22.95 (t), 23.02 (t), 23.7 (t), 25.5 (t), 27.25 (t), 27.26 (t), 38.8 (d), 40.6 (d), 65.2 (t), 67.0 (t), 77.2 (d), 77.4 (d), 166.3 (s), 166.5 (s); exact mass m/z calcd for $\text{C}_8\text{H}_{13}\text{NO}_2$ 155.09464, found 155.09474.

5-Iodopentanal *O*-(Triphenylmethyl)oxime (29).



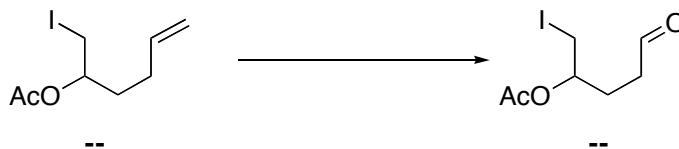
TrONH_2 (460 mg, 1.673 mmol) was added to a solution of 5-iodopentanal^{18,19,20} (355 mg, 1.675 mmol) in CH_2Cl_2 (15 mL) and the mixture was refluxed for 1 h. Evaporation of solvent and flash chromatography of the residue over silica gel (2 x 20 cm), using 20% EtOAc-hexane, gave **29** (694 mg, 89%) as an oil, which was a mixture of two isomers. The isomers were separated by preparative tlc (silica, 3% EtOAc-hexane, plate developed twice): FTIR (CH_2Cl_2 , cast, isomer mixture) 3056, 2930, 1953, 1813, 1596, 1447, 699 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) (less polar isomer, presumably *E*-isomer) δ 1.45-1.54 (m, 2 H), 1.58-1.67 (m, 2 H), 2.15 (ddd, $J = 7.0, 6.1, 6.1$ Hz, 2 H), 3.03 (t, $J = 6.8$ Hz, 2 H), 7.20-7.38 (m, 15 H), 7.54 (t, $J = 5.9$ Hz, 1 H); ^1H NMR (CDCl_3 , 400 MHz) (more polar isomer, presumably *Z*-isomer) δ 1.60-1.70 (m, 2 H), 1.82-1.92 (m, 2 H), 2.56 (ddd, $J = 7.6, 7.6, 5.4$ Hz, 2 H), 3.18 (t, $J = 6.9$ Hz, 2 H), 6.72 (t, $J = 5.5$ Hz, 1 H), 7.20-7.38 (m, 15 H); ^{13}C NMR (CDCl_3 , 100.6 MHz) (less polar isomer) δ 6.4 (t), 26.8 (t), 28.1 (t), 31.9 (t), 90.2 (s), 126.9 (d), 127.4 (d), 129.1 (d), 144.4 (s), 150.2 (d); ^{13}C NMR (CDCl_3 , 100.6 MHz) (more polar isomer) δ 6.0 (t), 25.1 (t), 26.9 (t), 32.9 (t), 90.3 (s), 127.0 (d), 127.5 (d), 128.9 (d), 144.5 (s), 151.2 (d); exact mass (electrospray) (less polar isomer) m/z calcd for $\text{C}_{24}\text{H}_{24}\text{INNaO}$ 492.07949, found 492.07974; exact mass (electrospray) (more polar isomer) m/z calcd for $\text{C}_{24}\text{H}_{24}\text{INNaO}$ 492.07949, found 492.07961.

Acetic Acid 1-(Iodomethyl)-4-[(triphenylmethyloxy)imino]butyl Ester (30). (a)
Acetic Acid (1-Iodomethyl)pent-4-enyl Ester.



Ac₂O (2.6 mL, 27.41 mmol) and then pyridine (2.22 mL, 27.41 mmol) were added successively to a stirred and cooled (0 °C) portion of 1-iodohex-5-en-2-ol³¹ (774 mg, 3.426 mmol). Stirring at 0 °C was continued for 4 h, the ice bath was left in place, but not recharged, and stirring was continued overnight. The solution was diluted with water, extracted with Et₂O, and washed with saturated aqueous NaHCO₃, water and brine. The combined organic extracts were dried (Na₂SO₄) and evaporated. Flash chromatography of the residue over silica gel (2 x 30 cm), using 30% EtOAc-hexane, gave acetic acid (1-iodomethyl)pent-4-enyl ester (763.9 mg, 83%) as an oil: FTIR (CH₂Cl₂, cast) 2924, 1740, 1235, 914; ¹H NMR (CDCl₃, 400 MHz) δ 1.68-1.80 (m, 2 H), 2.03-2.14 (m including s at δ at 2.08, 5 H), 3.29 (dddd, *J* = 10.6, 10.6, 10.6, 5.1 Hz, 2 H), 4.70 (dddd, *J* = 10.9, 5.1, 5.1, 5.1 Hz, 1 H), 4.94-5.06 (m, 2 H), 5.76 (dddd, *J* = 16.9, 10.2, 6.6, 6.6 Hz, 1 H); ¹³C NMR (CDCl₃, 100.6 MHz) δ 8.1 (t), 21.0 (q), 29.3 (t), 33.3 (t), 71.7 (d), 115.5 (t), 137.0 (d), 170.3 (s); exact mass (electrospray) *m/z* calcd for C₈H₁₃INaO₂ 290.98525, found 290.98494.

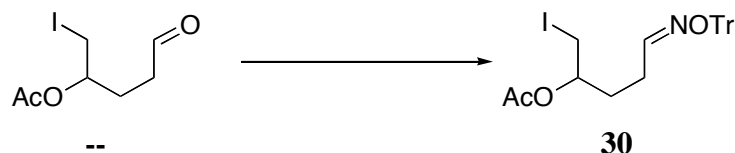
(b) Acetic Acid 1-(Iodomethyl)-4-oxobutyl Ester.



OsO₄ (0.1 M in PhMe, 2.85 mL, 0.285 mmol) was added in one portion to a stirred solution of acetic acid (1-iodomethyl)pent-4-enyl ester (763.9 mg, 2.85 mmol) in water (7 mL) and dioxane (21 mL). The solution was stirred at room temperature under Ar for 20 min. Then NaIO₄ was added in small portions over 20 min. The mixture was stirred at room temperature for an additional 2 h, quenched with water and extracted with EtOAc. The combined organic extracts were washed with brine and dried (MgSO₄). Evaporation of solvent and flash chromatography of the residue over silica gel (2 x 20 cm), using 30% EtOAc-hexane, gave acetic acid 1-(iodomethyl)-4-oxobutyl ester (614 mg, 79%) as an oil: FTIR (CH₂Cl₂, cast) 2830, 1734, 1720, 1235 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 1.80-2.20 (m including s at δ 2.05, 5 H), 2.42-2.54 (m, 2 H), 3.29 (dddd, *J* = 10.7, 10.7, 10.7, 5.4 Hz, 2 H), 4.73 (dddd, *J* = 5.3, 5.3, 4.2, 4.2 Hz, 1 H), 9.75 (t, *J* = 1.2 Hz, 1 H); ¹³C NMR (CDCl₃, 100.6 MHz) δ 7.1 (t), 20.8 (q), 26.5 (t), 39.4

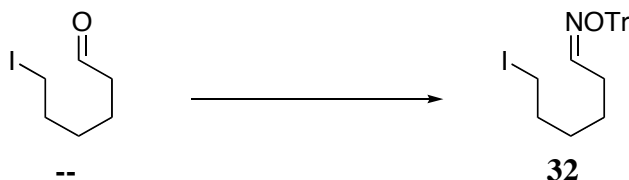
(t), 71.4 (d), 170.1 (s), 200.4 (d); exact mass (electrospray) m/z calcd for $C_7H_{11}INaO_3$ 292.96452, found 292.96427.

(c) Acetic Acid 1-(Iodomethyl)-4-[(triphenylmethyloxy)imino]butyl Ester (30).



TrONH₂ (623 mg, 2.265 mmol) was added to a stirred solution of acetic acid 1-(iodomethyl)-4-oxobutyl ester (611.4 mg, 2.265 mmol) in CH₂Cl₂ (40 mL) and stirring was continued for 1 h (Ar atmosphere). Evaporation of the solvent and flash chromatography of the residue over silica gel (2 x 30 cm), using 15% EtOAc-hexane, gave **30** (892.8 mg, 75%) as a mixture (ca 1:1) of *Z* and *E* isomers (¹H NMR): FTIR (CH₂Cl₂, cast) 3057, 1740, 1233, 700 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) (the spectrum of the isomer mixture is too complicated to be informative); one isomer had: δ 1.74 (ddd, *J* = 7.0, 7.0, 7.0 Hz, 2 H), 2.05 (s, 3 H), 2.17 (ddd, *J* = 7.0, 7.0, 7.0 Hz, 2 H), 3.01-3.20 (m, 2 H), 4.51 (dddd, *J* = 5.8, 5.8, 5.8, 5.8 Hz, 1 H), 7.10-7.48 (m, 15 H), 7.55 (t, *J* = 5.1 Hz, 1 H); ¹³C NMR (CDCl₃, 100.6 MHz) (isomer mixture) δ 7.3 (t), 8.1 (t), 21.01 (q), 21.04 (q), 22.3 (t), 25.5 (t), 29.7 (t), 30.5 (t), 30.7 (t), 71.3 (d), 72.0 (d), 90.5 (s), 90.7 (s), 127.1 (d), 127.2 (d), 127.5 (d), 127.6 (d), 127.9 (d), 129.0 (d), 129.2 (d), 144.4 (s), 144.5 (s), 149.4 (d), 150.3 (d), 170.1 (s), 170.2 (s); exact mass (electrospray) m/z calcd for C₂₆H₂₆INNaO₃ 550.08497, found 550.08463.

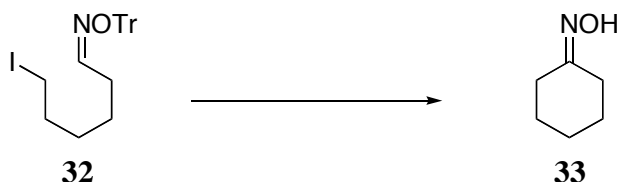
6-Iodoheptanal *O*-(Triphenylmethyl)oxime (32).



TrONH₂ (426 mg, 1.549 mmol) was added to a stirred solution of 6-iodoheptanal^{18,19,20} (350 mg, 1.549 mmol) in CH₂Cl₂ (20 mL) and stirring was continued for 2 h (Ar atmosphere). Evaporation of the solvent and flash chromatography of the residue over silica gel (3 x 30 cm), using 30% EtOAc-hexane, gave **32** (684.1 mg, 91%) as an oil, which was a mixture of two isomers (¹H NMR). These could be separated but only the less polar was obtained pure, and the

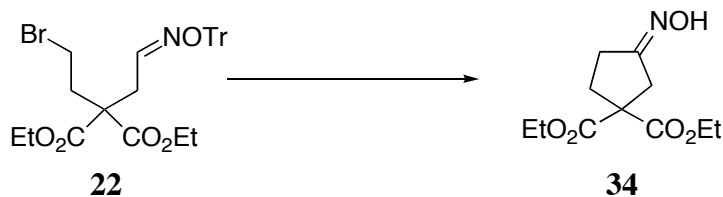
isomer mixture was used for radical cyclization: FTIR (CH₂Cl₂, cast) (isomer mixture) 2930, 1597, 699 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) (less polar isomer) δ 1.20-1.50 (m, 4 H), 1.73 (quintet, J = 7.2 Hz, 2 H), 2.15 (q, J = 7.0 Hz, 2 H), 3.09 (t, J = 7.0 Hz, 2 H), 7.20-7.40 (m, 15 H), 7.57 (t, J = 5.9 Hz, 1 H); ¹³C NMR (CDCl₃, 100.6 MHz) (less polar isomer) δ 6.8 (t), 25.3 (t), 29.2 (t), 29.5 (t), 33.2 (t), 90.2 (s), 127.0 (d), 127.5 (d), 129.2 (d), 144.6 (s), 150.8 (d); exact mass (electrospray) m/z calcd for C₂₅H₂₆INNaO 506.09514, found 506.09458.

Cyclohexanone Oxime (**33**).²⁵



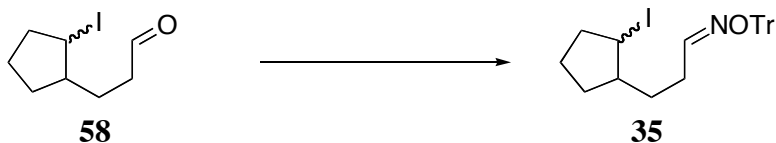
General procedure B for radical cyclization was followed using **32** (666 mg, 1.379 mmol), PhSeSePh (86.0 mg, 0.276 mmol) and Hünig's base (0.96 mL, 5.511 mmol) in THF (25 mL), Bu₃SnH (2.066 mmol) in THF (8 mL) and ABC (336 mg, 1.375 mL) in THF (8 mL). After evaporation of the solvent, flash chromatography of the residue over silica gel (2 x 30 cm), using 30% EtOAc-hexane, gave cyclohexanone oxime (**33**)²⁵ (136 mg, 87%).

3-(Hydroxyimino)cyclopentane-1,1-dicarboxylic Acid Diethyl Ester (**34**) (from **22**).



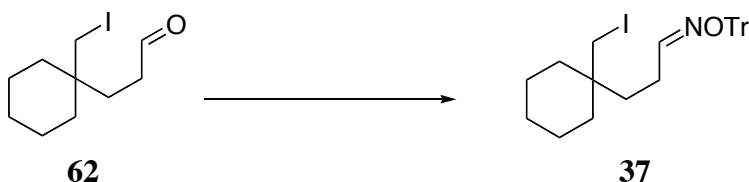
General procedure B for radical cyclization was followed, using **22** (110 mg, 0.194 mmol), PhSeSePh (12.2 mg, 0.0391 mmol) and Hünig's base (0.1 mL, 0.776 mmol) in dry THF (20 mL), Bu₃SnH (0.063 mL, 0.234 mmol) in THF (3 mL) and ABC (47.5 mg, 0.194 mmol) in THF (3 mL). After evaporation of the solvent, flash chromatography of the residue over silica gel (2 x 30 cm), using 30% EtOAc-hexane, gave **34** (36.4 mg, 77%) as a mixture (ca 1:1) of *Z* and *E* isomers, identical with material made from the iodide.

3-(2-Iodocyclopentyl)propanal *O*-(Triphenylmethyl)oxime (**35**).



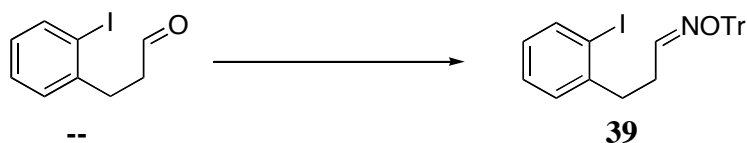
TrONH₂ (51.0 mg, 0.186 mmol) was added in one portion to a stirred solution of **58** (probably largely one isomer) (47.0 mg, 0.187 mmol) in dry CH₂Cl₂ (3 mL) and stirring was continued for 1 h. Evaporation of the solvent and flash chromatography of the residue over silica gel (1.5 x 25 cm), using 10% EtOAc-hexane, gave **35** (77.8 mg, 82%) as an oil, which was a mixture (1:1.3) of isomers (¹H NMR): FTIR (CH₂Cl₂, cast) 2954, 2922, 1954, 1887, 1811, 1631, 1448, 700 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 0.6-2.80 (m, 11 H), 4.31 (t, *J* = 4.3 Hz, 0.5 H), 4.52 (t, *J* = 4.3 Hz, 0.5 H), 6.75 (t, *J* = 5.6 Hz, 0.42 H), 7.16-7.44 (m, 15 H), 7.57 (t, *J* = 6.1 Hz, 0.57 H); ¹³C NMR (CDCl₃, 100.6 MHz) δ 21.6 (t), 21.8 (t), 24.8 (t), 27.9 (t), 28.7 (t), 29.0 (t), 33.205 (t), 33.213 (t), 38.9 (t), 44.3 (d), 45.1 (d), 45.2 (d), 46.4 (d), 90.2 (s), 90.4 (s), 127.0 (d), 127.1 (d), 127.5 (d), 127.6 (d), 129.0 (d), 129.2 (d), 144.56 (s), 144.58 (s), 150.5 (d), 151.6 (d). A satisfactory mass spectrum could not be obtained by electron impact or electrospray methods.

3-[(1-Iodomethyl)cyclohexyl]propanal *O*-(Triphenylmethyl)oxime (37**).**



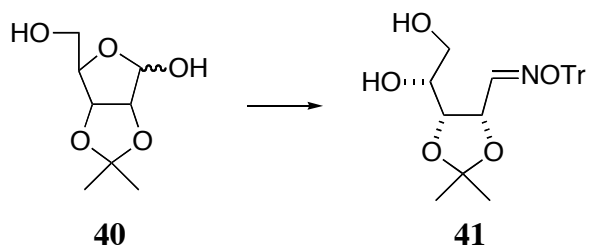
TrONH₂ (610 mg, 2.22 mmol) was added in one portion to a stirred solution of **62** (620 mg, 2.22 mmol) in dry CH₂Cl₂ (22 mL) containing 4Å molecular sieves. Stirring was continued for 4 h and the mixture was filtered and evaporated. Flash chromatography of the residue over silica gel (3 x 30 cm), using 10% EtOAc-hexane, gave **37** (1.12 g, 94%) as a mixture (1:1.3) of geometric isomers (¹H NMR): FTIR (neat film, microscope) 2926, 2853, 1953, 1886, 1810, 1723, 1449, 699 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 1.30-1.60 (m, 12 H), 2.0-2.10 (m, 1.43 H), 2.38-2.50 (m, 0.64 H), 3.10 (s, 1.31 H), 3.25 (s, 0.63 H), 6.75 (t, *J* = 5.6 Hz, 0.29 H), 7.15-7.40 (m, 15 H), 7.56 (t, *J* = 6.1 Hz, 0.65 H); ¹³C NMR (CDCl₃, 100.6 MHz) δ 20.2 (t), 20.9 (t), 21.2 (t), 21.7 (t), 21.8 (t), 23.7 (t), 25.96 (t), 26.01 (t), 34.83 (s/t), 34.85 (s/t), 35.05 (s/t), 35.07 (s/t), 90.2 (s), 90.4 (s), 126.9 (d), 127.1 (d), 127.4 (d), 127.6 (d), 129.0 (d), 129.2 (d), 144.5 (s), 144.6 (s), 151.4 (d), 151.8 (d); exact mass (electrospray) *m/z* calcd for C₂₉H₃₂INNaO 560.14209, found 560.14210.

3-(2-Iodophenyl)propanal *O*-(Triphenylmethyl)oxime (39**).**



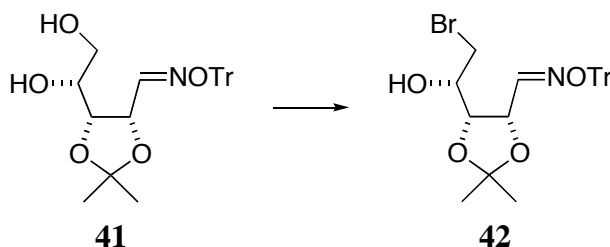
TrONH₂ (30 mg, 0.11 mmol) was added to a stirred solution of 3-(2-iodophenyl)propanal²¹ (29.1 mg, 0.11 mmol) in dry CH₂Cl₂ (2 mL) and stirring was continued for 2 h. Evaporation of the solvent and flash chromatography of the residue over silica gel (2 x 20 cm), using 10% EtOAc-hexane, gave **39** (0.054g, 94%) as an oil which was a mixture of *Z* and *E* isomers (¹H NMR), containing some impurities; the material was used directly in the next step.

2,3-*O*-(1-Methylethylidene)-D-ribose *O*-(Triphenylmethyl)oxime (41**).**



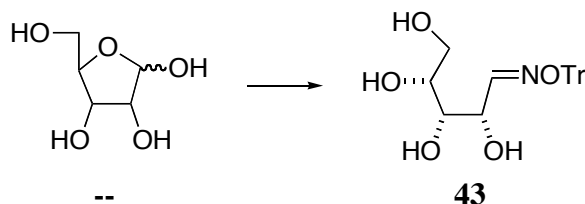
The general procedure for making *O*-trityl oximes was followed, using TrONH₂ (475.1 mg, 1.723 mmol) and 2,3-*O*-(1-methylethylidene)-D-ribose³² (**40**) (298.4 mg, 1.571 mmol) in THF (10 mL). Flash chromatography of the residue over silica gel (1.7 x 35 cm), using 40% EtOAc-hexane, gave **41** (604 mg, 86%) as a foam, which was a 2.8:1 mixture of geometric isomers (¹H NMR): FTIR (CH₂Cl₂ cast) 3438, 3057, 3033, 2986, 2934, 1597 cm⁻¹; ¹H NMR (CDCl₃, 500 MHz) δ 1.28, 1.38, 1.45, 1.50 (four s, 6 H in all), 1.63-2.01 (br s, 2 H), 3.31-3.72 (m, 3 H), 3.97-4.02 (m, 0.86 H), 4.33-4.41 (m, 0.32 H), 4.64 (t, *J* = 7.0 Hz, 0.76 H), 5.45 (t, *J* = 7.0 Hz, 0.27 H), 6.92 (d, *J* = 6.4 Hz, 0.26 H), 7.18-7.38 (m, 15 H), 7.62 (d, *J* = 6.4 Hz, 0.73 H); ¹³C NMR (CDCl₃, 125.7 MHz) δ 25.4 (q), 25.6 (q), 27.6 (q), 27.8 (q), 64.0 (t), 64.2 (t), 69.6 (d), 70.4 (d), 72.2 (d), 75.3 (d), 78.4 (d), 79.1 (d), 91.3 (s), 92.5 (s), 110.1 (s), 110.5 (s), 127.2 (d), 127.3 (d), 127.6 (d), 127.7 (d), 128.8 (d), 129.0 (d), 143.4 (s), 143.8 (s), 150.5 (d), 150.7 (d); exact mass (electrospray) *m/z* calcd for C₂₇H₂₉NNaO₅ 470.1943, found 470.1942.

5-Bromo-5-deoxy-2,3-*O*-(1-methylethylidene)-D-ribose *O*-(Triphenylmethyl)oxime (42).



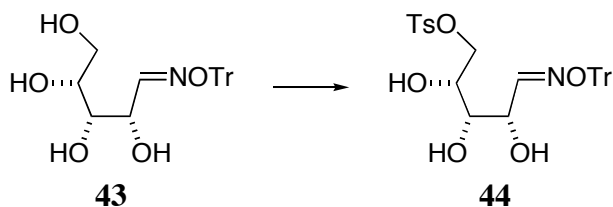
Ph₃P (3.1 g, 11.7 mmol) was added to a stirred and cooled (ice-water) solution of **41** (2.6 g, 5.9 mmol) in pyridine (30 mL), and then CBr₄ (2.14 g, 6.45 mmol) was added in several portions at the same temperature. After the addition, the mixture was heated at 65 °C for 2 h, cooled, and diluted with MeOH (10 mL). Evaporation of the solvent, and flash chromatography of the residue over silica gel (4 x 32 cm), using 10% EtOAc-hexane, gave **42** (2.65 g, 89%) as a foam. The material was a single isomer, but the oxime geometry was not determined: FTIR (CH₂Cl₂ cast) 3564, 3087, 3057, 3033, 2987, 2934, 1595, 1491 cm⁻¹; ¹H NMR (CDCl₃, 200 MHz) δ 1.30 (s, 3 H), 1.42 (s, 3 H), 1.92 (d, *J* = 6.0 Hz, 1 H), 3.33-3.61 (m, 3 H), 3.93-4.09 (m, 1 H), 4.72 (t, *J* = 6.0 Hz, 1 H), 7.21-7.51 (m, 15 H), 7.60 (d, *J* = 9.0 Hz, 1 H); ¹³C NMR (CDCl₃, 50.3 MHz) δ 25.5 (q), 27.7 (q), 36.8 (t), 68.8 (d), 75.1 (d), 79.1 (d), 91.2 (s), 110.5 (s), 127.3 (d), 127.8 (d), 128.9 (d), 143.9 (s), 149.8 (d); exact mass (electrospray) *m/z* calcd for C₂₇H₂₈⁷⁹BrNNaO₄ 532.1099, found 532.1100.

D-Ribose *O*-(Triphenylmethyl)oxime (43).



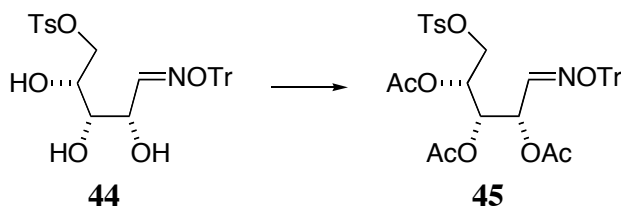
The general procedure for making *O*-trityl oximes was followed, using TrONH₂ (1.06 g, 3.86 mmol) and D-ribose (580 mg, 3.86 mmol) in THF (10 mL). Flash chromatography of the residue over silica gel (1.7 x 35 cm), using 80% EtOAc-hexane, gave **43** (1.438 g, 91%) as a foam. The material was used directly, without characterization.

D-Ribose 5-(4-Methylbenzenesulfonate) *O*-(Triphenylmethyl)oxime (44).



TsCl (460 mg, 2.41 mmol) was added to a stirred and cooled (0 °C) solution of **43** (890 mg, 2.19 mmol) in pyridine (5 mL), and stirring was continued overnight. Water (10 mL) was added to quench the reaction, and the mixture was extracted with Et₂O (2 x 10 mL). The combined organic extracts were dried (MgSO₄) and evaporated. Flash chromatography of the residue over silica gel (1.7 x 30 cm), using 50% EtOAc-hexane, gave **44** (658 mg, 54%) as a foam. The material was used directly, without characterization.

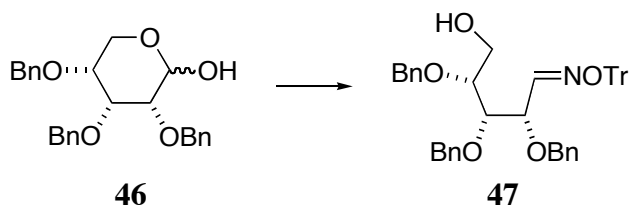
D-Ribose 2,3,4-Triacetate 5-(4-Methylbenzenesulfonate) O-(Triphenylmethyl)oxime (45).



Pyridine (4.6 mL, 80 mmol) was added to a stirred and cooled (0 °C) solution of **44** (405 mg, 0.722 mmol) in Ac₂O (10.6 mL, 144 mmol), and stirring was continued overnight. Water (15 mL) was added and the mixture was extracted with Et₂O (3 x 10 mL). The combined organic extracts were washed with saturated aqueous NaHCO₃ and water (10 mL), dried (MgSO₄), and evaporated. Flash chromatography of the residue over silica gel (1.7 x 30 cm), using 20% EtOAc-hexane, gave **45** (459 mg, 93%) as a foam. The material, which contained small impurities, appeared to be a single isomer, but was used with only partial characterization (¹H NMR): ¹H NMR (CDCl₃, 300 MHz) δ 1.86 (s, 3 H), 1.91 (s, 6 H), 2.4 (s, 3 H), 4.01-4.3 (m, 2 H), 4.94-5.09 (m, 1 H), 5.22-5.48 (m, 2 H), 7.18-7.42 (m, 17 H), 7.49 (d, *J* = 7 Hz, 1 H), 7.72 (d, *J* = 8 Hz, 2 H).

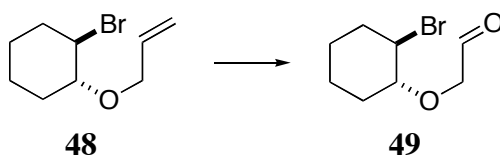
The oxime geometry was not determined.

2,3,4-Tris-O-(phenylmethyl)-D-ribose O-(Triphenylmethyl)oxime (47).

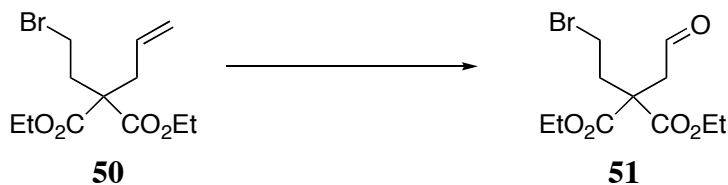


The general procedure for making *O*-trityl oximes was followed, using TrONH_2 (80 mg, 0.29 mmol) and **46**³³ (110 mg, 0.26 mmol) in THF (5 mL). Flash chromatography of the residue over silica gel (1.7 x 25 cm), using 15% EtOAc-hexane, gave **47** (167 mg, 94%) as a foam, which was a 5:1 mixture of geometrical isomers (^1H NMR): FTIR (CH_2Cl_2 cast) 3462, 3060, 2869, 1597 cm^{-1} ; ^1H NMR (CDCl_3 , 500 MHz) (in some regions the multiplets include AB quartet signals) δ 3.24-3.31 (m, 1 H), 3.60-3.78 (m, 2.3 H), 3.85 (dd, $J = 7.0, 2.8$ Hz, 0.93 H), 3.95 (dd, $J = 7.0, 2.8$ Hz, 0.25 H), 4.01-4.22 (m, 3.6 H), 4.37-4.58 (m, 2 H), 4.62-4.89 (m, 2 H), 5.24 (dd, $J = 6.0, 2.0$ Hz, 0.2 H), 6.84 (d, $J = 7.0$ Hz, 0.2 H), 6.98-7.44 (m, 30 H), 7.66 (d, $J = 7.0$ Hz, 0.98 H); ^{13}C NMR (CDCl_3 , 125.7 MHz) δ 61.0 (t), 70.5 (t), 72.4 (t), 74.5 (t), 77.1 (d), 78.5 (d), 80.4 (d), 90.8 (s), 127.0 (d), 127.46 (d), 127.49 (d), 127.53 (d), 127.55 (d), 127.57 (d), 127.6 (d), 127.7 (d), 127.81 (d), 127.84 (d), 128.1 (d), 128.21 (d), 128.23 (d), 128.3 (d), 128.31 (d), 128.34 (d), 129.0 (d), 129.1 (d), 137.6 (s), 137.7 (s), 138.0 (s), 144.2 (s), 148.9 (d); exact mass m/z calcd for $\text{C}_{45}\text{H}_{43}\text{NO}_5$ 678.3219, found 678.3217.

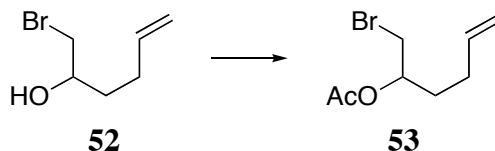
***Trans*-2-[2-Bromocyclohexyl]oxy]acetaldehyde (**49**).**



Ozone was bubbled through a stirred and cooled ($-78\text{ }^\circ\text{C}$) solution of **48**³⁴ (697.4 mg, 3.184 mmol) in CH_2Cl_2 (15 mL) for 1 h. The mixture was then flushed with O_2 for 10 min, and Ph_3P (2.5 g, 9.6 mmol) was added. Stirring was continued overnight, the cold bath being left in place, but not recharged. Evaporation of the solvent and flash chromatography of the residue over silica gel (1.7 x 30 cm), using 15% EtOAc-hexane, gave **49** (609 mg, 87%) as a colorless oil: FTIR (CH_2Cl_2 cast) 3450, 2937, 2860, 1735 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 1.19-2.42 (m, 8 H), 3.29-3.43 (m, 1 H), 3.82-4.11 (m, 1 H), 4.21 (s, 2 H), 9.79 (s, 1 H); ^{13}C NMR (CDCl_3 , 100.6 MHz) δ 22.9 (t), 25.1 (t), 30.9 (t), 35.5 (t), 54.8 (d), 75.3 (t), 83.3 (d), 200.8 (d); exact mass m/z calcd for $\text{C}_8\text{H}_{13}^{79}\text{BrNaO}_2$ 242.9996, found 242.9991.

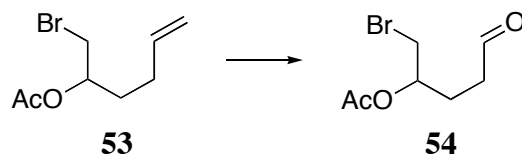
Diethyl (2-Bromoethyl)(2-oxoethyl)propanedioate (51).

OsO₄ (0.1 M in PhMe, 0.19 mL, 0.019 mmol) was added in one portion to a stirred solution of **50**³⁵ (59.0 mg, 0.192 mmol) in a mixture of dioxane (3 mL) and water (1 mL). The mixture was stirred for 30 min and then NaIO₄ (102.7 mg, 0.48 mmol) was added in several small portions. The resulting solution was stirred for 2 h, then quenched with water and extracted with EtOAc. The combined organic extracts were washed with brine and dried (Na₂SO₄). Evaporation of the solvent and flash chromatography of the residue over silica gel (1.5 x 20 cm), using 30% EtOAc-hexane, gave aldehyde **51**³⁶ (56 mg, 94%) as an oil: ¹H NMR (CDCl₃, 300 MHz) δ 1.22 (t, *J* = 7.1 Hz, 6 H), 2.52-2.62 (m, 2 H), 3.02 (apparent d, *J* = 1.3 Hz, 2 H), 3.32-3.42 (m, 2 H), 4.22 (q, *J* = 7.1 Hz, 4 H), 9.74 (t, *J* = 1.3 Hz, 1 H).

(1-Bromomethyl)-4-penten-1-yl Acetate (53).

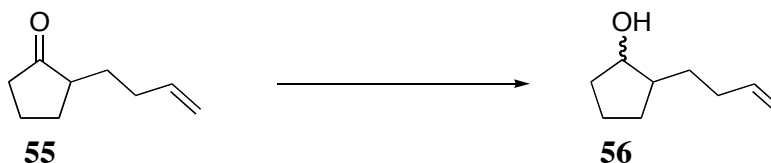
Pyridine (1 mL) was added to a stirred and cooled (0 °C) mixture of Ac₂O (5 mL) and **52**³⁷ (310 mg, 1.73 mmol), and stirring was continued overnight. Water (15 mL) was added and the mixture was extracted with Et₂O (3 x 10 mL). The combined organic extracts were washed with saturated aqueous NaHCO₃, and water (1 x 10 mL), dried, and evaporated. Flash chromatography of the residue over silica gel (1.7 x 30 cm), using 5% EtOAc-hexane, gave **53** (365 mg, 96%) as a colorless oil: FTIR (CH₂Cl₂ cast) 3465, 3078, 2977, 2924, 1742, 1641 cm⁻¹; ¹H NMR (CDCl₃, 500 MHz) δ 1.73-1.82 (m, 2 H), 2.02-2.14 (m, including a singlet at δ 2.07, 5 H in all), 3.38-3.57 (m, 2 H), 4.87-5.09 (m, 3 H), 5.70-5.82 (m, 1 H); ¹³C NMR (CDCl₃, 125.7 MHz) δ 21.1 (q), 29.4 (t), 31.8 (t), 34.1 (t), 71.8 (d), 115.5 (t), 136.9 (d), 170.2 (s). A satisfactory mass spectrum could not be obtained by electron impact or electrospray methods.

(1-Bromomethyl)-4-oxobutyl Acetate (54).



OsO₄ (2.5% w/w in *t*-BuOH, 2.0 mL, 0.16 mmol) was added to a stirred mixture of **53** (358 mg, 1.62 mmol), water (8 mL), CCl₄ (8 mL) and *t*-BuOH (4 mL). After 20 min, the mixture had become black. NaIO₄ (870 mg, 4.05 mmol) was then added in one portion and the resulting mixture was stirred for 6 h. Brine (10 mL) was added, and the mixture was extracted with Et₂O (3 x 10 mL). The combined organic extracts were washed with water (10 mL) and aqueous NaHSO₃ (10%, 15 mL), dried (MgSO₄), and evaporated. Flash chromatography of the residue over silica gel (1.7 x 28 cm), using 20% EtOAc-hexane, gave **54** (289 mg, 80%) as a colorless oil. The material appeared to be a mixture of the aldehyde and its hydrate (¹H and ¹³C NMR): FTIR (CH₂Cl₂ cast) 3459, 2966, 2936, 1739 cm⁻¹; ¹H NMR (CDCl₃, 500 MHz) δ 1.44-2.56 (m, including a singlet at δ 2.07, 7 H in all), 3.37-3.49 (m, 2 H), 4.82-5.11 (m, 1.6 H), 9.76 (t, *J* = 1.3 Hz, 0.3 H); ¹³C NMR (CDCl₃, 100.6 MHz) δ 20.8 (q), 20.9 (q), 25.0 (t), 26.4 (t), 29.7 (t), 33.4 (t), 33.8 (t), 39.5 (t), 71.5 (d), 71.8 (d), 100.5 (d), 170.2 (s), 170.3 (s), 200.4 (d); exact mass (electrospray) *m/z* calcd for C₇H₁₂⁷⁹BrO (M + H) 222.9969, found 222.9967.

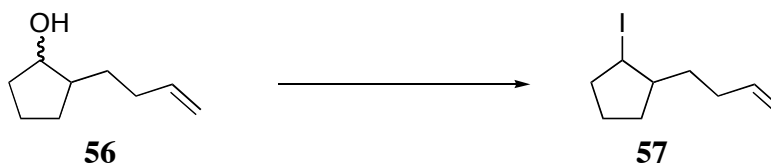
2-(But-3-enyl)cyclopentanol (**56**).



NaBH₄ (340 mg, 8.99 mmol) was added to a stirred and cooled (0 °C) **55**³⁸ (618 mg, 4.478 mmol) and dry MeOH (40 mL). Stirring was continued for 5 h, and the mixture was quenched with saturated aqueous NH₄Cl and extracted with EtOAc. The combined organic extracts were washed with brine, dried (Na₂SO₄) and evaporated. Flash chromatography of the residue over silica gel (2 x 30 cm), using 30% EtOAc-hexane, gave **56**³⁹ (590 mg, 94%) as an oil, which was a mixture of *cis* and *trans* isomers, with one of these greatly predominating (¹H NMR): FTIR (CH₂Cl₂, cast) 3339, 2954, 2925, 2872, 1640 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 1.03-1.32 (m, 2 H), 1.44-1.75 (m, 6 H), 1.78-2.20 (m, 4 H), 3.80 (q, *J* = 5.5 Hz, 1 H), 4.87-5.05 (m, 2 H), 5.80 (dddd, *J* = 16.9, 10.2, 6.7, 6.7 Hz, 1 H); ¹³C NMR (CDCl₃, 100.6 MHz) (major isomer only) δ 21.7 (t), 29.8 (t), 32.3 (t), 32.9 (t), 34.6 (t), 47.7 (d), 79.0 (d), 114.2 (t), 138.8 (d);

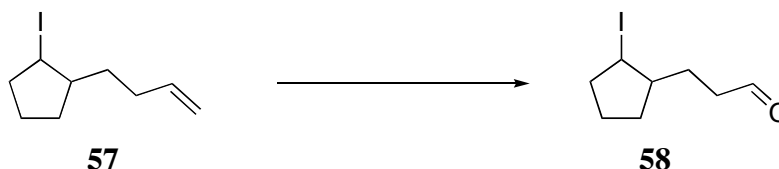
exact mass m/z calcd for $C_9H_{16}O$ 140.12012, found 140.11971.

1-(But-3-enyl)-2-iodocyclopentane (57**).**



Imidazole (698 mg, 10.25 mmol) was added to a stirred solution of **56** (377.8 mg, 2.699 mmol) in THF (30 ml). The mixture was cooled to 0 °C and stirred for 10 min. Ph_3P (1.91 g, 7.282 mmol) was added in one portion and stirring was continued for additional 10 min. I_2 (1.78 g, 7.013 mmol) was then added in one portion and stirring at 0 °C was continued for 2 h. The ice bath was left in place, but not recharged, and stirring was continued overnight. The mixture was quenched with saturated aqueous $Na_2S_2O_3$ and extracted with Et_2O . The combined organic extracts were washed with water and brine, dried (Na_2SO_4) and evaporated. Flash chromatography of the residue over silica gel (2 x 20 cm), using hexane, gave **57** (383.6 mg, 57%) as an oil, which appeared to be a single isomer (NMR): FTIR (CH_2Cl_2 , cast) 2959, 2924, 2867, 910 cm^{-1} ; 1H NMR ($CDCl_3$, 300 MHz) δ 0.84-0.99 (m, 1 H), 1.09-2.43 (m, 10 H), 3.79 (q, $J = 8.2$ Hz, 0.1 H), 4.55 (ddd, $J = 4.1, 4.1, 4.1$ Hz, 0.9 H), 4.92-5.10 (m, 2 H), 5.83 (dddd, $J = 16.7, 10.2, 6.7, 6.7$ Hz, 1 H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 21.7 (t), 28.9 (t), 32.2 (t), 35.9 (t), 38.9 (t), 45.2 (d), 46.2 (d), 114.7 (t), 138.5 (d); exact mass m/z calcd for C_9H_{15} (M - I) 123.11738, found 122.11701.

3-(2-Iodocyclopentyl)propanal (58**).**



OsO_4 in $PhMe$ (0.1 M, 0.40 mL, 0.04 mmol) was added in one portion to a stirred solution of **57** (100 mg, 0.40 mmol) in water (1 mL) and dioxane (3 mL). Stirring was continued for 20 min and then $NaIO_4$ (220 mg, 1.029 mmol) was added in small portions over 10 min. The mixture was stirred for an additional 2 h, diluted with $EtOAc$ and washed with water and brine, dried ($MgSO_4$) and evaporated. Flash chromatography of the residue over silica gel (1.5 x 30

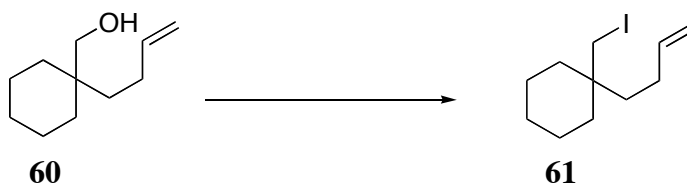
cm), using 10% EtOAc-hexane, gave **58** (57.2 mg, 57%) as an oil, which was largely a single isomer (^1H NMR): FTIR (CH_2Cl_2 , cast) 2952, 2865, 1723, 668 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 0.84-2.60 (m, 11 H), 3.70 (q, $J = 8.3$ Hz, 0.11 H), 4.51 (ddd, $J = 4.3, 4.3, 4.3$ Hz, 0.89 H), 9.78 (t, $J = 1.7$ Hz, 1 H); ^{13}C NMR (CDCl_3 , 100.6 MHz) δ 21.7 (t), 28.9 (t), 29.1 (t), 38.7 (t), 42.1 (t), 43.7 (d), 45.9 (d), 201.9 (d); exact mass m/z calcd for $\text{C}_8\text{H}_{13}\text{O}$ (M - I) 125.09664, found 125.09652.

[1-(But-3-enyl)cyclohexyl]methanol (60**).**



DIBAL (1 M, 15.6 mL, 15.6 mmol) was added dropwise to a stirred and cooled ($-78\text{ }^{\circ}\text{C}$) solution of ester **59**⁴⁰ (1.17 g, 5.97 mmol) in dry CH_2Cl_2 (40 mL). The mixture was stirred at $-78\text{ }^{\circ}\text{C}$ for 4 h. The mixture was quenched with solid sodium potassium tartrate (ca 3 g) and glycerol (ca 6 mL) and the cold bath was removed and stirring was continued overnight, by which stage two layers had formed. The mixture was extracted with EtOAc (3 times) and the combined organic extracts were washed with brine, dried (Na_2SO_4) and evaporated. Flash chromatography of the residue over silica gel (3 x 20 cm), using 10% EtOAc-hexane, gave **60** (780 mg, 78%) as a colorless oil: FTIR (CH_2Cl_2 cast) 3345, 2926, 2853, 1454 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz) δ 1.23-1.51 (m, 12 H), 1.92-2.05 (m, 2 H), 3.43 (d, $J = 4.5$ Hz, 2 H), 4.87-5.09 (m, 2 H), 5.84 (dddd, $J = 16.8, 10.1, 6.6, 6.6$ Hz, 1 H); ^{13}C NMR (CDCl_3 , 100.6 MHz) δ 21.4 (t), 26.3 (t), 27.4 (t), 32.3 (t), 33.9 (t), 36.9 (s), 68.2 (t), 113.9 (t), 139.6 (d); exact mass m/z calcd for $\text{C}_{11}\text{H}_{18}$ (M- H_2O) 150.14085, found 150.14043.

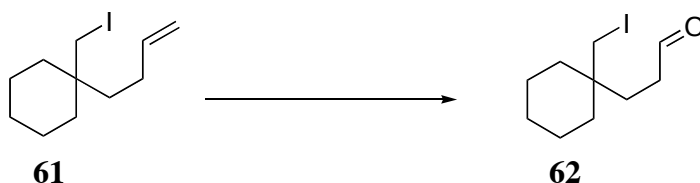
1-(But-3-enyl)-1-(iodomethyl)cyclohexane (61**).**



Imidazole (110 mg, 1.67 mmol) was added to a stirred solution of **60** (74 mg, 0.44 mmol) in THF (5 mL) and the mixture was cooled to $0\text{ }^{\circ}\text{C}$. Stirring was continued until all the

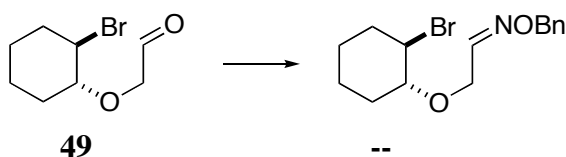
imidazole had dissolved and then Ph_3P (312 mg, 1.19 mmol) was added. Stirring was continued for an additional 10 min, I_2 (290 mg, 1.14 mmol) was added in one portion and stirring was continued for 2 h. The cold bath was left in place, but not recharged, and stirring was continued overnight. The mixture was quenched with saturated aqueous $\text{Na}_2\text{S}_2\text{O}_3$ and extracted with Et_2O (3 times). The combined organic extracts were washed with brine, dried (Na_2SO_4) and evaporated. Flash chromatography of the residue over silica gel (2 x 25 cm), using 10% EtOAc -hexane, gave **61** (120 mg, 100%): FTIR (neat film microscope) 2926, 2851, 1641, 910 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 1.26-1.55 (m, 12 H), 1.86-2.03 (m, 2 H), 3.25 (s, 2 H), 4.91-5.13 (m, 2 H), 5.85 (dddd, $J = 16.8, 10.2, 6.6, 6.6$ Hz, 1 H); ^{13}C NMR (CDCl_3 , 100.6 MHz) δ 21.7 (t), 21.8 (t), 26.0 (t), 27.0 (t), 34.9 (t), 34.9 (t), 37.1 (s), 114.2 (t), 138.8 (d); exact mass m/z calcd for $\text{C}_{11}\text{H}_{19}\text{I}$ 278.05316, found 278.05280.

3-[(1-Iodomethyl)cyclohexyl]propanal (62).



OsO_4 (0.1 M in PhMe , 3.2 mL, 0.32 mmol) was added in one portion to a stirred solution of alkene **61** (896 mg, 3.22 mmol) in water (8 mL) and dioxane (24 mL). The mixture was stirred for 20 min and then NaIO_4 (1.72 g, 8.0 mmol) was added in small portions and stirring was continued for 2 h. The mixture was diluted with water and extracted with EtOAc (3 times). The combined organic extracts were washed with brine, dried (Na_2SO_4) and evaporated. Flash chromatography of the residue over silica gel (2 x 20 cm), using 10% EtOAc -hexane, gave **62** (636 mg, 71%): FTIR (neat film microscope) 2927, 2854, 1725 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 1.32-1.54 (br s, 10 H), 1.64-1.75 (m, 2 H), 2.26-2.40 (m, 2 H), 3.20 (s, 2 H), 9.80 (t, $J = 1.8$ Hz, 1 H); ^{13}C NMR (CDCl_3 , 100.6 MHz) δ 20.4 (t), 21.7 (t), 25.9 (t), 30.1 (s), 34.6 (t), 34.8 (t), 37.8 (t), 202.0 (d); exact mass m/z calcd for $\text{C}_{10}\text{H}_{17}\text{IO}$ 280.03241, found 280.03232.

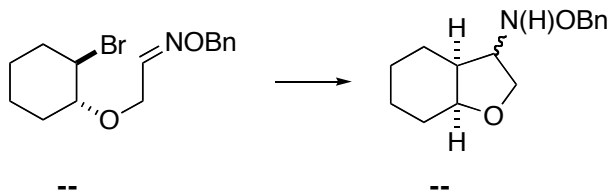
***Trans*-2-[(2-Bromocyclohexyl)oxy]acetaldehyde *O*-(Phenylmethyl)oxime.**



O-Benzylhydroxylamine hydrochloride (330 mg, 2.66 mmol) was added to a stirred solution of **49** (490 mg, 2.22 mmol) in THF (10 mL). Pyridine (350 mg, 4.44 mmol) was then added, and the mixture was refluxed for 10 h, cooled, filtered and evaporated. Flash chromatography of the residue over silica gel (1.7 x 32 cm), using 8% EtOAc-hexane, gave *trans*-2-[(2-bromocyclohexyl)oxy]acetaldehyde *O*-(phenylmethyl)oxime (632 mg, 88%) as a light yellow oil, which was a mixture of *Z* and *E* isomers (^1H NMR): FTIR (CH_2Cl_2 cast) 3087, 3063, 2937, 2860, 1452 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 1.19-2.4 (m, 8 H), 3.27-3.39 (m, 1 H), 3.88-3.99 (m, 1 H), 4.12-4.51 (m, 2 H), 5.09 (d, J = 1.6 Hz, 2 H), 6.87 (t, J = 4.0 Hz, 0.42 H), 7.23-7.59 (m, 5 H), 7.53 (t, J = 4.0 Hz, 0.5 H); ^{13}C NMR (CDCl_3 , 100.6 MHz) δ 23.2 (t), 25.4 (t), 30.7 (t), 30.9 (t), 35.6 (t), 54.9 (d), 55.2 (d), 64.0 (t), 66.4 (t), 75.9 (t), 76.3 (t), 81.5 (s), 82.4 (s), 127.8 (d), 127.9 (d), 128.0 (d), 128.1 (d), 128.4 (d), 137.4 (s), 137.5 (s), 147.9 (d), 151.1 (d); exact mass (electrospray) m/z calcd for $\text{C}_{15}\text{H}_{20}^{79}\text{BrNNaO}_2$ 348.0575, found 348.0570.

The oxime geometry was not established.

***O*-Benzyl-*N*-[(3 α ,7 α)octahydrobenzofuran-3-yl]hydroxylamine.**



General procedure A for radical cyclization was followed, using *trans*-2-[(2-bromocyclohexyl)oxy]acetaldehyde *O*-(phenyl-methyl)oxime (281 mg, 0.865 mmol) in THF (50 mL), Bu_3SnH (370 mg, 1.30 mmol) in THF (5 mL), and ABC (4 mg, 0.02 mmol) in THF (5 mL). Flash chromatography of the residue over silica gel (1.7 x 20 cm), using 20% EtOAc-hexane, gave *O*-benzyl-*N*-[(3 α ,7 α)octahydrobenzofuran-3-yl]hydroxylamine (129 mg, 61%) as a foam, which was a mixture of two isomers (^1H NMR): FTIR (CH_2Cl_2 cast) 3242, 3086, 3062, 2931 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 1.12-2.23 (m, 9 H), 3.41-3.46 (m, 0.68 H), 3.48-3.57 (m, 1 H), 3.91-4.20 (m, 1.8 H), 4.12 (dd, J = 10.3, 6.4 Hz, 0.7 H), 4.69, 4.70 (two s, 2 H in all), 4.83-5.92 (br s, 1 H), 7.22-7.43 (m, 5 H); ^{13}C NMR (CDCl_3 , 100.6 MHz) δ 20.4 (t), 20.5 (t), 20.9 (t), 24.1 (t), 24.2 (t), 26.1 (t), 27.7 (t), 28.2 (t), 39.7 (d), 41.4 (d), 64.0 (d), 67.1 (d), 68.3 (t), 69.8 (t), 75.6 (d), 76.0 (t), 76.6 (t), 77.3 (d), 127.8 (d), 127.9 (d), 128.3 (d), 128.4 (d), 128.5 (d), 137.7 (s); exact mass m/z calcd for $\text{C}_{15}\text{H}_{22}\text{NO}_2$ ($M + \text{H}$) 248.1650, found 248.1654.

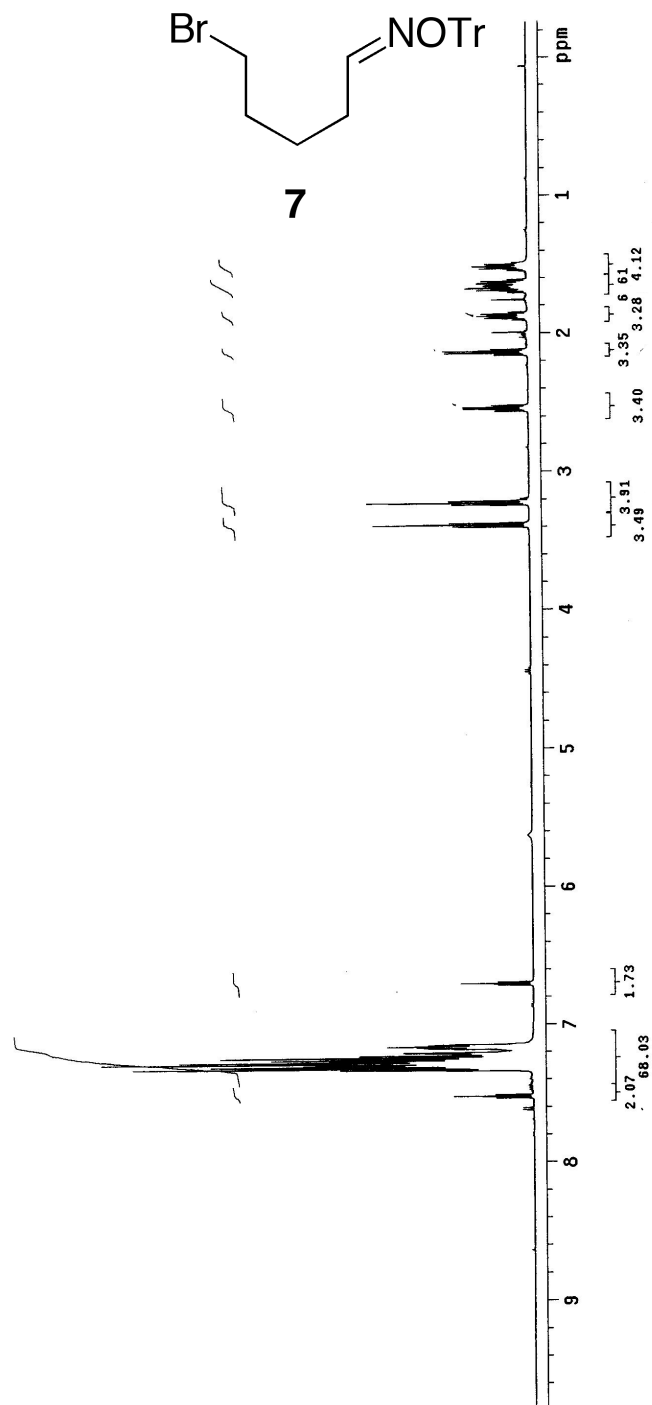
References and footnotes

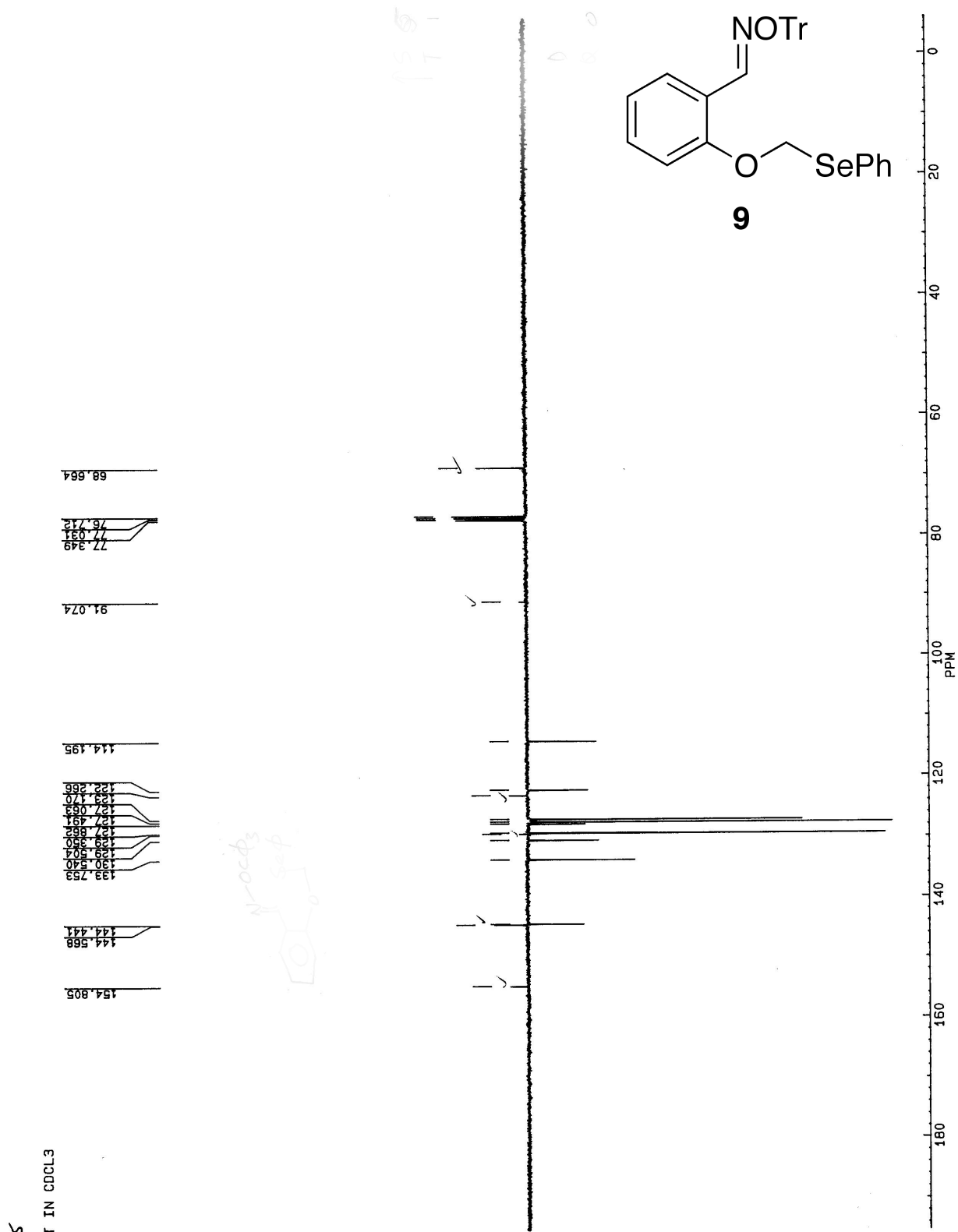
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- (40) Nuhrich, A.; Moulines, J. *Tetrahedron* **1991**, *47*, 3075-3088. Our material had: FTIR 2936, 2854, 1731, 1455 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 1.14-1.40 (m, 5 H), 1.50-1.65 (m, 5 H), 1.89-2.00 (m, 2 H), 2.03-2.15 (m, 2 H), 3.68 (s, 3 H), 4.88-5.03 (m, 2 H), 5.76 (dddd, *J* = 16.8, 10.2, 6.6, 6.6 Hz, 1 H); ¹³C NMR (CDCl₃, 100.6 MHz) δ 23.2 (t), 25.9 (t), 28.4 (t), 34.1 (t), 39.6 (t), 46.8 (s), 51.4 (q), 114.4 (t), 138.4 (d), 177.0 (s); exact mass *m/z* calcd for C₁₂H₂₀O₂ 196.14633, found 196.14612.

RSF-70

Pulse Sequence: s2pu1

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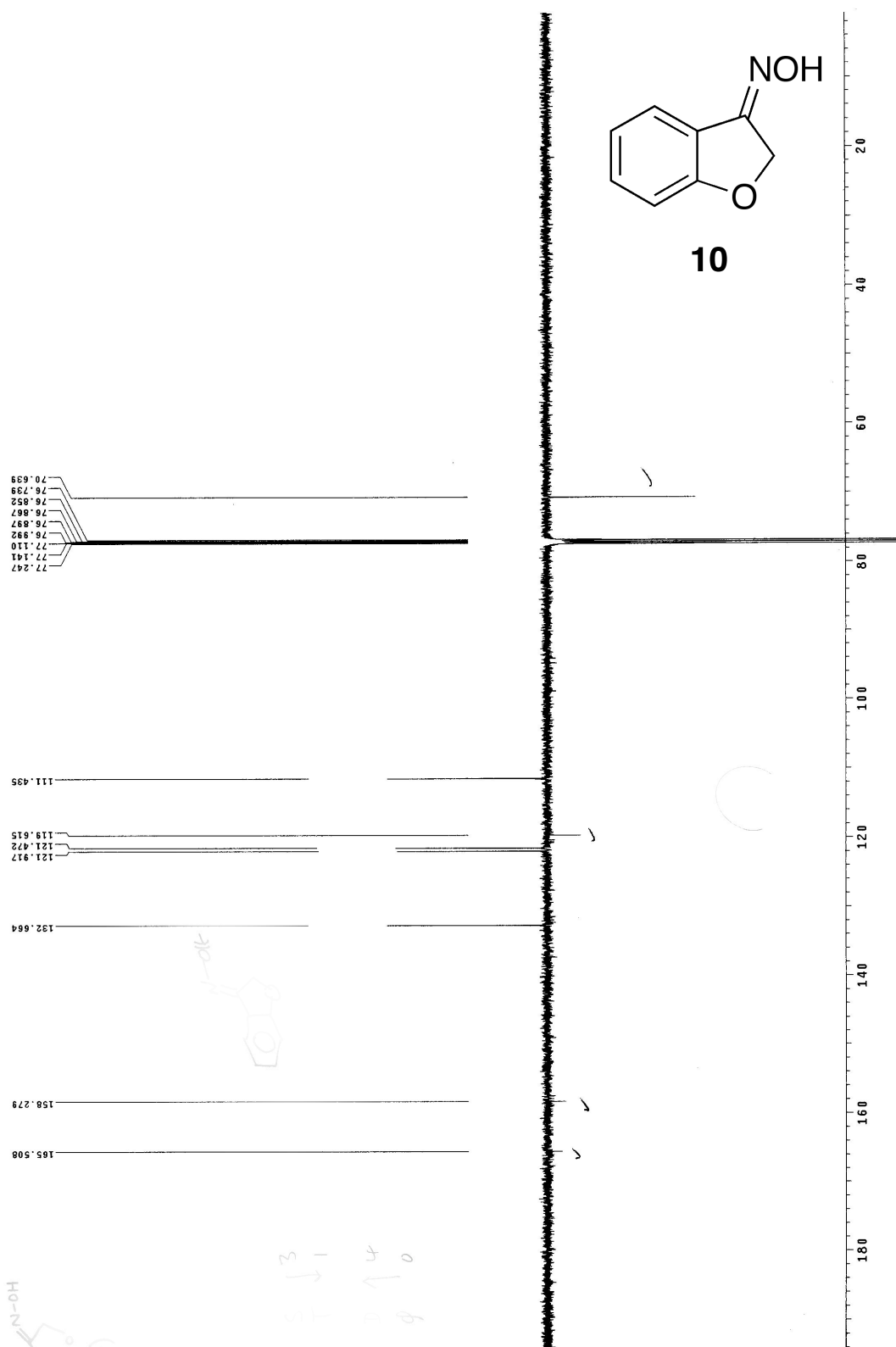




RSF-54

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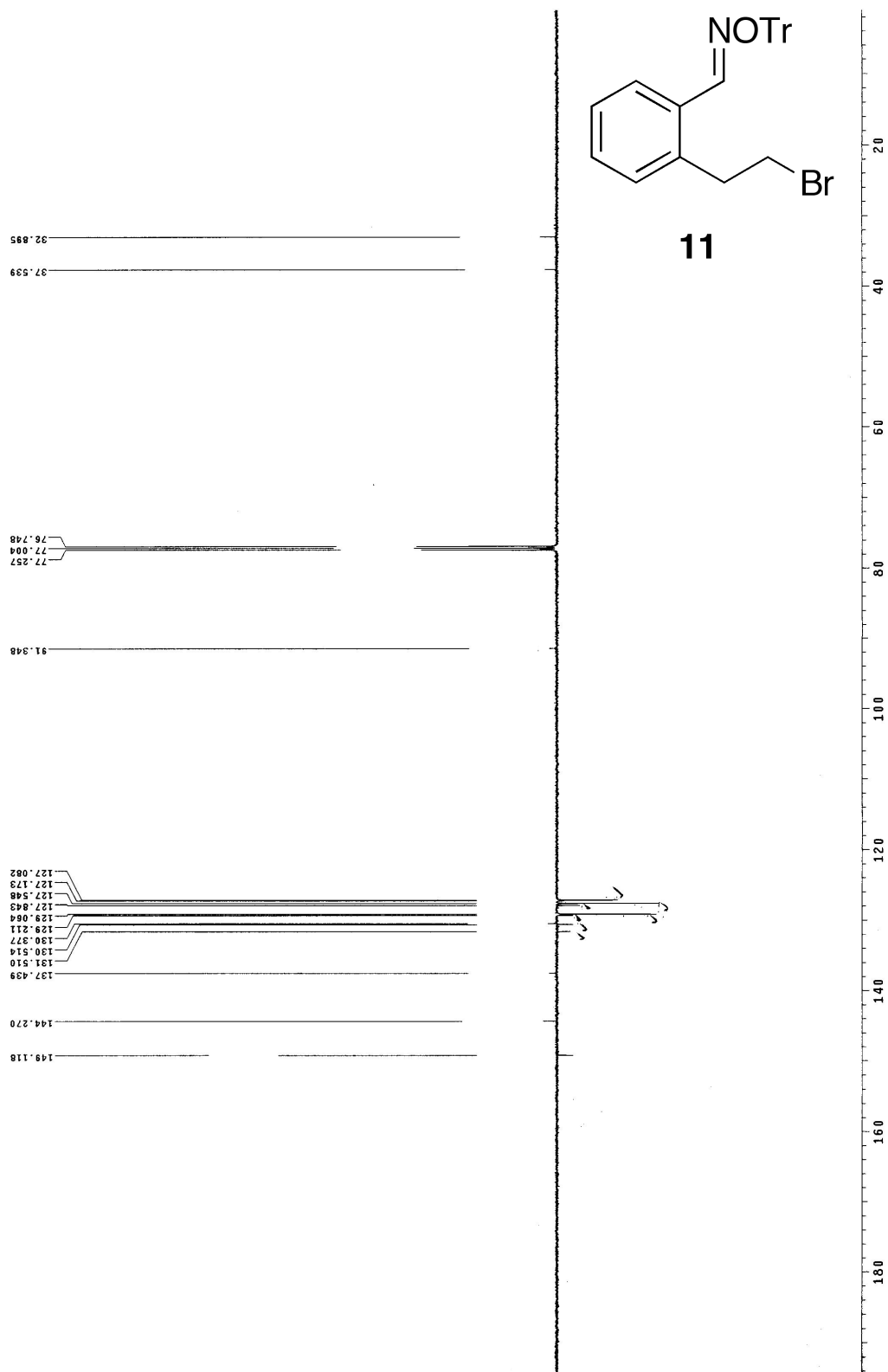
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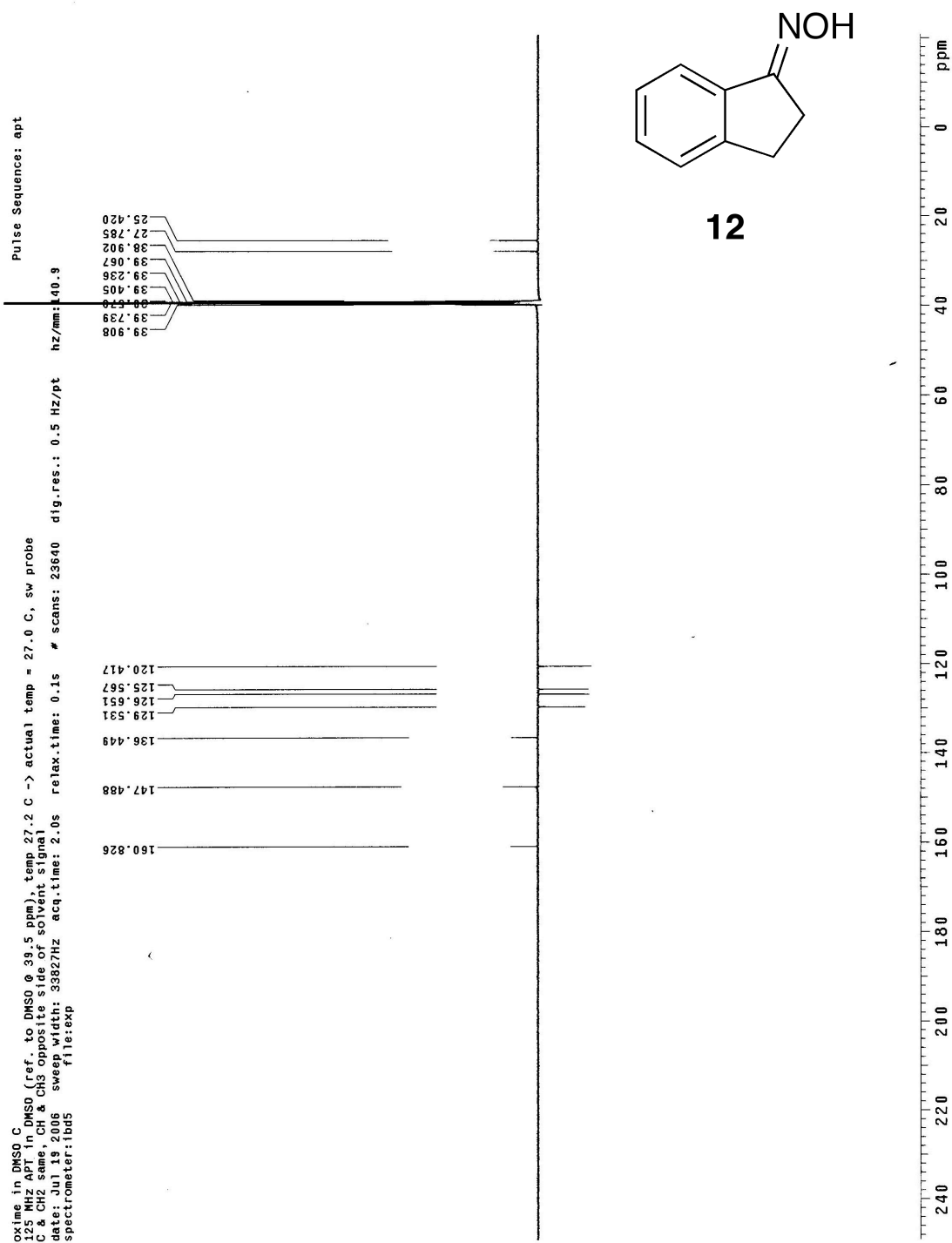


25F-57

25F-57
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 25F-57
 125 MHz in CDCl₃ (ref. to CDCl₃ at 77.0 ppm), temp 27.2 °C → actual temp = 27.0 °C, sw probe
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Pulse Sequence: aq

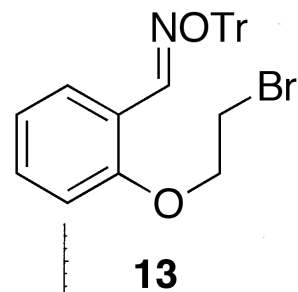
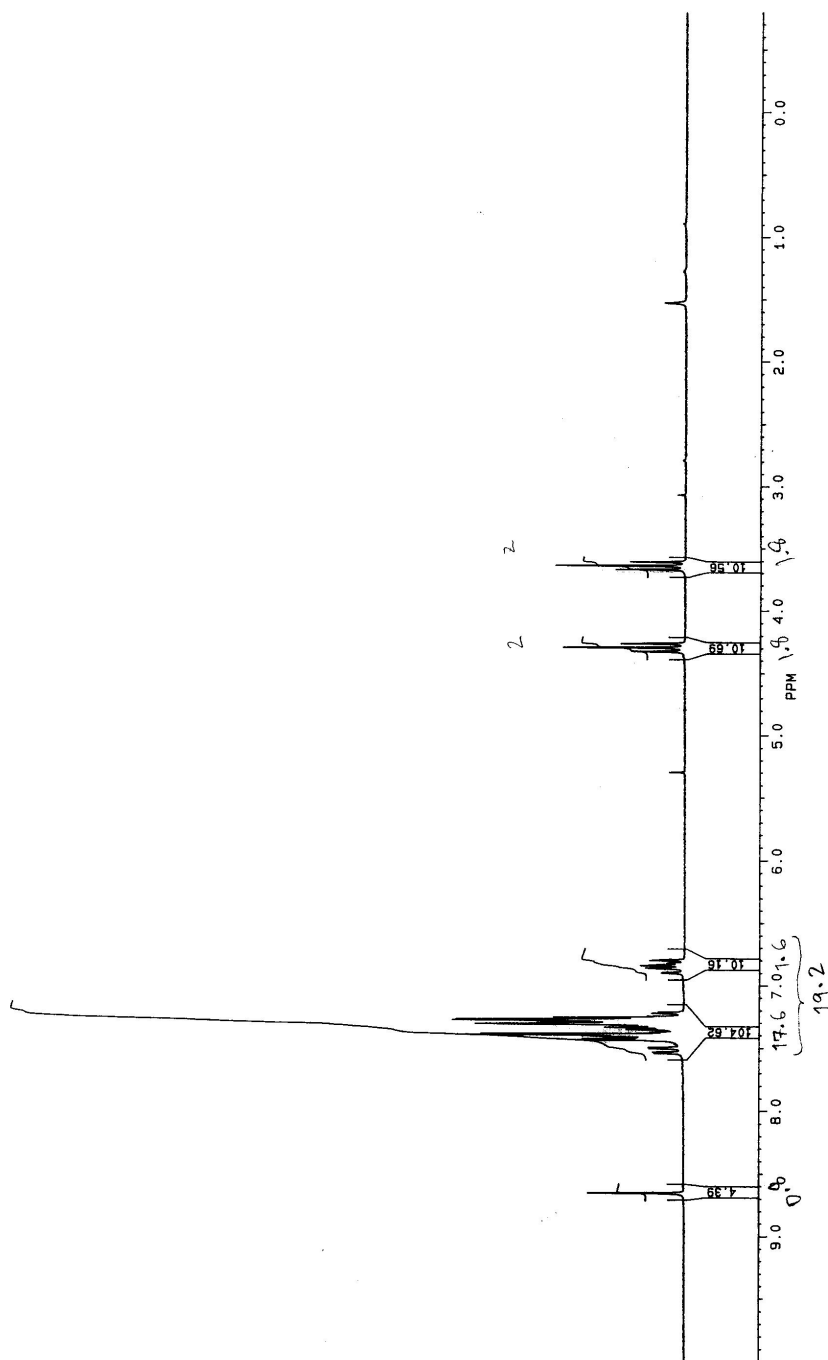




KS F-44

¹H IN CDCl₃~~BIOSER~~

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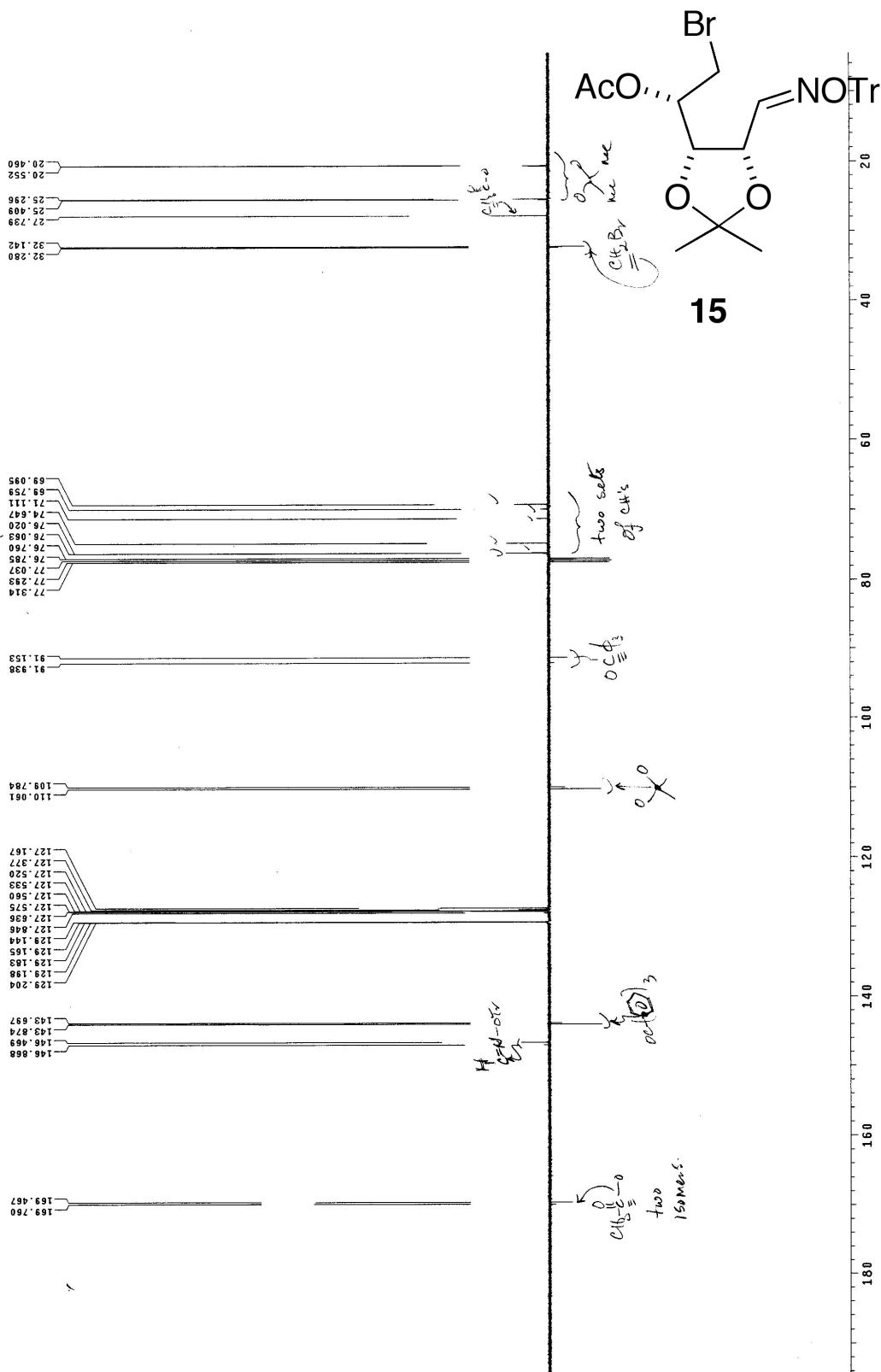



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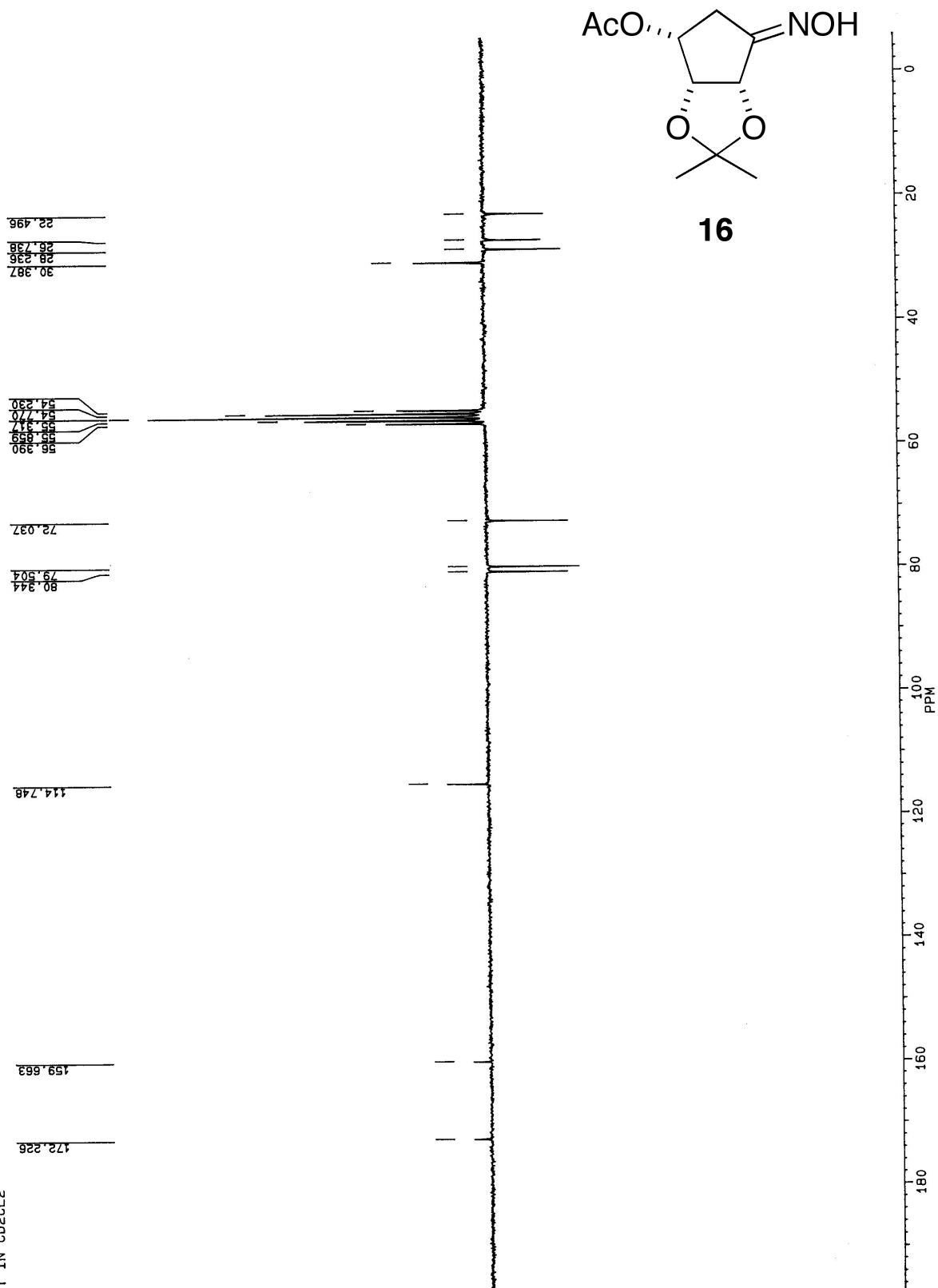
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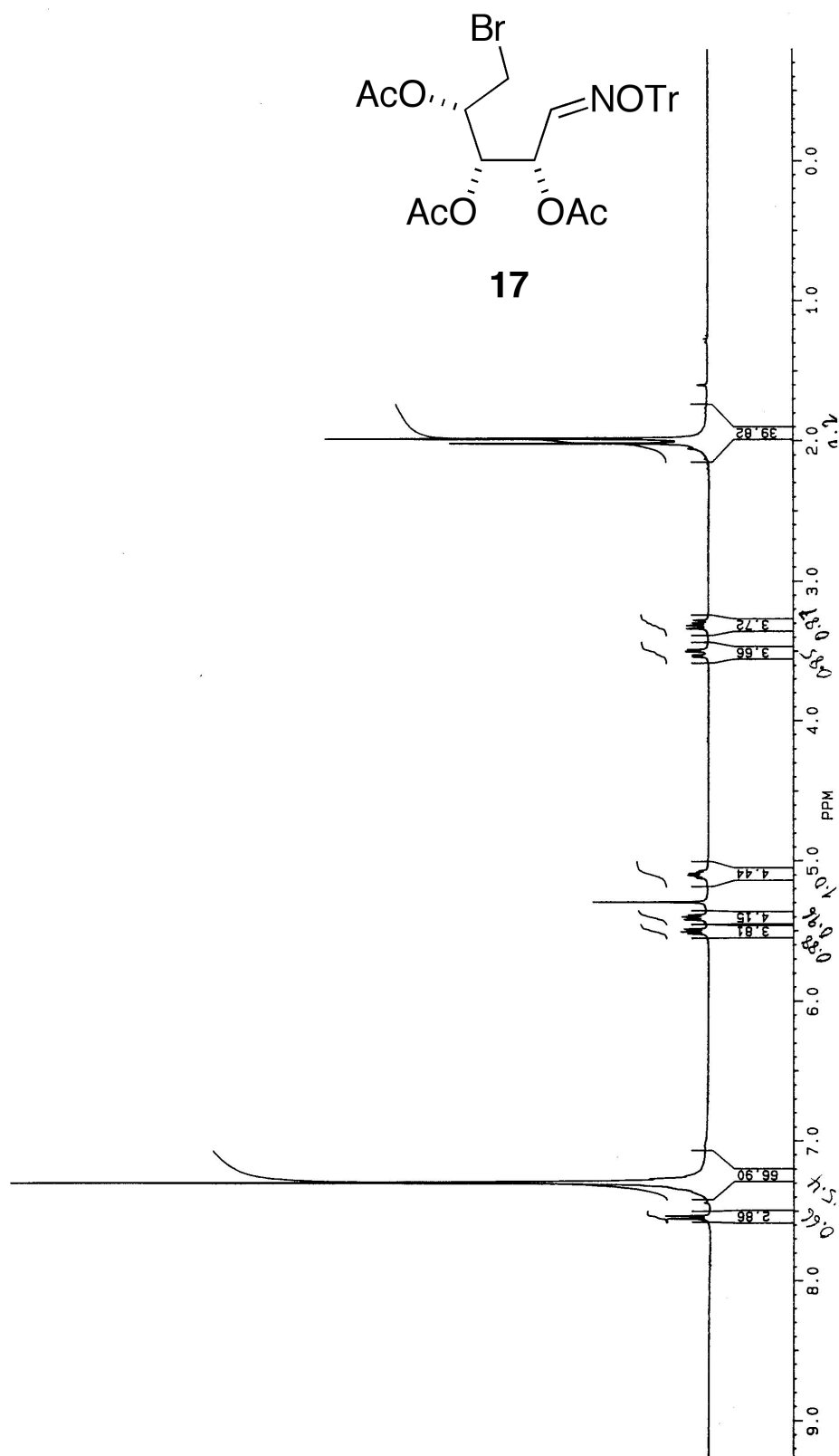
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Pulse Sequence



G-22
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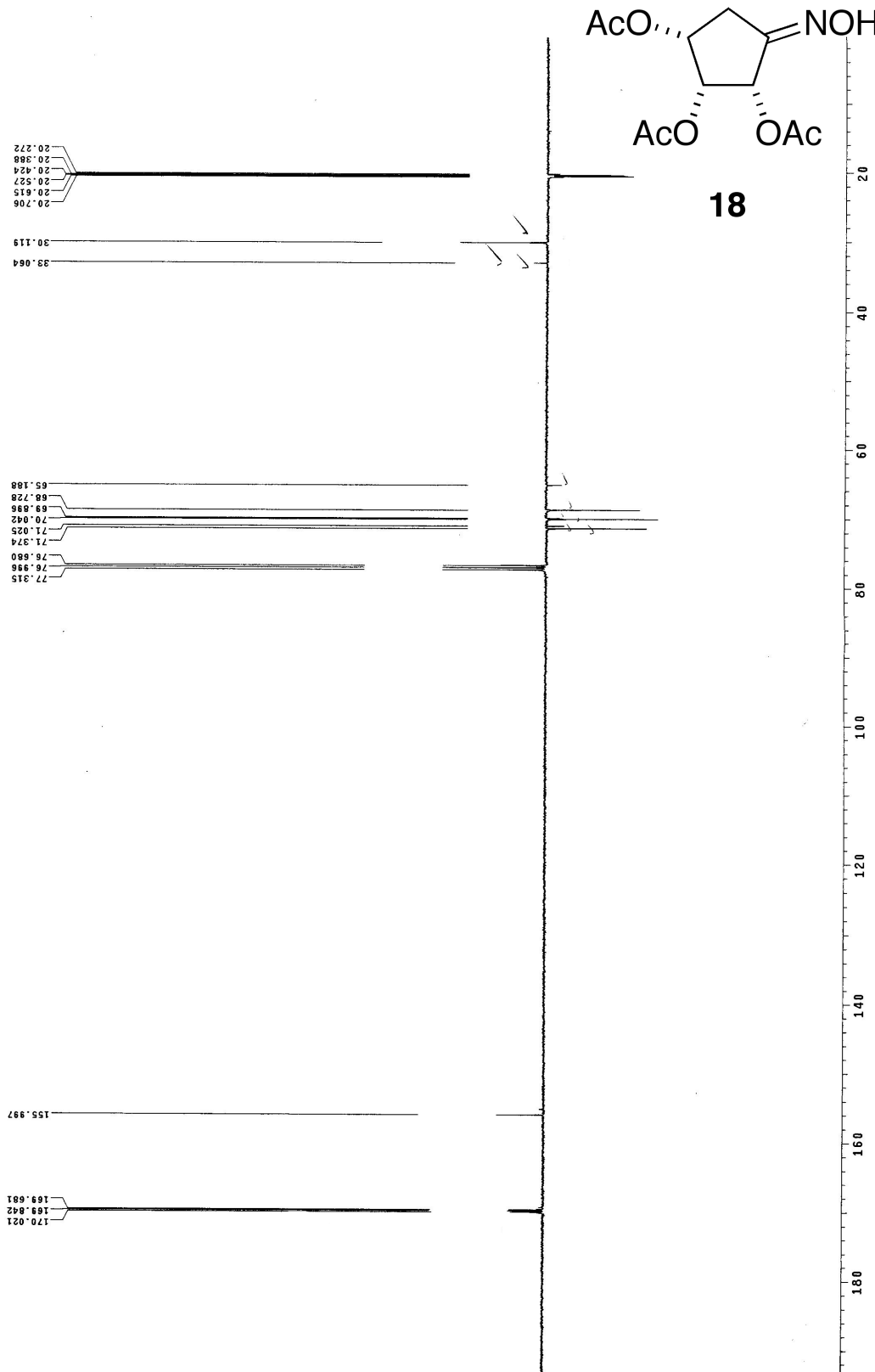
30CL3 RS6-58

v. 44

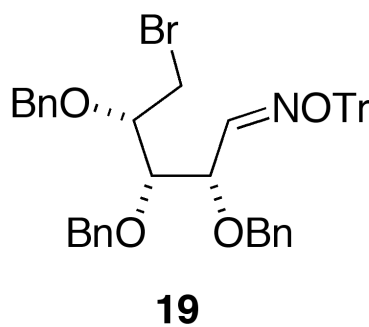
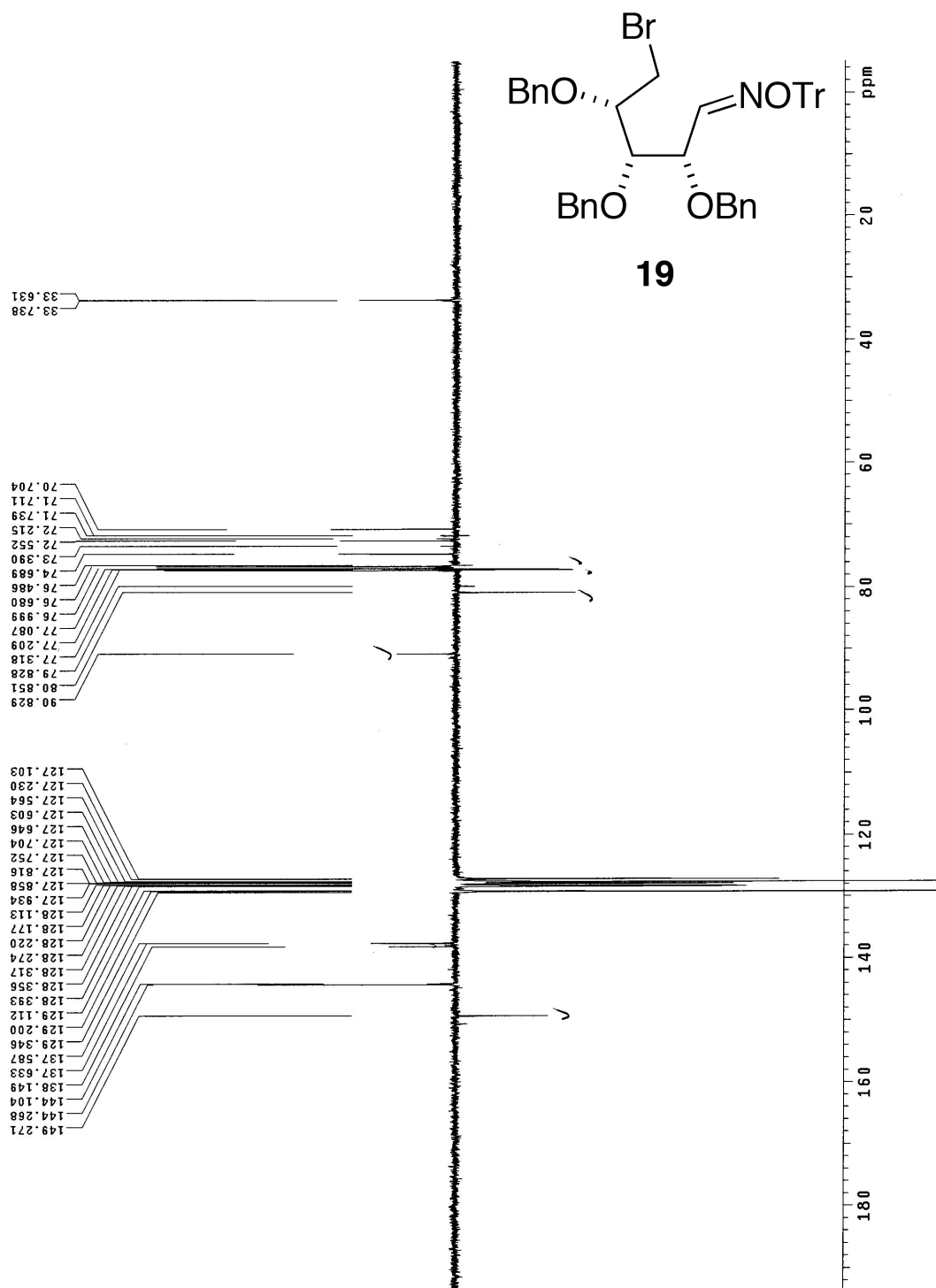
RSH-34

MZ APT in CDCl₃ (ref. to CDCl₃ @ 77.0 ppm), temp 27.8 C -> actual temp = 27.0 C, acq4000 probe
 CH₂ pos: 1.000, CH₃ pos: 0.000, CH₂ neg: 0.000, CH₃ neg: 0.000, CH₂ neg: 0.000, CH₃ neg: 0.000
 time [s]: 2.0, relax. time [s]: 0.31, sweep width [Hz]: 20000, dig.res. [Hz/p2]: 0.31, spectrometer: 400

Pulse Sequence: a1

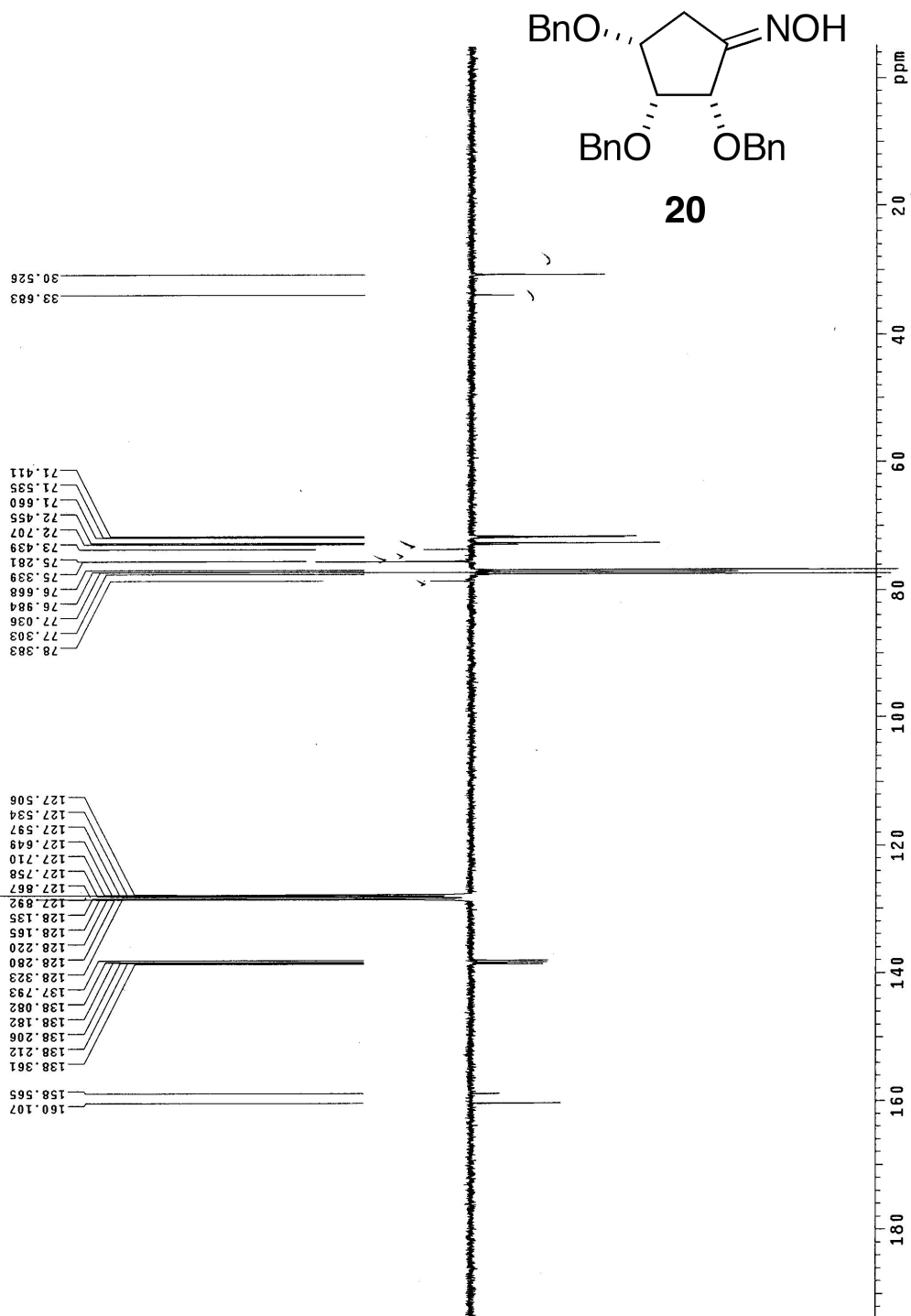


```
100 MHz APT [in CDC13 (ref. to CDC13 @ 77.0 ppm), temp 27.8 C -> actual temp = 27.0 C, asw400 probe
CH, CH3 positive; C, CH2 negative
date: Jul 31, 2001 seqfil: apt          hz/mm: 83.33      spectrometer: 400      file: exp
acq.time [s]: 2.0      relax.time [s]: 0.5      sweep width [Hz]: 20000      d1g.res. [Hz/pt]: 0.31
```



Pulse Sequence: apt

100 MHz APT in CDCl₃ (ref. to CDCl₃ @ 77.0 ppm), temp 27.8 C -> actual temp = 27.0 C, asw400 probe
 CH, CH3 positive; C, CH2 negative
 acq: Aug 9, 2003
 date: Aug 9, 2003
 file: apt
 spectrometer: 400
 hz/mm: 89.33
 relaxation time [s]: 0.5
 sweep width [Hz]: 20000
 dig.res. [Hz/pt]: 0.31

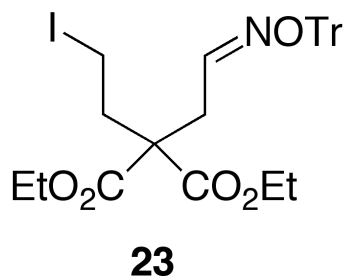
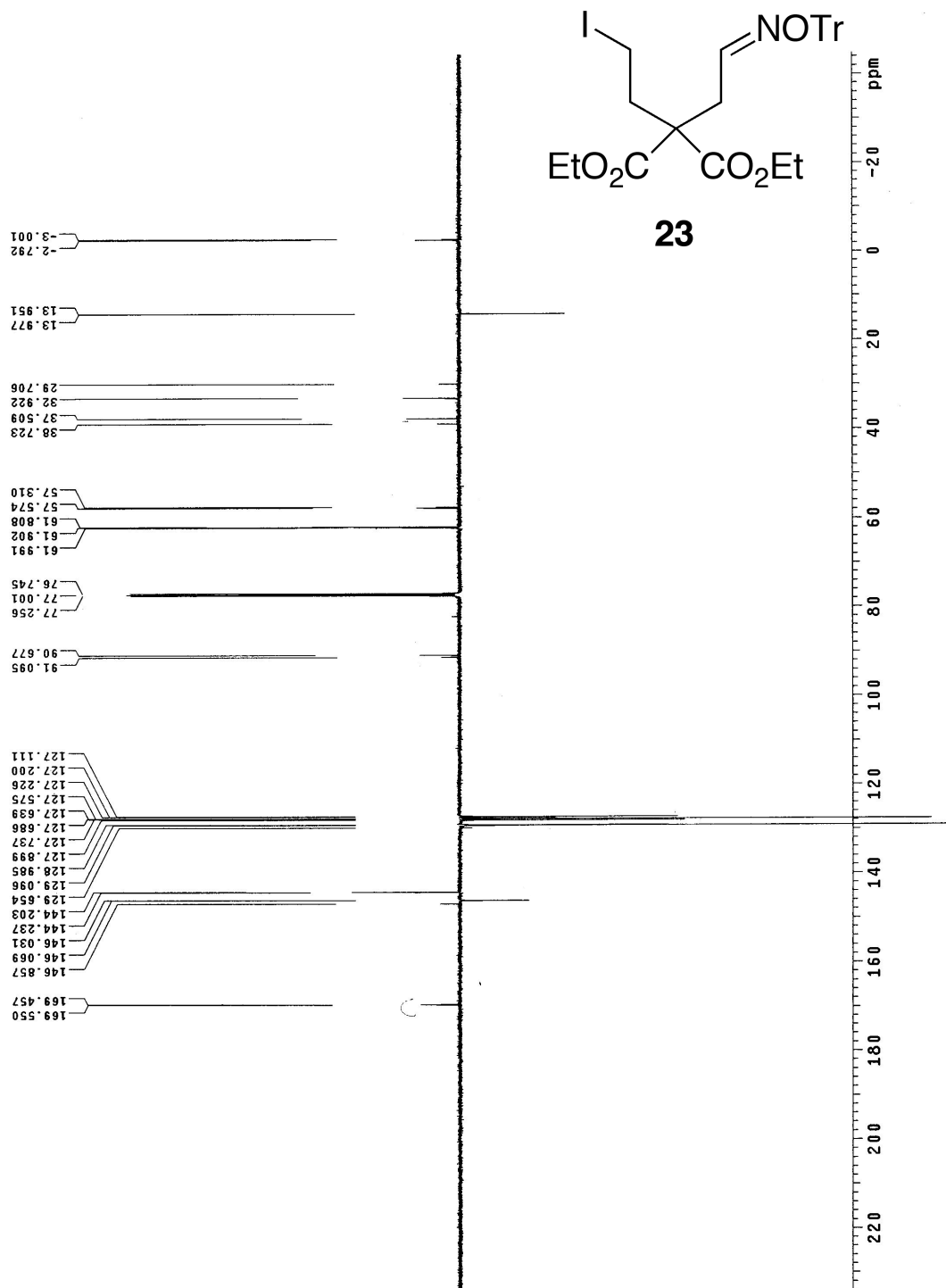


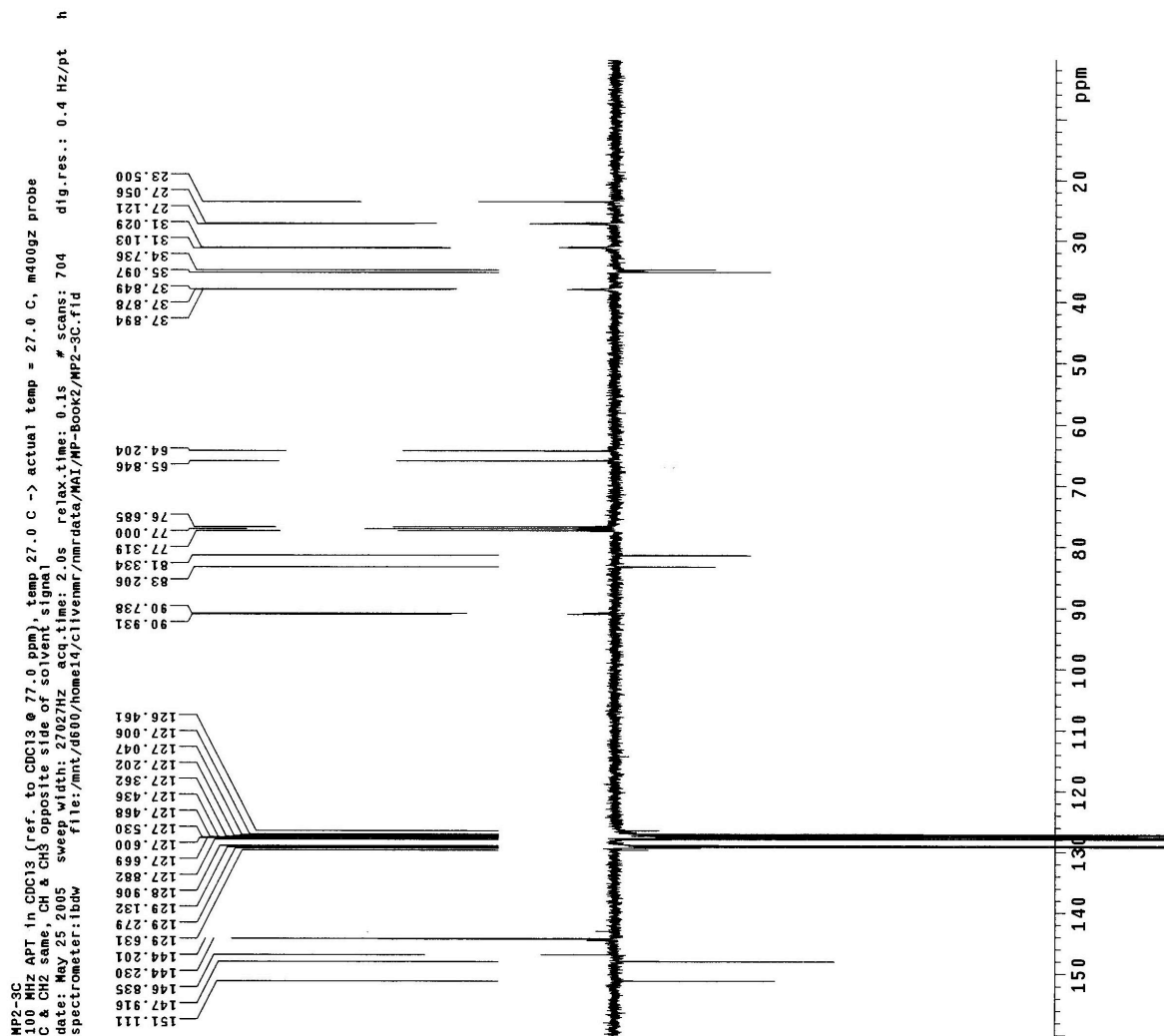
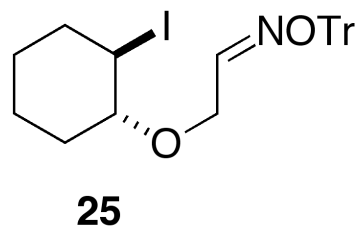

```

125 MHz APT in CDC13 (ref. to CDC13 @ 77.0 ppm), temp 27.2 C -> actual temp = 27.0 C, Sw probe
CH, CH3 positive; C, CH2 negative
date: Jul 29 2001 seqfil: apt          hz/mm: 145.69      spectrometer: lbd5
acq.time [s]: 2.0    relax.time [s]: 0.5   sweep width [Hz]: 34985     file: exp
                                     [hz/prob] : 0.53

```

Pulse Sequence: apt

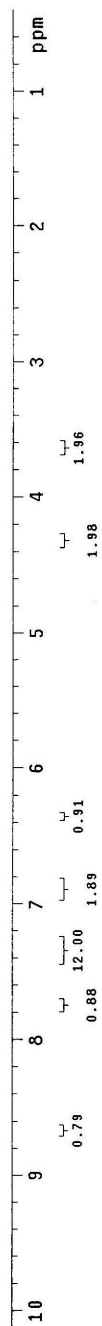
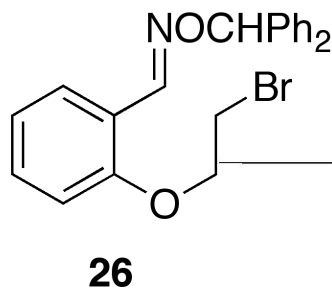


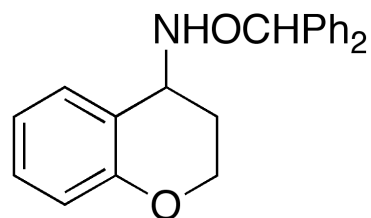


MPI-137H
 300 MHz 1D in CDCl₃ (ref. to CDCl₃ at 2.2
 6 ppm), temp 27.5 C -> actual temp 27.5
 0 C, 1d probe

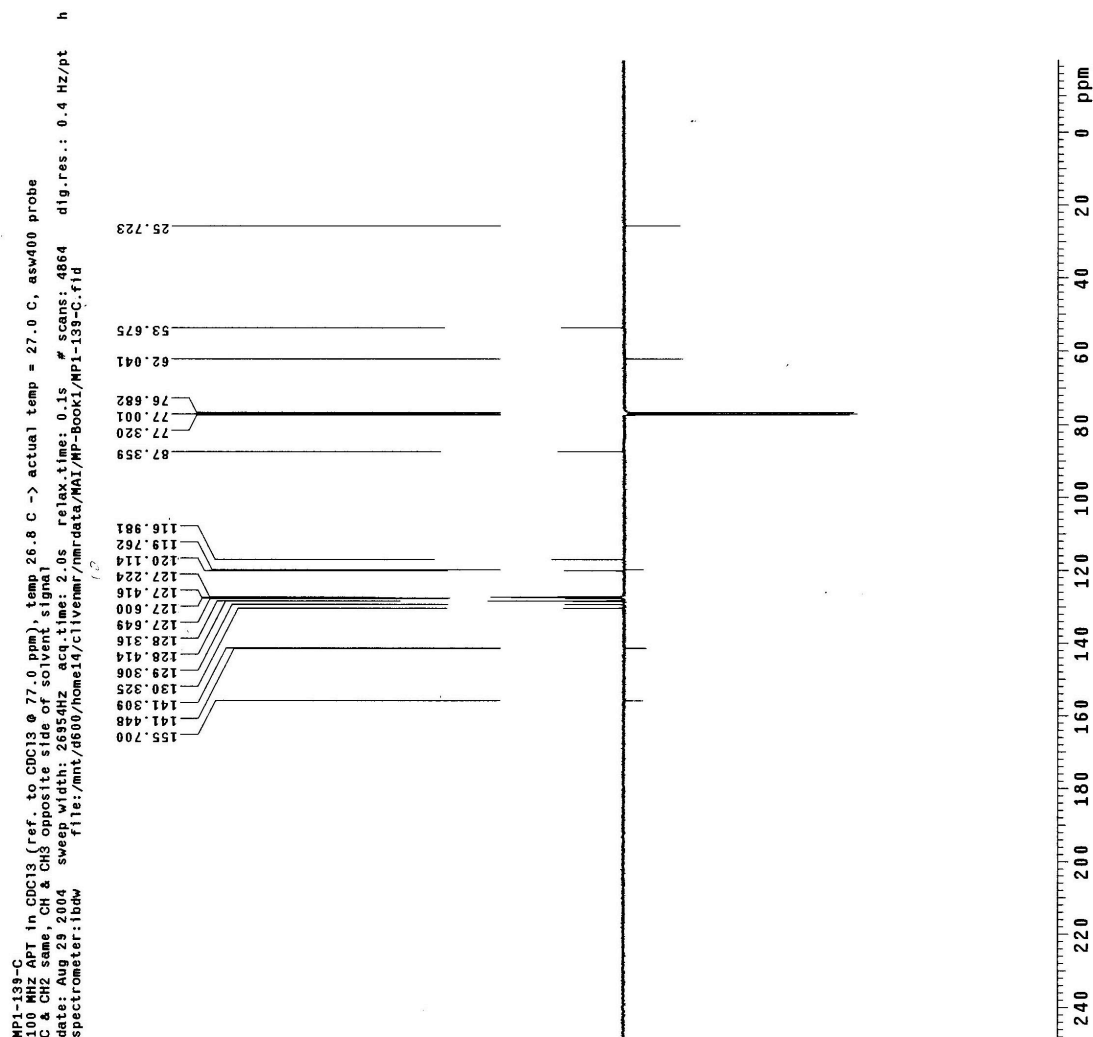
exp2 s2pul

SAMPLE		DEC. & VT	
date	Aug 26 2006	dfrq	299.971
solvent	cdcl3	dn	H1
file	exp	dpwr	3
ACQUISITION		dof	0
sfrq	299.971	dm	nm
tn	H1	dmm	C
at	4.999	dmg	200
ns	3600.7	dng	1.0
sw	3600.7	drg	1.0
fb	not used	homo	n
bs	4	temp	27.5
ss	2	PROCESSING	
tpwr	53	gf	not used
pw	3.8	gfs	not used
ql	0.100	wtfile	
nt	673550	proc	lp
ct	36	math	f
alock	16	werr	
gain	n	wexp	
ll	n	wbs	
in	n	wnt	wft
ds	y		
hs	nm		
DISPLAY			
sp	120.1		
wp	3080.1		
vs	47		
sc	0		
wdw	248		
h2nm	12.83		
ls	28187.75		
rfl	2598.2		
rfp	2171.8		
th	3		
ins	12.000		
nm			
ph			



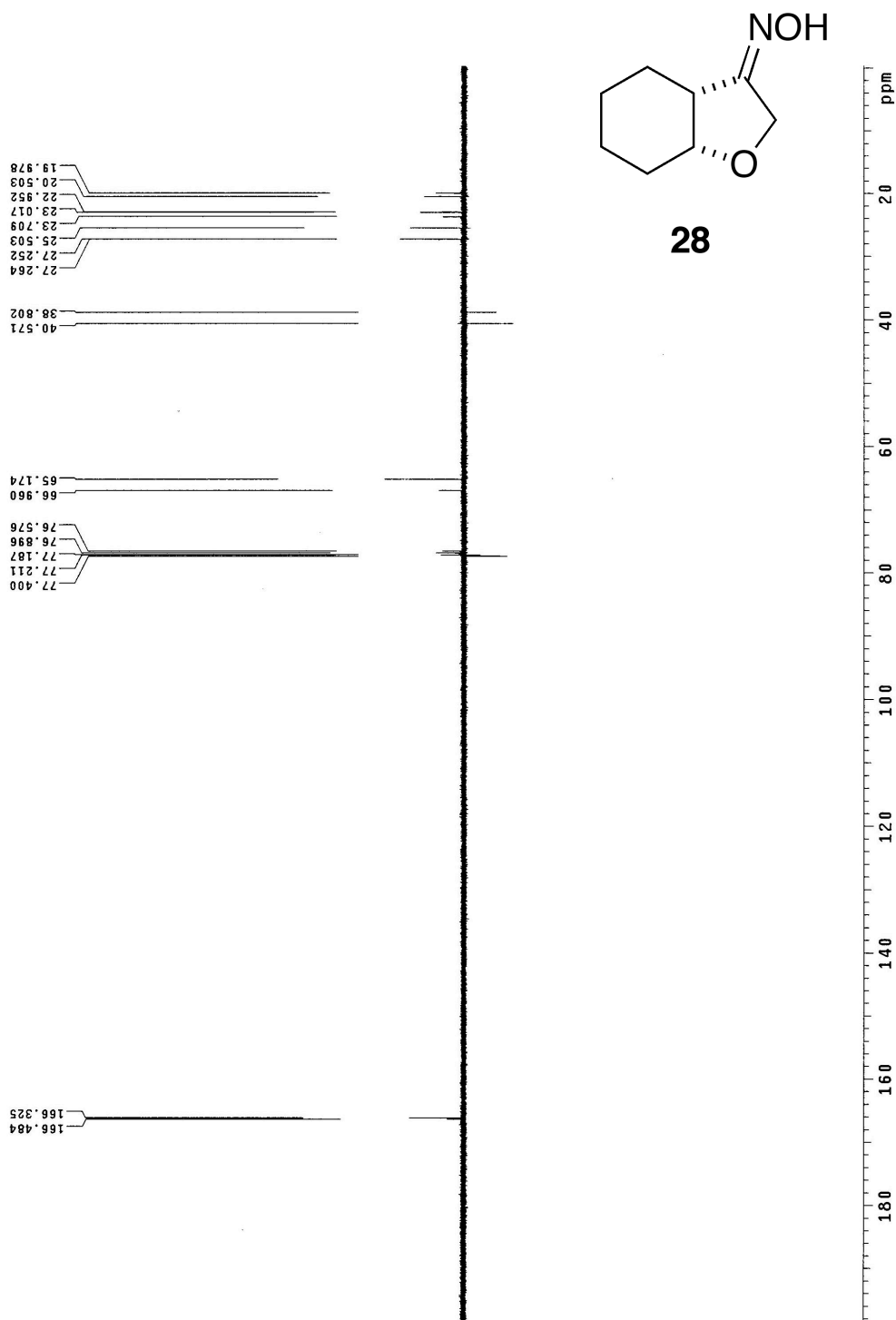


27



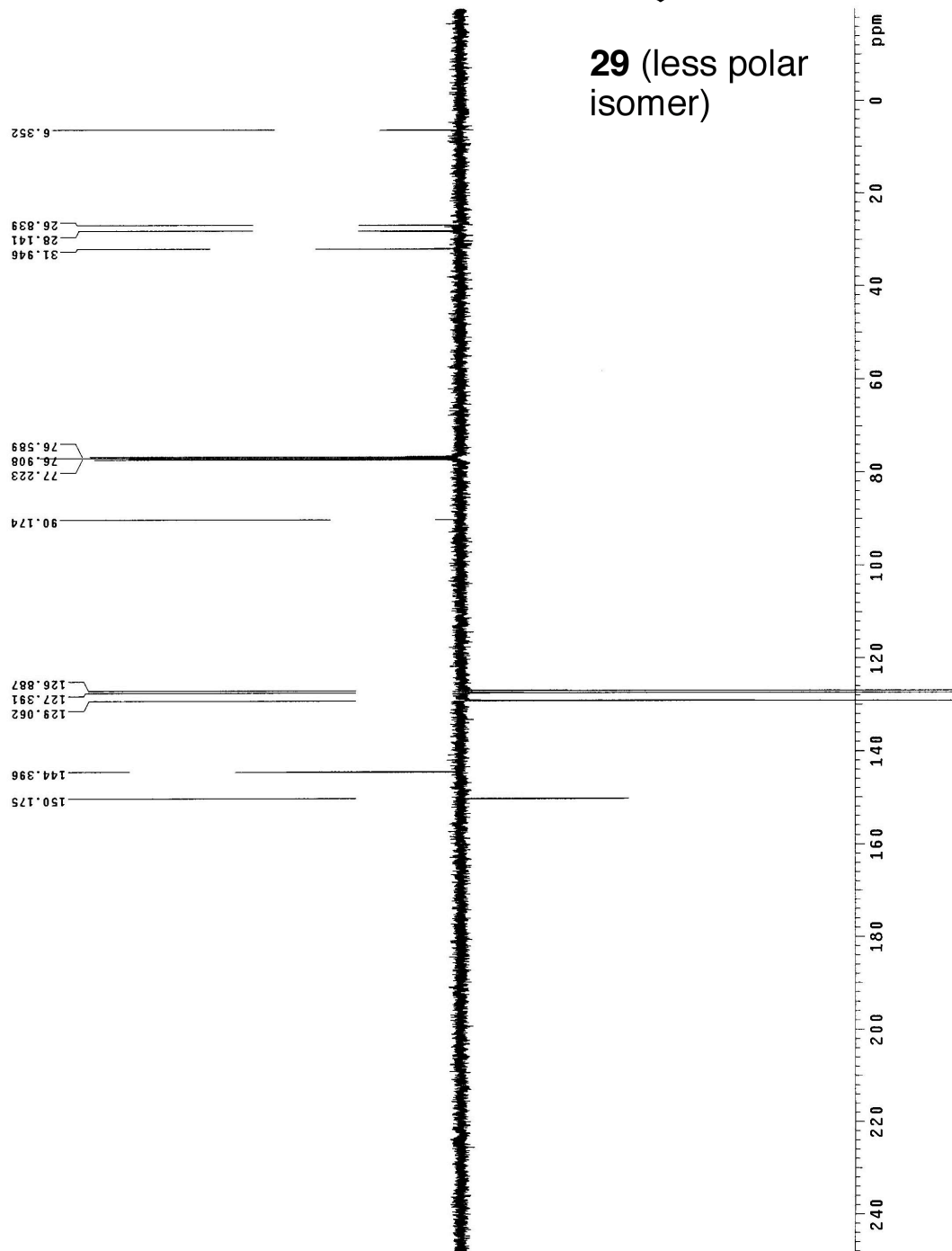
MP2-85C
100 MHz APT in CDCl₃ (ref. to CDCl₃ @ 77.0 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
C & CH₂ same, CH & CH₃ opposite side of solvent signal
date: Jul 11 2005 sweep width: 27027Hz acq.time: 2.0s relax.time: 0.1s # scans: 192 dfg.res.: 0.4 Hz/pt
spectrometer:m400 file:exp

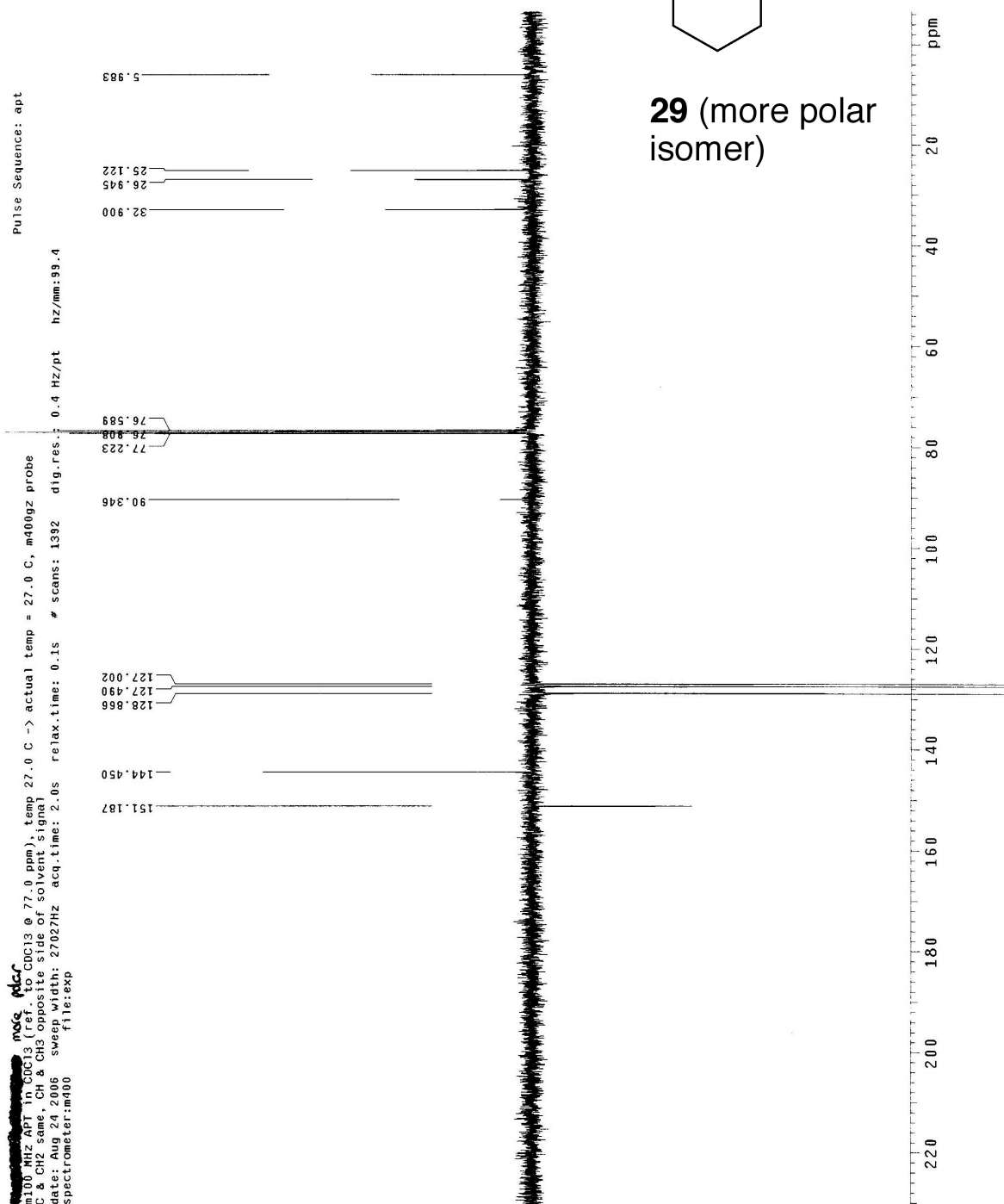
Pulse Sequence: apt

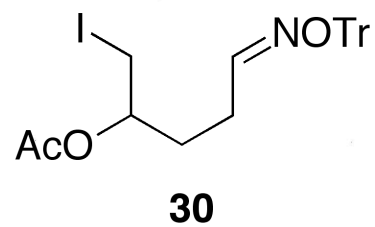


Pulse Sequence: apt

m100 MHz APT in CDCl₃ (ref. to CDCl₃ @ 77.0 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
C & CH₂ same, CH & CH₃ opposite side of solvent signal
date: Aug 24 2006 sweep width: 27027Hz acq.time: 2.0s relax.time: 0.1s # scans: 1560 dig.res.: 0.4 Hz/pt
spectrometer: lbd5 file:/mnt/d600/home14/cilvenmr/nmrdata/NAI/S-Iodo-o-tritylamineCl.fid



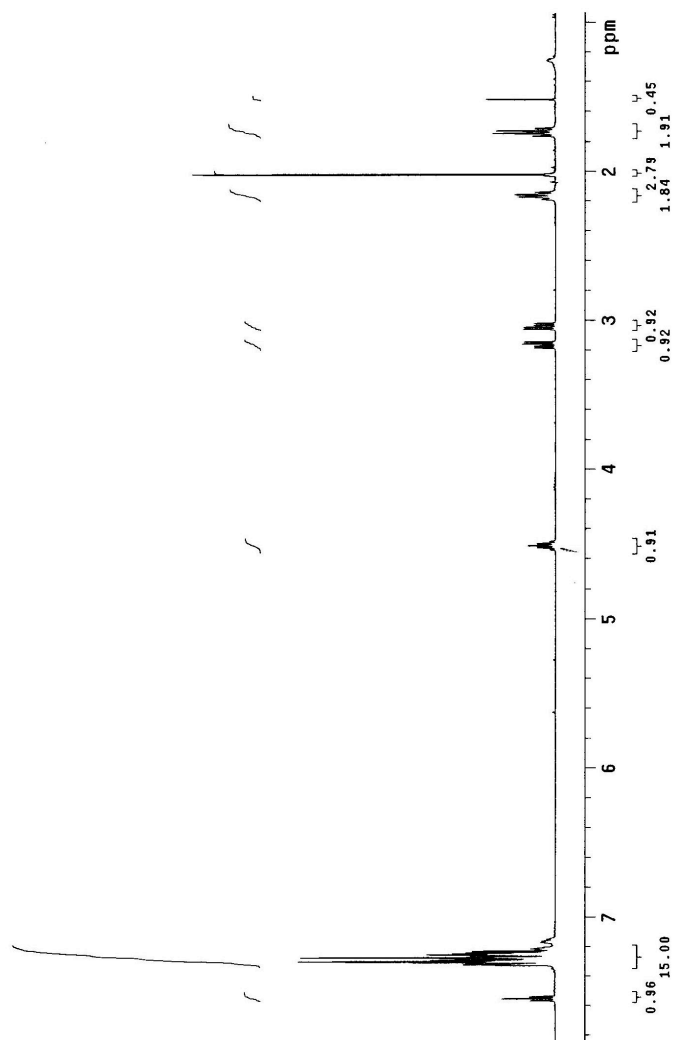


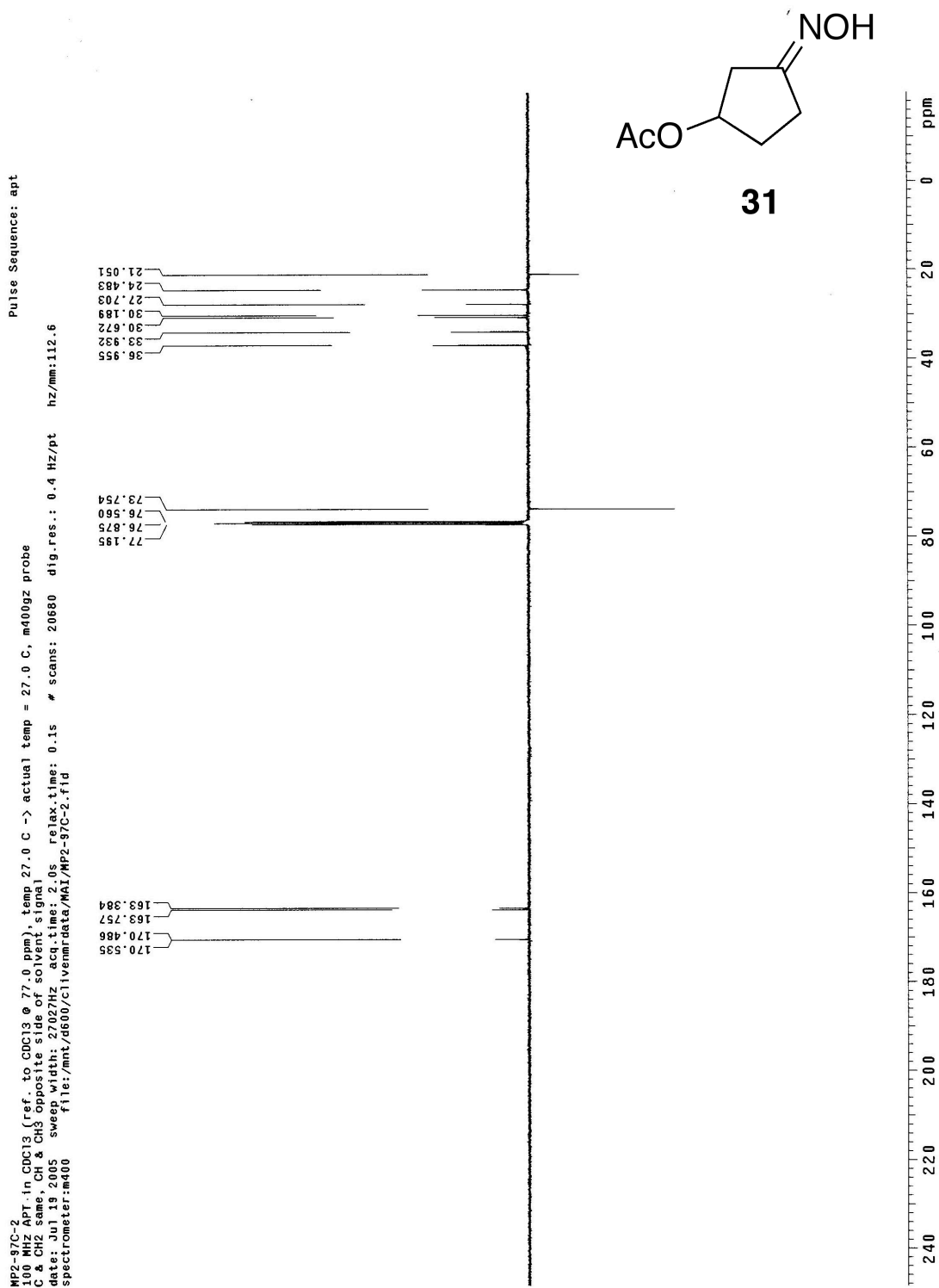


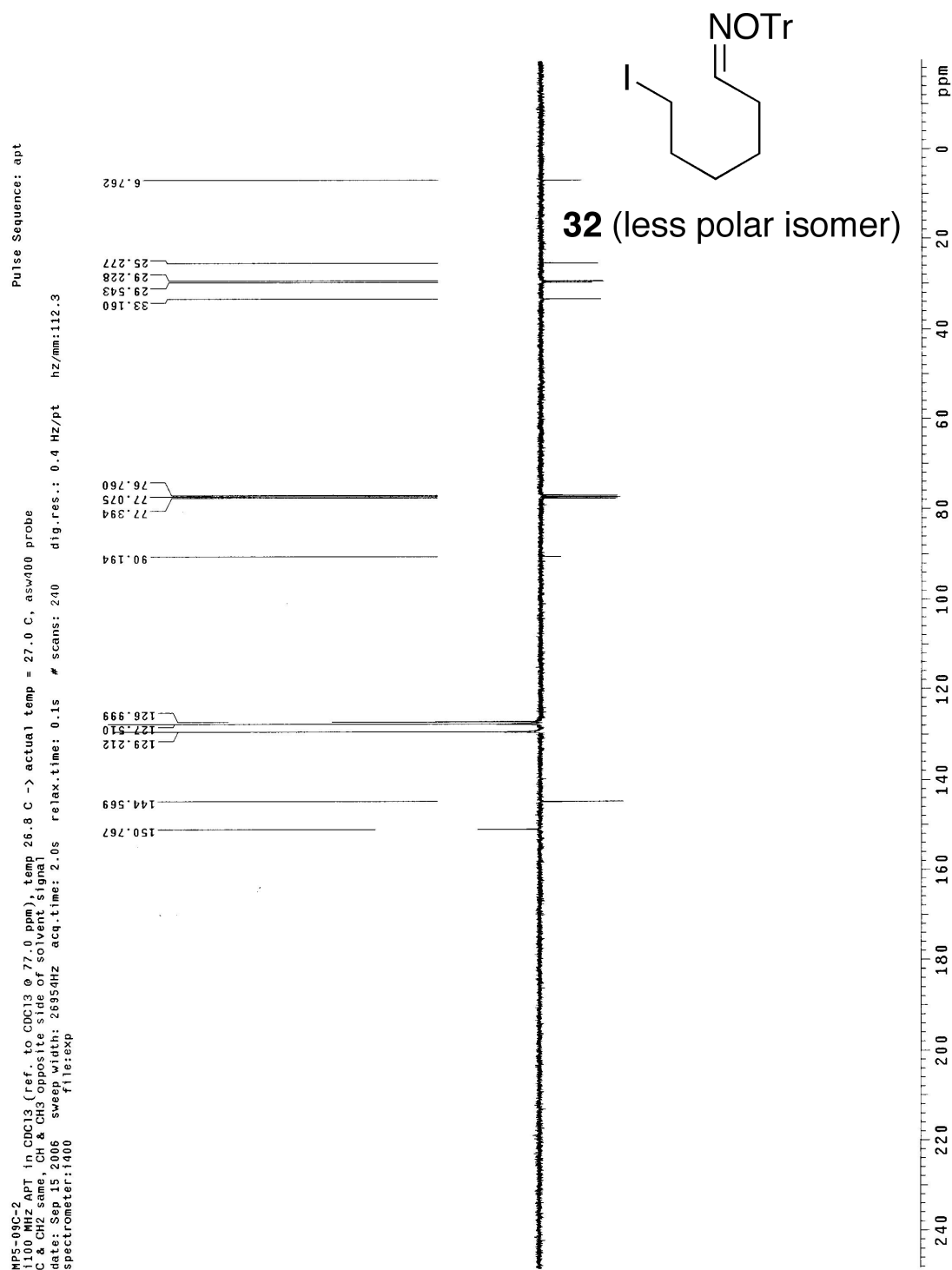
MP2-95H
400 MHz 1D in CDC13 (ref. to CDC13 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

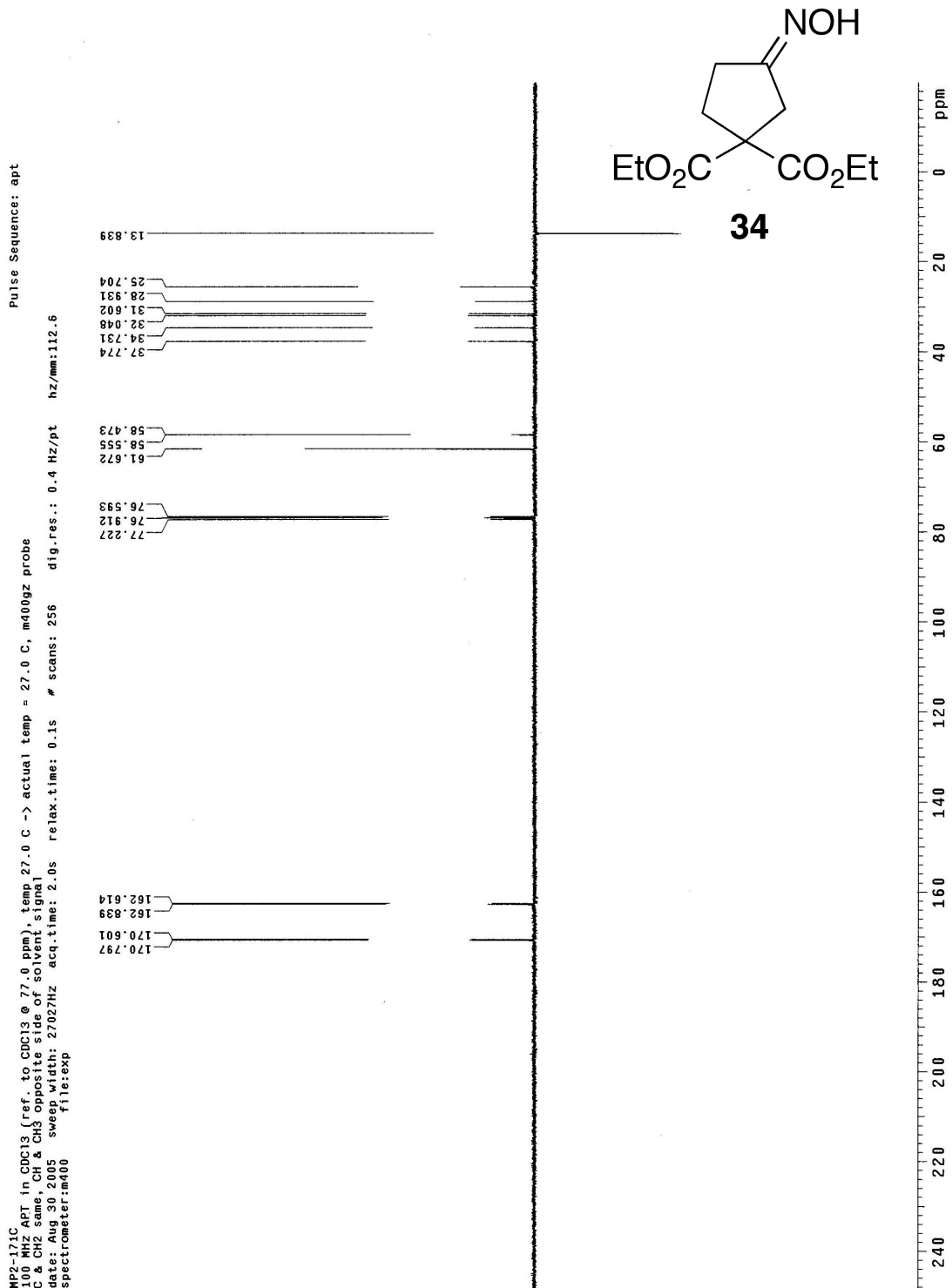
Solvent: cdc13
Temp: 27.0 C / 300.1 K
File: MP2-95H
INOVA-500 "1bdw"

Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 1.999 sec
Date_ 20110216
Time 14:11:02
OBSERVE H1 400.3981759 MHz
DATA PROCESSING
FT size 131072
Total time 1 min, 30 sec







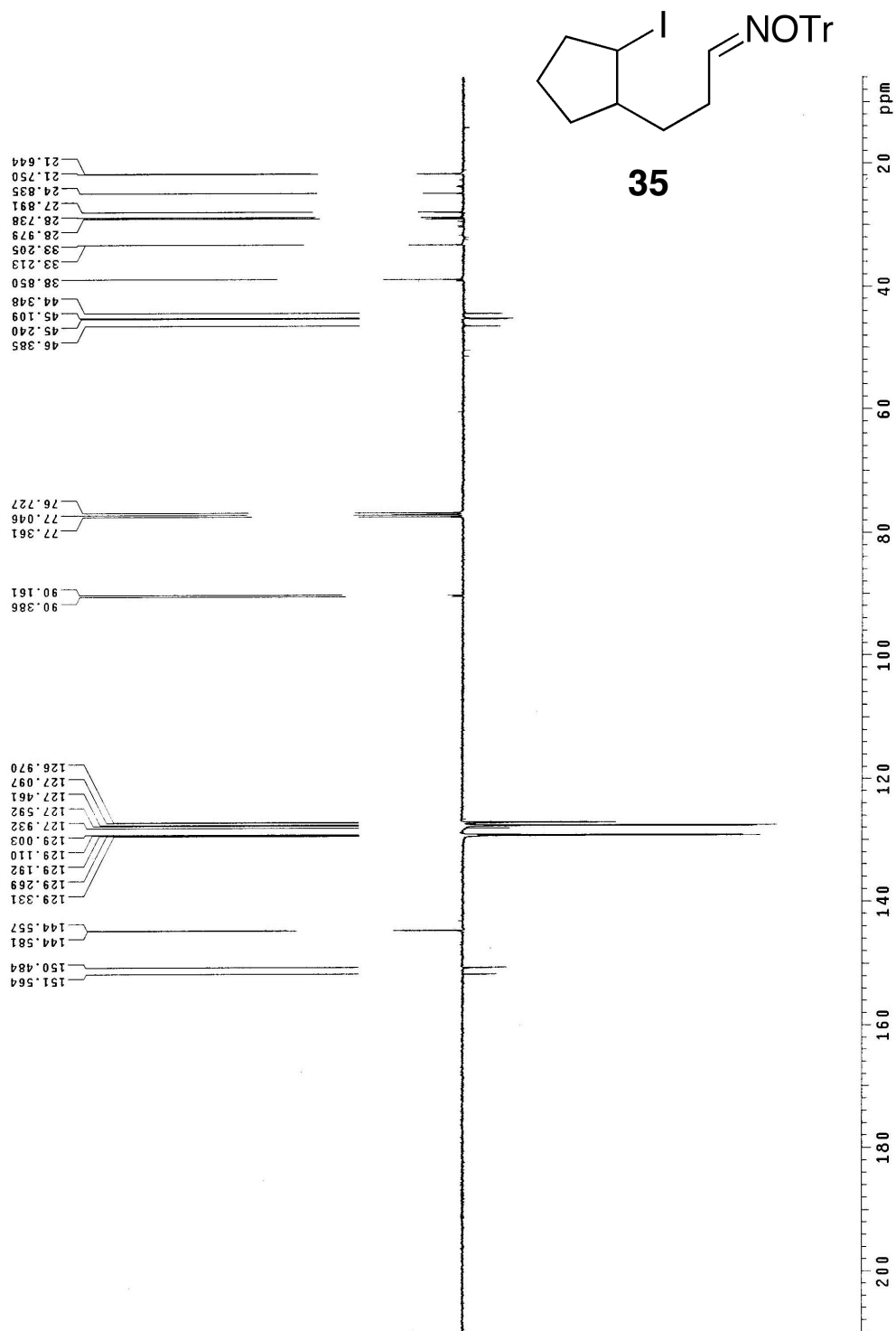


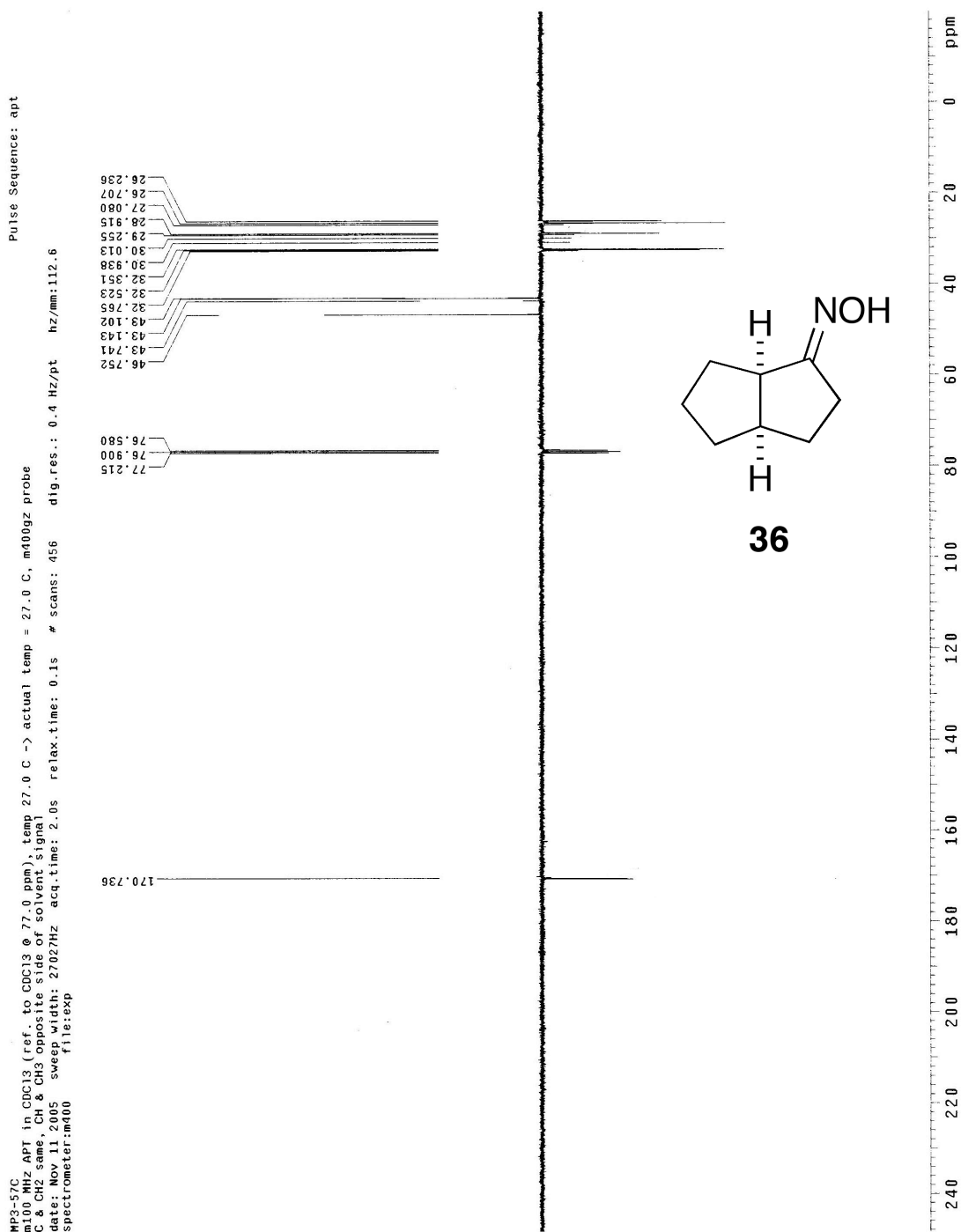
NP3-55C

100 MHz APT in CDCl₃ (ref. to CDCl₃ @ 77.0 ppm), temp 26.8 C -> actual temp = 27.0 C, asw400 probe
C & CH₂ same, CH & CH₃ opposite side of solvent signal

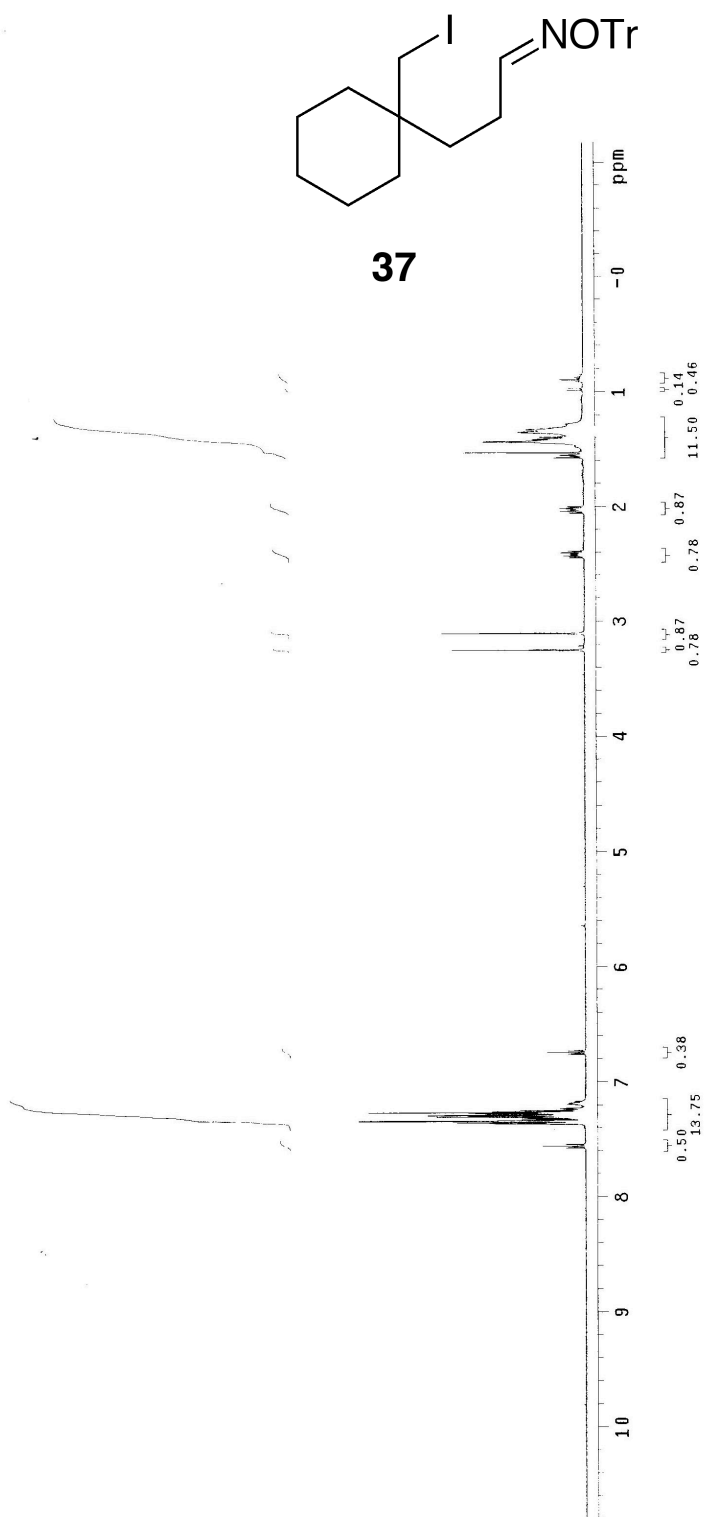
Pulse Sequence: apt

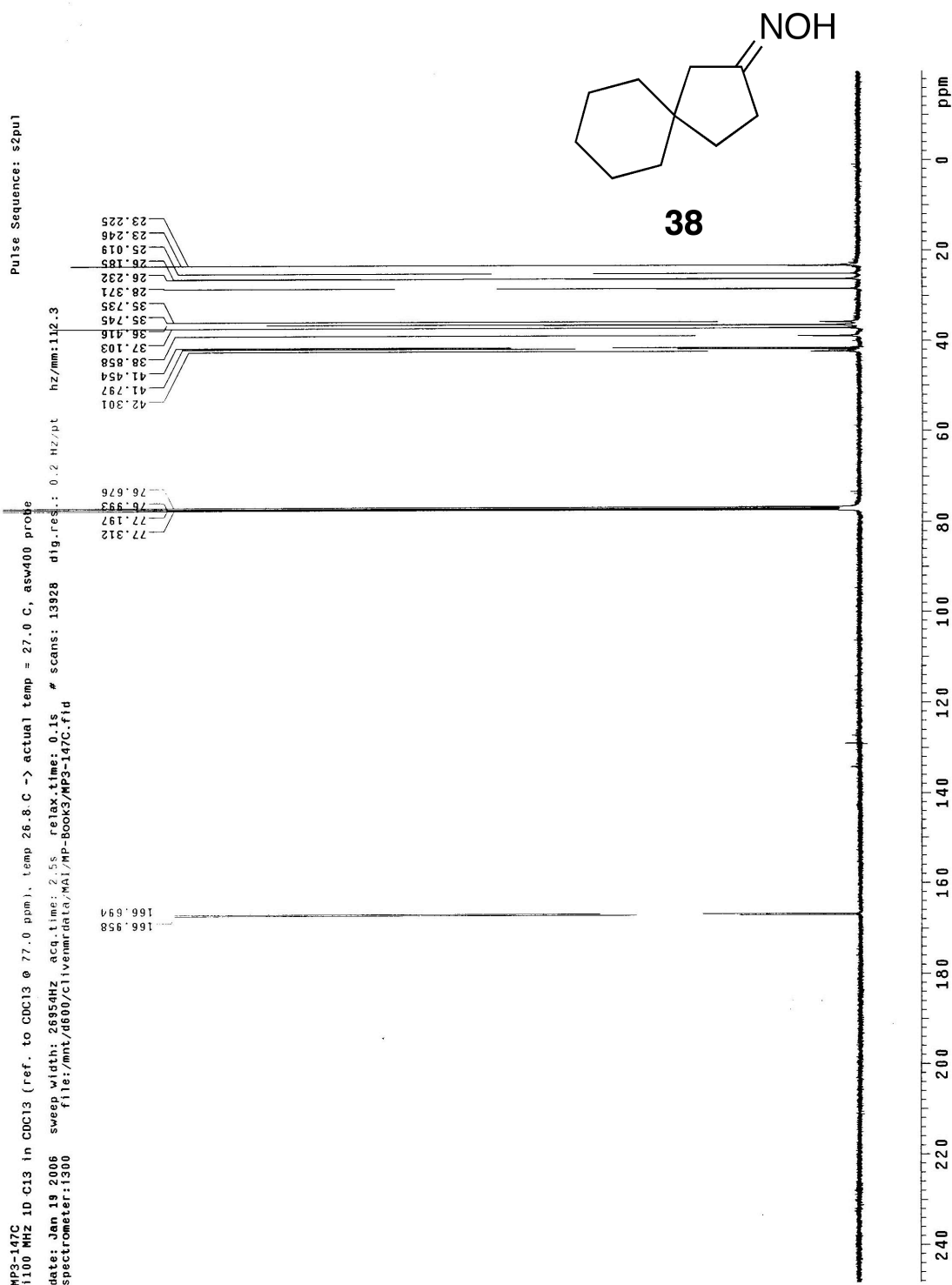
date: Nov 10 2005 sweep width: 26954Hz acq.time: 2.0s relax.time: 0.1s # scans: 1456 dig.res.: 0.4 Hz/pt hz/mm: 85.6
spectrometer: 400 file: exp





MP3-145H
m500 MHz 1D in CDCl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Jan 17 2006 sweep width: 4803Hz acq time: 2.0s relax time: 3.0s # scans: 16 dig.res.: 0.1 Hz/pt hz/mm: 20.0
spectrometer: m400 file: exp Pulse Sequence: s2pul

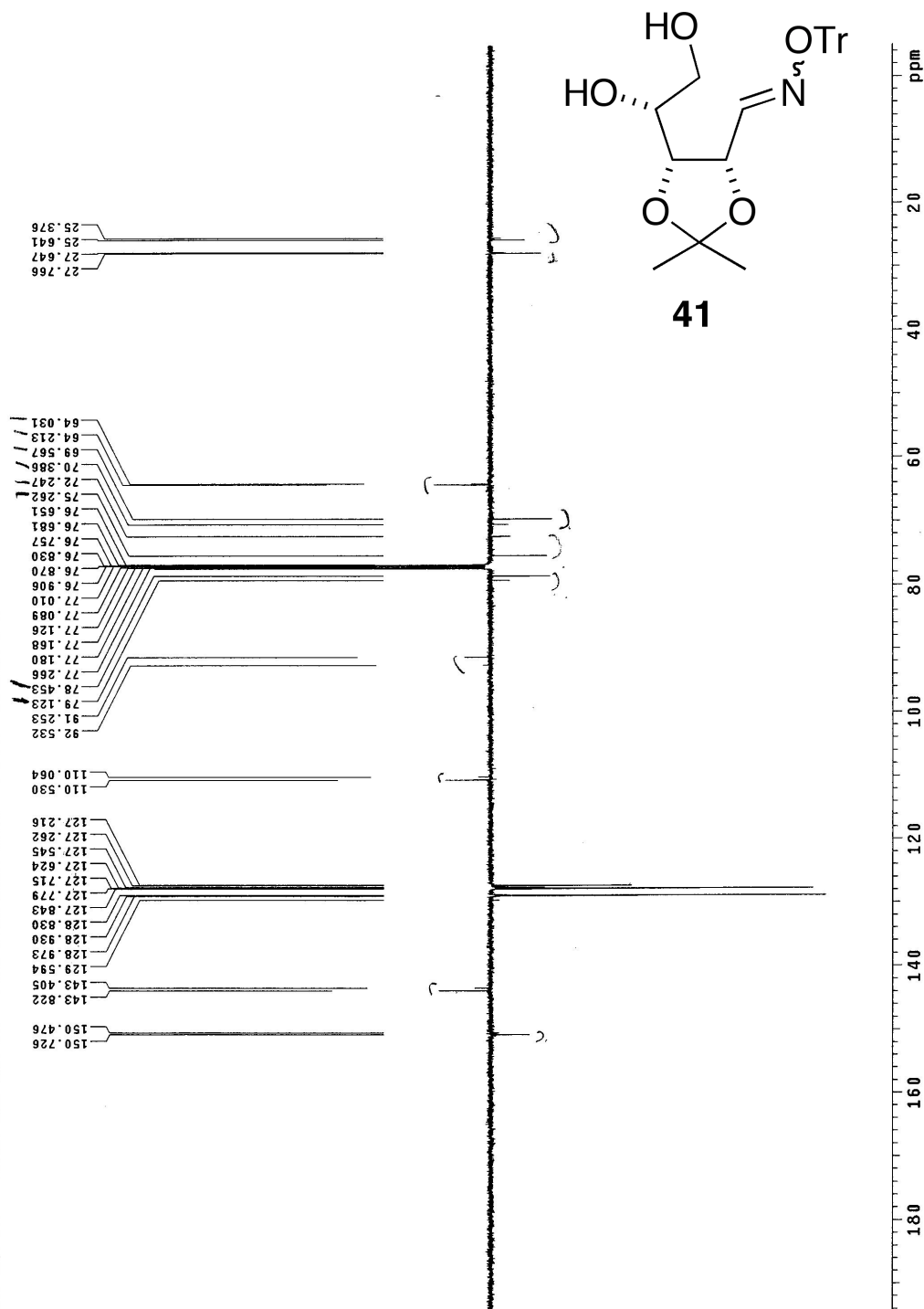




RS G-16

125 MHz APT in CDCl₃ (ref. to CDCl₃ @ 77.0 ppm), temp 27.2 C -> actual temp = 27.0 C, sw probe CH, CH3 positive; C, CH2 negative
 date: Jul 13, 2001 seq: 104.17 spectrometer: hds5 f1la: e3g
 acq: time [s]: 2.0 seq: relax: time [s]: 0.5 sweep width [Hz]: 25000 d1: 0.38

Pulse Sequence: apt



6-20
1 IN CDCl₃

27.667
25.526

36.814

79.074
77.035
76.369
75.107
69.827

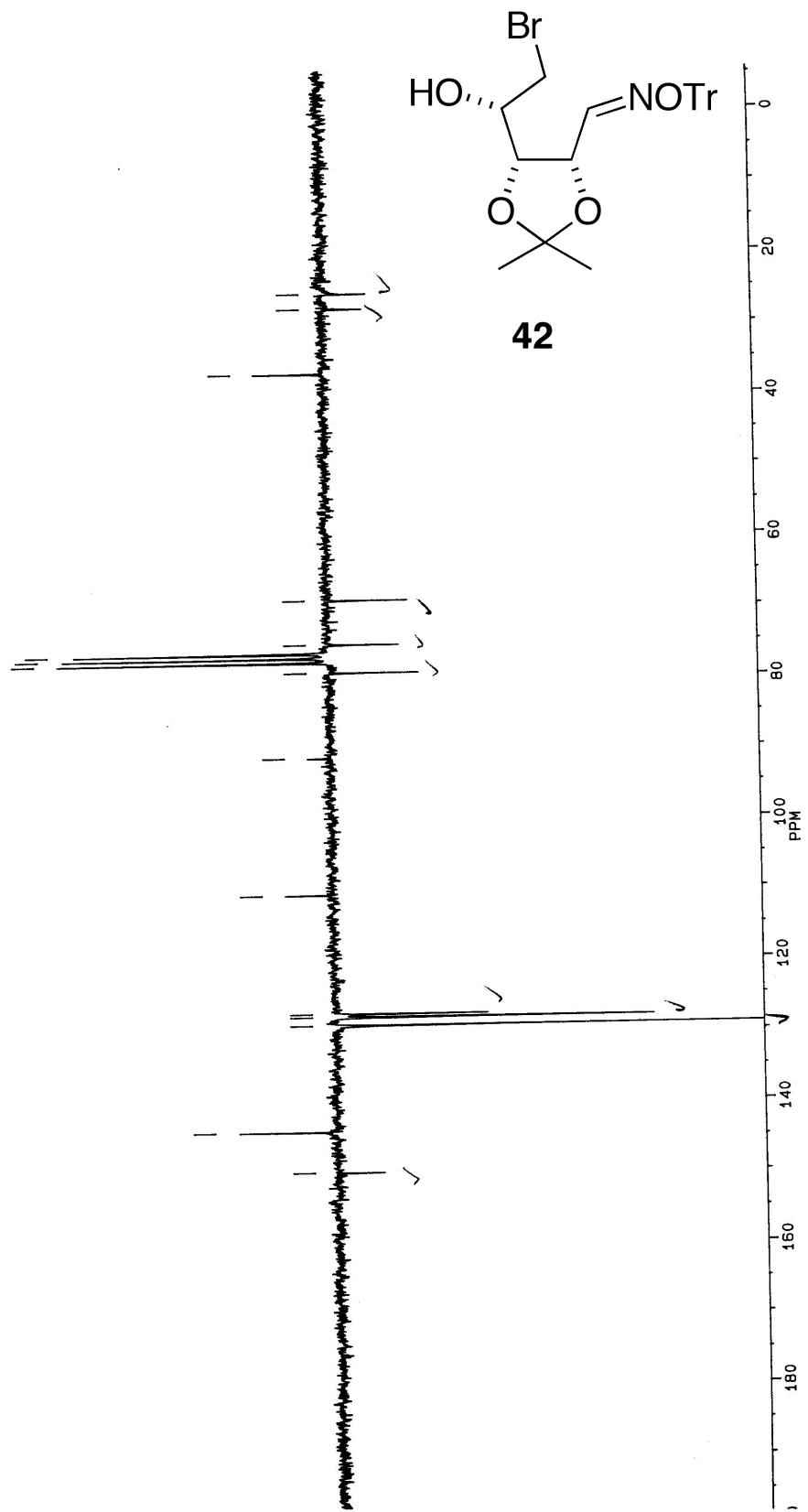
91.228

110.512

128.925
127.766
127.510

143.982

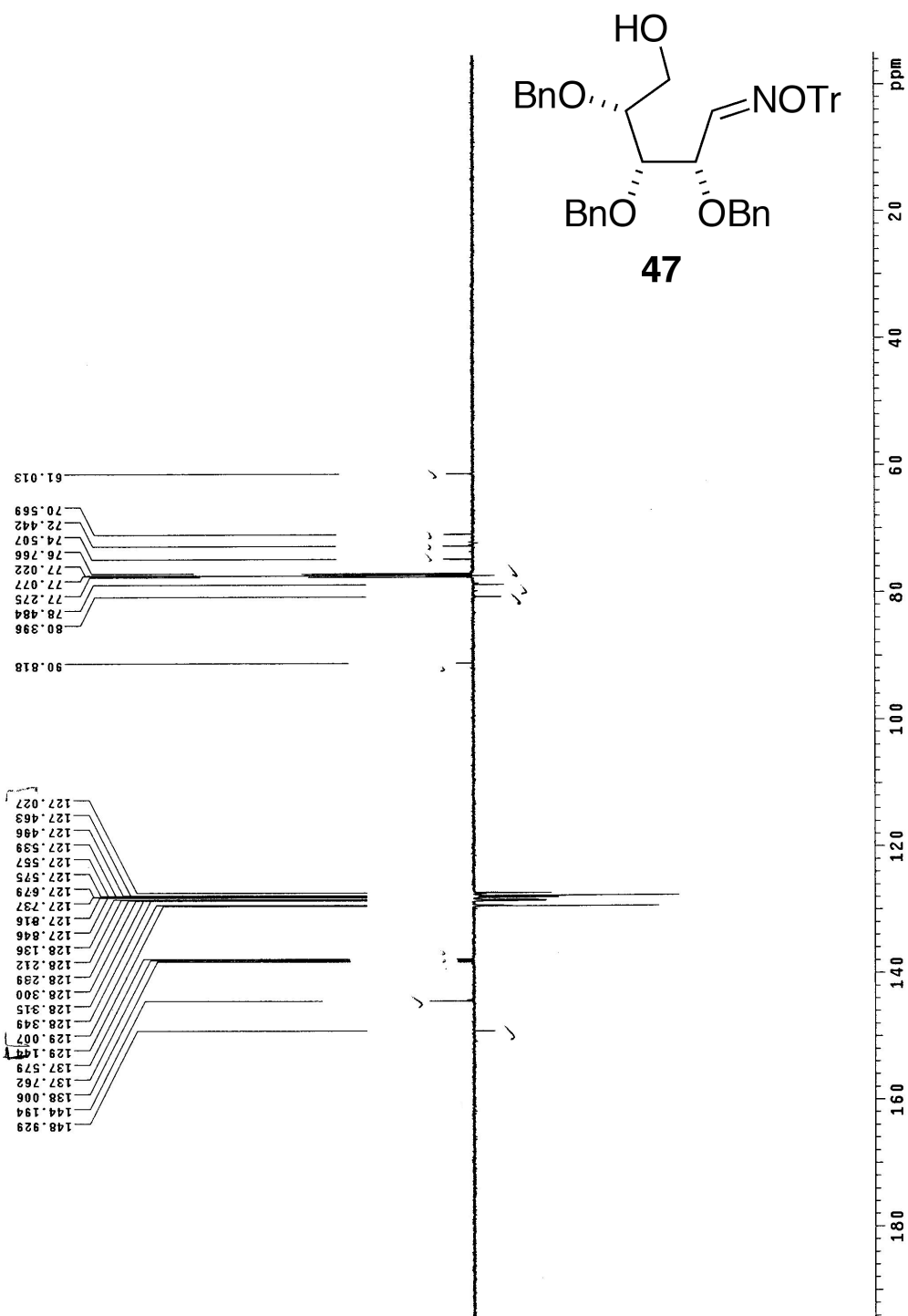
149.757



RS H-23

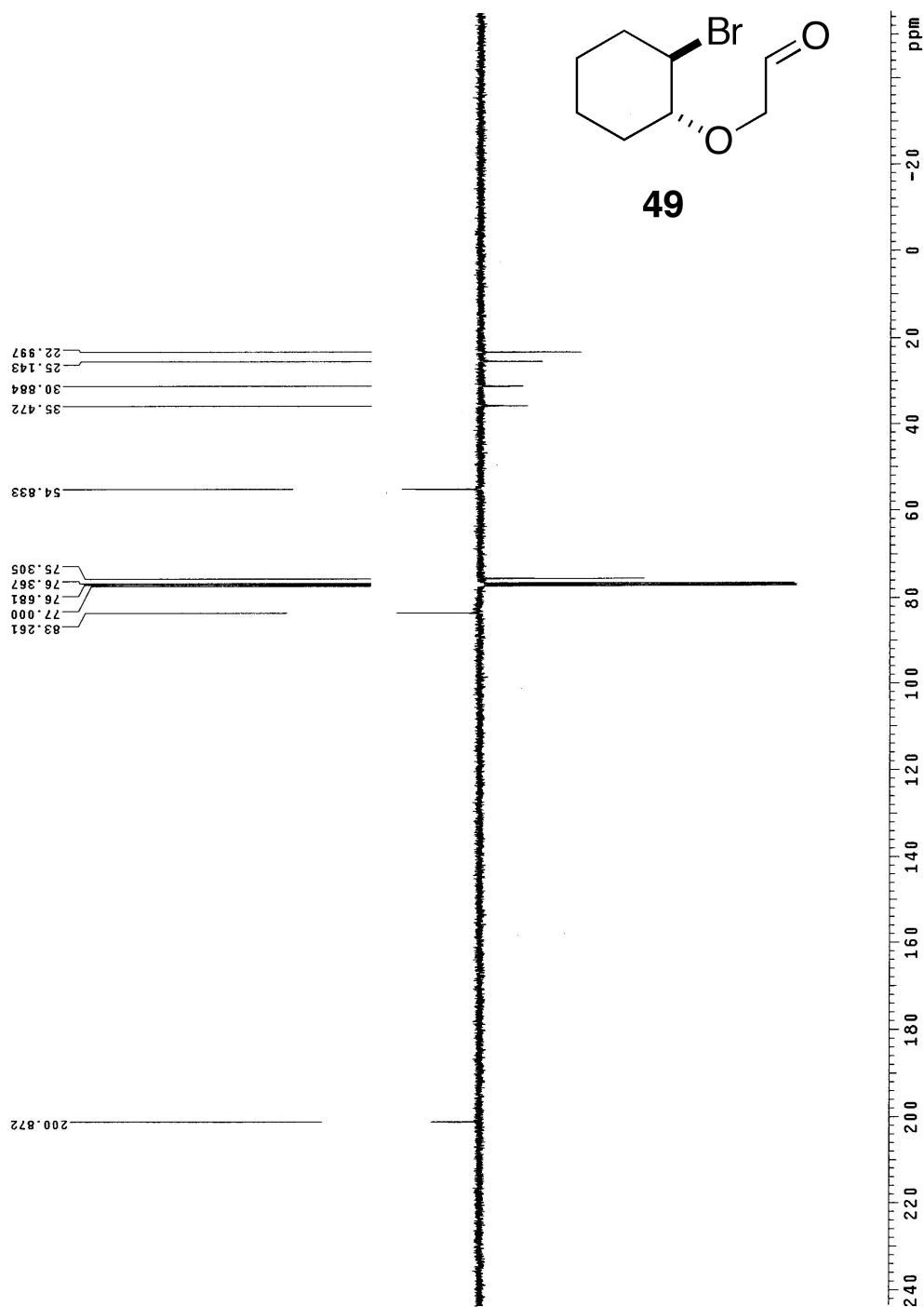
Pulse Sequence: apt

125 MHz APT in CDCl₃ (ref. to CDCl₃ @ 77.0 ppm), temp 27.2 C -> actual temp = 27.0 C, sw probe CH, CH3 positive; C, CH2 negative
 date: Jul 23 2006 seq: 104.17 spectrometer: jbd5 file: ex8
 aq: time [s]: 2.00 relax: time [s]: 0.5 sweep width [Hz]: 25000 dig: res. [Hz/pt]: 0.38

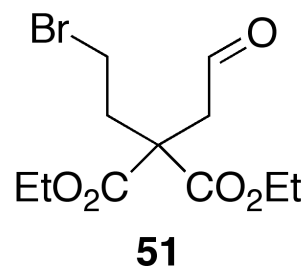
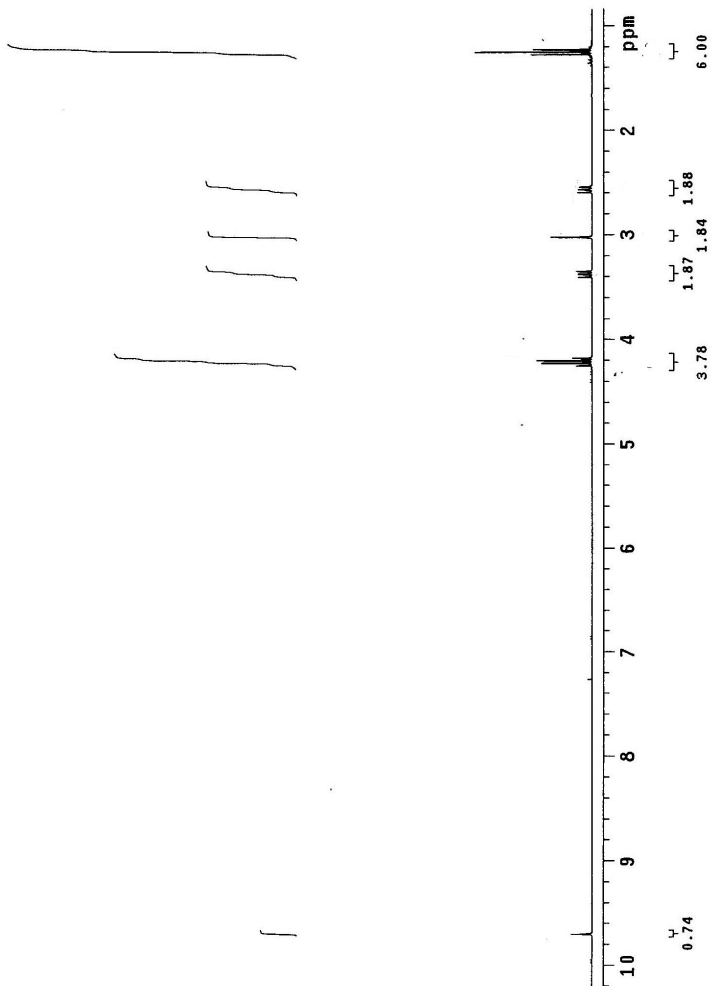


RS G-71

Pulse Sequence: apt
100 MHz APT (in CDCl₃ (ref. to CDCl₃ @ 77.0 ppm), temp 27.8 C -> actual temp = 27.0 C, asw400 probe
CH, CH3 positive; C, CH2 negative
acq time [s]: 2.00 relax time [s]: 0.50 sweep width [Hz]: 125.08 spectrometer: 400
file: /mnt/d600/nmr/abdata/Clive/Subedi/Bromocyclohexane-aldehyde-cl3.fld
dig.res. [Hz/pt]: 0.48



MP2-151H-1
300 MHz ID in CDCl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.5 C -> actual temp = 27.0 C, id probe
date: Aug 19 2005 sweep width: 3598Hz acq.time: 2.06 relax.time: 3.0s # scans: 16 d1g.res.: 0.1 Hz/pt h
file:/mnt/d600/cilivenardata/NA1/MP-Book2/MP2-151H-1.f1d
spectrometer: lbdw



RS G-91

Pulse Sequence: apt

125 MHz APT in CDCl₃ (ref. to CDCl₃ @ 77.0 ppm), temp 27.2 C -> actual temp = 27.0 C, sw probe

CH CH3 positive; C, CH2 negative

date: Jun 28 2001 seqfil: apt

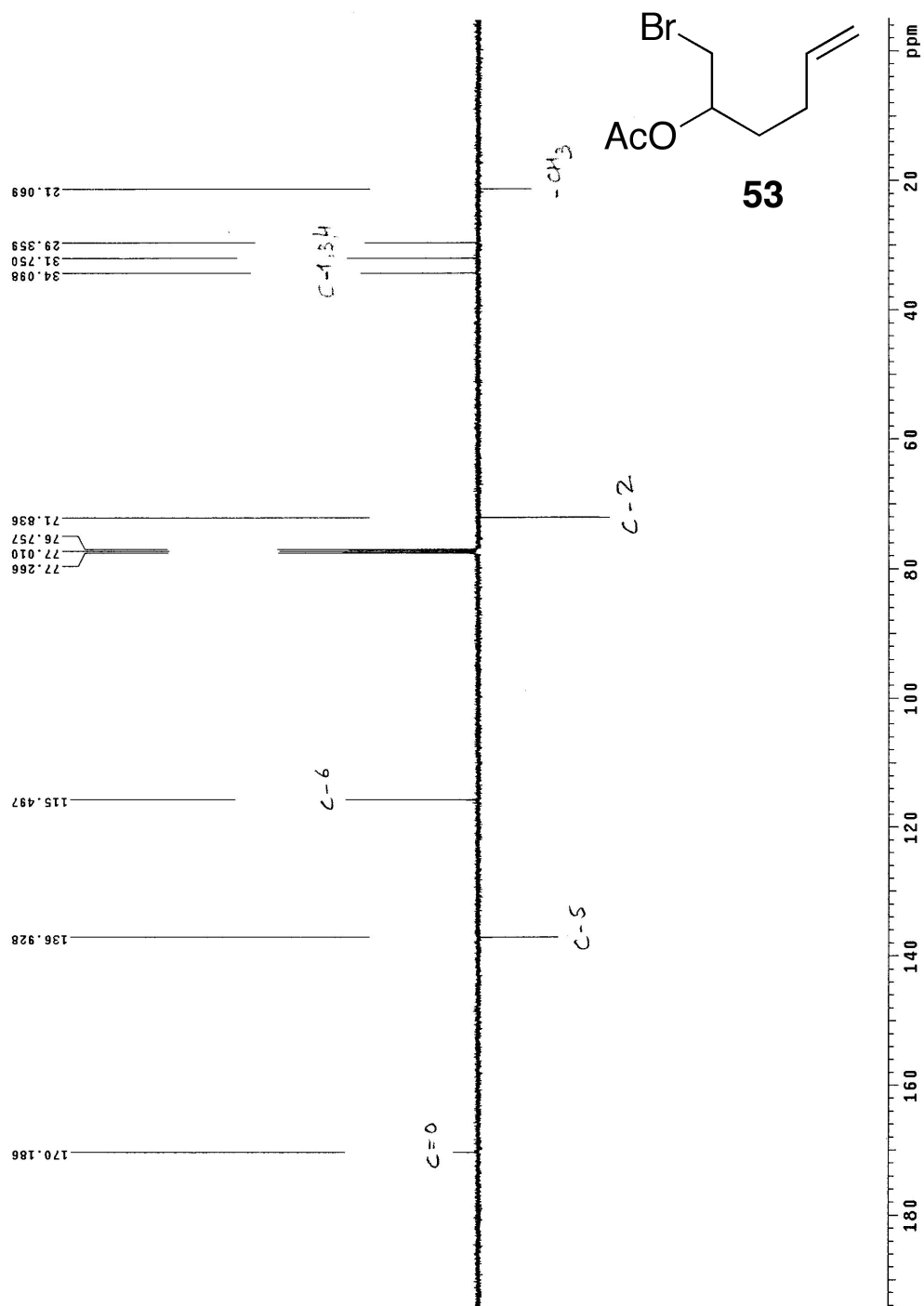
acq.time [s]: 2.0

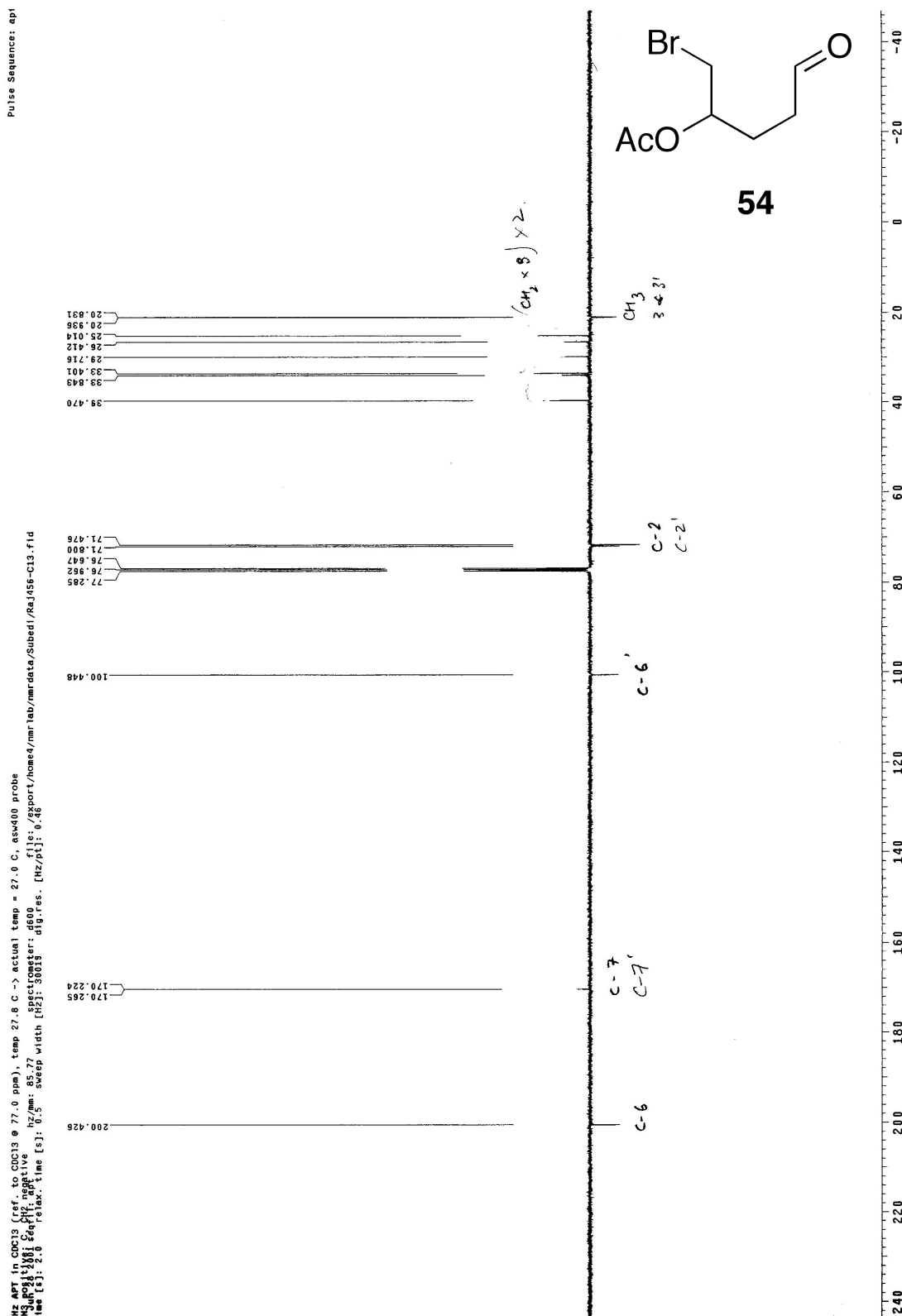
relax.time [s]: 0.5

sweep width [Hz]: 25000

dig.res. [Hz/pt]: 0.38

file: /mnt/d600/nmr/labdata/Subset1/RSG-91-C13.fid

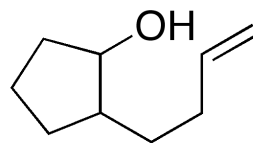
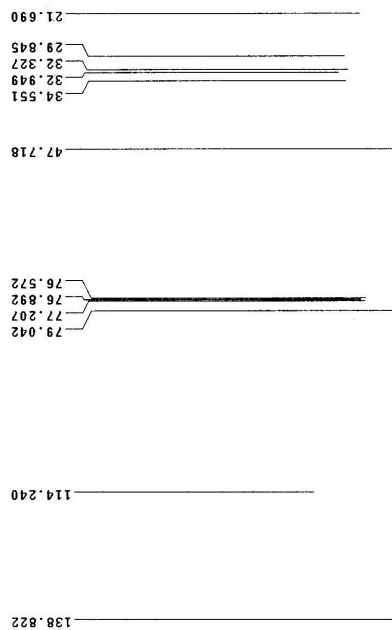




MP3-15C-md
100 MHz APT in CDCl₃ (ref. to CDCl₃ @ 77.0 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
C & CH₂ same, CH & CH₃ opposite side of solvent signal
date: Sep 26 2005 sweep width: 27027Hz acq.time: 2.0s relax.time: 0.1s # scans: 1248 dig.res.: 0.4 Hz/pt
spectrometer:m400 file:exp

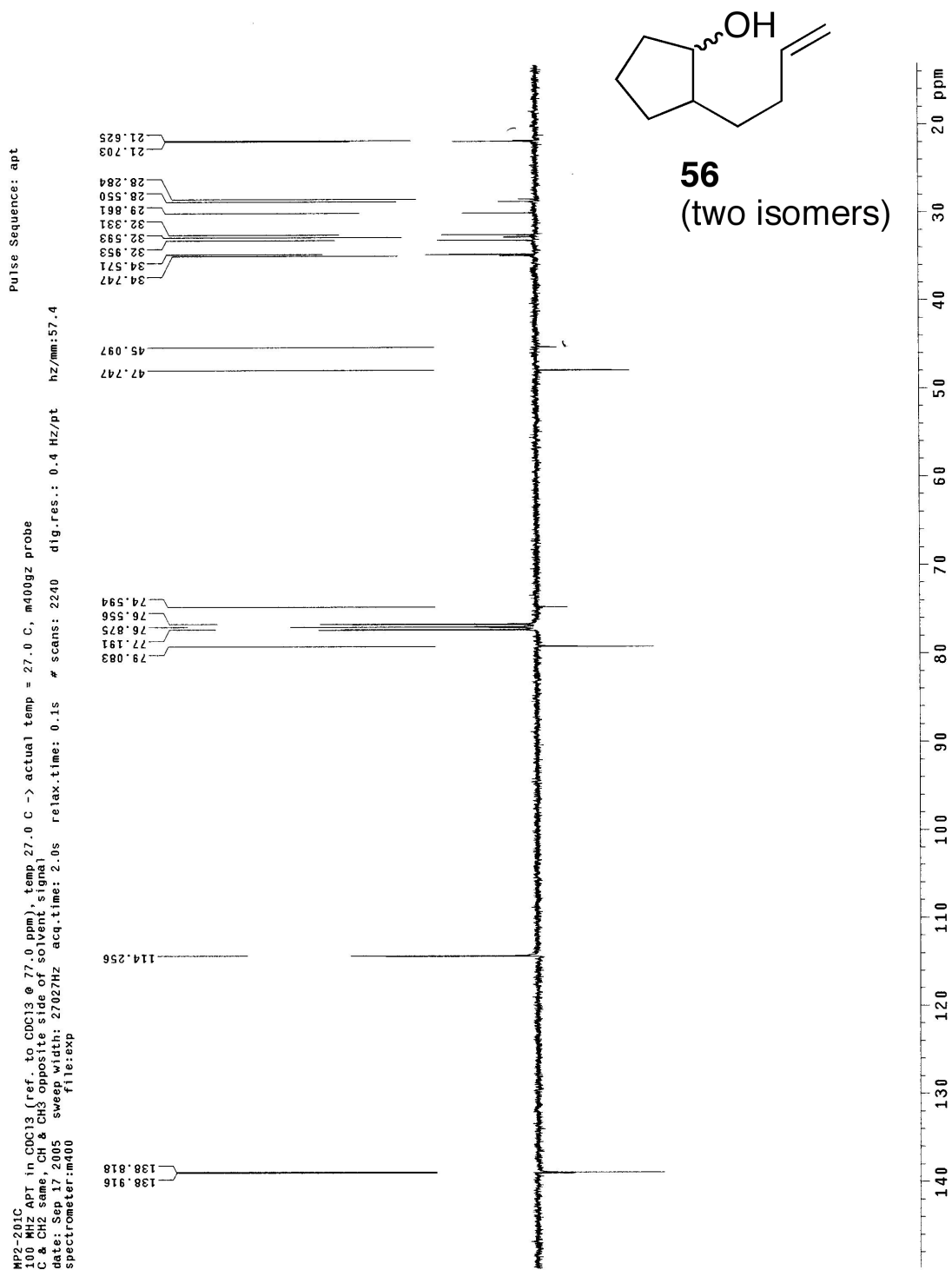
Pulse Sequence: apt

hz/mm:112.6



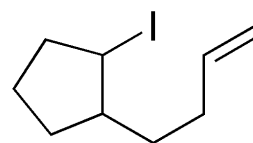
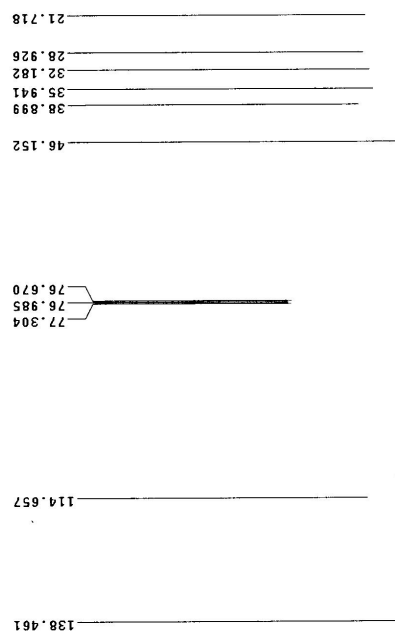
56 (single isomer)





MP3-35C-2
1100 MHz APT in CDCl₃ (ref. to CDCl₃ @ 77.0 ppm), temp 26.8 C -> actual temp = 27.0 C, asw400 probe
C & CH₂ same, CH & CH₃ opposite side of solvent signal
date: Oct 5 2005 sweep width: 26954Hz acq.time: 2.0s relax.time: 0.1s # scans: 256 dig.res.: 0.4 Hz/pt
spectrometer:1400 file:exp

Pulse Sequence: apt



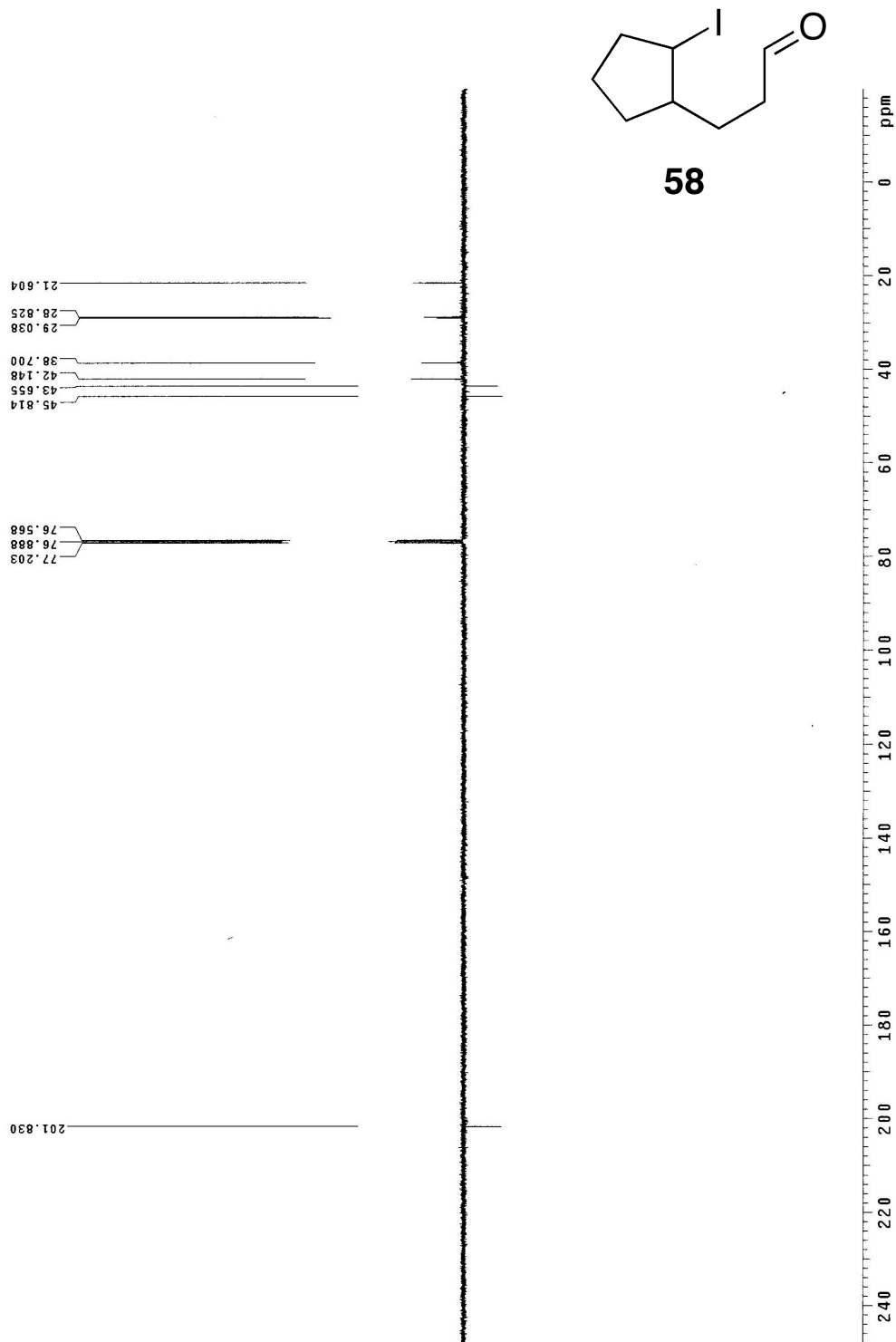
57



MP3-33C
m100 MHz APT in CDCl₃ (ref. to CDCl₃ @ 77.0 ppm), temp 27.0 C -> actual temp = 27.0 C, m400g2 probe
C & CH2 same, CH & CH3 opposite side of solvent signal
date: Oct 6 2005 sweep width: 27027Hz acq.time: 2.0s relax.time: 0.1s # scans: 400 dig.res.: 0.4 Hz/pt
spectrometer:m400 file:exp

Pulse Sequence: apt

hz/mm:112.6



MF3-125C
m100 MHz API in CDCl3 (ref. to CDCl3 @ 77.0 ppm), temp 27.0 C -> actual temp = 27.0 C, m400qz probe
C & CH2 same, CH & CH3 opposite side of solvent signal
date: Jan 7 2006 sweep width: 27027Hz acq.time: 2.0s relax.time: 0.1s # scans: 872 dig.res.: 0.4 Hz/pt
spectrometer:m400 file:exp

Pulse sequence: apt

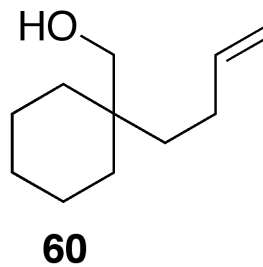
hz/mm:112.6

36.889
33.879
32.323
27.420
26.294
21.358

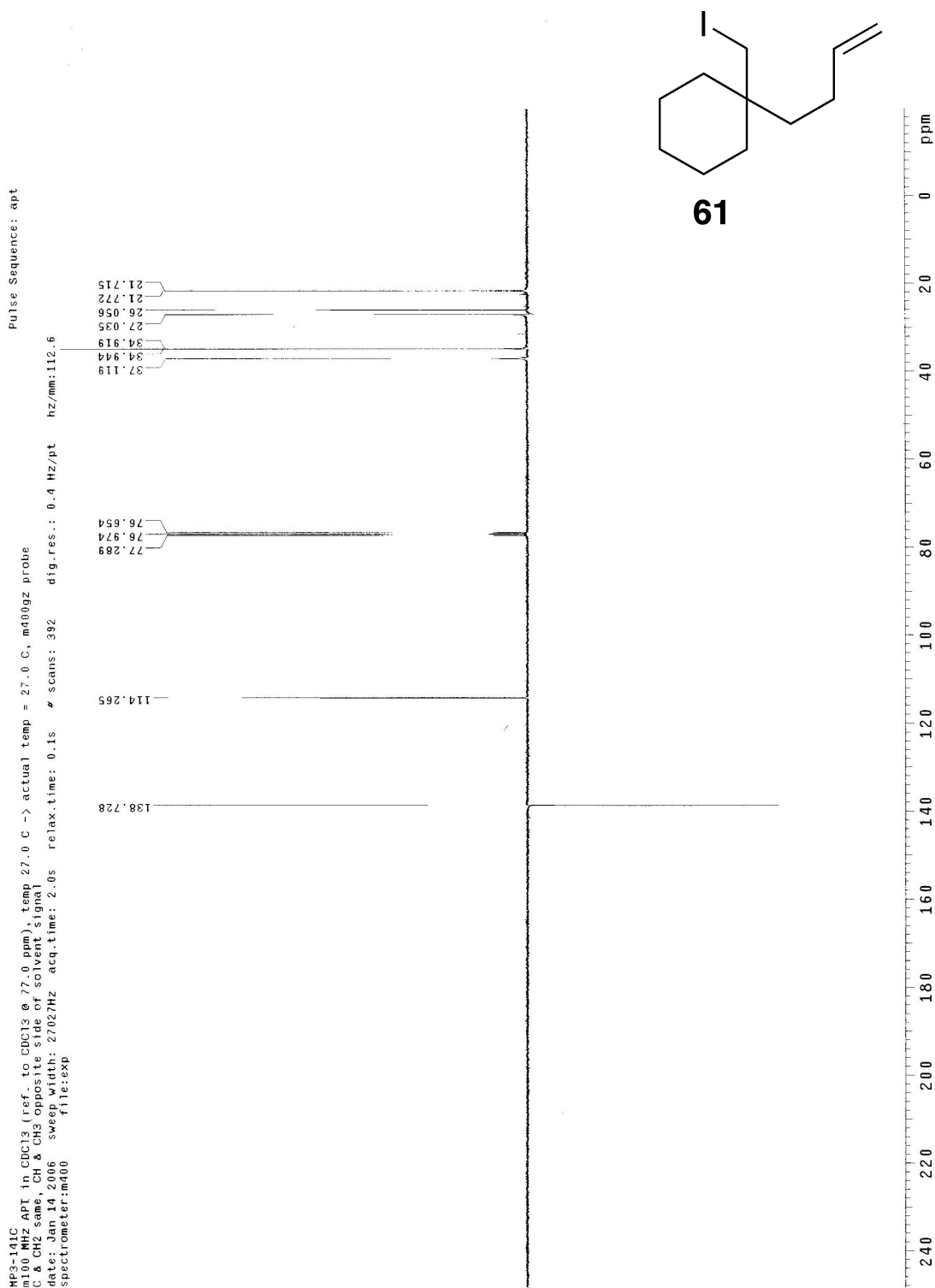
77.232
76.916
76.597
68.205

113.876

139.551



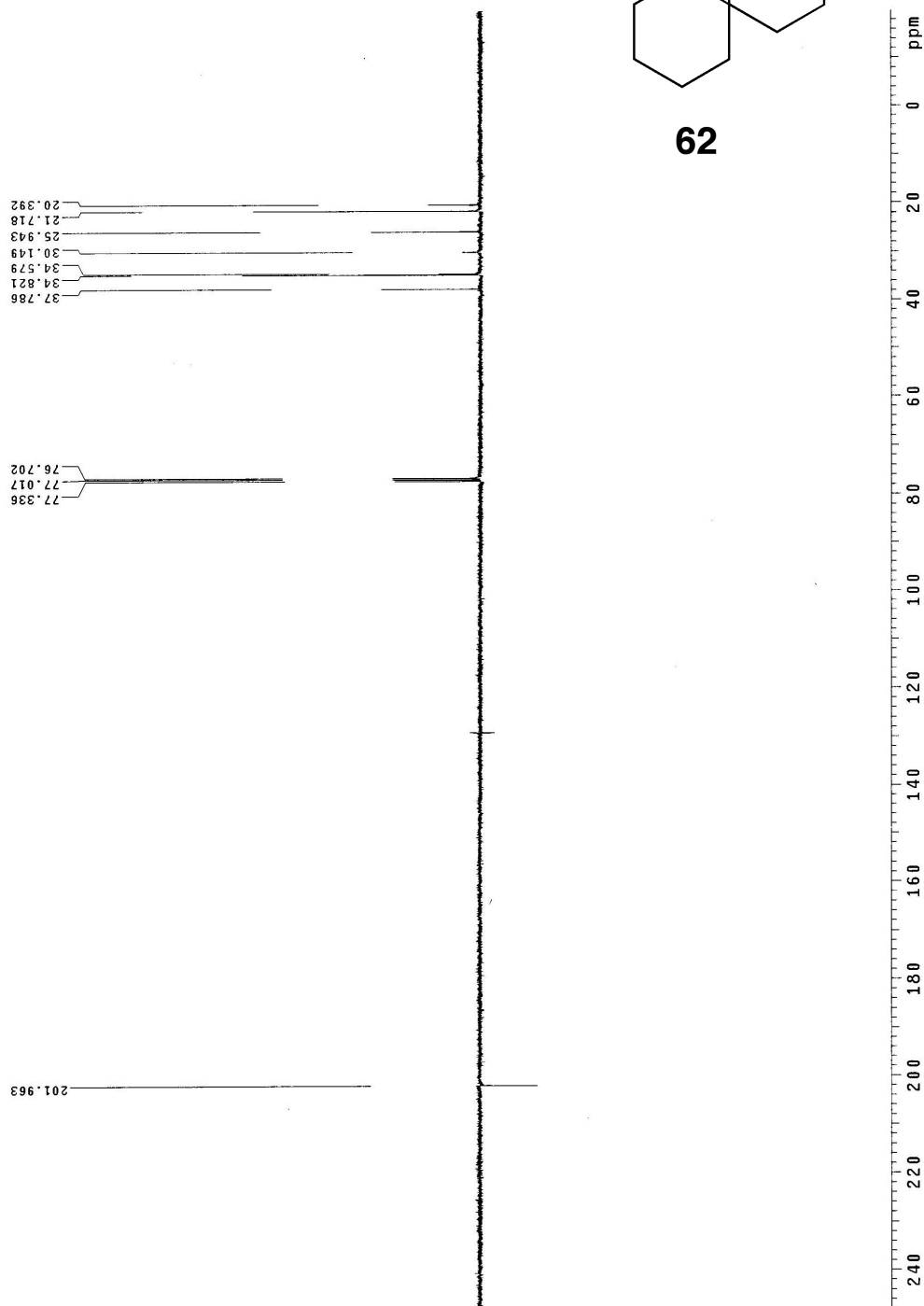
240 220 200 180 160 140 120 100 80 60 40 20 0 ppm

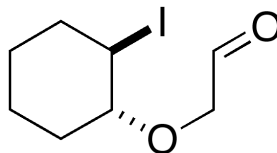


MP3-143C
1100 MHz APT in CDCl₃ (ref. to CDC13 @ 77.0 ppm), temp 26.8 C -> actual temp = 27.0 C, asw400 probe
C & CH2 same, CH & CH3 opposite side of solvent signal
date: Jan 17 2006 sweep width: 26954Hz acq.time: 0.1s # scans: 336 dfg.res.: 0.4 Hz/pt
spectrometer:1400 file:exp

Pulse Sequence: apt

hz/mm:112.3



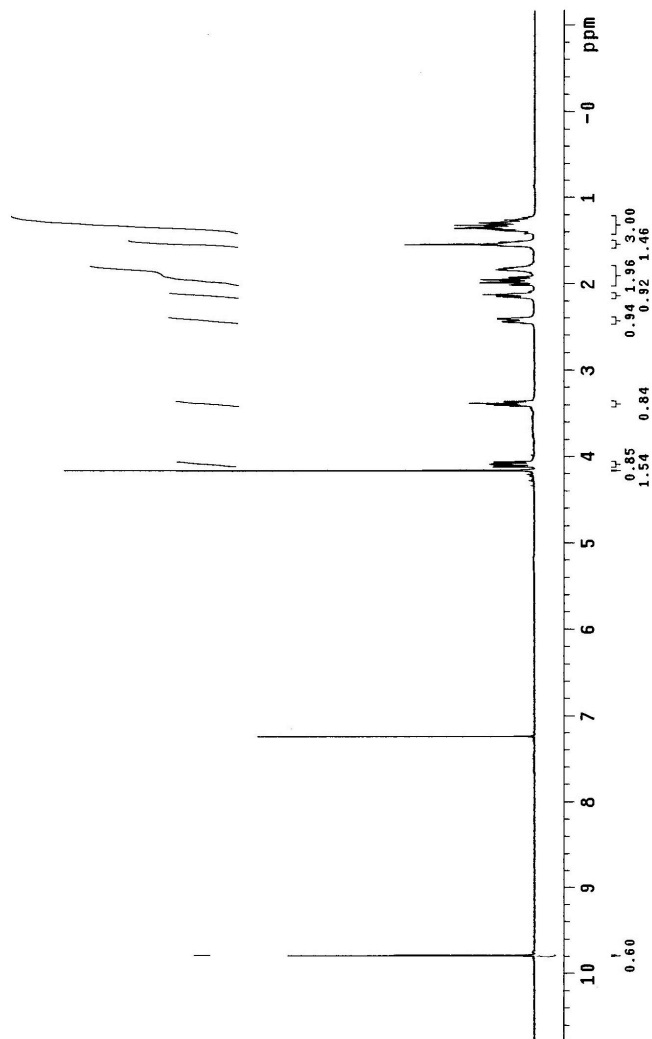


Trans-[(2-iodocyclohexyl)oxy]acetaldehyde

MPI-199H
400 MHz 1D in CDC13 (ref. to CDC13 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Solvent: cdc13
Temp: 27.0 C / 300.1 K
File: MPI-199H
INOVIA-500 "1bow"

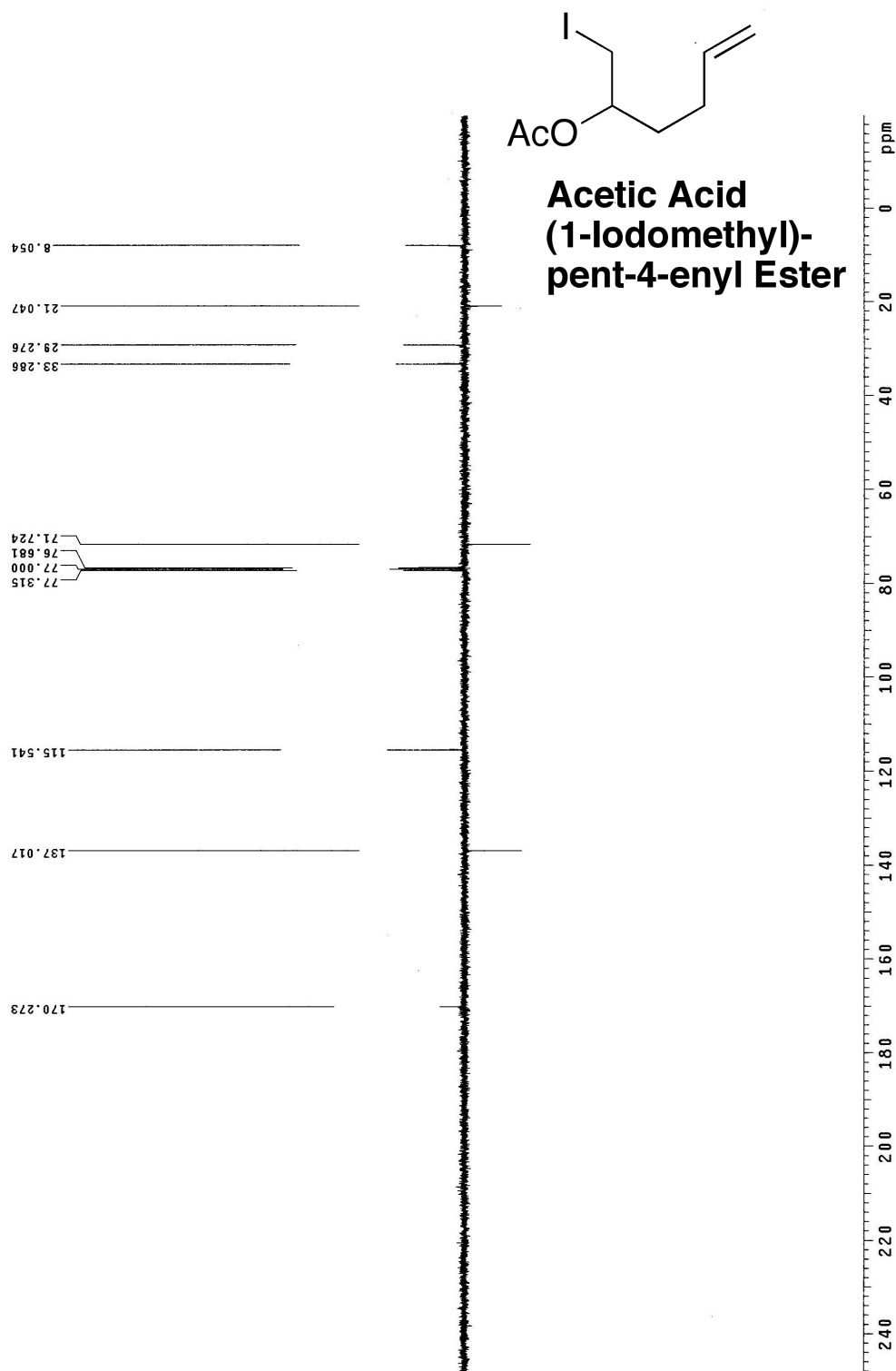
Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 1.99 sec
Width 4001 Hz
16 repetitions
OBSERVE H1 400.3981759 MHz
DATA PROCESSING
FT size 131072
Total time 1 min, 30 sec



MP2-55C
100 MHz APT in CDCl₃ (ref. to CDCl₃ @ 77.0 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
C & CH₂ same, CH & CH₃ opposite side of solvent signal
date: Jun 27 2005 sweep width: 27027Hz acq.time: 2.0s relax.time: 0.1s # scans: 152 dfg.res.: 0.4 Hz/pt
spectrometer:m400 file:exp

Pulse Sequence: apt

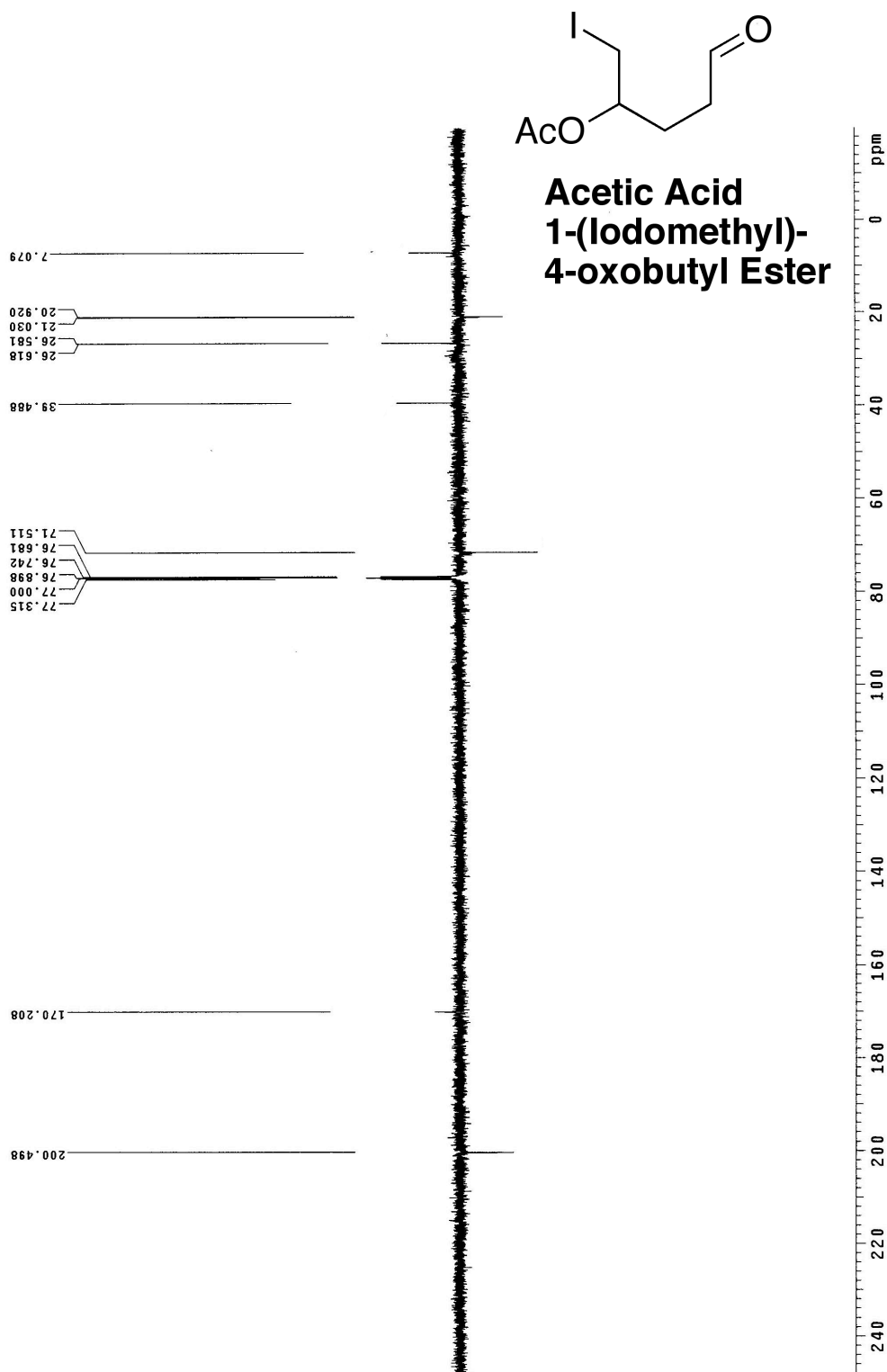
hz/mm:112.6

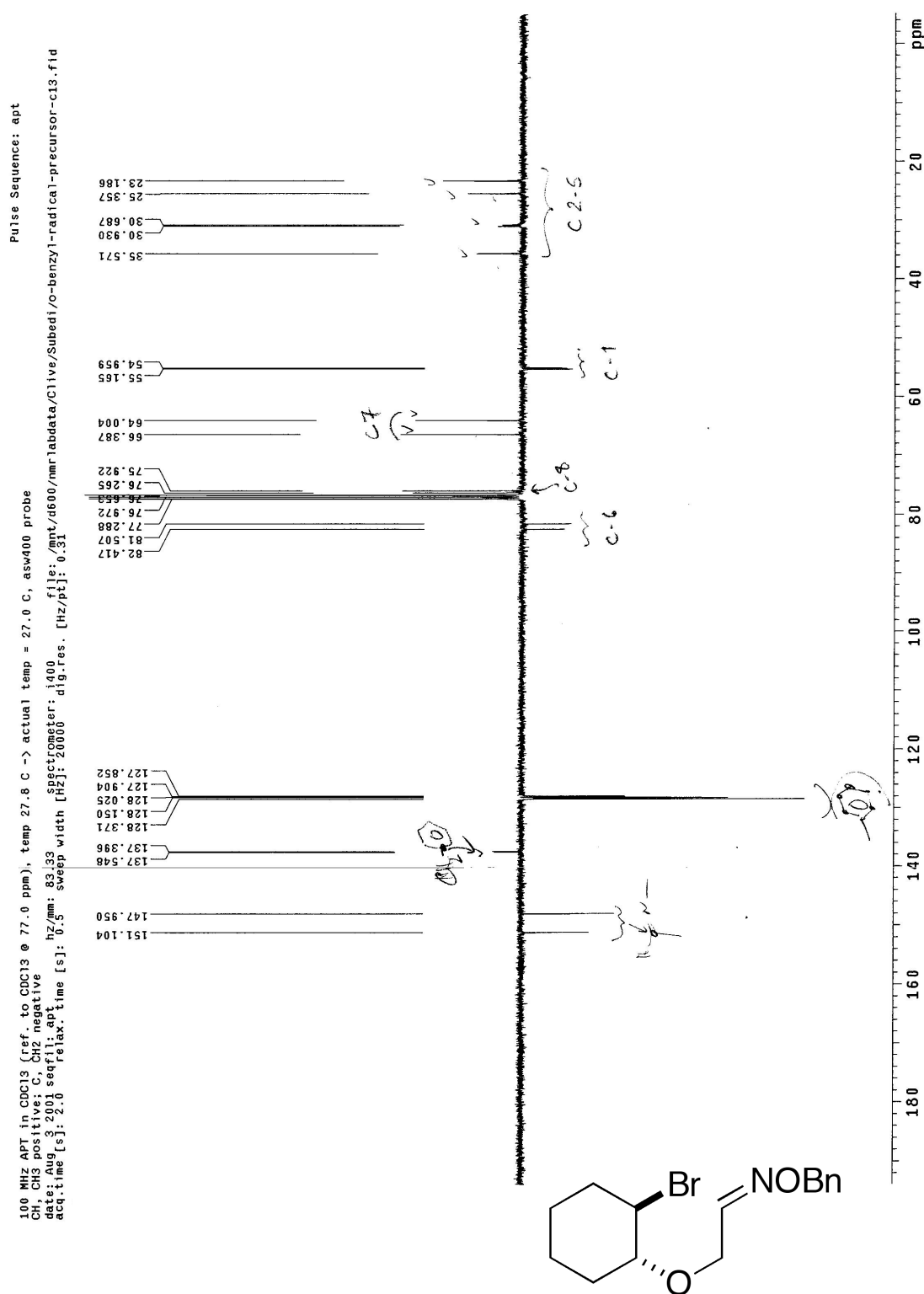


100 MHz APT in CDCl₃ (ref. to CDCl₃ @ 77.0 ppm), temp 27.0 C -> actual temp = 27.0 C, m400g2 probe
C & CH₂ same, CH & CH₃ opposite side of solvent signal
date: Jul 2 2005 sweep width: 27027Hz acq.time: 2.0s relax.time: 0.1s # scans: 254 dig.res.: 0.4 Hz/pt
spectrometer:m400 file:exp

Pulse Sequence: apt

hz/mm:112.6



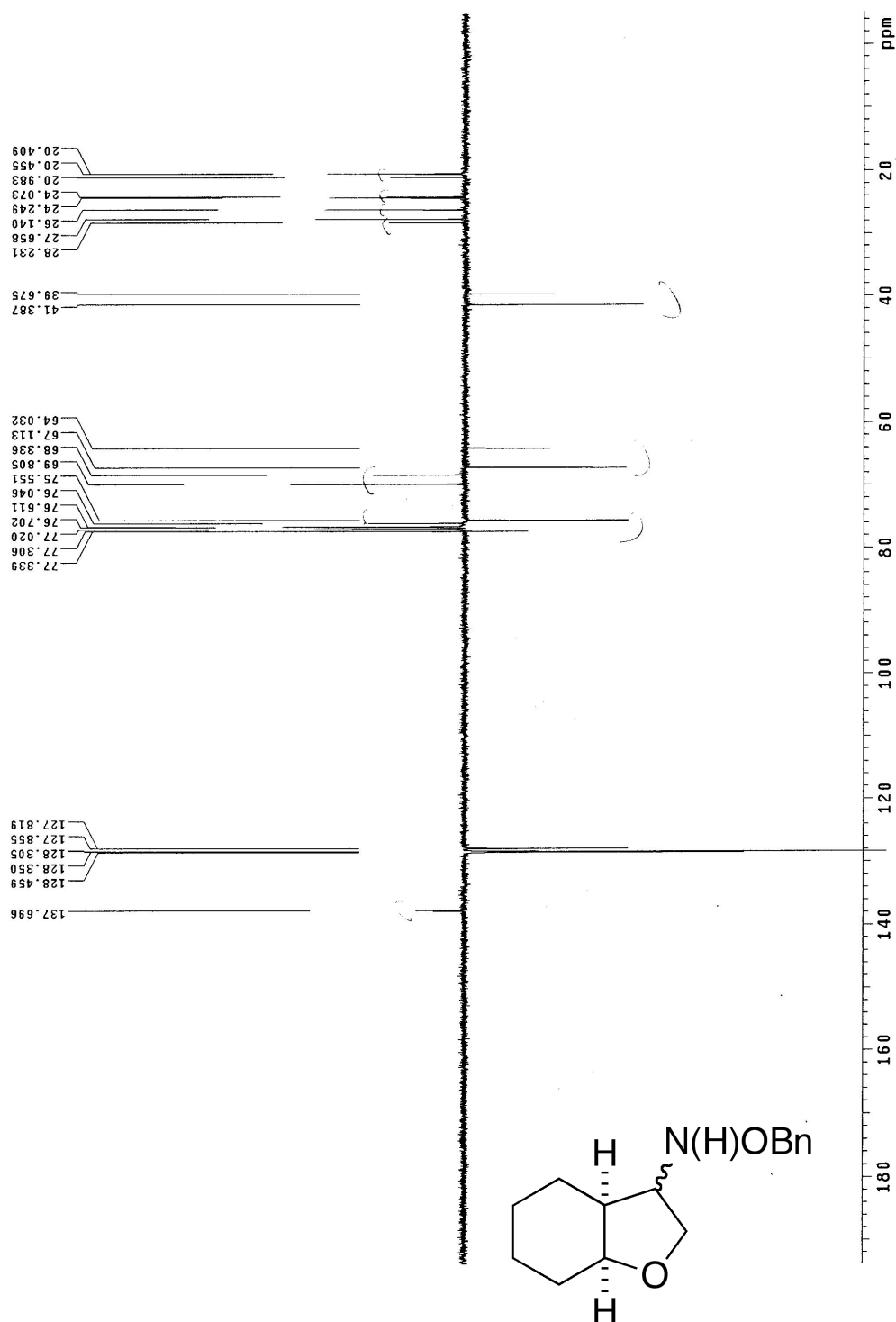


**Trans-2-[(2-bromocyclohexyl)oxy]-
acetaldehyde *O*-(phenylmethyl)oxime**

(Handwritten signature)

100 MHz APT in CDCl₃ (ref. to CDCl₃ @ 77.0 ppm), temp 27.8 C -> actual temp = 27.0 C, asw400 probe
 CH, CH₃ positive; C, CH₂ negative
 date: Aug. 2003 seq: 111, apt: time [s]: 83.33 spectrometer: (400
 acq: time [s]: 2.0 relax: time [s]: 0.5 sweep width [Hz]: 20000 dig: res. [Hz/pt]: 0.31

Pulse Sequence: apt



O-Benzyl-N-[(3a α ,7a α)octahydro-benzofuran-3-yl]hydroxylamine