Continuum of Mechanisms for the Nucleophilic Substitution of Cyclic Acetals

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Supporting Information

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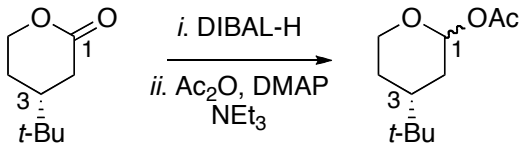
I. General Experimental Considerations

^{1}H NMR and ^{13}C NMR spectra were recorded at ambient temperature at 400 or 500 MHz and 100 or 125 MHz, respectively, using a Bruker DRX 400 or DRX 500 spectrometer. Chemical shift data are reported in ppm downfield from tetramethylsilane and are referenced to the proton or carbon peaks for this standard. NMR resonance multiplicities are reported with the following abbreviations: br = broad, s = singlet, d = doublet, t = triplet, q = quartet, and m = multiplet, and coupling constants are reported in Hz. Infrared (IR) spectra were obtained using a MIDAC Prospect FT-IR spectrometer, a Perkin-Elmer Paragon 1000PC FT-IR, or a React-IR AS-1000 spectrometer. High resolution mass spectra were acquired on a VG Analytical 7070E, Fisons Autospec, or Waters LCT Premier spectrometer, and were obtained by peak matching. Microanalyses were performed by Atlantic Microlab Inc, Norcross, GA. Analytical gas-liquid

chromatography (GC) analyses were performed on an Agilent 6850 series chromatograph, equipped with an Agilent 6850 auto-sampler and a flame-ionization detector. A fused silica capillary column (30 m x 0.32 mm x 0.25 μm) wall-coated with DB-1 (J & W Scientific) was used with helium as the carrier gas (25 psi column head pressure). Method of chromatography is as follows: Start temperature = 50 °C; ramp = 10 °C/min; final temperature = 250 °C. Reported melting points are uncorrected. Liquid chromatography was performed using forced flow (flash chromatography) of the indicated solvent system on silica gel (SiO_2) 60 (230–400 mesh). All reactions were performed under nitrogen atmosphere in dry glassware. Glassware was dried in one of the following methods: oven-dried for at least eight hours at 150 °C, or flame-dried under dynamic vacuum, or flame-dried under a stream of nitrogen. Unless otherwise noted, all reagents were obtained from commercial suppliers and, where appropriate, purified prior to use. Acetal **1**,[1] lactone **S1**,[2] lactol **S2**,[1] enoxy silane **8**,[3,4] and silyl-ketene acetals **9**,[4,5] **10**,[4,6] and **11**[4,7] were prepared by reported methods. THF, Et_2O, and CH_2Cl_2 were dried by filtration through alumina according to the procedure of Grubbs.[8]

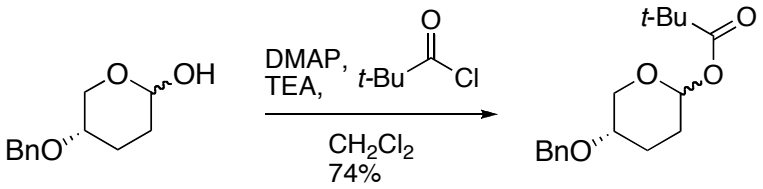
II. Substrate Preparation



S1 **12**

C3 *t*-Bu Acetal 12. To a cooled (–78 °C) solution of lactone **S1**[2] (1.00 g, 6.40) in CH_2Cl_2 (90 mL) was added diisobutylaluminum hydride (4.48 mL of a 1.5 M solution in toluene, 6.72 mmol) over 40 minutes. After stirring at –78 °C for 3 h, acetic anhydride

(2.66 mL, 28.2 mmol) and pyridine (1.81 mL, 22.4 mmol) were added to the reaction mixture dropwise followed by dimethylaminopyridine (0.820 g, 6.72 mmol) in one portion. The reaction mixture was allowed to slowly warm to room temperature and stir overnight. After this time, saturated aqueous ammonium chloride (50 mL) and methyl *tert*-butyl ether (300 mL) were added to the reaction mixture. The resulting emulsion was filtered through Celite and the layers were separated. The organic layer was washed with saturated aqueous Na_2HPO_4 (2 x 40 mL), saturated aqueous NaH_2PO_4 (2 x 40 mL) and saturated aqueous $CuSO_4$ (2 x 40 mL). The organic layer was dried over Na_2SO_4, filtered, and concentrated by rotary evaporation to yield a clear yellow oil (3.40 g). The residue was purified by flash chromatography (80:20 hexane/EtOAc) to afford **12** as a clear colorless oil (0.857 g, 67%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.59 (dd, *J* = 9.5, 2.5, 1.0H), 4.09 (ddd, *J* = 11.6, 4.4, 1.7, 1.0H), 3.55 (dt, *J* = 11.6, 2.5, 1.1H), 2.11 (s, 3.1H), 1.85 (m, 1.0H), 1.54 (m, 1.1H), 1.40–1.23 (m, 3.4H), 0.87 (s, 9.5H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 169.6, 121.5, 95.3, 66.6, 44.4, 32.2, 31.8, 27.2, 26.2, 21.3; IR (thin film) 1754, 1232, 1060 cm^{-1}; HRMS (TOF MS ES+) *m* / *z* calcd for $C_{11}H_{20}O_3Na$ $(M + Na)^+$ 223.1310, found 223.1304. Anal. Calcd for $C_{11}H_{20}O_3$: C, 65.97; H, 10.07. Found: C, 65.69; H, 10.23.



S2 **17**

Pivaloate Acetal 17. To a cooled (0 °C) solution of dimethylaminopyridine (DMAP, 0.368 g, 3.01 mmol), triethylamine (4.2 mL, 30 mmol), 4-benzyloxytetrahydropyran-1-ol[1] (**S2**, 1.57 g, 7.53 mmol) in CH_2Cl_2 (35 mL) was added

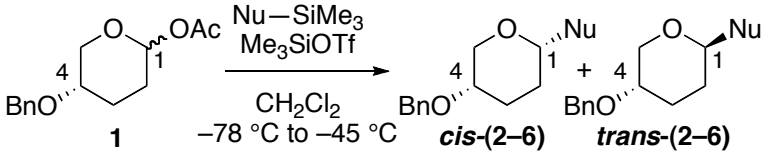
pivaloyl chloride (1.5 mL, 11 mmol) dropwise by syringe. The reaction mixture was then warmed to room temperature and stirred for 2 h. After this time, the reaction mixture was poured onto 160 mL of a saturated aqueous ammonium chloride solution. The layers were separated, and the aqueous phase was extracted with CH_2Cl_2 (4 x 200 mL). The combined organic extracts were dried ($MgSO_4$), filtered, and concentrated by rotary evaporation to yield a colorless oil, which formed white crystals of **17** (4.2 g) upon standing. The crystals were washed with pentane (2 x 10 mL), then further purified by flash chromatography (95:5 → 90:10 hexane/EtOAc) to afford **17** (as a 1: 1.5 mixture of anomers) as a white solid (1.6 g, 74%): mp 51–58 °C ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39–7.29 (m, 13.2H), 6.05 (t, *J* = 2.9, 1H), 6.00 (br, 1.5H), 4.60 (m, 5.2H), 3.93 (dd, *J* = 12.4, 2.4, 1H), 3.87–3.78 (m, 2.6H), 3.68–3.56 (m, 4.2H), 2.22 (m, 1H), 2.06–1.97 (m, 2.7H), 1.94–1.89 (m, 5.7H), 1.64–1.59 (m, 2.3H), 1.25 (s, 22.6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 138.4, 138.3, 128.6, 128.5, 127.9, 127.74, 127.71, 127.66, 92.0, 90.6, 72.0, 70.9, 70.5, 70.4, 64.5, 64.1, 39.15, 39.13, 27.7, 27.1, 24.9, 24.4, 22.7; IR (thin film) 1736, 1155, 1128, 1097, 1027 cm^{-1}; HRMS (TOF MS ES+) *m* / *z* calcd for $C_{15}H_{20}O_2Na$ $(M + Na)^+$ 315.1572, found 315.1566. Anal. Calcd for $C_{17}H_{24}O_4$: C, 69.84; H, 8.27. Found: C, 69.50; H, 8.24.

III. Nucleophilic Substitution

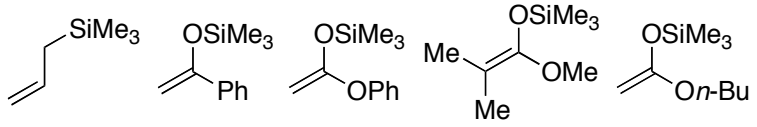
General Procedure for Nucleophilic Substitution Reactions of Acetal 1 and 12: To a cooled (–78 °C) 0.1 M solution of acetal **1** (0.40–0.61 mmol, 1.0 equiv) and nucleophile (2.0–4.0 equiv) in CH_2Cl_2 was added the Lewis acid (1.4–1.6 equiv) dropwise. After stirring at –78 °C for 1–5 min, the reaction mixture was maintained at –

45 °C for 1–2 h. A saturated aqueous solution of $NaHCO_3$ (4.0 mL) was added and the solution was warmed to 22 °C. The resulting biphasic mixture was diluted with CH_2Cl_2 (2 mL) and H_2O (2 mL) and the layers were separated. The aqueous layer was extracted with CH_2Cl_2 (3 x 2 mL) and the combined organic layers were dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Diastereomeric ratios were determined by GC and confirmed by GC/MS and ^1H NMR spectroscopy. Reported yields refer to purified material, and examples with each nucleophile are described in the text; those tables are reproduced here (Table 1 and Table 2).

Table 1. Nucleophile Screen with Me_3SiOTf.



Nu—$SiMe_3$ =

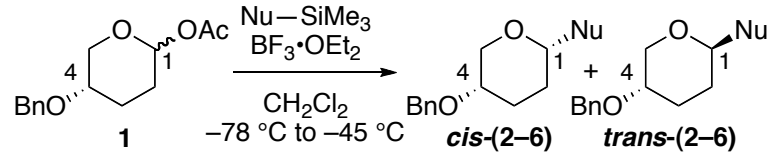


7 **8** **9** **10** **11**

entry	Nu–$SiMe_3$	N[a]	product	*cis*:*trans*[b,c]	yield (%)[d]
1	**7**	1.8	**2**	6:94	96
2	**8**	6.2	**3**	10:90	95
3	**9**	8.2	**4**	71:29	83
4	**10**	9.0	**5**	85:15	93
5	**11**	10.2	**6**	89:11	96

[a] N = nucleophilicity parameter; see ref 9. [b] Determined by GC and ^1H NMR spectra the unpurified reaction mixtures. [c] Diastereoselectivities were independent of starting anomer ratio. [d] Isolated yield.

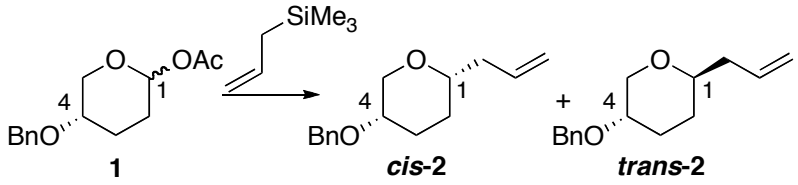
Table 2. Nucleophile screen with $BF_3{\bullet}OEt_2$.



entry	Nu–$SiMe_3$	N[a]	product	*cis*:*trans*[b,c]	yield (%)[d]
1	**7**	1.8	**2**	8:92	82
2	**8**	6.2	**3**	8:92	87

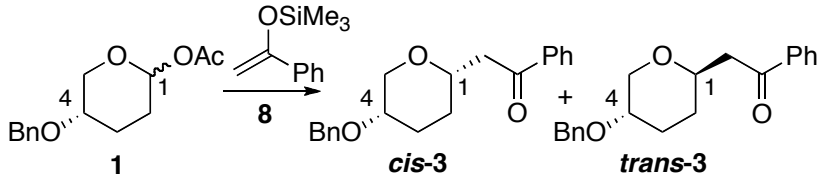
3	**9**	8.2	**4**	50:50	88
4	**10**	9.0	**5**	58:42	80
5	**11**	10.2	**6**	60:40	86

[a] *N* = nucleophilicity parameter; see ref 9. [b] Determined by GC and ^{1}H NMR spectra of the unpurified reaction mixtures. [c] Diastereoselectivities were independent of starting anomer ratio. [d] Isolated yield.



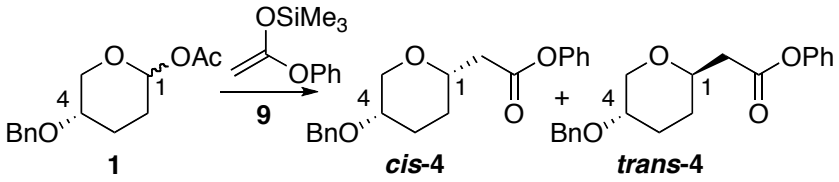
Allyl tetrahydropyran 2: (Table 1, Entry 1 and Table 2, Entry 1).[1] The standard procedure for nucleophilic substitution was followed with acetal **1** (1.0 equiv), allyltrimethylsilane (4.0 equiv), and Lewis acid (BF_3•OEt_2 or Me_3SiOTf; 1.6 equiv in each case). Purification by flash chromatography (97.5:2.5 hexanes/EtOAc) afforded the product as a colorless, clear oil. Product yields and diastereoselectivities are reported in Table 1 and Table 2. The major isomer *trans*-**2** was isolated as a pure sample. Characterization data (^{1}H NMR, ^{13}C NMR) matches reported values.[1]

trans-**2.** ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35–7.24 (m, 5H), 5.80 (ddt, *J* = 17.2, 10.2, 7.0, 1H), 5.10–5.01 (m, 2H), 4.58 (d, *J* = 11.9, 1H), 4.52 (d, *J* = 11.9, 1H), 4.09 (ddd, *J* = 10.8, 4.7, 2.3, 1H), 3.43 (tt, *J* = 10.3, 4.6, 1H), 3.33–3.25 (m, 1H), 3.18 (t, *J* = 10.5, 1H), 2.30–2.22 (m, 1H), 2.22–2.12 (m, 2H), 1.78–1.70 (m, 1H), 1.49–1.38 (m, 1H), 1.36–1.25 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 138.7, 135.0, 128.6, 127.82, 127.78, 117.0, 77.1, 73.2, 70.94, 70.92, 40.4, 30.3, 30.2; HRMS (TOF MS ES+) *m* / *z* calcd for $C_{15}H_{20}O_2Na$ $(M + Na)^+$ 255.1361, found 255.1369.



Ketone 3 (Table 1, Entry 2 and Table 2, Entry 2).[10-12] The standard procedure for nucleophilic substitution was followed with acetal **1** (1.0 equiv), enoxy silane **8** (4.0–4.1 equiv), and BF_3•OEt_2 (1.6 equiv) or Me_3SiOTf (1.4 equiv). Purification by flash chromatography (90:10 hexanes/EtOAc) afforded the product as an oily white solid. Product yields and diastereoselectivities are reported in Table 1 and Table 2. The major isomer *trans*-**3** was isolated as a pure sample.

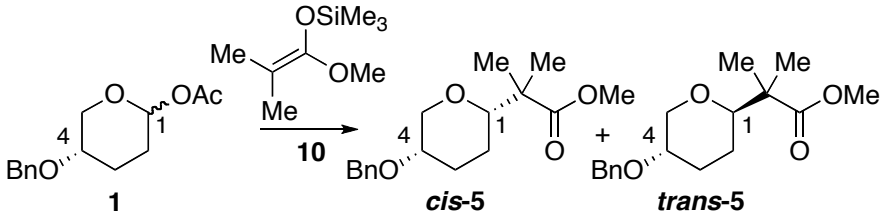
trans-**3.** mp 59–60.5 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.95 (d, *J* = 7.9, 2H), 7.57 (t, *J* = 7.3, 1H), 7.46 (t, *J* = 7.7, 2H), 7.37–7.27 (m, 5H), 4.59 (d, *J* = 11.9, 1H), 4.53 (d, *J* = 11.9, 1H), 4.06 (ddd, *J* = 10.8, 4.6, 2.2, 1H), 3.98–3.88 (m, 1H), 3.48 (tt, *J* = 10.0, 4.5, 1H), 3.29 (dd, *J* = 16.3, 6.7, 1H), 3.24 (t, *J* = 10.6, 1H), 2.93 (dd, *J* = 16.3, 5.8, 1H), 2.27–2.18 (m, 1H), 1.97–1.87 (m, 1H), 1.60–1.48 (m, 1H), 1.46–1.35 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 198.2, 138.7, 137.3, 133.4, 128.8, 128.6, 128.4, 127.9, 127.8, 74.2, 73.0, 71.0, 70.9, 44.7, 30.8, 30.2; IR (thin film) 3061, 2860, 2352, 1681, 1596 cm^{-1}; HRMS (TOF MS ES+) *m* / *z* calcd for $C_{20}H_{22}O_3Na$ $(M + Na)^+$ 333.1467, found 333.1460. Anal. Calcd for $C_{20}H_{22}O_3$: C, 77.39; H, 7.14. Found: C, 77.14; H, 7.07.



Ester 4 (Table 1, Entry 3 and Table 2, Entry 3). The standard procedure for nucleophilic substitution was followed with acetal **1**, silyl ketene acetal **9** (2.0 equiv for the BF_3•OEt_2-mediated reaction, 4.0 equiv for the Me_3SiOTf-mediated reaction), and Lewis acid (BF_3•OEt_2 or Me_3SiOTf; 1.6 equiv in each case). Purification by flash chromatography (80:20 hexanes/EtOAc) afforded the product as a colorless, clear oil. Product yields and diastereoselectivities are reported in Table 1 and Table 2. Isomers *cis*-**4** and *trans*-**4** were isolated as pure samples for analytical purposes.

cis-**4.** ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40–7.17 (m, 8H), 7.07 (d, *J* = 7.9, 2H), 4.62–4.54 (m, 2H), 4.11 (d, *J* = 12.5, 1H), 3.92 (dddd, *J* = 10.4, 7.5, 5.3, 2.0, 1H), 3.54 (dd, *J* = 12.5, 1.2, 1H), 3.42 (br s, 1H), 2.86 (dd, *J* = 15.5, 7.9, 1H), 2.67 (dd, *J* = 15.5, 5.2, 1H), 2.12–2.06 (m, 1H), 1.86 (m, 1H), 1.71–1.63 (m, 1H), 1.62–1.55 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.0, 150.8, 138.8, 129.5, 128.5, 127.8, 127.7, 125.9, 121.8, 74.1, 70.4, 70.2, 69.7, 41.4, 27.0, 26.4; IR (thin film) 3066, 3031, 2944, 2858, 1756, 1594 cm $^{-1}$.

trans-**4.** ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40–7.19 (m, 8H), 7.08 (d, *J* = 7.7, 2H), 4.60 (d, *J* = 11.9, 1H), 4.54 (d, *J* = 11.9, 1H), 4.12 (ddd, *J* = 10.8, 4.7, 2.2, 1H), 3.84 (dddd, *J* = 10.6, 7.6, 5.2, 2.1, 1H), 3.49 (tt, *J* = 10.1, 4.5, 1H), 3.26 (t, *J* = 10.5, 1H), 2.74 (dd, *J* = 15.2, 7.9, 1H), 2.65 (dd, *J* = 15.2, 5.2, 1H), 2.27–2.19 (m, 1H), 1.92–1.84 (m, 1H), 1.57–1.40 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 169.9, 150.8, 138.6, 129.6, 128.7, 127.9, 127.8, 126.1, 121.8, 74.2, 72.8, 71.0, 70.9, 41.1, 30.5, 30.1; IR (thin film) 3064, 3033, 2939, 2863, 1756, 1594 cm^{-1}; HRMS (TOF MS ES+) *m* / *z* calcd for $C_{20}H_{22}O_4Na$ $(M + Na)^+$ 349.1416, found 349.1420. Anal. Calcd for $C_{20}H_{22}O_4$: C, 73.60; H, 6.79. Found: C, 73.22; H, 6.80.

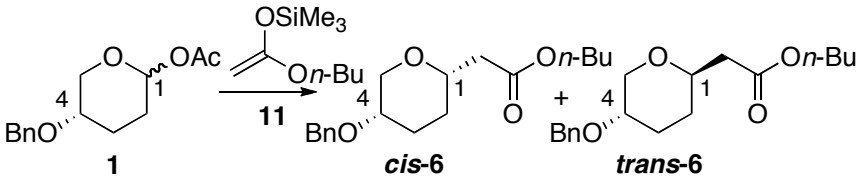


Ester 5 (Table 1, Entry 4 and Table 2, Entry 4). The standard procedure for nucleophilic substitution was followed with acetal **1**, silyl ketene acetal **10** (5.0 equiv for the BF_3•OEt_2-mediated reaction, 4.3 equiv for the Me_3SiOTf-mediated reaction), and Lewis acid (BF_3•OEt_2 or Me_3SiOTf; 1.6 equiv in each case). Purification by flash chromatography (90:10 hexanes/EtOAc) afforded the product as a colorless, clear oil. Product yields and diastereoselectivities are reported in Table 1 and Table 2. Isomers *cis*-**5** and *trans*-**5** were isolated as pure samples for analytical purposes.

cis-**5.** ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38–7.24 (m, 5H), 4.59 (d, *J* = 12.3, 1H), 4.51 (d, *J* = 12.3, 1H), 4.12 (d, *J* = 12.5, 1H), 3.68 (s, 3H), 3.55 (dd, *J* = 11.4, 1.7, 1H), 3.45 (dd, *J* = 12.5, 1.3, 1H), 3.38 (br s, 1H), 2.10 (dt, *J* = 13.9, 3.1, 1H), 1.81 (qd, *J* = 13.1, 3.7, 1H), 1.67–1.57 (m, 1H), 1.31 (br d, *J* = 12.8, 1H), 1.23 (s, 3H), 1.14 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 177.7, 139.1, 128.5, 127.64, 127.58, 82.4, 70.8, 69.9, 69.8, 52.0, 46.9, 27.8, 21.3, 20.64, 20.56; IR (neat) 2950, 2858, 1730, 1454, 1143, 1115 cm^{-1}; HRMS (TOF MS ES+) *m* / *z* calcd for $C_{17}H_{24}O_4Na$ $(M + Na)^+$ 315.1572, found 315.1569. Anal. Calcd for $C_{17}H_{24}O_4$: C, 69.84; H, 8.27. Found: C, 70.06; H, 8.32.

trans-**5.** ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38–7.24 (m, 5H), 4.57 (d, *J* = 12.0, 1H), 4.52 (d, *J* = 12.0, 1H), 4.09 (ddd, *J* = 10.7, 4.7, 2.3, 1H), 3.67 (s, 3H), 3.46–3.37 (m, 2H), 3.16 (t, *J* = 10.5, 1H), 2.26–2.18 (m, 1H), 1.66–1.60 (m, 1H), 1.50–1.34 (m, 2H), 1.16 (s, 3H), 1.11 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 177.4, 138.8, 128.6, 127.9, 127.8, 82.3, 73.2, 71.3, 71.0, 52.1, 46.4, 30.3, 24.7, 21.7, 20.7; IR (neat) 2948, 2864, 1734, 1454, 1275,

1093 cm^{-1}; HRMS (TOF MS ES+) *m* / *z* calcd for $C_{17}H_{24}O_4Na$ $(M + Na)^+$ 315.1572, found 315.1577. Anal. Calcd for $C_{17}H_{24}O_4$: C, 69.84; H, 8.27. Found: C, 70.00; H, 8.31.

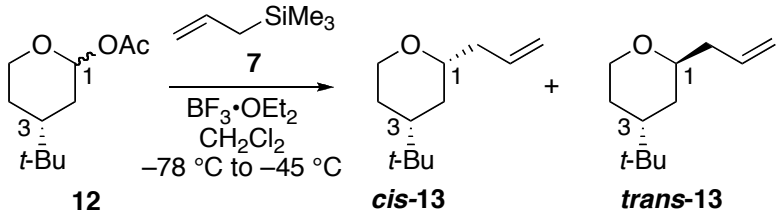


Ester 6 (Table 1, Entry 5 and Table 2, Entry 5). The standard procedure for nucleophilic substitution was followed with acetal **1**, silyl ketene acetal **11** (2.0 equiv for the BF_3•OEt_2-mediated reaction, 3.8 equiv for the Me_3SiOTf-mediated reaction), and BF_3•OEt_2 (1.5 equiv) or Me_3SiOTf (1.6 equiv). Purification by flash chromatography (95:5 hexanes/EtOAc) afforded the product as a colorless, clear oil. Product yields and diastereoselectivities are reported in Table 1. Isomers *cis*-**6** and *trans*-**6** were isolated as pure samples for analytical purposes.

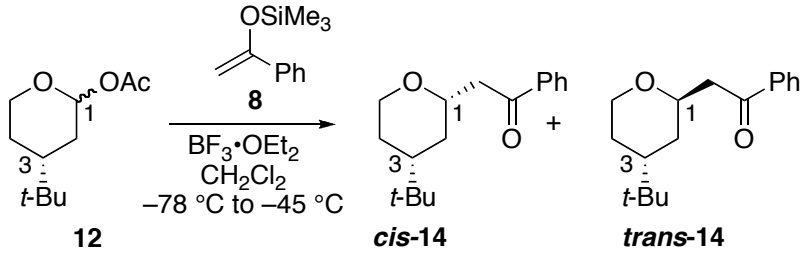
cis-**6.** ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38–7.24 (m, 5H), 4.60–4.53 (m, 2H), 4.12–4.03 (m, 3H), 3.85–3.77 (m, 1H), 3.51 (dd, *J* = 12.5, 1.1, 1H), 3.40 (br s, 1H), 2.62 (dd, *J* = 15.5, 7.7, 1H), 2.41 (dd, *J* = 15.5, 5.4, 1H), 2.10–2.01 (m, 1H), 1.77 (qd, *J* = 13.2, 3.6, 1H), 1.69–1.57 (m, 3H), 1.51 (d, *J* = 12.0, 1H), 1.36 (sextet, *J* = 7.6, 2H), 0.92 (t, *J* = 7.5, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.6, 138.8, 128.5, 127.7, 127.6, 74.2, 70.4, 70.1, 69.6, 64.5, 41.4, 30.8, 27.0, 26.4, 19.2, 13.9; IR (thin film) 2958, 2871, 1733, 1455, 1115 cm^{-1}; HRMS (TOF MS ES+) *m* / *z* calcd for $C_{18}H_{26}O_4Na$ $(M + Na)^+$ 329.1729, found 329.1718. Anal. Calcd for $C_{18}H_{26}O_4$: C, 70.56; H, 8.55. Found: C, 70.39; H, 8.48.

trans-**6.** ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35–7.25 (m, 5H), 4.58 (d, *J* = 11.9, 1H), 4.52 (d, *J* = 11.8, 1H), 4.14–4.04 (m, 3H), 3.75–3.68 (m, 1H), 3.45 (tt, *J* = 10.0, 4.6, 1H), 3.21

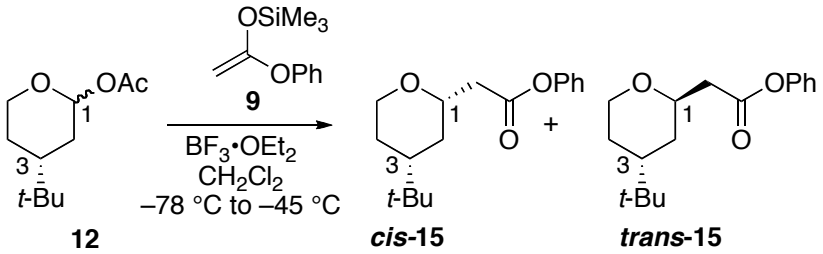
(t, J = 10.5, 1H), 2.49 (dd, J = 15.1, 7.8, 1H), 2.39 (dd, J = 15.1, 5.2, 1H), 2.19 (br d, J = 12.4, 1H), 1.79 (br d, J = 12.9, 1H), 1.61 (quintet, J = 6.8, 2H), 1.54–1.44 (m, 1H), 1.42–1.32 (m, 3H), 0.92 (t, J = 7.5, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.4, 138.6, 128.6, 127.9, 127.8, 74.2, 72.8, 71.0, 70.9, 64.6, 41.2, 30.8, 30.4, 30.1, 19.3, 13.9; IR (thin film) 2960, 2867, 1733, 1455, 1090 cm^{-1}; HRMS (TOF MS ES+) m / z calcd for $C_{18}H_{26}O_4Na$ $(M + Na)^+$ 329.1729, found 329.1720. Anal. Calcd for $C_{18}H_{26}O_4$: C, 70.56; H, 8.55. Found: C, 70.49; H, 8.70.



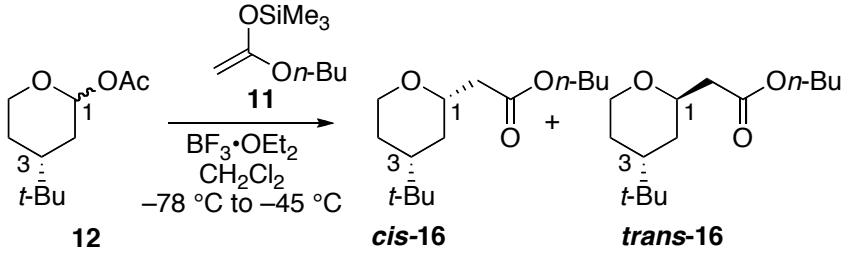
Allyl tetrahydropyran 13: The standard procedure for nucleophilic substitution was followed with acetal **12** (0.041 g, 0.21 mmol), allyltrimethylsilane (0.130 mL, 0.82 mmol), and BF_3•OEt_2 (0.040 mL, 0.32 mmol). Analysis of the crude mixture by GC revealed a 99:1 *trans*-**13** to *cis*-**13** ratio. Purification by flash chromatography (98:2 pentane/Et_2O) afforded *trans*-**13** as a colorless, clear oil (0.024 g, 67%): ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.80 (ddt, J = 17.1, 10.1, 7.0, 1H), 5.12–5.04 (m, 2H), 4.04–3.98 (m, 1H), 3.73 (ddd, J = 11.7, 4.9, 2.0, 1H), 3.63 (td, J = 11.9, 2.7, 1H), 2.60–2.52 (m, 1H), 2.29 (dt, J = 14.3, 7.2, 1H), 1.58–1.46 (m, 3H), 1.45–1.33 (m, 2H), 0.84 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 135.8, 116.7, 72.9, 61.4, 39.5, 35.2, 32.3, 29.2, 27.6, 27.2; IR (thin film) 3078, 2944, 2860, 1642, 1364, 1125 cm^{-1}; HRMS (TOF MS APCI) m / z calcd for $C_{12}H_{23}O$ $(M + H)^+$ 183.1749, found 183.1741. Anal. Calcd for $C_{12}H_{22}O$: C, 79.06; H, 12.16. Found: C, 79.02; H, 12.09.



Ketone 14: The standard procedure for nucleophilic substitution was followed with acetal **12** (0.040 g, 0.20 mmol), enoxy silane **8** (0.160 g, 0.83 mmol), and BF_3•OEt_2 (0.040 mL, 0.32 mmol). Analysis of the crude mixture by GC revealed a 98:2 *trans*-**14** to *cis*-**14** ratio. Purification by flash chromatography (95:5 hexane/EtOAc) afforded *trans*-**14** as a white solid (0.043 g, 83%): mp 74.5–75.0 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.99–7.96 (m, 2H), 7.59–7.54 (m, 1H), 7.49–7.45 (m, 2H), 4.70–4.64 (m, 1H), 3.79 (ddd, J = 11.7, 4.9, 2.1, 1H), 3.68 (td, J = 11.9, 2.7, 1H), 3.44 (dd, J = 15.3, 6.5, 1H), 3.22 (dd, J = 15.4, 7.4, 1H), 1.67–1.56 (m, 3H), 1.49–1.33 (m, 2H), 0.84 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 198.8, 137.2, 133.3, 128.8, 128.4, 70.1, 62.2, 40.2, 39.8, 32.3, 30.0, 27.4, 27.2; IR (thin film) 2956, 2865, 1683 cm^{-1}; HRMS (TOF MS ES+) m / z calcd for $C_{17}H_{24}O_2Na$ $(M + Na)^+$ 283.1674, found 283.1680. Anal. Calcd for $C_{17}H_{24}O_2$: C, 78.42; H, 9.29. Found: C, 78.24; H, 9.31.



Ester 15: The standard procedure for nucleophilic substitution was followed with acetal **12** (0.042 g, 0.21 mmol), silyl ketene acetal **9** (0.160 g, 0.77 mmol), and BF_3•OEt_2 (0.040 mL, 0.32 mmol). Analysis of the crude mixture by GC revealed a 83:17 *trans*-**15** to *cis*-**15** ratio. Purification by flash chromatography (95:5 hexane/EtOAc) afforded a mixture of *trans*-**15** and *cis*-**15** as a colorless, clear oil (0.051 g, 93%). The *trans*-**15** isomer was isolated as a pure sample for analytical purposes: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 (t, *J* = 7.6, 2H), 7.22 (t, *J* = 7.5, 1H), 7.09 (d, *J* = 7.6, 2H), 4.67–4.60 (m, 1H), 3.86–3.80 (m, 1H), 3.73 (td, *J* = 11.9, 2.4, 1H), 3.11 (dd, *J* = 14.3, 8.8, 1H), 2.73 (dd, *J* = 14.3, 6.4, 1H), 1.70–1.58 (m, 3H), 1.49–1.34 (m, 2H), 0.87 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.3, 150.9, 129.6, 126.0, 121.8, 70.5, 62.0, 39.8, 36.7, 32.3, 29.9, 27.4, 27.2; IR (thin film) 2954, 2867, 1756, 1594, 1493, 1196 cm^{-1}; HRMS (TOF MS ES+) *m* / *z* calcd for $C_{17}H_{24}O_3Na$ $(M + Na)^+$ 299.1623, found 299.1621. Anal. Calcd for $C_{17}H_{24}O_3$: C, 73.88; H, 8.75. Found: C, 74.14; H, 8.87.

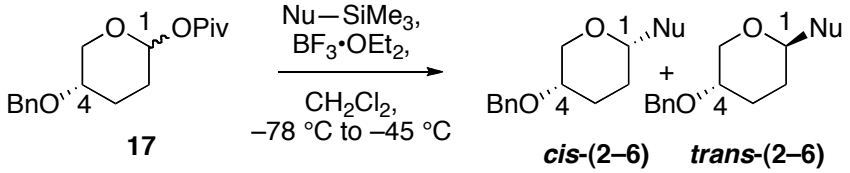


Ester 16: The standard procedure for nucleophilic substitution was followed with acetal **12** (0.040 g, 0.20 mmol), silyl ketene acetal **11** (0.154 g, 0.818 mmol), and BF_3•OEt_2 (0.040 mL, 0.32 mmol). Analysis of the crude mixture by GC revealed a 66:34 *trans*-**16** to *cis*-**16** ratio. Purification by flash chromatography (97.5:2.5 hexane/EtOAc)

afforded a mixture of *trans*-**16** and *cis*-**16** as a colorless, clear oil (0.035 g, 69%). Isomers *cis*-**16** and *trans*-**16** were isolated as pure samples for analytical purposes.

cis-**16.** ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.10 (t, *J* = 6.7, 2H), 4.05–4.01 (m, 1H), 3.71 (dddd, *J* = 10.3, 7.4, 5.2, 1.9, 1H), 3.41 (td, *J* = 11.5, 2.0, 1H), 2.53 (dd, *J* = 15.0, 7.9, 1H), 2.40 (dd, *J* = 15.0, 5.2, 1H), 1.67–1.58 (m, 3H), 1.53 (dd, *J* = 9.3, 2.0, 1H), 1.43–1.35 (m, 2H), 1.33–1.24 (m, 2H), 1.08–0.99 (m, 1H), 0.93 (t, *J* = 7.4, 3H), 0.85 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.7, 74.8, 68.7, 64.5, 45.8, 42.2, 33.0, 32.3, 30.8, 27.3, 27.1, 19.3, 13.9; IR (thin film) 2958, 2869, 1737, 1468, 1364 cm^{-1}; HRMS (TOF MS ES+) *m* / *z* calcd for $C_{15}H_{29}O_3$ $(M + H)^+$ 257.2117, found 257.2116. Anal. Calcd for $C_{15}H_{28}O_3$: C, 70.27; H, 11.01. Found: C, 70.44; H, 11.25.

trans-**16.** ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.52–4.45 (m, 1H), 4.14–4.04 (m, 2H), 3.78–3.73 (m, 1H), 3.63 (td, *J* = 11.7, 2.4, 1H), 2.85 (dd, *J* = 14.4, 8.5, 1H), 2.49 (dd, *J* = 14.4, 6.5, 1H), 1.64–1.50 (m, 5H), 1.42–1.31 (m, 4H), 0.93 (t, *J* = 7.4, 3H), 0.84 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.9, 70.4, 64.6, 61.8, 39.7, 36.6, 32.2, 30.9, 29.9, 27.4, 27.1, 19.3, 13.9; IR (thin film) 2958, 2869, 1737, 1468, 1366 cm^{-1}; HRMS (TOF MS ES+) *m* / *z* calcd for $C_{15}H_{29}O_3$ $(M + H)^+$ 257.2117, found 257.2125. Anal. Calcd for $C_{15}H_{28}O_3$: C, 70.27; H, 11.01. Found: C, 70.08; H, 11.21.



General Procedure for Nucleophilic Substitution Reactions of Pivaloate Acetal 17: To a cooled (–78 °C) 0.1 M solution of **17** (0.40–0.69 mmol, 1.0 equiv) and

nucleophile (3.5 – 4.0 equiv) in CH_2Cl_2 was added BF_3•OEt_2 (1.6–3.5 equiv) dropwise. After stirring at –78 °C for 1–5 min, the reaction mixture was maintained at –45 °C for 1–2 h. Upon warming to 22 °C, a saturated aqueous solution of $NaHCO_3$ (4.0 mL) was added. The resulting biphasic mixture was diluted with CH_2Cl_2 (5 mL) and H_2O (2 mL), and the layers were separated. The aqueous layer was extracted with CH_2Cl_2 (3 x 8 mL) and the combined organic layers were dried over $MgSO_4$, filtered, and concentrated *in vacuo*. Diastereomeric ratios (Table S1) were determined by GC and confirmed by ^1H NMR spectroscopy. Reported yields (Table S1) refer to purified material.

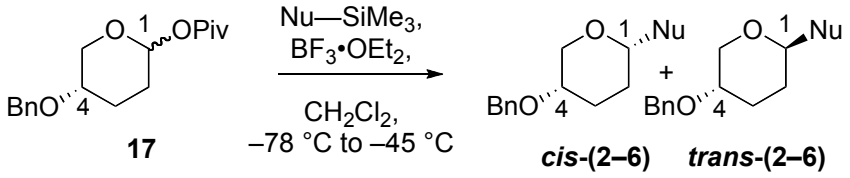
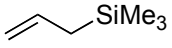
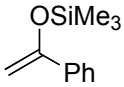
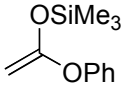
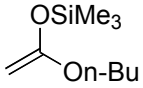


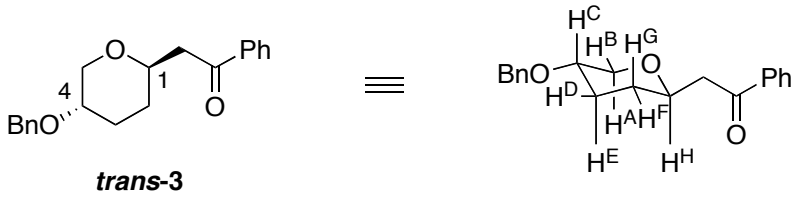
Table S1. *Nucleophilic Substitution Reactions with Acetal* ***17****.*

entry	Nu—$SiMe_3$	product	*cis* : *trans*[a]	yield (%)[b]
1		**2**	12 : 88	78
2		**3**	10: 90	67
3		**4**	49 : 51	47
4		**6**	52 : 48	18

[a]Measured by GC analysis of unpurified reaction mixture.
[b]Based on purified products.

IV. Stereochemical Proofs of Nucleophilic Substitution Products

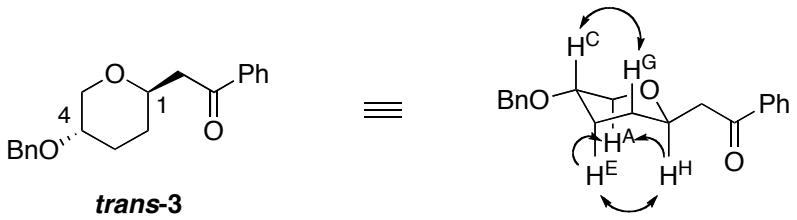
Figure S1. Diagnostic $^{1}H–^{1}H$ coupling constants for *trans*-**3**.



***trans*-3**

H^A: t, J = 10.6 Hz (gem ax^A-eq^B, ax^A-ax^C)
H^C: tt, J = 10.0 Hz (ax^C-ax^A, ax^C-ax^E),
4.5 Hz (ax^C-eq^B, ax^C-eq^D)

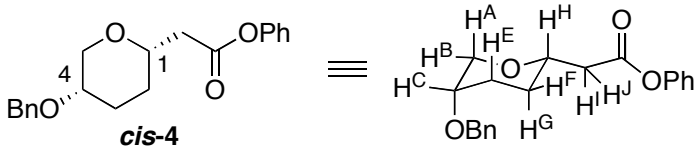
Figure S2. Relevant DPFGSE-NOE data (mixing time 2.0 s) for *trans*-**3**.



***trans*-3**

H^C irradiated: H^G (3.7%)
H^E irradiated: H^A (3.8%), H^H (8.3%)
H^G irradiated: H^C (8.2%)
H^H irradiated: H^A (9.3%), H^E (4.1%)

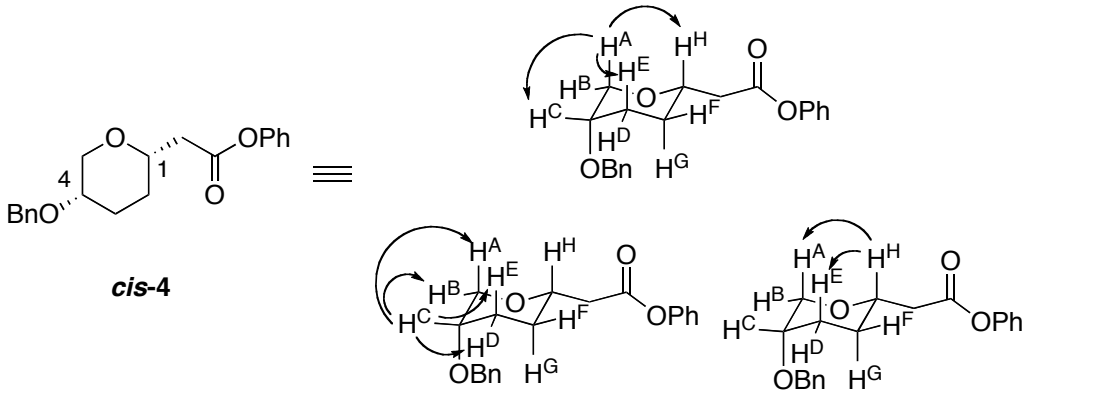
Figure S3. Diagnostic $^{1}H–^{1}H$ coupling constants for *cis*-**4**.



***cis*-4**

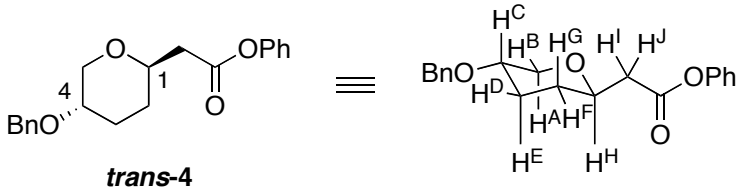
H^A: dd, J = 12.5 Hz (gem ax^A-eq^B), 1.2 Hz (ax^A-eq^C)
H^C: br s
H^H: dddd, J = 10.4 Hz (ax^H-ax^G), 7.5 Hz (ax^H-H^I),
5.3 Hz (ax^H-H^J), 2.0 Hz (ax^H-eq^F)

Figure S4. Relevant DPFGSE-NOE data (mixing time 2.0 s) for *cis*-**4**.



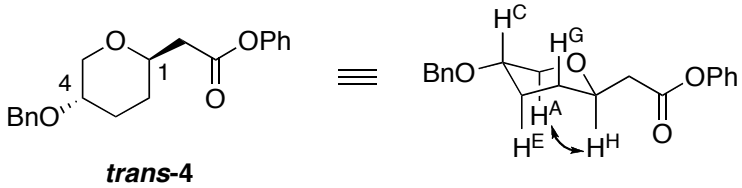
H^A irradiated: H^C (13.0%), H^E (2.4%), H^H (13.9%)
H^C irradiated: H^A (7.0%), H^B (3.2%), H^D (2.1%), H^E (4.9%)
H^H irradiated: H^A (6.5%), H^E (3.9%)

Figure S5. Diagnostic ^1H–^1H coupling constants for *trans*-**4**.



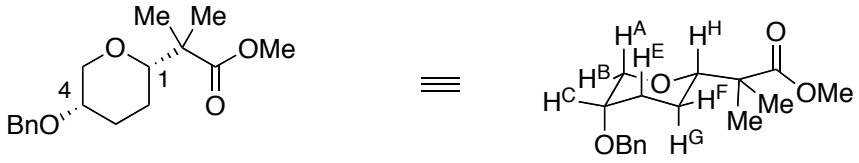
H^A: t, J = 10.5 Hz (gem ax^A-eq^B, ax^A-ax^C)
H^H: dddd, J = 10.6 Hz (ax^H-ax^G), 7.6 Hz (ax^H-H^I),
5.2 Hz (ax^H-H^J), 2.1 (ax^H-eq^F)
H^C: tt, J = 10.1 Hz (ax^C-ax^A, ax^C-ax^E),
4.5 Hz (ax^C-eq^B, ax^C-eq^D)

Figure S6. Relevant DPFGSE-NOE data (mixing time 2.0 s) for *trans*-**4**.



H^A irradiated: H^H (13.4%)
H^H irradiated: H^A (8.3%)

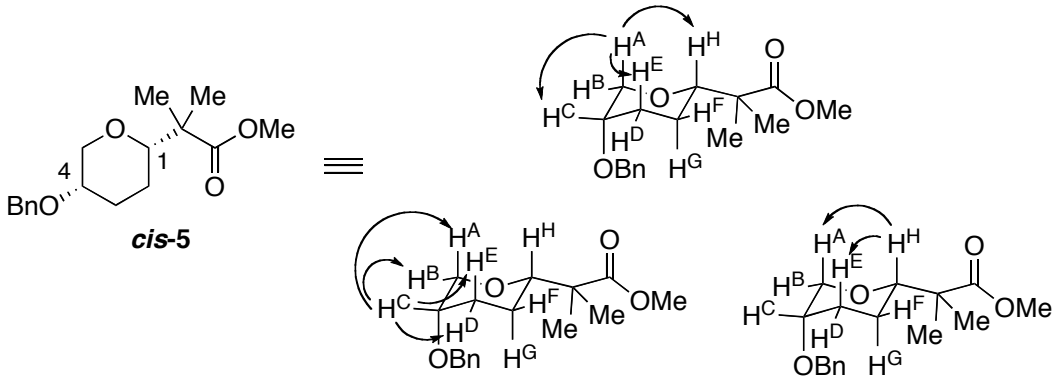
Figure S7. Diagnostic $^{1}H–^{1}H$ coupling constants for *cis*-**5**.



***cis*-5**

H^{A}: dd, J = 12.5 Hz (gem ax^{A}-eq^{B}), 1.3 Hz (ax^{A}-eq^{C})
H^{C}: br s
H^{H}: dd, J = 11.4 Hz (ax^{H}-ax^{G}), 1.7 Hz (ax^{H}-eq^{F})

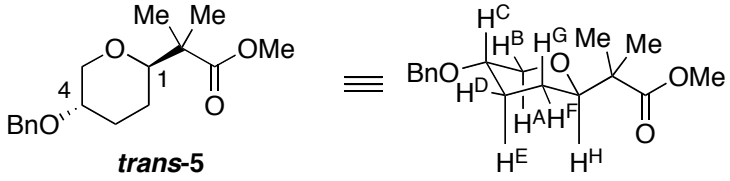
Figure S8. Relevant DPFGSE-NOE data (mixing time 2.0 s) for *cis*-**5**.



***cis*-5**

H^{A} irradiated: H^{C} (7.7%), H^{E} (2.9%), H^{H} (12.1%)
H^{C} irradiated: H^{A} (4.1%), H^{B} (3.2%), H^{D} (2.1%), H^{E} (4.7%)
H^{H} irradiated: H^{A} (6.3%), H^{E} (3.8%)

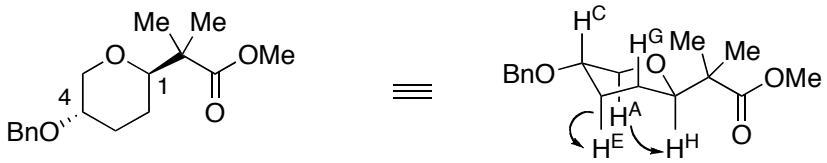
Figure S9. Diagnostic $^{1}H–^{1}H$ coupling constants for *trans*-**5**.



***trans*-5**

H^{A}: t, J = 10.5 Hz (gem ax^{A}-eq^{B}, ax^{A}-ax^{C})

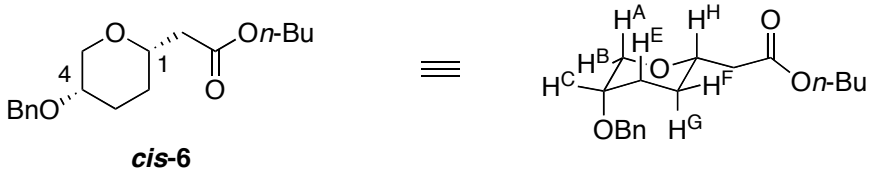
Figure S10. Relevant DPFGSE-NOE data (mixing time 2.0 s) for *trans*-**5**.



***trans*-5**

H^A irradiated: H^H (14.0%), H^E (3.6%)

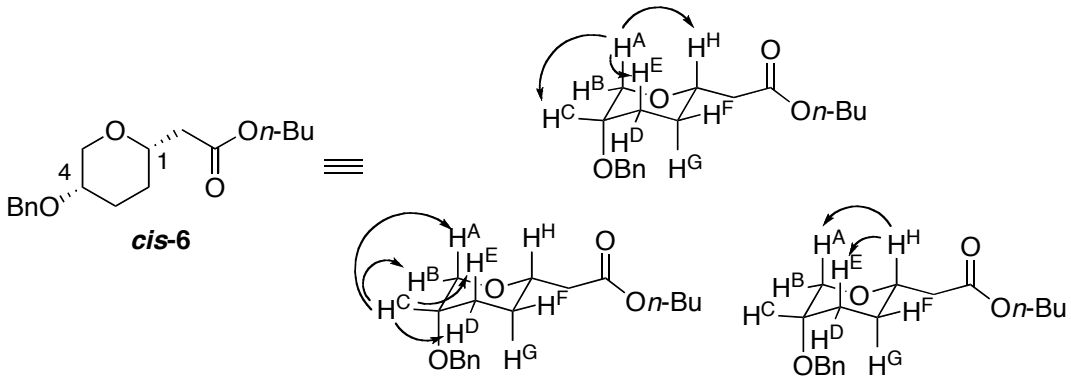
Figure S11. Diagnostic $^1H–^1H$ coupling constants for *cis*-**6**.



***cis*-6**

H^A: dd, J = 12.5 Hz (gem ax^A-eq^B), 1.1 Hz (ax^A-eq^C)
H^C: br s

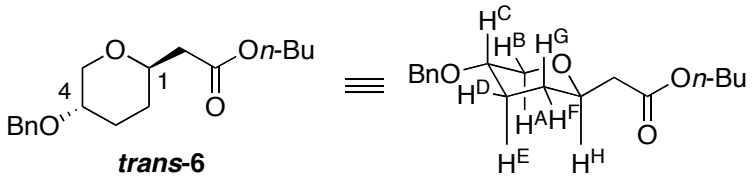
Figure S12. Relevant DPFGSE-NOE data (mixing time 2.0 s) for *cis*-**6**.



***cis*-6**

H^A irradiated: H^C (11.8%), H^E (2.4%), H^H (13.1%)
H^C irradiated: H^A (6.4%), H^B (3.1%), H^D (2.0%), H^E (4.6%)
H^H irradiated: H^A (6.5%), H^E (3.8%)

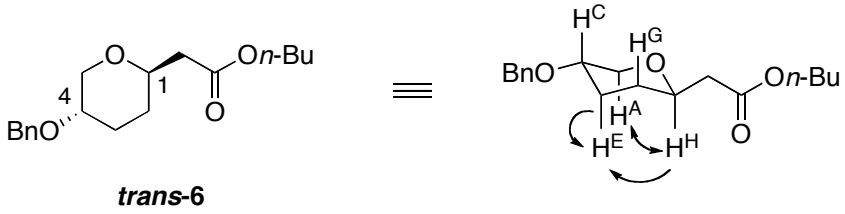
Figure S13. Diagnostic $^{1}H–^{1}H$ coupling constants for *trans*-**6**.



***trans*-6**

H^A: t, J = 10.5 Hz (gem ax^A-eq^B, ax^A-ax^C)
H^C: tt, J = 10.0 Hz (ax^C-ax^A, ax^C-ax^E),
4.6 Hz (ax^C-eq^B, ax^C-eq^D)

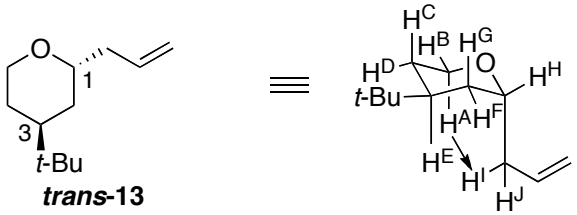
Figure S14. Relevant DPFGSE-NOE data (mixing time 2.0 s) for *trans*-**6**.



***trans*-6**

H^A irradiated: H^H (12.1%), H^E (2.8%)
H^H irradiated: H^A (8.0%), H^E (4.1%)

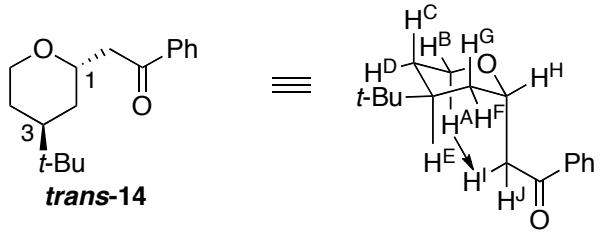
Figure S15. Relevant DPFGSE-NOE data (mixing time 2.0 s) for *trans*-**13** (analysis assumes an equatorial *tert*-butyl group).



***trans*-13**

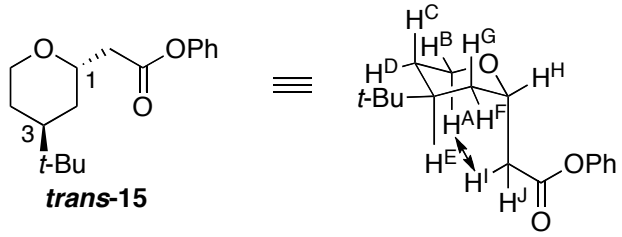
H^A irradiated: H^I (0.7%), H^J (0.6%)

Figure S16. Relevant DPFGSE-NOE data (mixing time 2.0 s) for *trans*-**14** (analysis assumes an equatorial *tert*-butyl group).



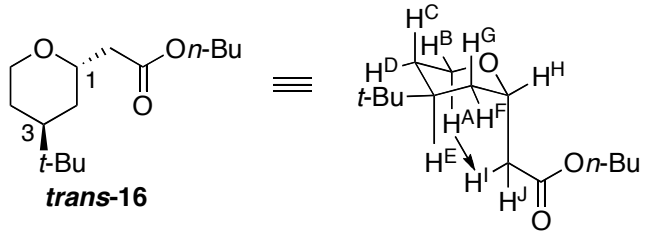
H^A irradiated: H^I (4.3%), H^J (4.3%)
H^I irradiated: H^A (3.6%), H^H (3.5%)

Figure S17. Relevant DPFGSE-NOE data (mixing time 2.0 s) for *trans*-**15** (analysis assumes an equatorial *tert*-butyl group).



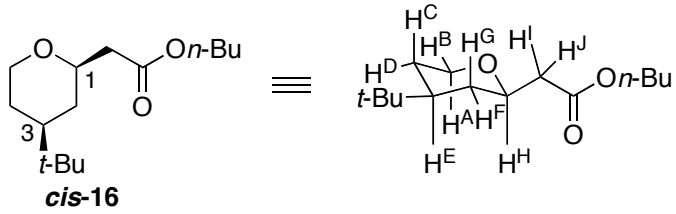
H^A irradiated: H^I (1.3%), H^J (0.9%)
H^I irradiated: H^A (1.0%)

Figure S18. Relevant DPFGSE-NOE data (mixing time 2.0 s) for *trans*-**16** (analysis assumes an equatorial *tert*-butyl group).



H^A irradiated: H^I (4.1%), H^J (2.7%)
H^I irradiated: H^A (3.2%)

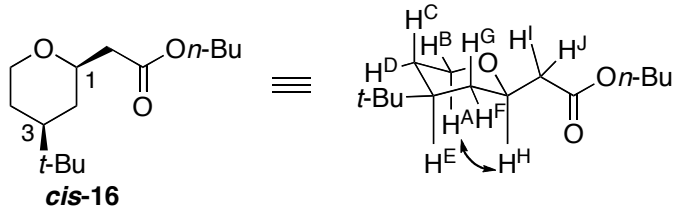
Figure S19. Diagnostic ^{1}H–^{1}H coupling constants for *cis*-**16** (analysis assumes an equatorial *tert*-butyl group).



***cis*-16**

H^H: dddd, J = 10.3 Hz (ax^H-ax^G), 7.4 Hz (ax^H-H^I), 5.2 Hz (ax^H-H^J), 1.9 Hz (ax^H-eq^F)
H^I: dd, J = 15.0 Hz (gem H^I-H^J), 7.9 Hz (ax^H-H^I)
H^J: dd, J = 15.0 Hz (gem H^I-H^J), 5.2 Hz (ax^H-H^I)

Figure S20. Relevant DPFGSE-NOE data (mixing time 2.0 s) for *cis*-**16** (analysis assumes an equatorial *tert*-butyl group).



***cis*-16**

H^A irradiated: H^H (7.8%)
H^H irradiated: H^A (5.1%)

V. Competition Experiments

Standard Competition Experiment Conditions. A solution of acetal (0.20 mmol, 1.0 equiv.) and nucleophiles (1.0 mmol each, 5.0 equiv. each) in anhydrous CH_2Cl_2 (2.0 mL) was prepared. An aliquot (0.050 mL) was removed, diluted with $CDCl_3$ and analyzed by ^{1}H NMR spectroscopy to confirm the molar ratio of the acetal and nucleophiles prior to addition of Lewis acid. Additional quantities of nucleophiles or

acetal were added to adjust the stoichiometry as needed. The reaction mixture was then cooled to –78 °C , and Lewis acid (0.32 mmol, 1.6 equiv) was added dropwise. After stirring at –78 °C for 5 min, the reaction mixture was warmed to –45 °C for 1–2 h before quenching. A saturated aqueous solution of $NaHCO_3$ (4.0 mL) was added, and the solution was warmed to 22 °C, diluted with CH_2Cl_2 (2 mL) and H_2O (2 mL), and the layers were separated. The aqueous layer was extracted with CH_2Cl_2 (3 x 2 mL) and the combined organic layers were dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Chemoselectivity values were determined by GC after addition of dodecane standard. Chemoselectivity data reported are based on response factor calibration curves generated from authentic samples of pure products with dodecane as internal standards. Diastereoselctivities are reported as uncorrected values.

VI. Bibliography

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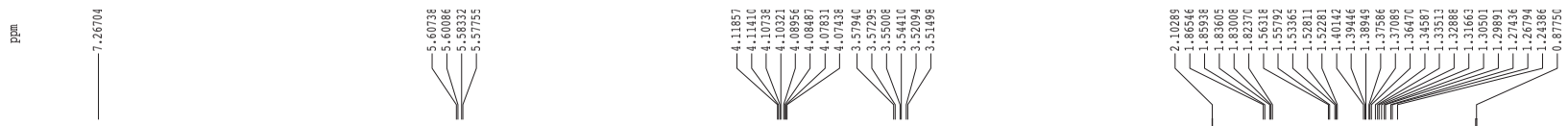
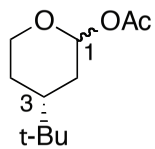
(10) Compound 3 has been synthesized previously, however the reported analytical data was in error. The correct analytical data is presented here.
(11) Ayala, L.; Lucero, C. G.; Romero, J. A. C.; Tabacco, S. A.; Woerpel, K. A. J. Am. Chem. Soc. 2003, 125, 15521-15528.
(12) Shenoy, S.; Woerpel, K. A. Org. Lett. 2005, 7, 1157-1160.





12

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PROCNO 1

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PULPROG zg30
TD 65536
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 1290.2
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 400.1300192 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 70.00 cm
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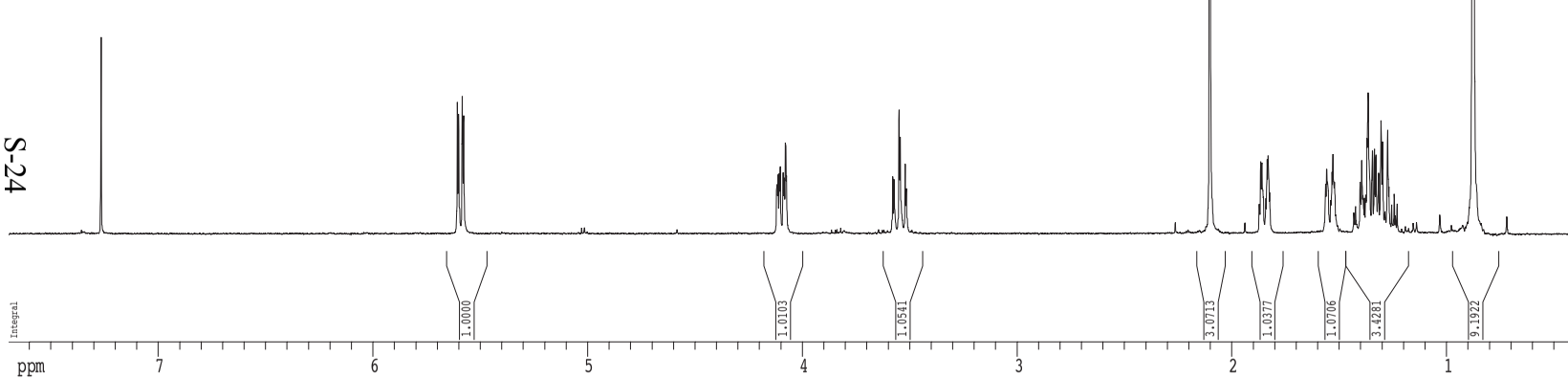
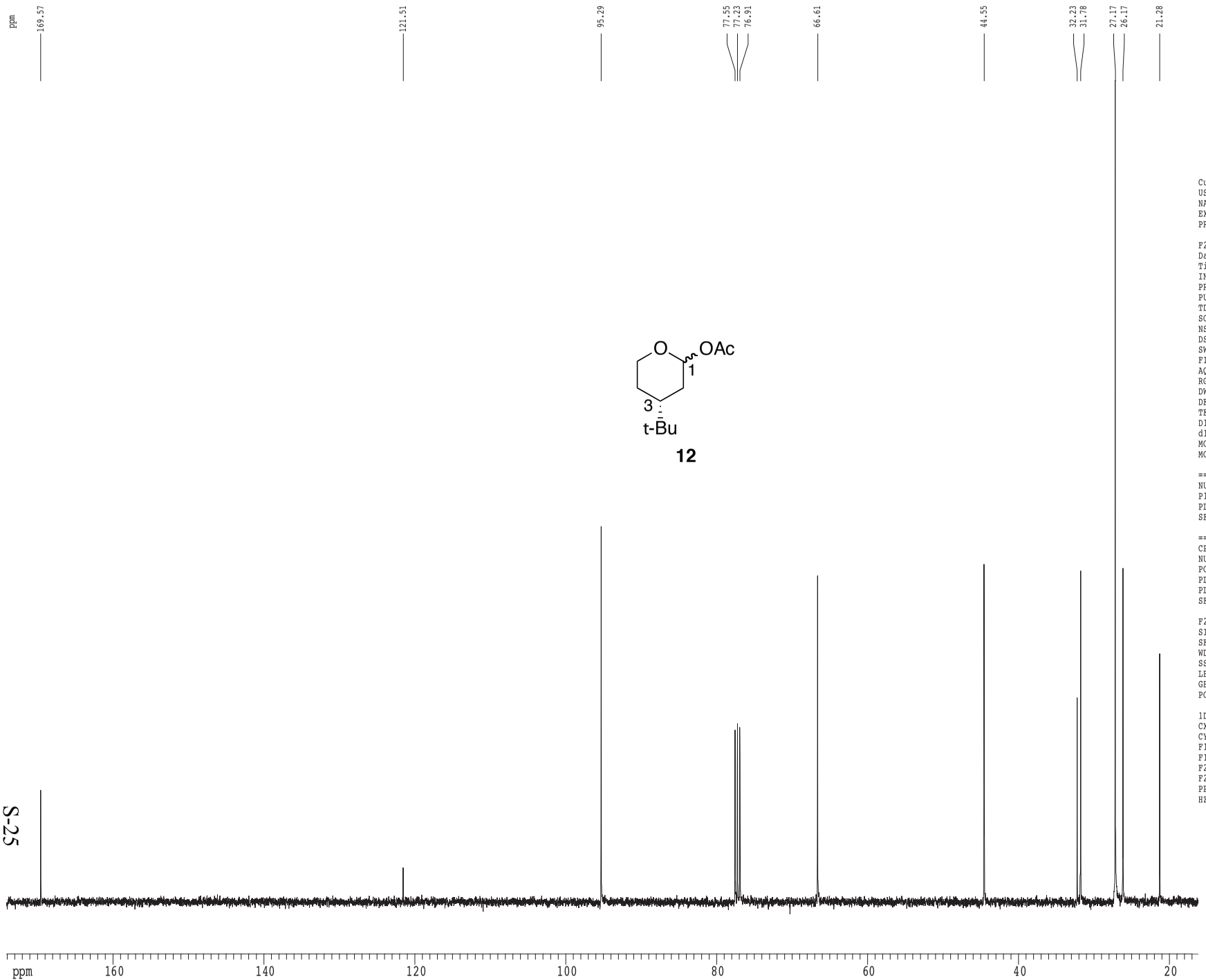
F1 3078.83 Hz
F2P 0.020 ppm
F2 8.13 Hz
PPMCM 0.33659 ppm/cm
HZCM 134.67969 Hz/cm



jrk-2-71 purified C3-tBu acetate
13C spectrum with 1H decoupling



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NAME jrk-2-71
EXPNO 14
PROCNO 1

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Time 10.26
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 122
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 9195.2
DW 20.700 usec
DE 20.39 usec
TE 298.0 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

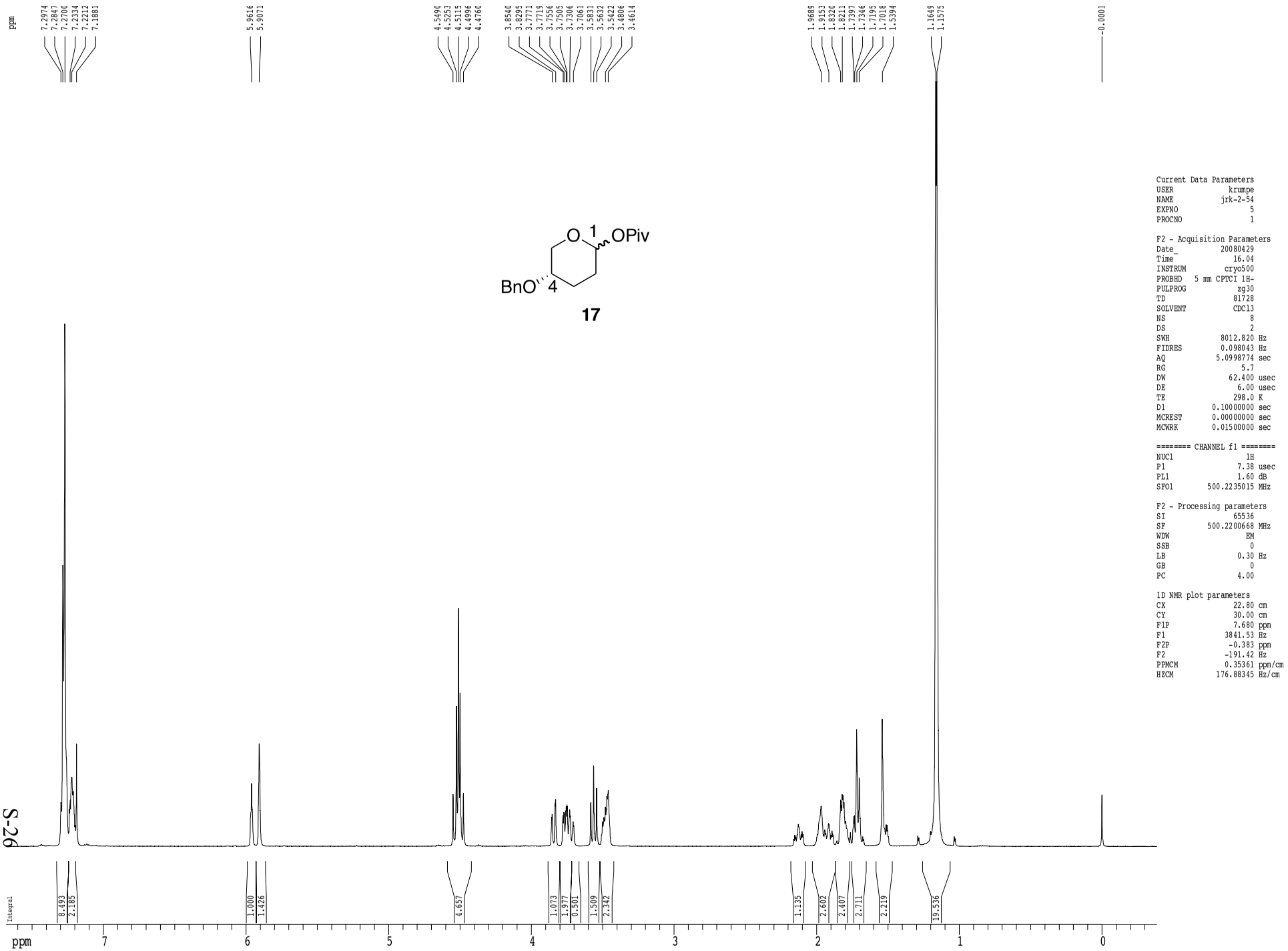
======= CHANNEL f1 =======
NUC1 13C
P1 10.30 usec
PL1 0.00 dB
SFO1 100.6237964 MHz

======= CHANNEL f2 =======
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SFO2 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 100.6127576 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.80 cm
CY 20.00 cm
F1P 174.107 ppm
F1 17517.35 Hz
F2P 16.177 ppm
F2 1627.57 Hz
PPMCM 6.92675 ppm/cm
HZCM 696.91992 Hz/cm

jrk-2-54 pivaloate



17

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NAME            jrk-2-54
EXPNO                  5
PROCNO                 1

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Time               16.04
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG             zg30
TD                 81728
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             8012.820 Hz
FIDRES          0.098043 Hz
AQ              5.0998774 sec
RG                   5.7
DW                62.400 usec
DE                 6.00 usec
TE                298.0 K
D1           0.10000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.38 usec
PL1                1.60 dB
SFO1         500.2235015 MHz

F2 - Processing parameters
SI                 65536
SF           500.2200668 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 4.00

1D NMR plot parameters
CX                22.80 cm
CY                30.00 cm
F1P               7.680 ppm
F1              3841.53 Hz
F2P              -0.383 ppm
F2              -191.42 Hz
PPMCM           0.35361 ppm/cm
HZCM          176.88345 Hz/cm
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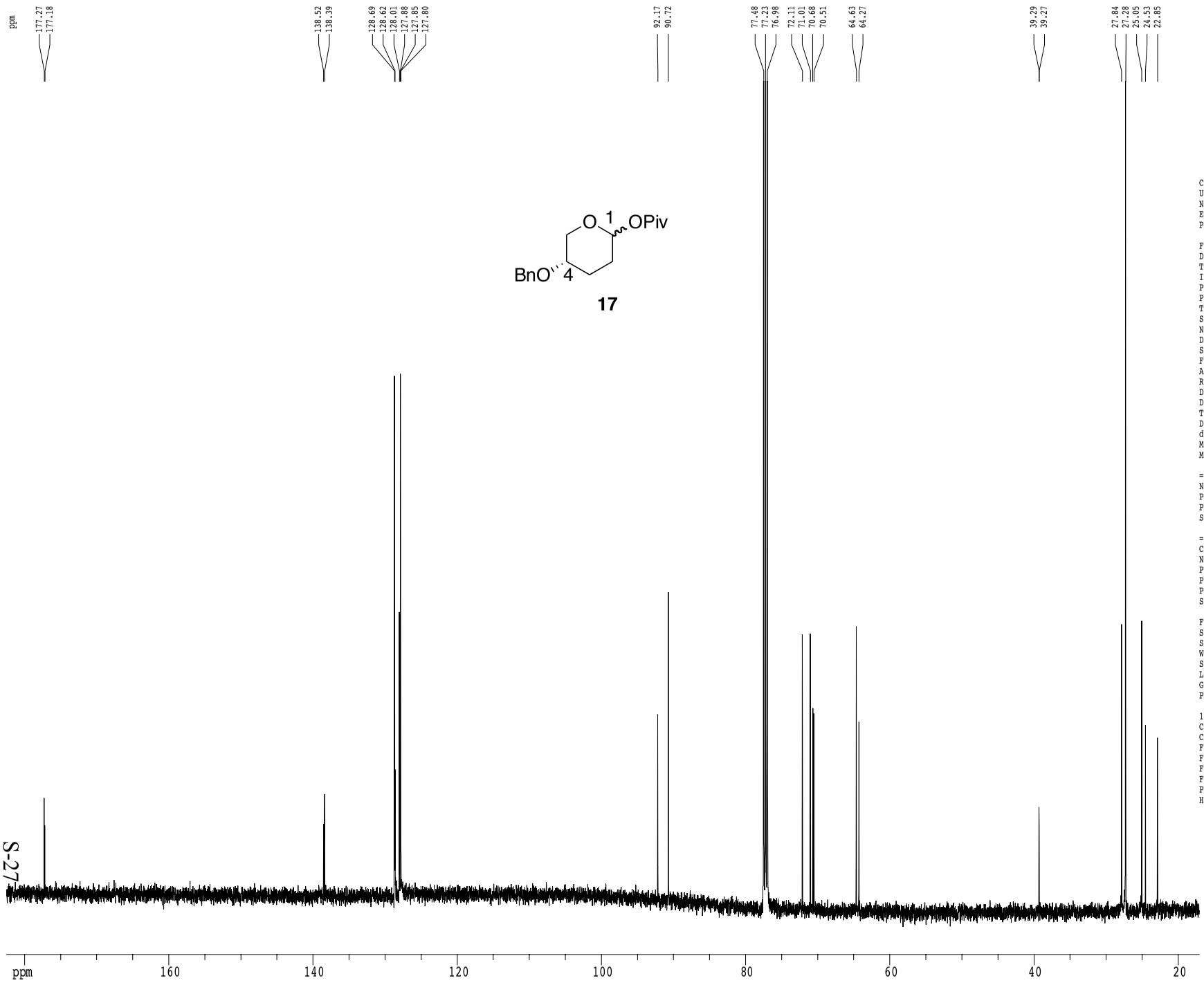
jrk-2-54 pivaloate 13C



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NAME jrk-2-54
EXPNO 114
PROCNO 1

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Time 16.06
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65536
SOLVENT CDCl3T
NS 113
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 13004
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

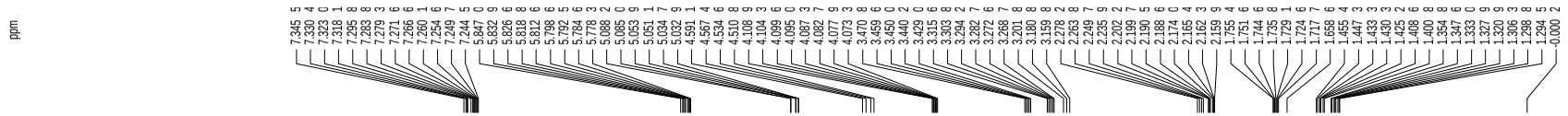
======== CHANNEL f1 ========
NUC1 13C
P1 15.00 usec
PL1 -1.00 dB
SFO1 125.7942548 MHz

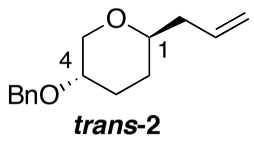
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 23.54 dB
SFO2 500.2225011 MHz

F2 - Processing parameters
SI 65536
SF 125.7804011 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 35.00 cm
F1P 182.472 ppm
F1 22951.37 Hz
F2P 17.072 ppm
F2 2147.31 Hz
PPMCM 7.25438 ppm/cm
HZCM 912.45911 Hz/cm

WAS-III-177-23
1H spectrum





***trans*-2**

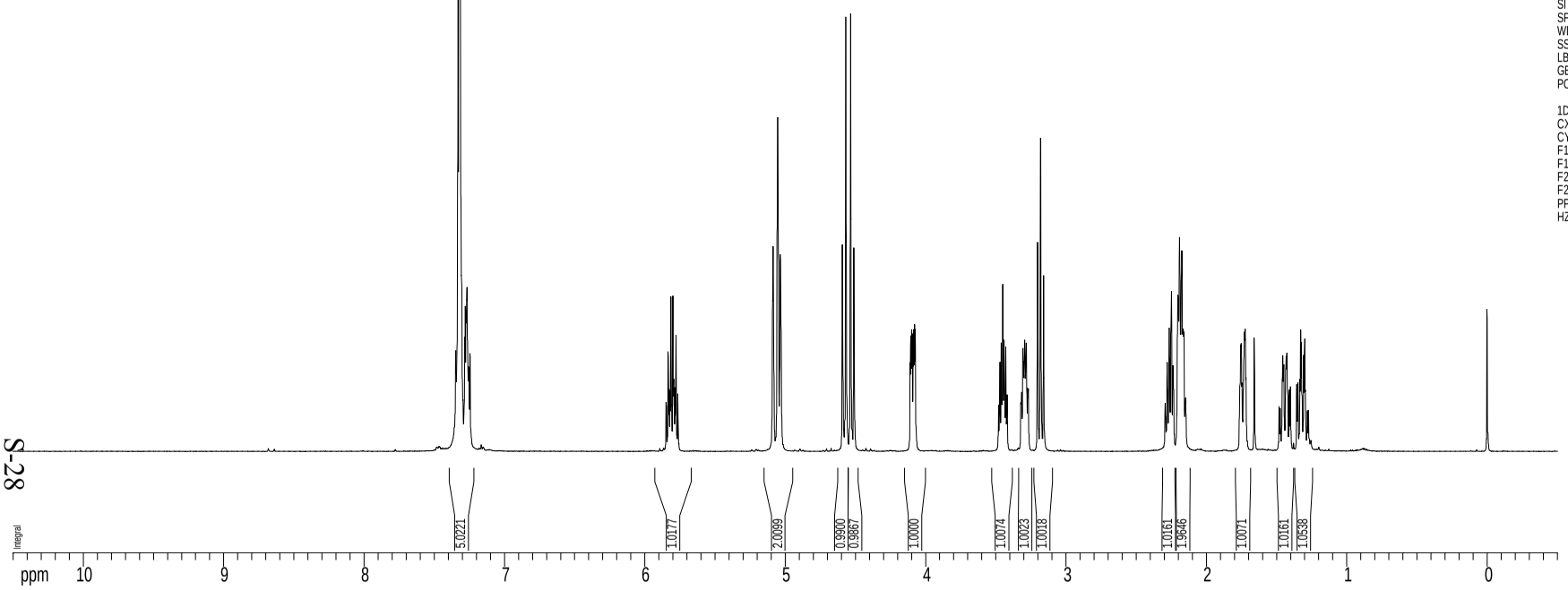
Current Data Parameters
USER walter
NAME WAS-III-177-23
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20071030
Time 17.58
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3T
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0999398 sec
RG 4
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

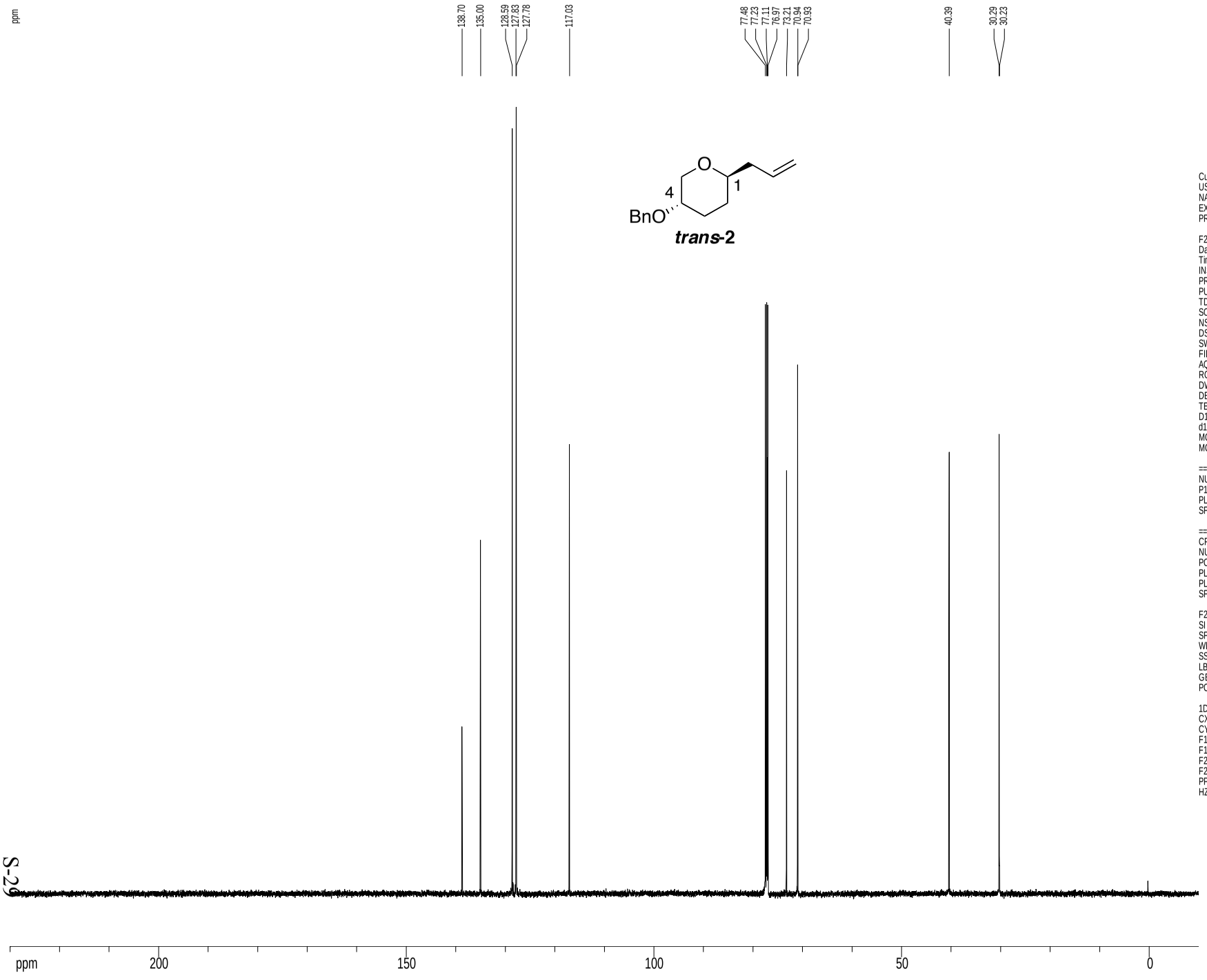
======== CHANNEL f1 ========
NUC1 1H
P1 8.00 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200386 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 10.500 ppm
F1 5252.31 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.48246 ppm/cm
HZCM 241.33423 Hz/cm



WAS-III-177-23
13C spectrum with 1H decoupling



Current Data Parameters
USER walter
NAME WAS-III-177-23
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20071030
Time 18.01
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65418
SOLVENT CDCl3
NS 65
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794635 sec
RG 8192
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

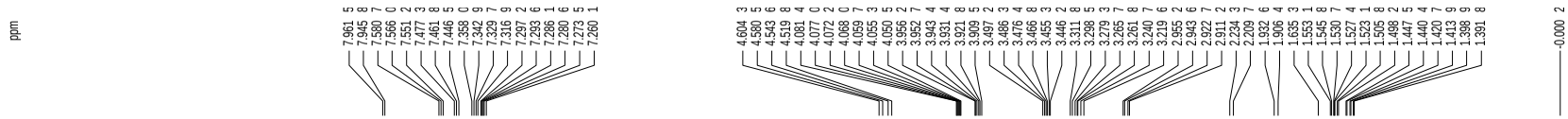
======== CHANNEL f1 ========
NUC1 13C
P1 15.00 usec
PL1 -1.00 dB
SFO1 125.7942548 MHz

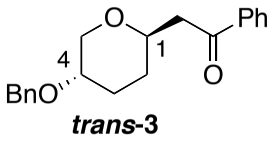
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 23.54 dB
SFO2 500.2225011 MHz

F2 - Processing parameters
SI 65536
SF 125.7804062 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 230.000 ppm
F1 28929.49 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 10.52632 ppm/cm
HZCM 1324.00427 Hz/cm

WAS-III-31-25
1H spectrum





***trans*-3**

Current Data Parameters
USER walter
NAME WAS-III-31-25
EXPNO 31
PROCNO 1

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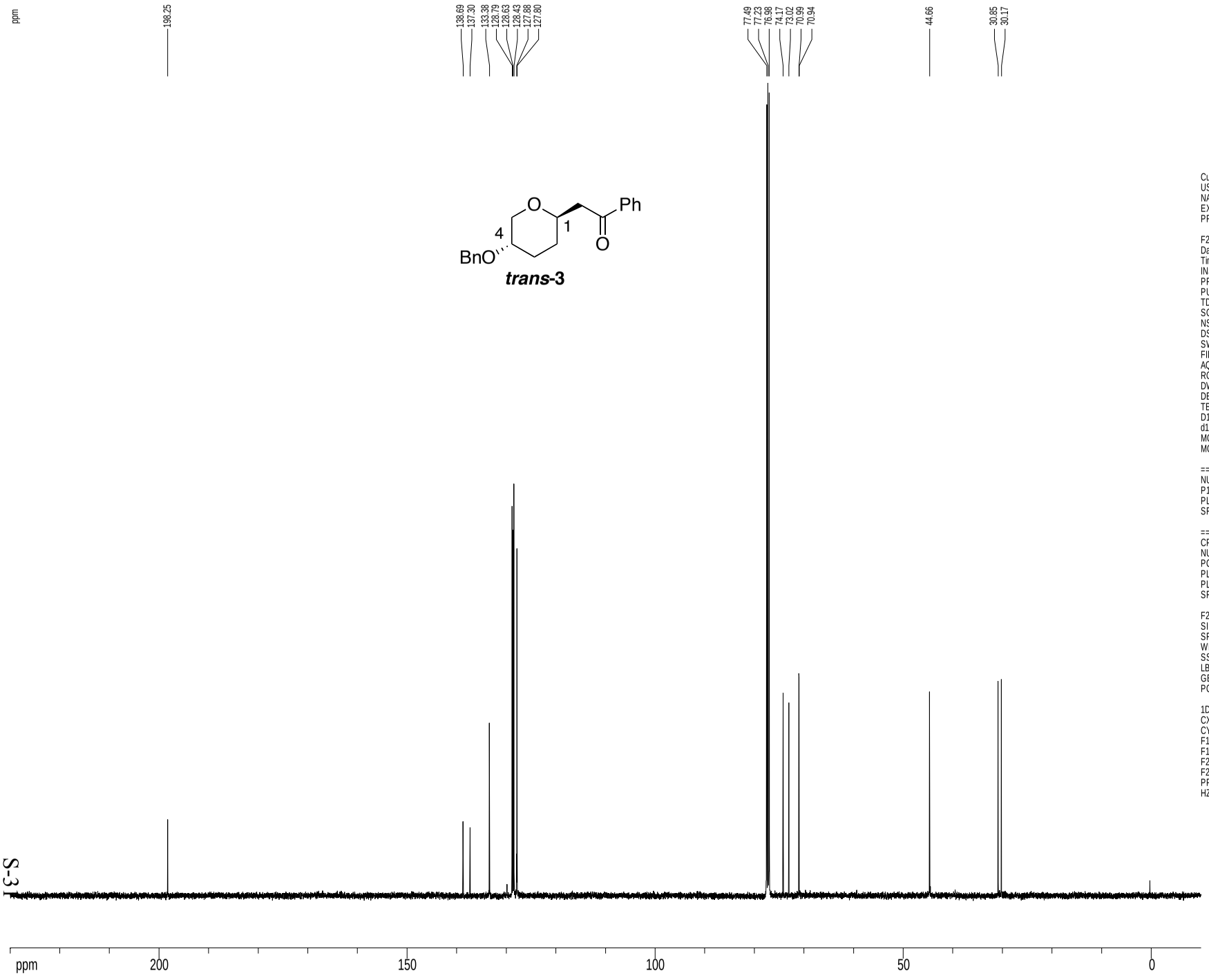
Date_ 20070621
Time 9.45
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3T
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 4.5
DW 62.400 usec
DE 6.00 usec
TE 290.1 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 8.00 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200319 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 10.500 ppm
F1 5252.31 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.48246 ppm/cm
HZCM 241.33423 Hz/cm

WAS-III-31-25
13C spectrum with 1H decoupling



Current Data Parameters
USER walter
NAME WAS-III-31-25
EXPNO 33
PROCNO 1

F2 - Acquisition Parameters
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Time 9.53
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65418
SOLVENT CDCl3
NS 225
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 11585.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

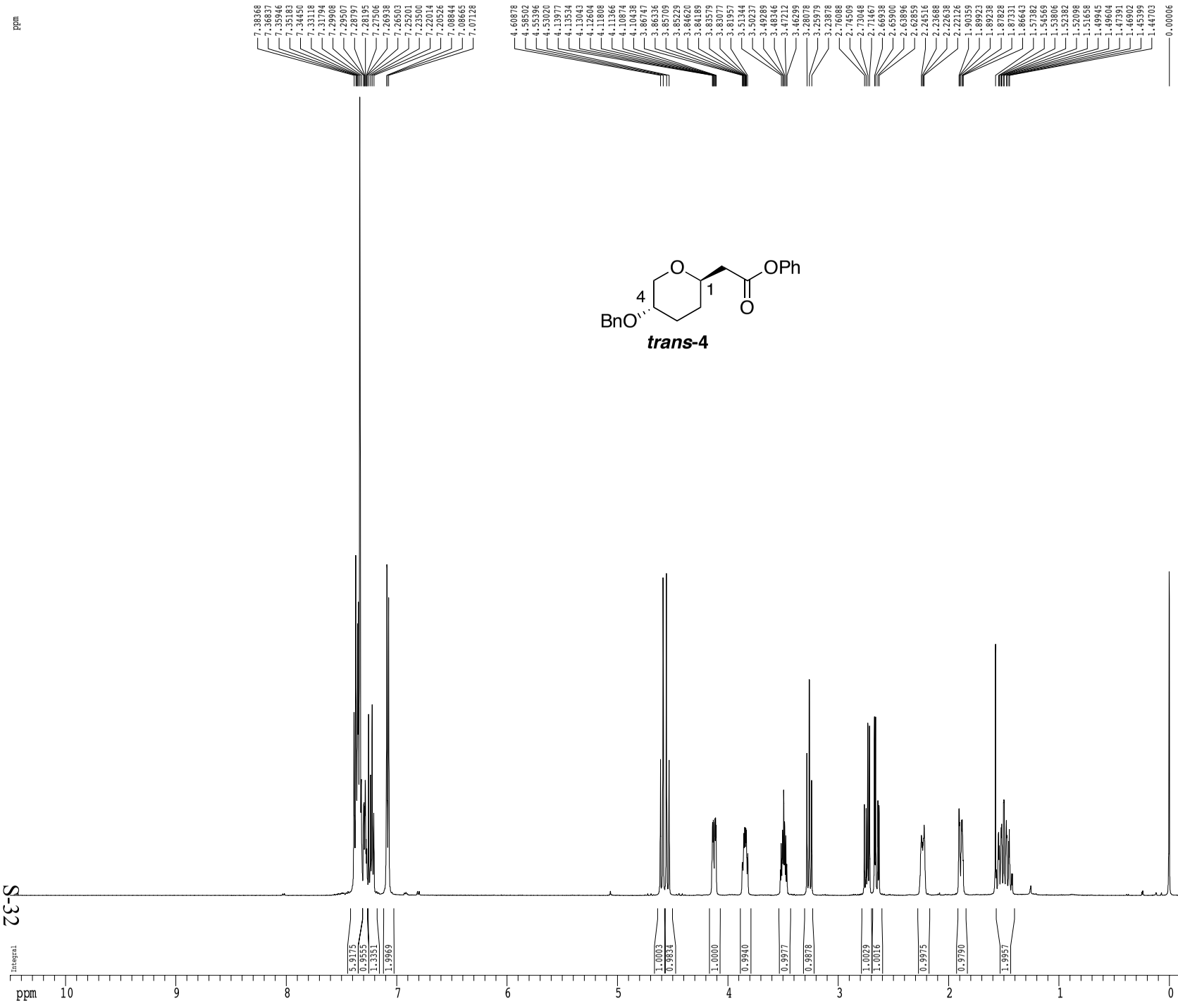
======== CHANNEL f1 =======
NUC1 13C
P1 15.00 usec
PL1 -1.00 dB
SFO1 125.7942548 MHz

======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 23.54 dB
SFO2 500.2225011 MHz

F2 - Processing parameters
SI 65536
SF 125.7804015 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 230.000 ppm
F1 28929.49 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 10.52632 ppm/cm
HZCM 1324.00427 Hz/cm

WAS-III-154-28
1H spectrum

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PROCNO                  1

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PULPROG              zg30
TD                  81728
SOLVENT           CDCl3T
NS                      8
DS                      0
SWH              8012.820 Hz
FIDRES           0.098043 Hz
AQ               5.0999398 sec
RG                      5
DW                 62.400 usec
DE                  6.00 usec
TE                298.0 K
D1             0.10000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

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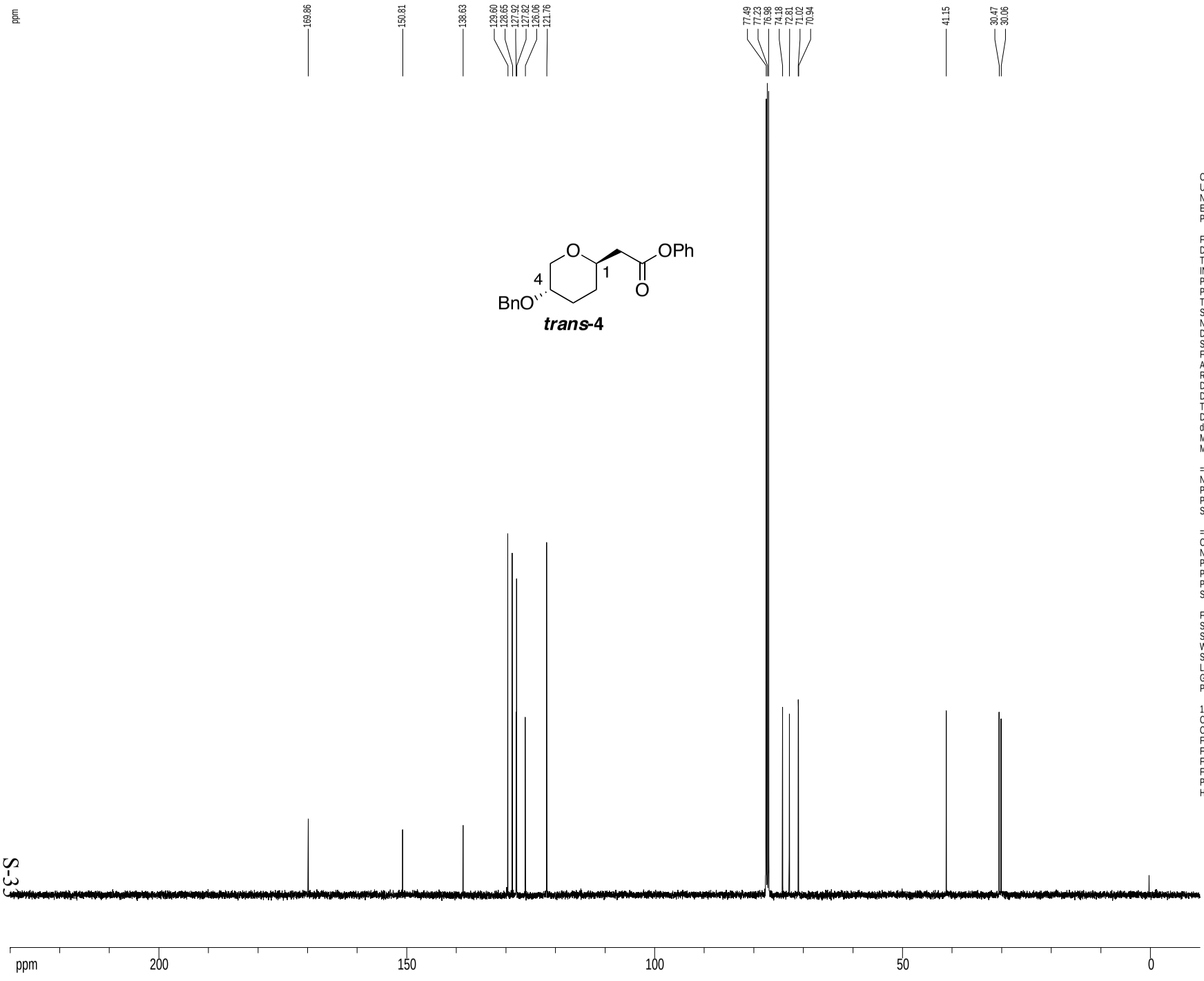
NUC1                   1H
P1                  8.00 usec
PL1                 1.60 dB
SFO1         500.2235015 MHz

F2 - Processing parameters
SI                  65536
SF           500.2200351 MHz
WDW                    EM
SSB                     0
LB                  0.30 Hz
GB                      0
PC                  4.00

1D NMR plot parameters
CX                  22.80 cm
CY                  15.00 cm
F1P                10.500 ppm
F1                5252.31 Hz
F2P                -0.500 ppm
F2               -250.11 Hz
PPMCM             0.48246 ppm/cm
HZCM            241.33423 Hz/cm
```

WAS-III-154-28
13C spectrum with 1H decoupling



Current Data Parameters
USER walter
NAME WAS-III-154-28
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20071002
Time 16.37
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65418
SOLVENT CDCl3T
NS 112
DS 0
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794635 sec
RG 10321.3
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

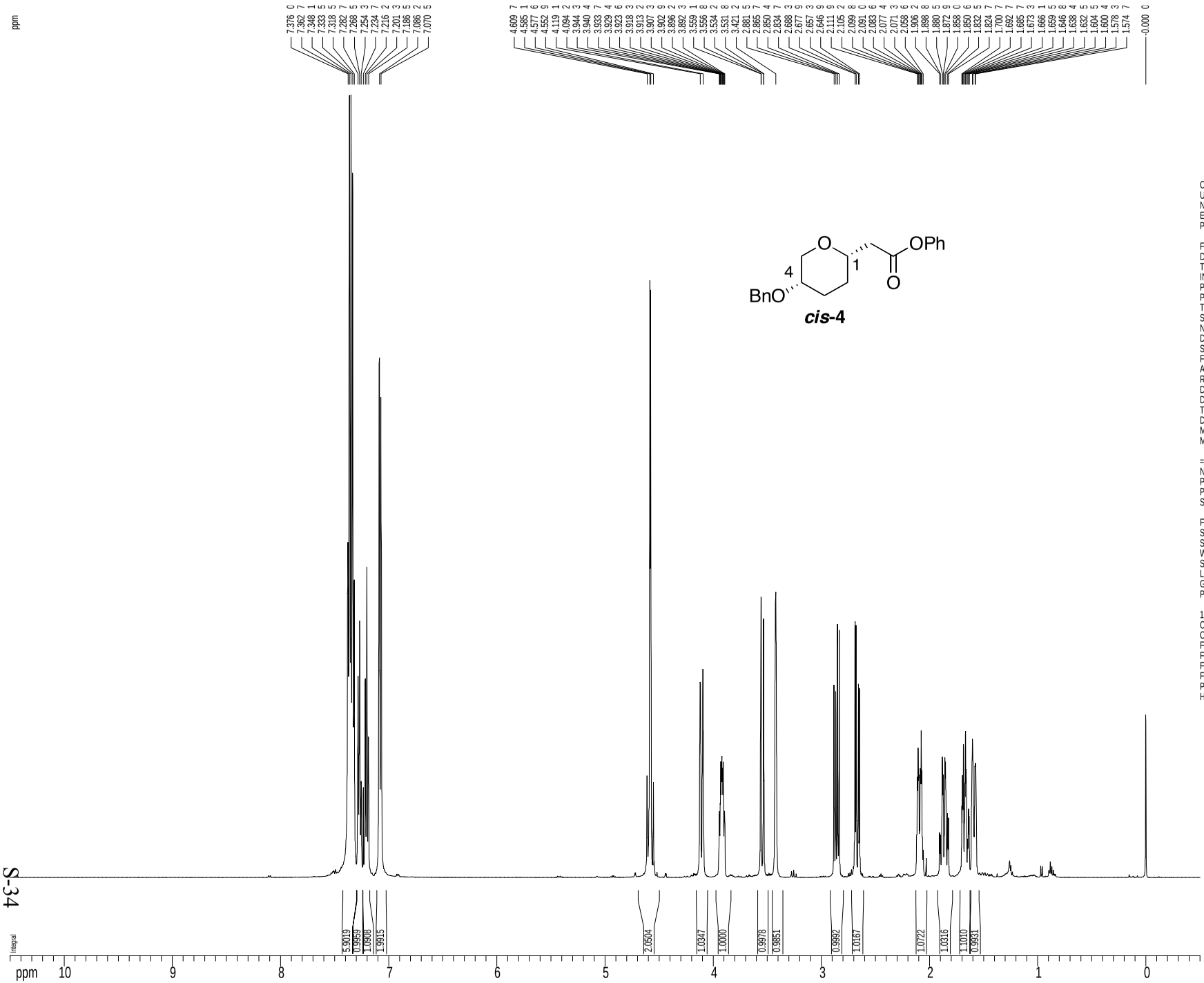
======== CHANNEL f1 =======
NUC1 13C
P1 15.00 usec
PL1 -1.00 dB
SFO1 125.7942548 MHz

======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 23.54 dB
SFO2 500.2225011 MHz

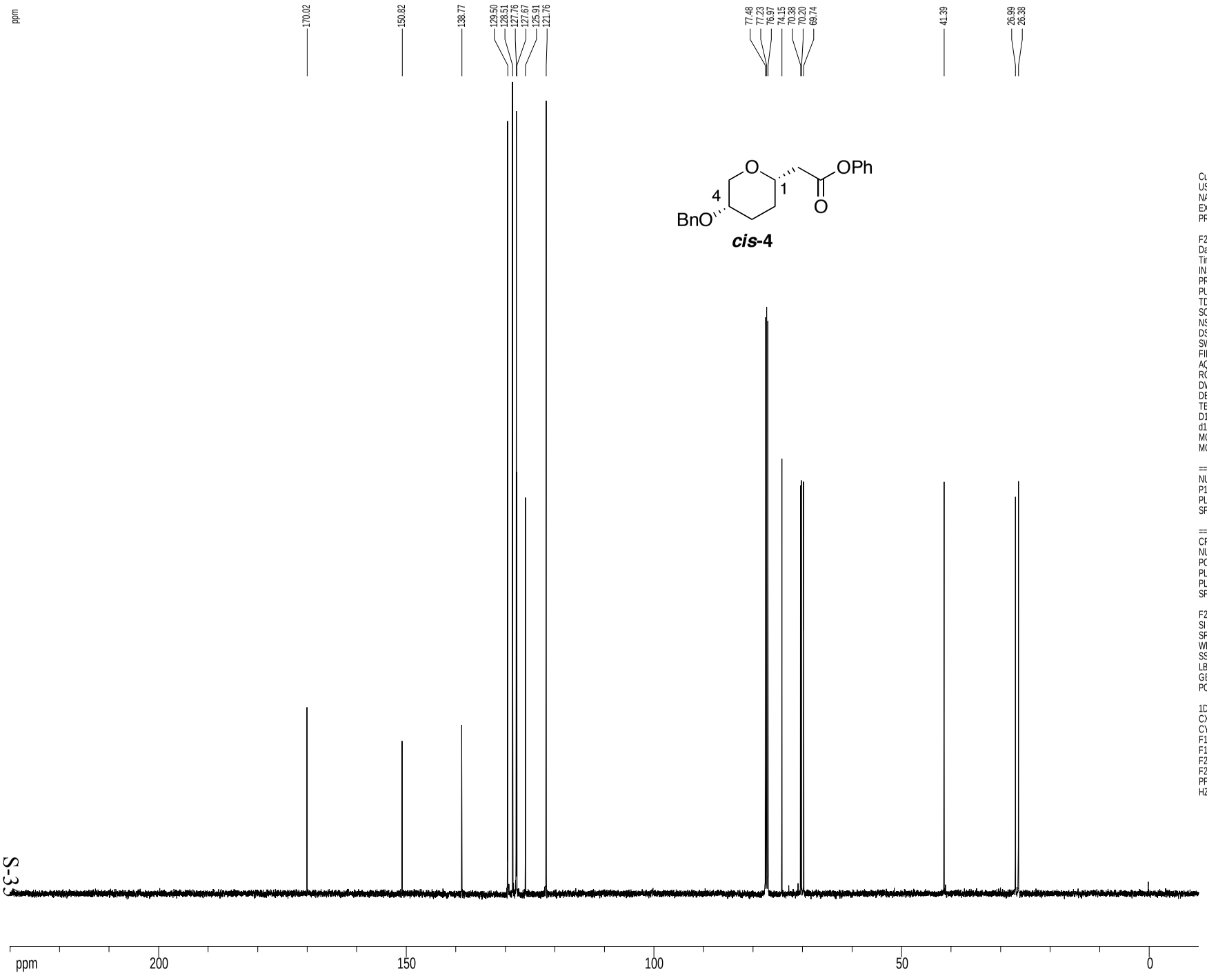
F2 - Processing parameters
SI 65536
SF 125.7804015 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 230.000 ppm
F1 28929.49 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 10.52632 ppm/cm
HZCM 1324.00427 Hz/cm

WAS-III-154-30
1H spectrum



WAS-III-154-30
13C spectrum with 1H decoupling



Current Data Parameters
USER walter
NAME WAS-III-154-30
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20071002
Time 16.47
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65418
SOLVENT CDCl3T
NS 76
DS 0
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794635 sec
RG 13004
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

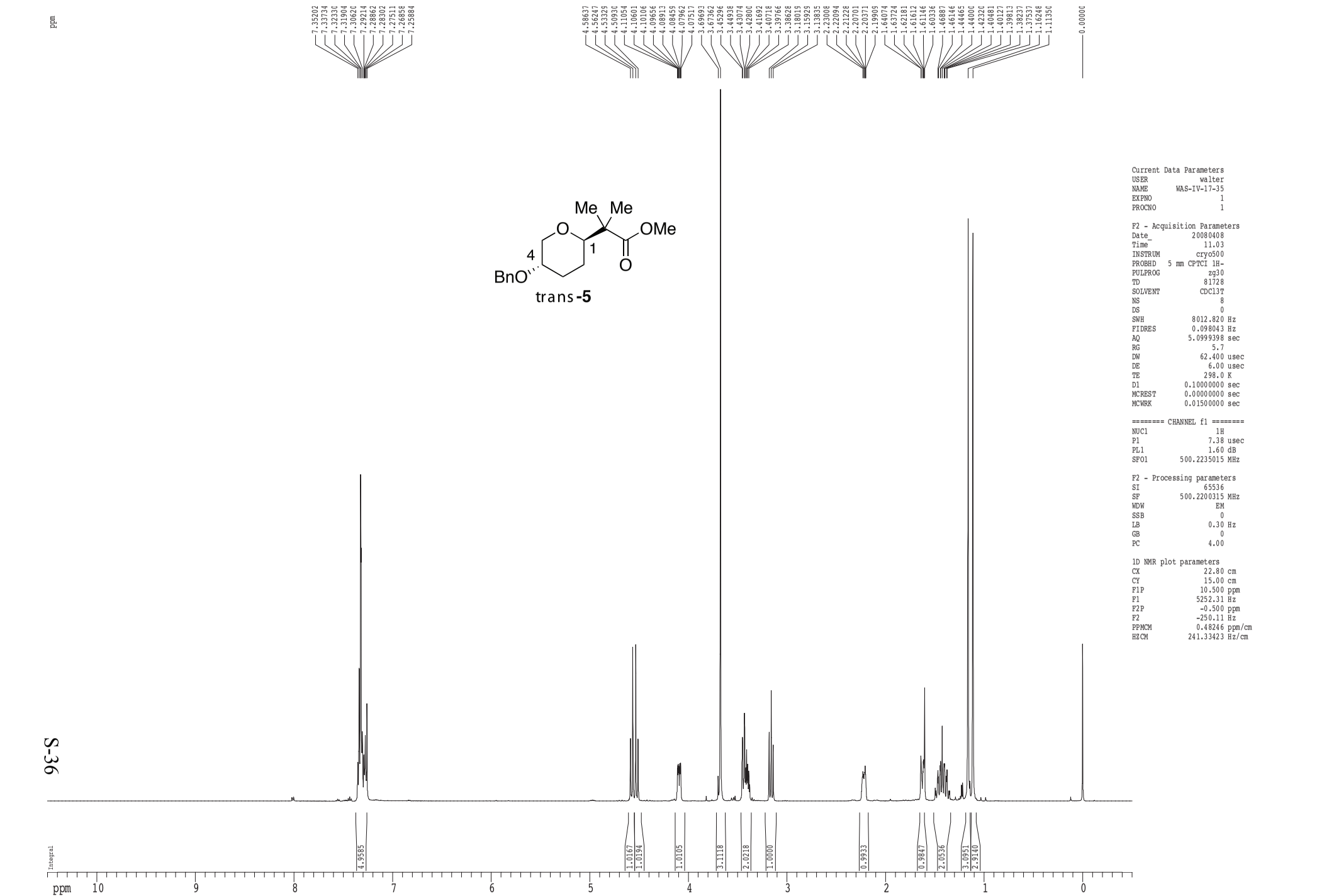
======== CHANNEL f1 ========
NUC1 13C
P1 15.00 usec
PL1 -1.00 dB
SFO1 125.7942548 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 23.54 dB
SFO2 500.2225011 MHz

F2 - Processing parameters
SI 65536
SF 125.7804112 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 230.000 ppm
F1 28929.49 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 10.52632 ppm/cm
HZCM 1324.00439 Hz/cm

WAS-IV-17-35
1H spectrum



Current Data Parameters

USER	walter
NAME	WAS-IV-17-35
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20080408
Time	11.03
INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	81728
SOLVENT	CDCl3T
NS	8
DS	0
SWH	8012.820 Hz
FIDRES	0.098043 Hz
AQ	5.0999398 sec
RG	5.7
DW	62.400 usec
DE	6.00 usec
TE	298.0 K
D1	0.10000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	1H
P1	7.38 usec
PL1	1.60 dB
SFO1	500.2235015 MHz

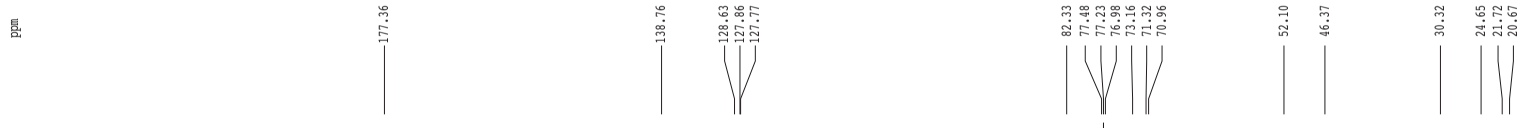
F2 - Processing parameters

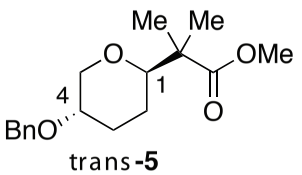
SI	65536
SF	500.2200315 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	22.80 cm
CY	15.00 cm
F1P	10.500 ppm
F1	5252.31 Hz
F2P	-0.500 ppm
F2	-250.11 Hz
PPMCM	0.48246 ppm/cm
HZCM	241.33423 Hz/cm

WAS-IV_17-35
13C spectrum with 1H decoupling





Current Data Parameters
USER walter
NAME WAS-IV-17-35
EXPNO 2
PROCNO 1

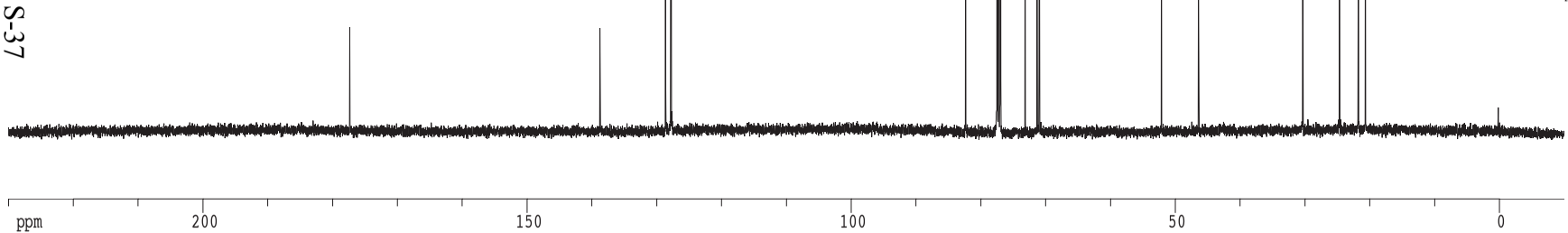
F2 - Acquisition Parameters
Date_ 20080408
Time 11.07
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 106
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0814105 sec
RG 13004
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 14.75 usec
PL1 -1.00 dB
SFO1 125.7942548 MHz

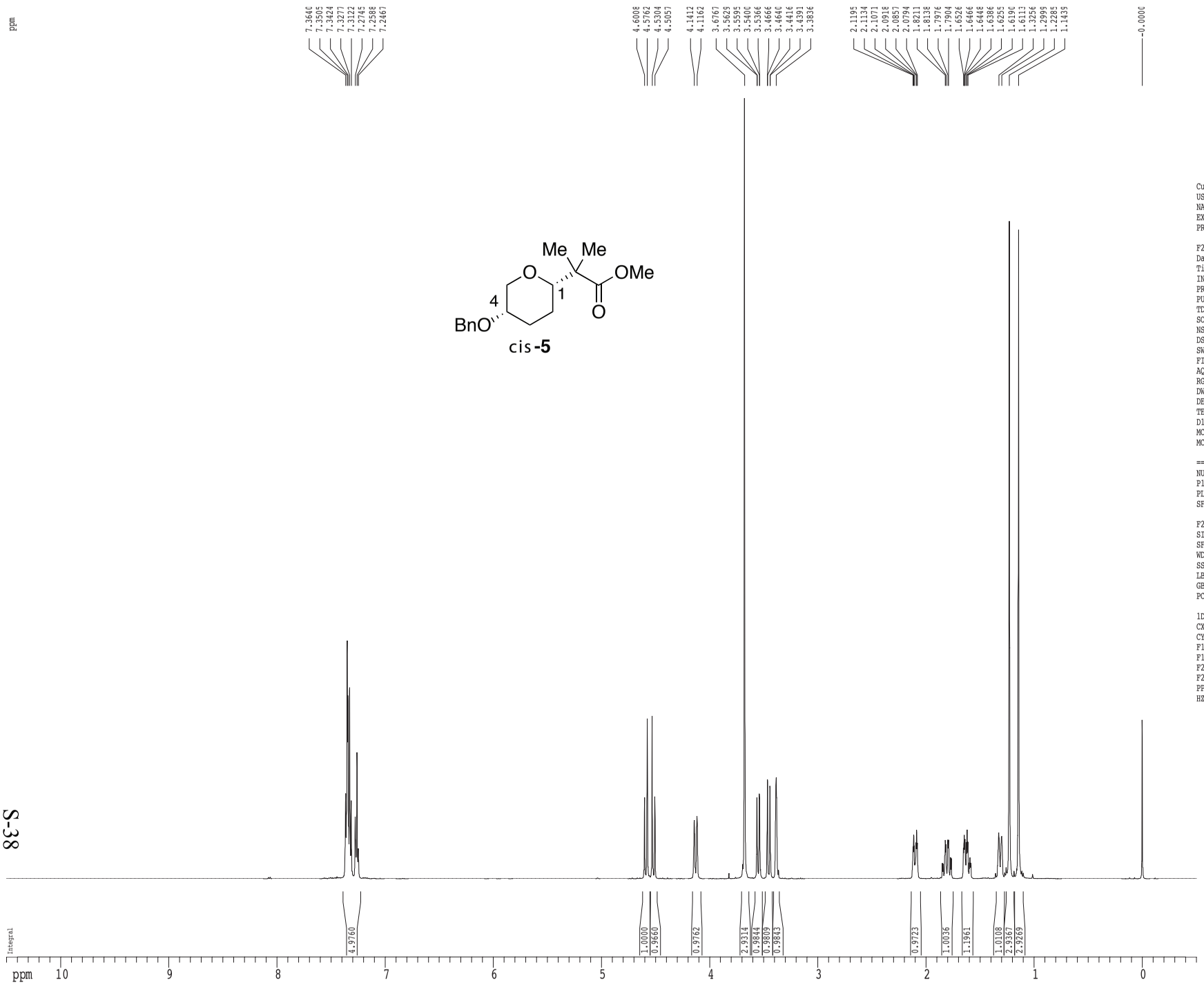
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.80 dB
SFO2 500.2225011 MHz

F2 - Processing parameters
SI 65536
SF 125.7804006 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 230.000 ppm
F1 28929.49 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 10.52632 ppm/cm
HZCM 1324.00427 Hz/cm



WAS-IV-18-33
1H spectrum



Current Data Parameters
USER walter
NAME WAS-IV-18-33
EXPNO 1
PROCNO 1

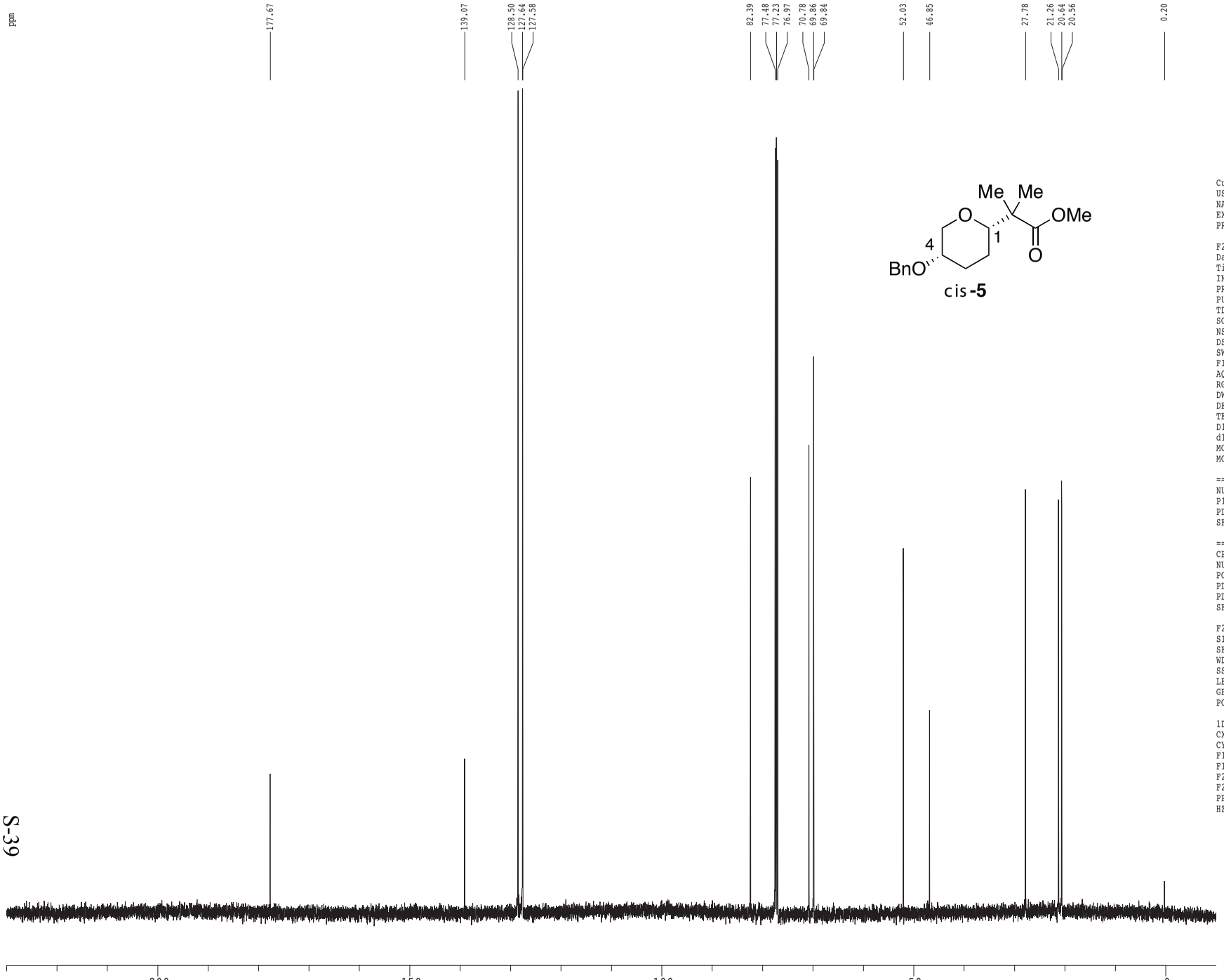
F2 - Acquisition Parameters
Date_ 20080408
Time 11.11
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3T
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0999398 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.38 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200315 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 10.500 ppm
F1 5252.31 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.48246 ppm/cm
HZCM 241.33423 Hz/cm

WAS-IV-18-33
13C spectrum with 1H decoupling



Current Data Parameters
USER walter
NAME WAS-IV-18-33
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080408
Time 11.14
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 77
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0814105 sec
RG 9195.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

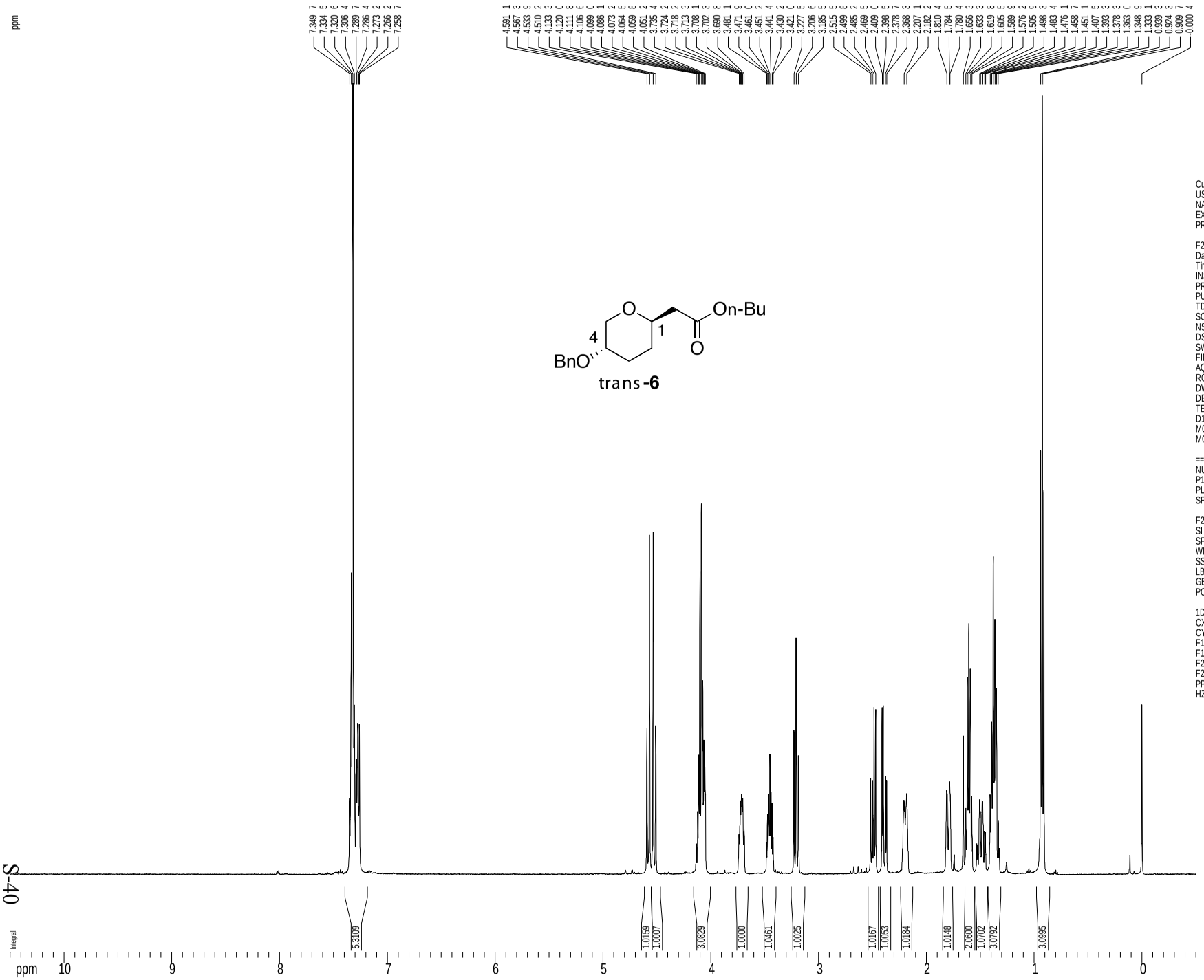
======= CHANNEL f1 =======
NUC1 13C
P1 14.75 usec
PL1 -1.00 dB
SFO1 125.7942548 MHz

======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.80 dB
SFO2 500.2225011 MHz

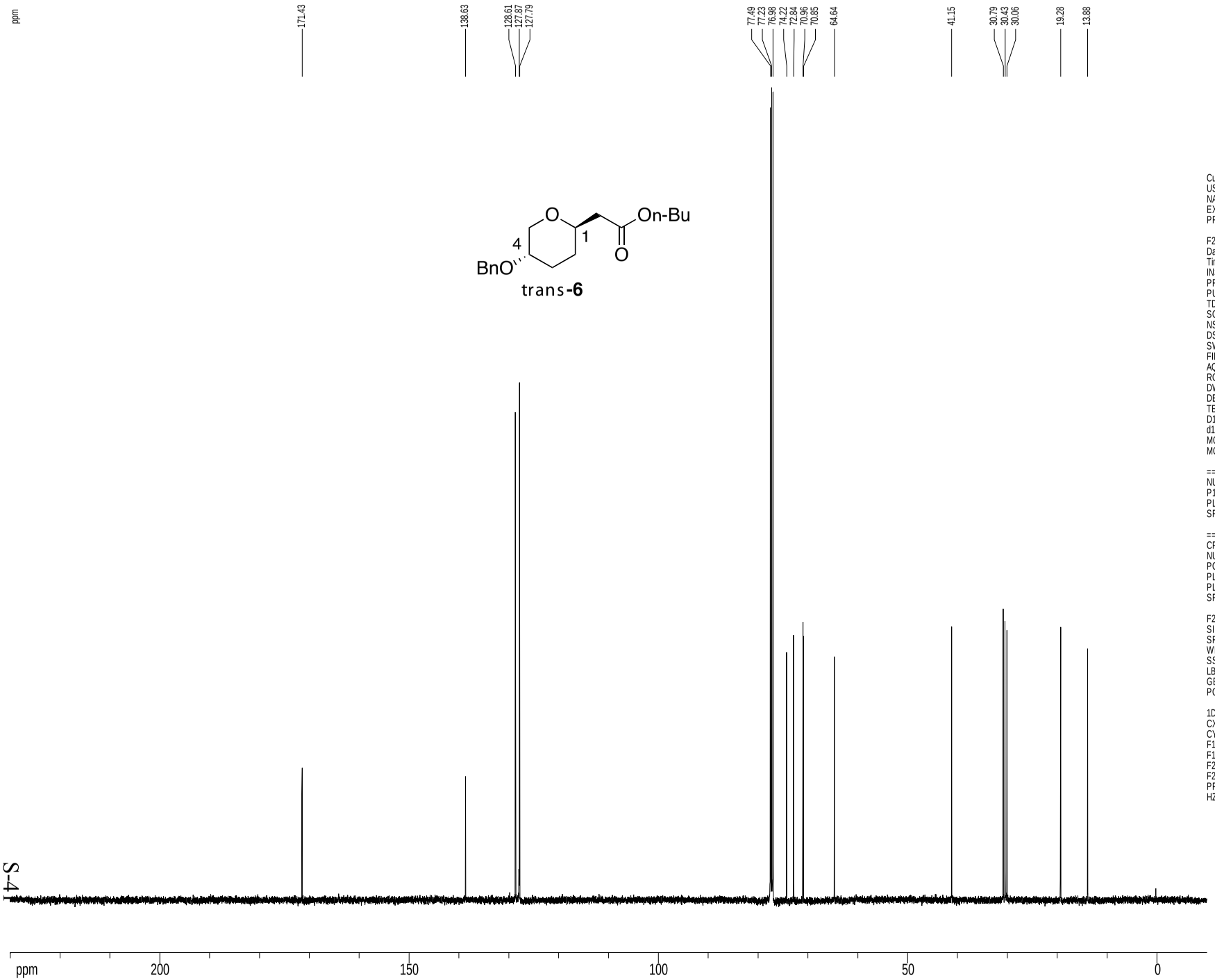
F2 - Processing parameters
SI 65536
SF 125.7804015 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 230.000 ppm
F1 28929.49 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 10.52632 ppm/cm
HZCM 1324.00427 Hz/cm

WAS-III-97-25
1H spectrum



WAS-III-97-25
13C spectrum with 1H decoupling



Current Data Parameters
USER walter
NAME WAS-III-97-25
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070516
Time 14.47
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65418
SOLVENT CDCl3
NS 94
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 11585.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

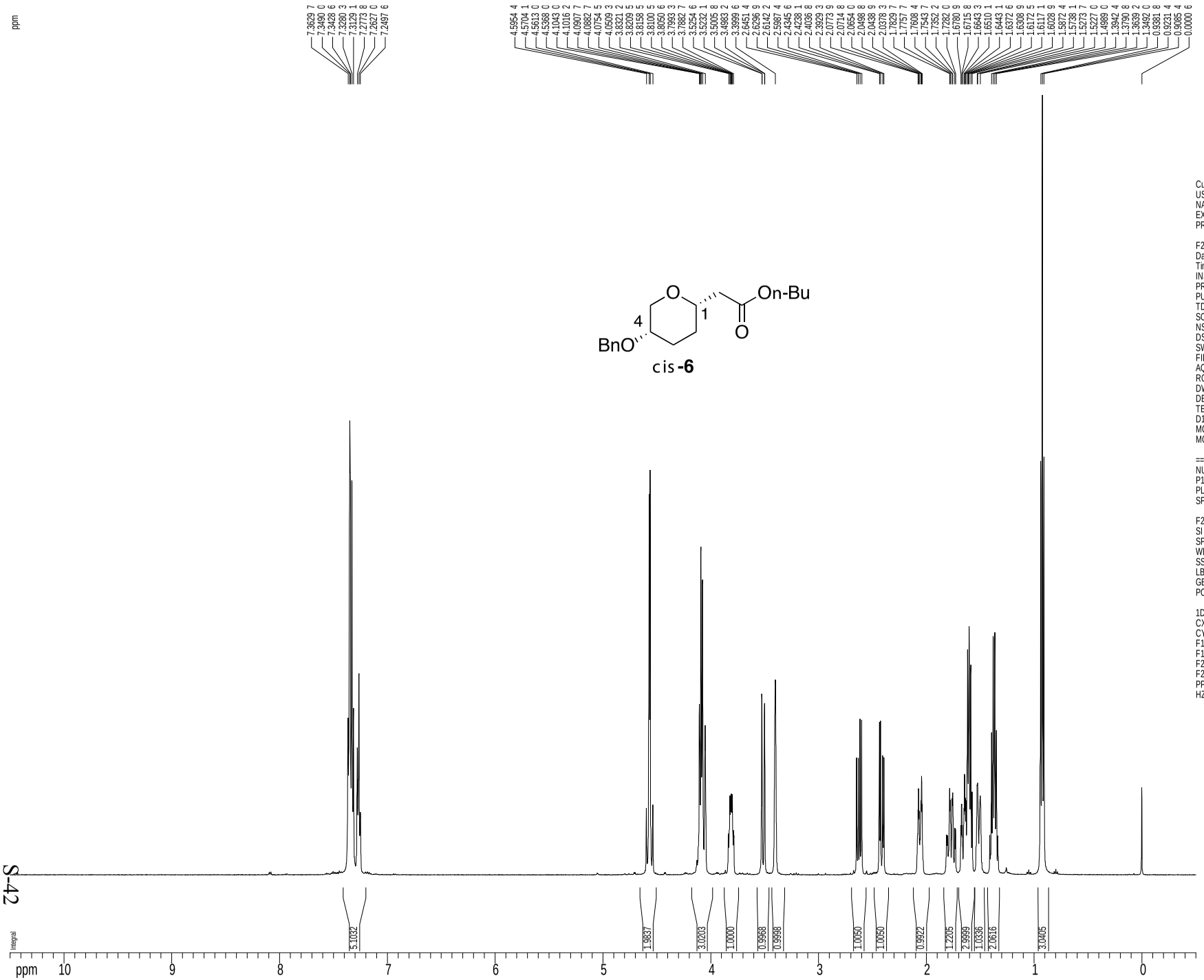
======== CHANNEL f1 =======
NUC1 13C
P1 15.00 usec
PL1 -1.00 dB
SFO1 125.7942548 MHz

======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 23.54 dB
SFO2 500.2225011 MHz

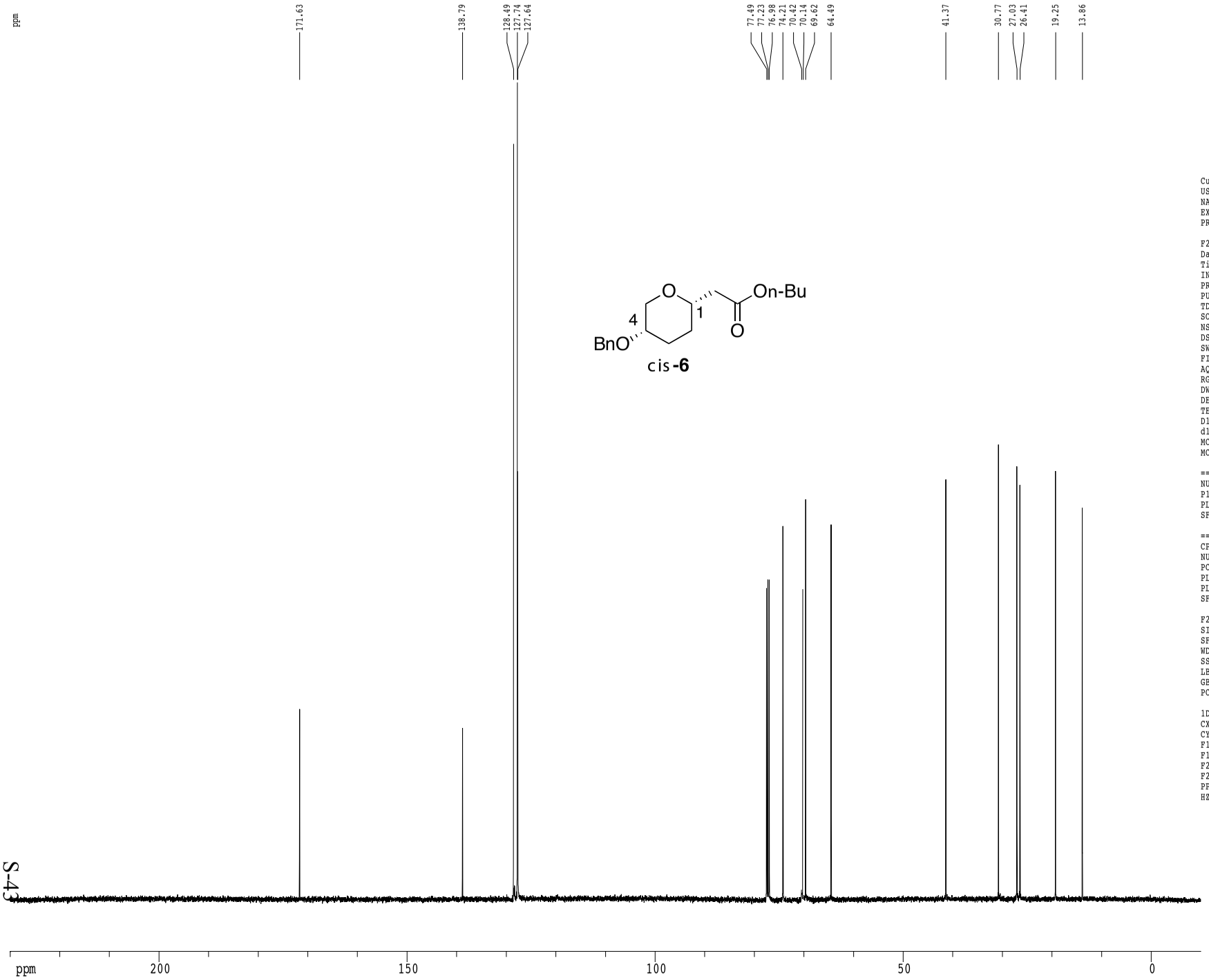
F2 - Processing parameters
SI 65536
SF 125.7804034 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 230.000 ppm
F1 28929.49 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 10.52632 ppm/cm
HZCM 1324.00427 Hz/cm

WAS-III-97-27
1H spectrum



WAS-III-97-27
13C spectrum with 1H decoupling



Current Data Parameters
USER walter
NAME WAS-III-97-27
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070516
Time 14.38
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 118
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 8192
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

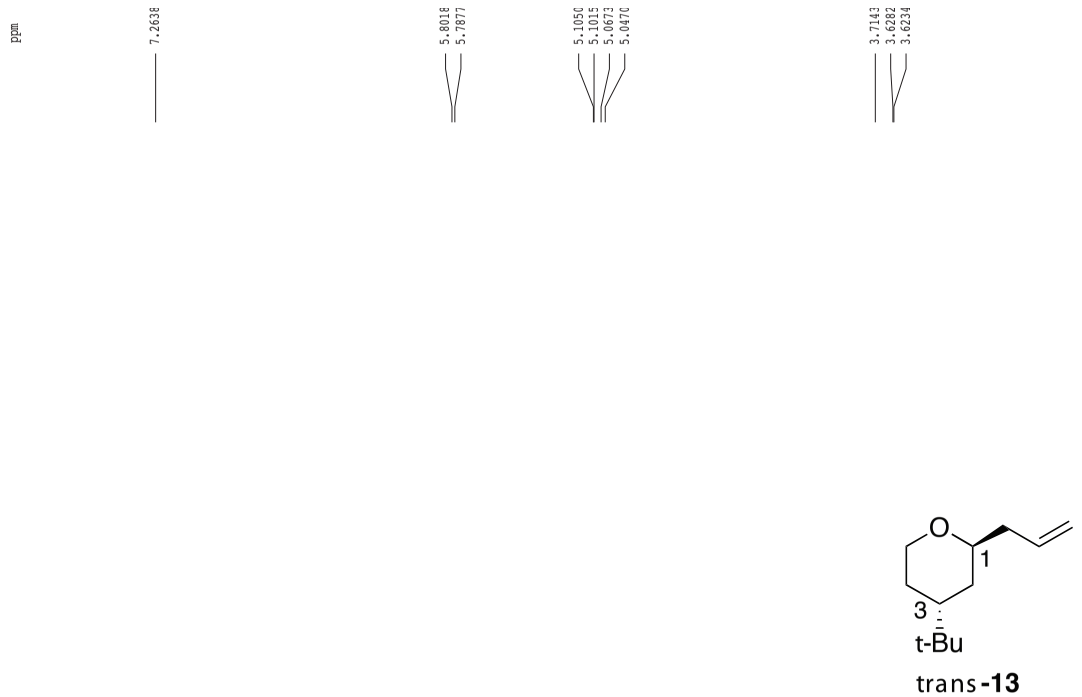
======== CHANNEL f1 ========
NUC1 13C
P1 15.00 usec
PL1 -1.00 dB
SFO1 125.7942548 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 23.54 dB
SFO2 500.2225011 MHz

F2 - Processing parameters
SI 65536
SF 125.7804080 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 230.000 ppm
F1 28929.49 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 10.52632 ppm/cm
HZCM 1324.00439 Hz/cm

WAS-III-58-27
1H spectrum



trans-**13**

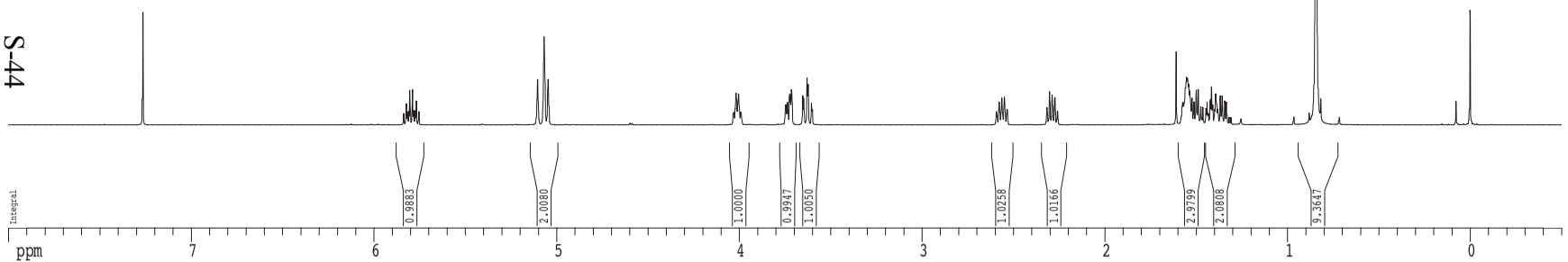
Current Data Parameters
USER walter
NAME WAS-III-58-27
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070212
Time 17.18
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3T
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

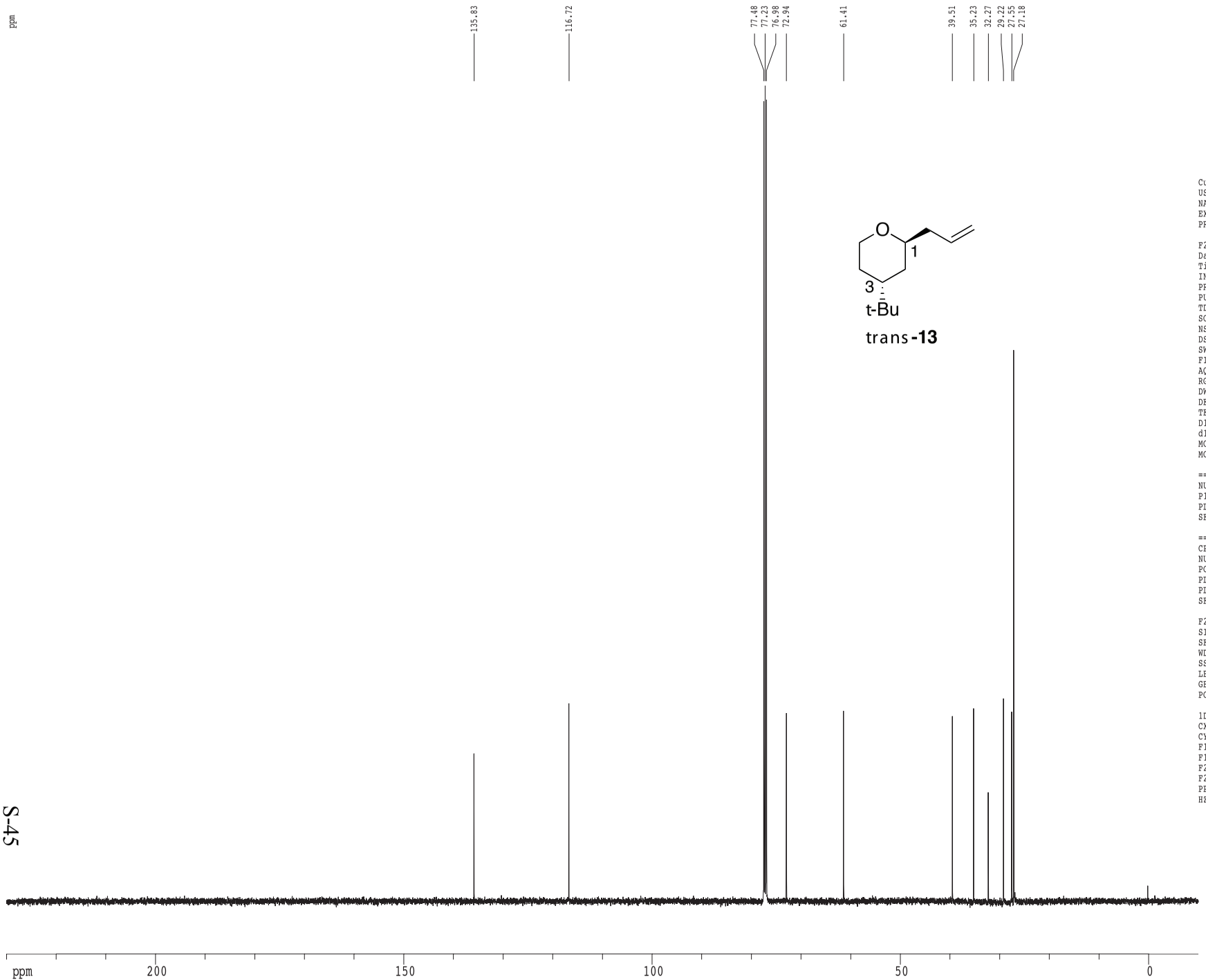
======== CHANNEL f1 ========
NUC1 1H
P1 8.00 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200305 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 8.000 ppm
F1 4001.76 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.37281 ppm/cm
HZCM 186.48555 Hz/cm



WAS-III-58-27
13C spectrum



Current Data Parameters
USER walter
NAME WAS-III-58-27
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070212
Time 17.22
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65418
SOLVENT CDCl3
NS 150
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 9195.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

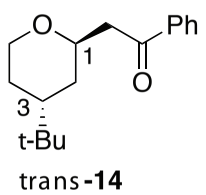
======== CHANNEL f1 ========
NUC1 13C
P1 15.00 usec
PL1 -1.00 dB
SFO1 125.7942548 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 23.54 dB
SFO2 500.2225011 MHz

F2 - Processing parameters
SI 65536
SF 125.7803997 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 230.000 ppm
F1 28929.49 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 10.52632 ppm/cm
HZCM 1324.00427 Hz/cm

WAS-III-62-26
1H spectrum



trans-**14**

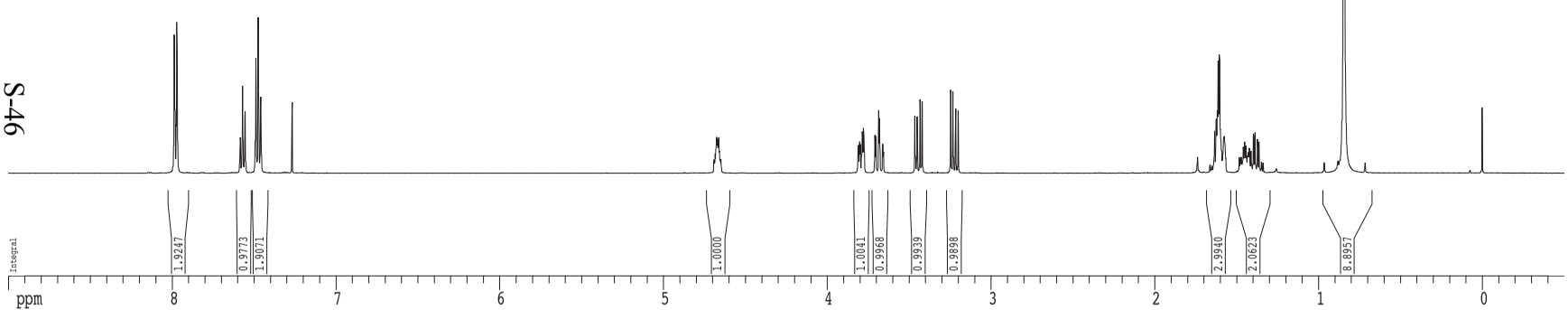
Current Data Parameters
USER walter
NAME WAS-III-62-26
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070222
Time 17.38
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3T
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

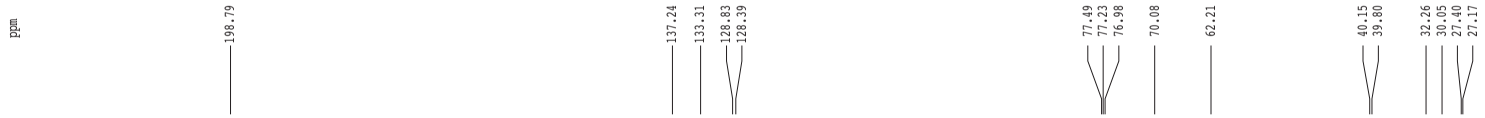
======== CHANNEL f1 ========
NUC1 1H
P1 8.00 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

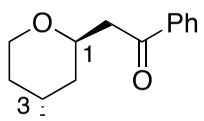
F2 - Processing parameters
SI 65536
SF 500.2200278 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm



WAS-III-62-26
13C spectrum with 1H decoupling





trans-**14**


```
Current Data Parameters
USER              walter
NAME       WAS-III-62-26
EXPNO                2
PROCNO               1

F2 - Acquisition Parameters
Date_          20070222
Time              17.42
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG          zgdc30
TD                65536
SOLVENT           CDCl3
NS                  222
DS                    4
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ             1.0813940 sec
RG               11585.2
DW                16.500 usec
DE                 6.00 usec
TE                298.0 K
D1            0.25000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

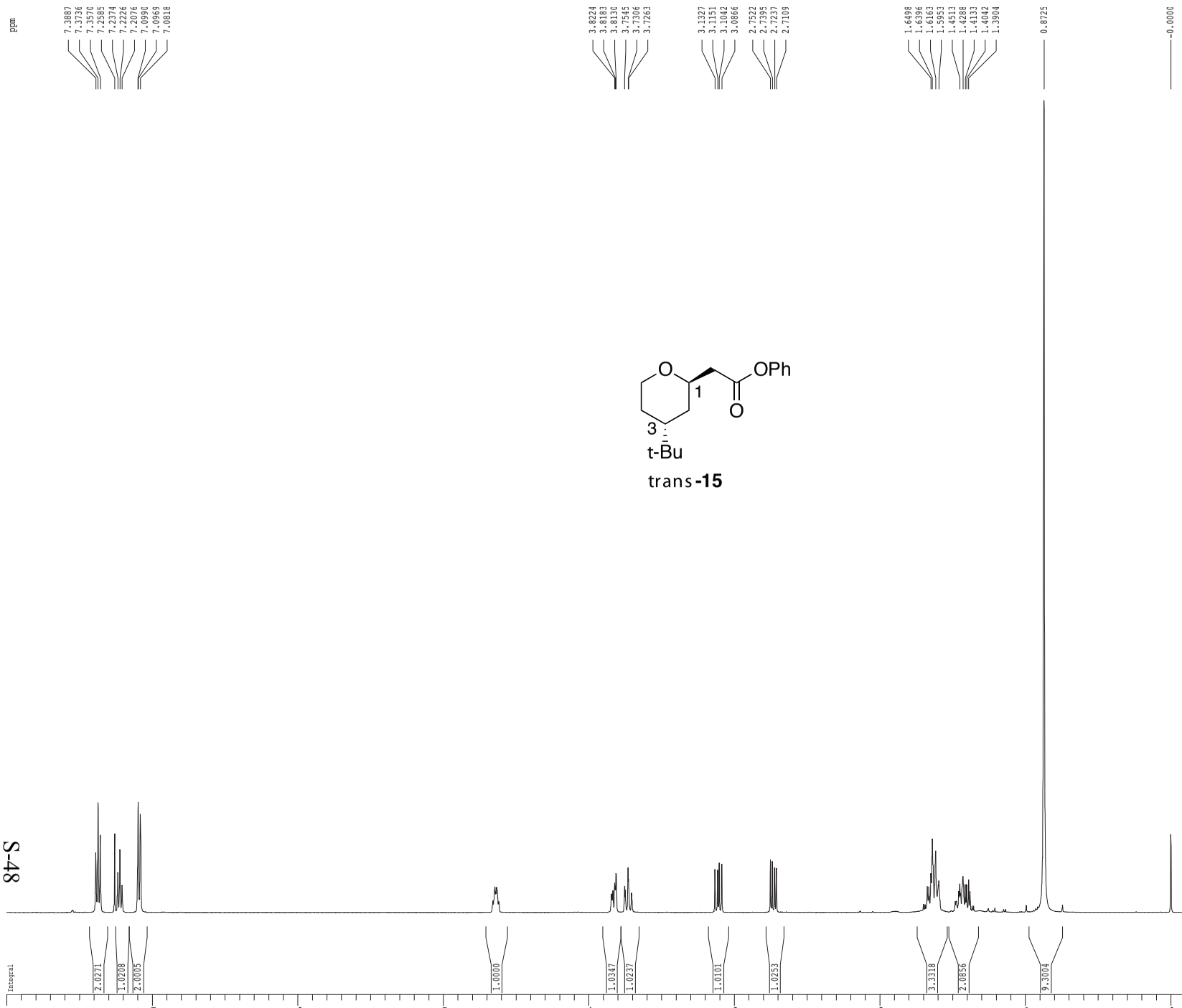
======== CHANNEL f1 ========
NUC1                13C
P1                15.00 usec
PL1               -1.00 dB
SFO1         125.7942548 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             100.00 usec
PL2                1.60 dB
PL12              23.54 dB
SFO2         500.2225011 MHz

F2 - Processing parameters
SI                  65536
SF           125.7804034 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   2.00

1D NMR plot parameters
CX                  22.80 cm
CY                  15.65 cm
F1P               230.000 ppm
F1              28929.49 Hz
F2P               -10.000 ppm
F2              -1257.80 Hz
PPMCM           10.52632 ppm/cm
HZCM          1324.00427 Hz/cm
```

WAS-III-59-27
1H spectrum



Current Data Parameters
USER walter
NAME WAS-III-59-27
EXPNO 3
PROCNO 1

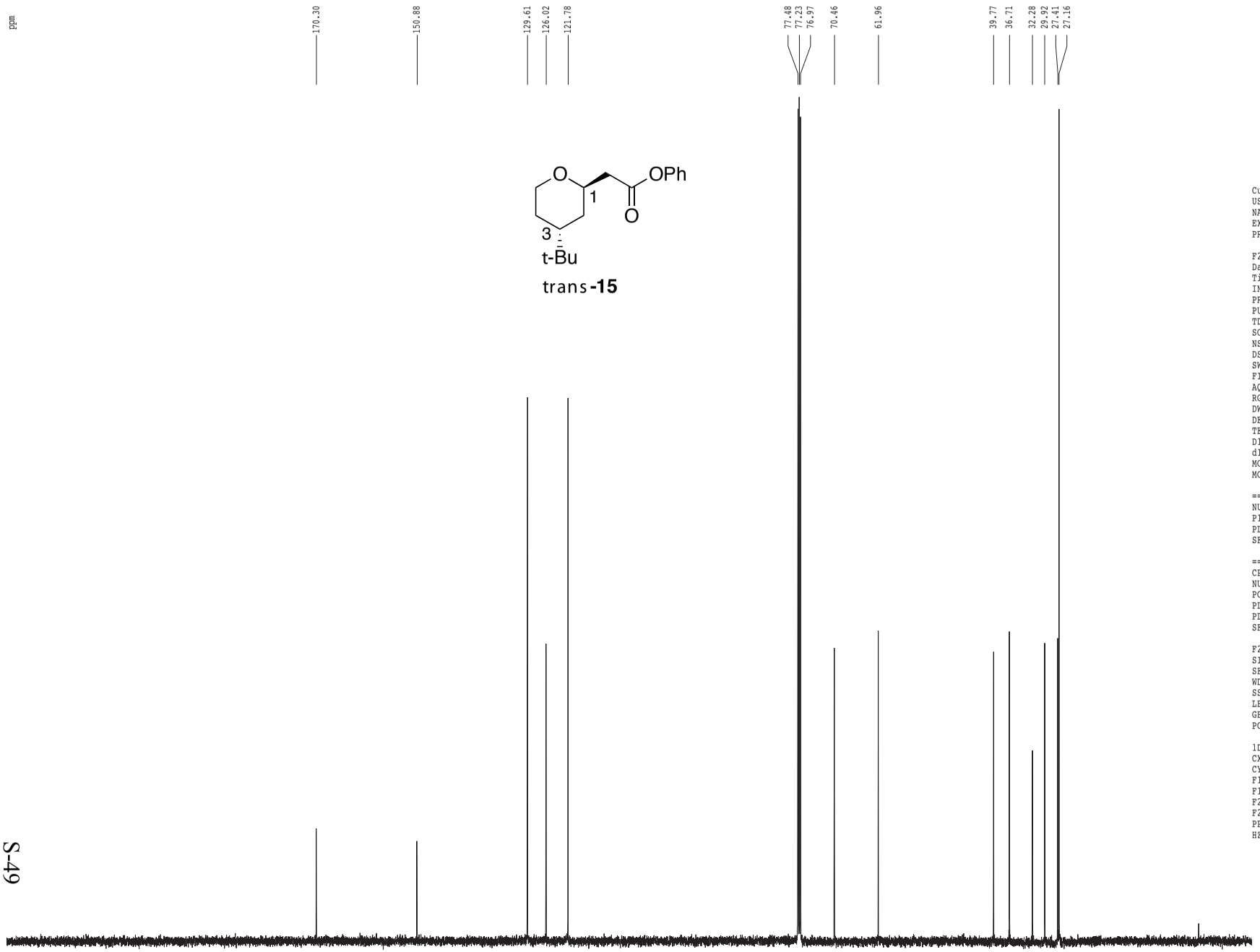
F2 - Acquisition Parameters
Date_ 20070213
Time 9.11
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3T
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 4.5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 8.00 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200331 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 8.000 ppm
F1 4001.76 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.37281 ppm/cm
HZCM 186.48555 Hz/cm

WAS-III-59-27
13C spectrum with 1H decoupling



Current Data Parameters
USER walter
NAME WAS-III-59-27
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070213
Time 9.14
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65418
SOLVENT CDCl3
NS 94
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 13004
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

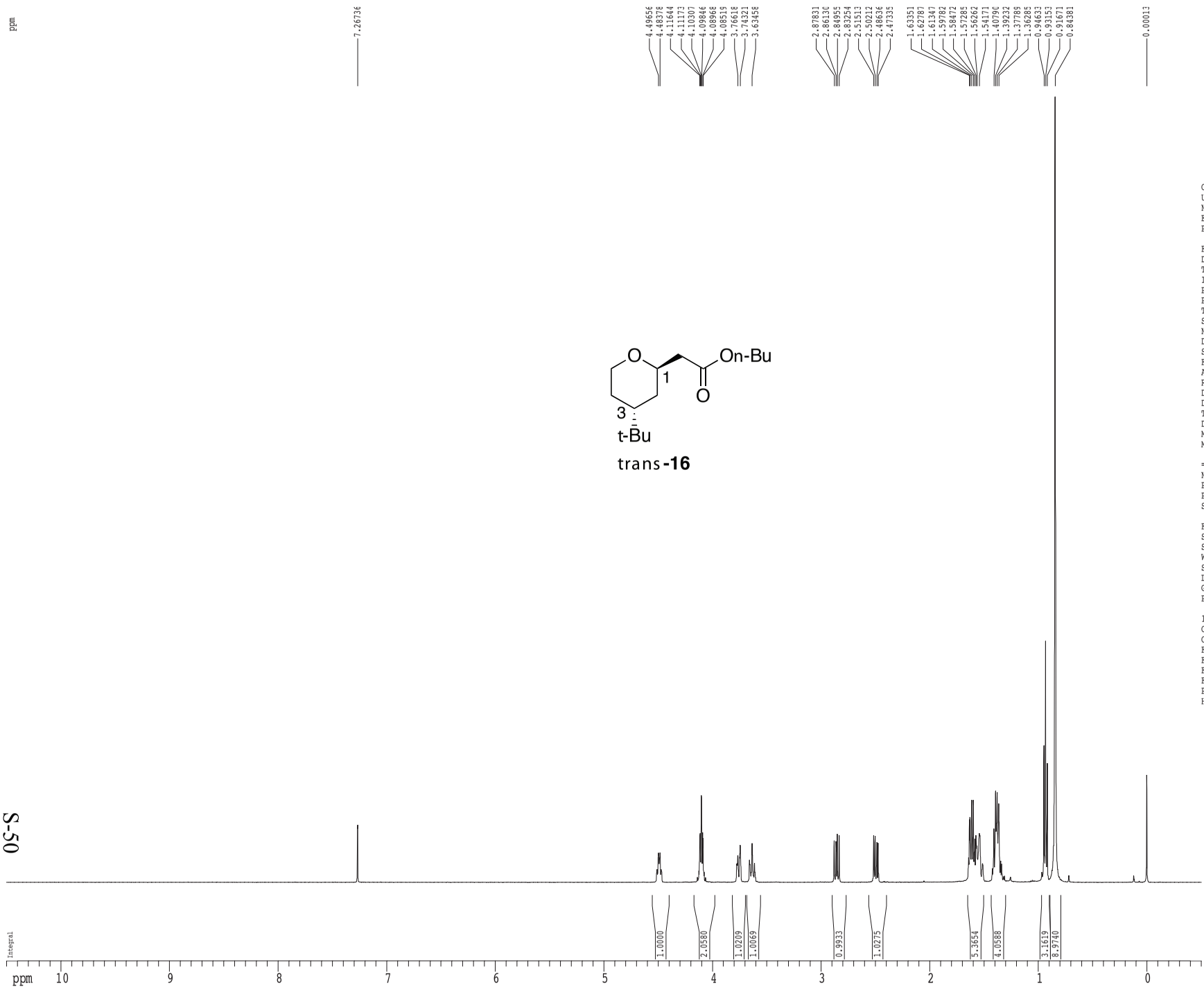
======== CHANNEL f1 ========
NUC1 13C
P1 15.00 usec
PL1 -1.00 dB
SFO1 125.7942548 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 23.54 dB
SFO2 500.2225011 MHz

F2 - Processing parameters
SI 65536
SF 125.7804020 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 230.000 ppm
F1 28929.49 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 10.52632 ppm/cm
HZCM 1324.00427 Hz/cm

WAS-III-166-27
1H spectrum



Current Data Parameters
USER walter
NAME WAS-III-166-27
EXPNO 3
PROCNO 1

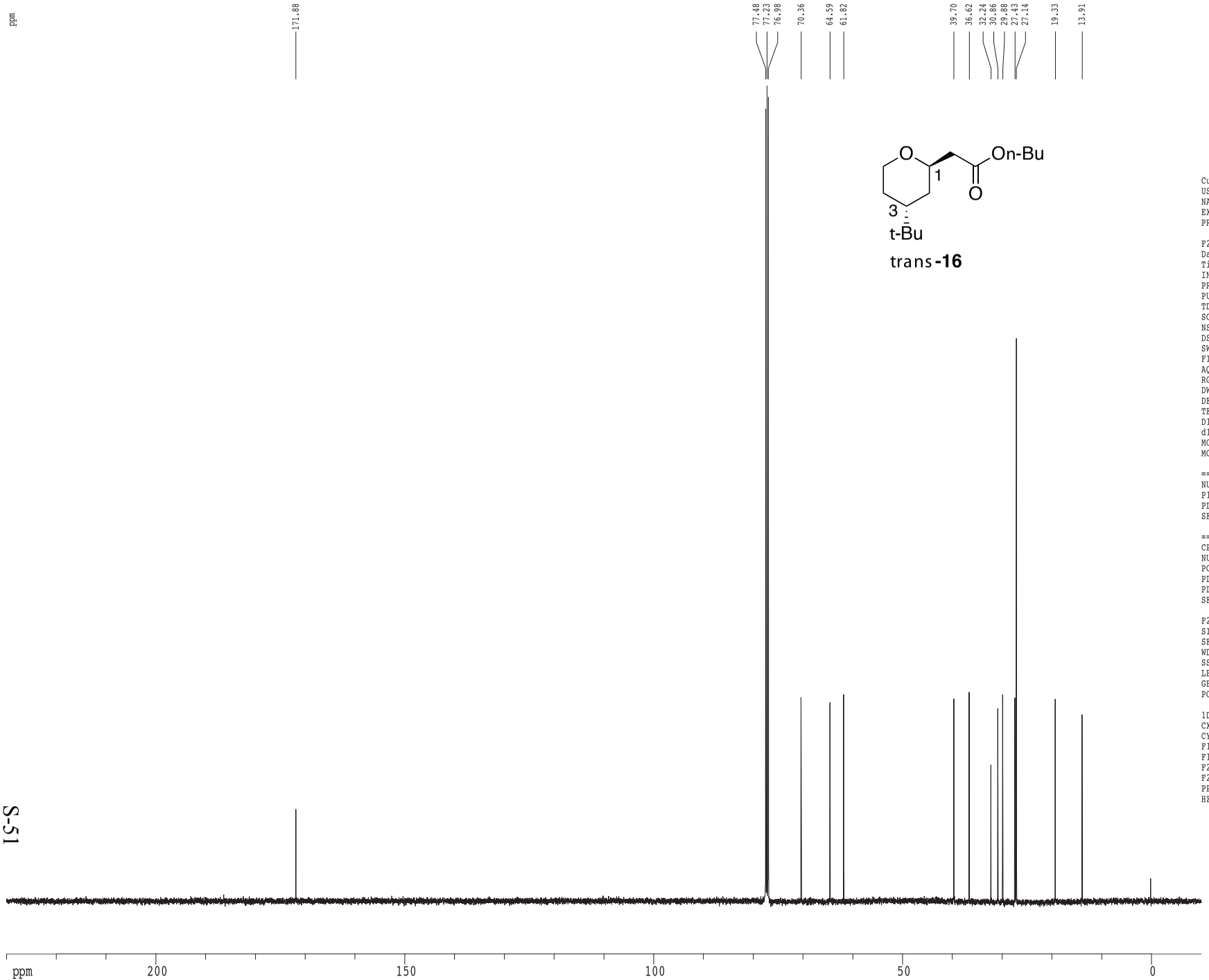
F2 - Acquisition Parameters
Date_ 20071002
Time 22.04
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3T
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0999398 sec
RG 4
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======= CHANNEL f1 =======
NUC1 1H
P1 8.00 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200275 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 10.500 ppm
F1 5252.31 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.48246 ppm/cm
HZCM 241.33423 Hz/cm

WAS-III-166-27
13C spectrum with 1H decoupling



Current Data Parameters
USER walter
NAME WAS-III-166-27
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20071002
Time 16.51
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65418
SOLVENT CDCl3T
NS 179
DS 0
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794635 sec
RG 13004
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

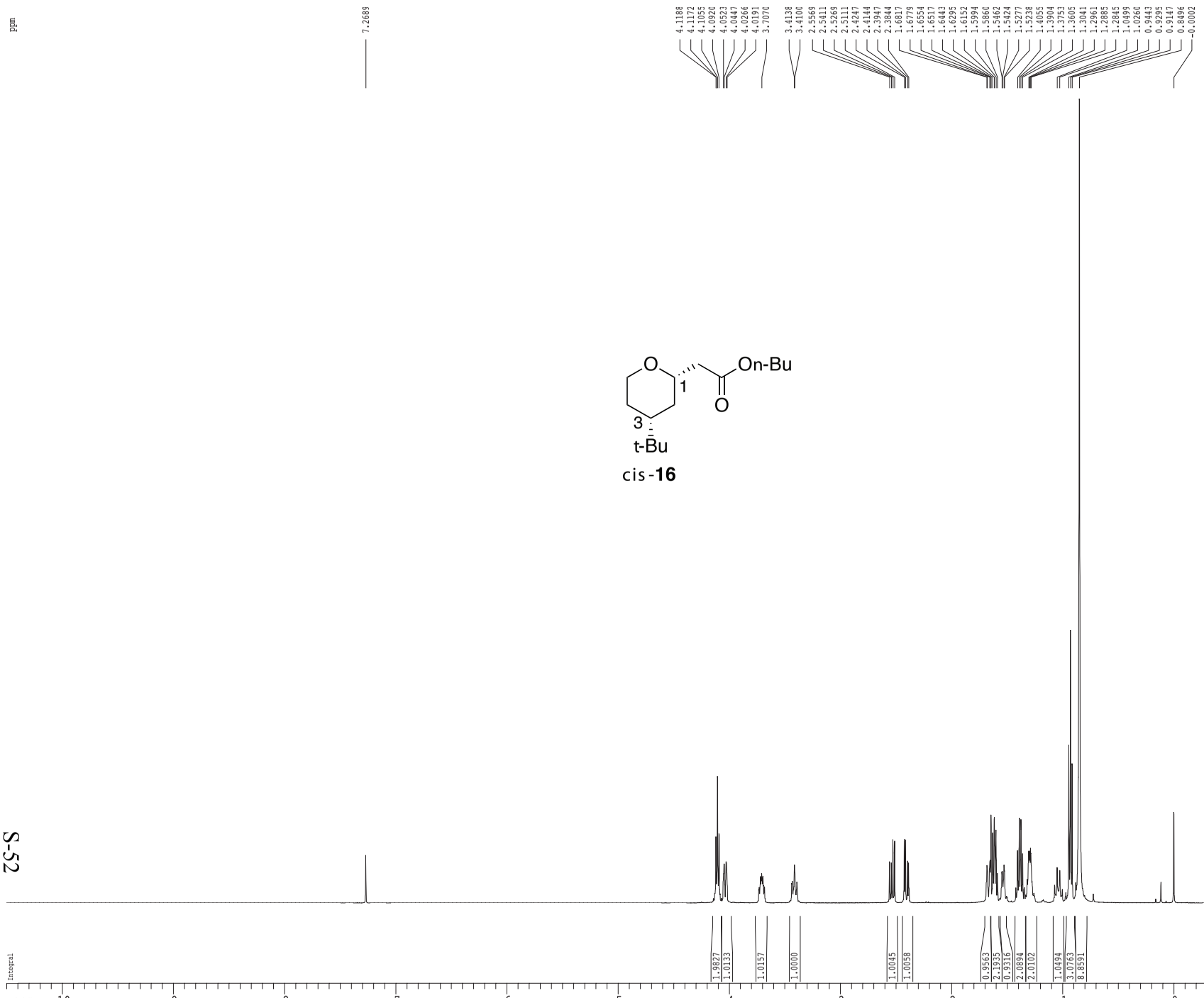
======== CHANNEL f1 ========
NUC1 13C
P1 15.00 usec
PL1 -1.00 dB
SFO1 125.7942548 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 23.54 dB
SFO2 500.2225011 MHz

F2 - Processing parameters
SI 65536
SF 125.7803992 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 230.000 ppm
F1 28929.49 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 10.52632 ppm/cm
HZCM 1324.00415 Hz/cm

WAS-III-166-26
1H spectrum



Current Data Parameters
USER walter
NAME WAS-III-166-26
EXPNO 5
PROCNO 1

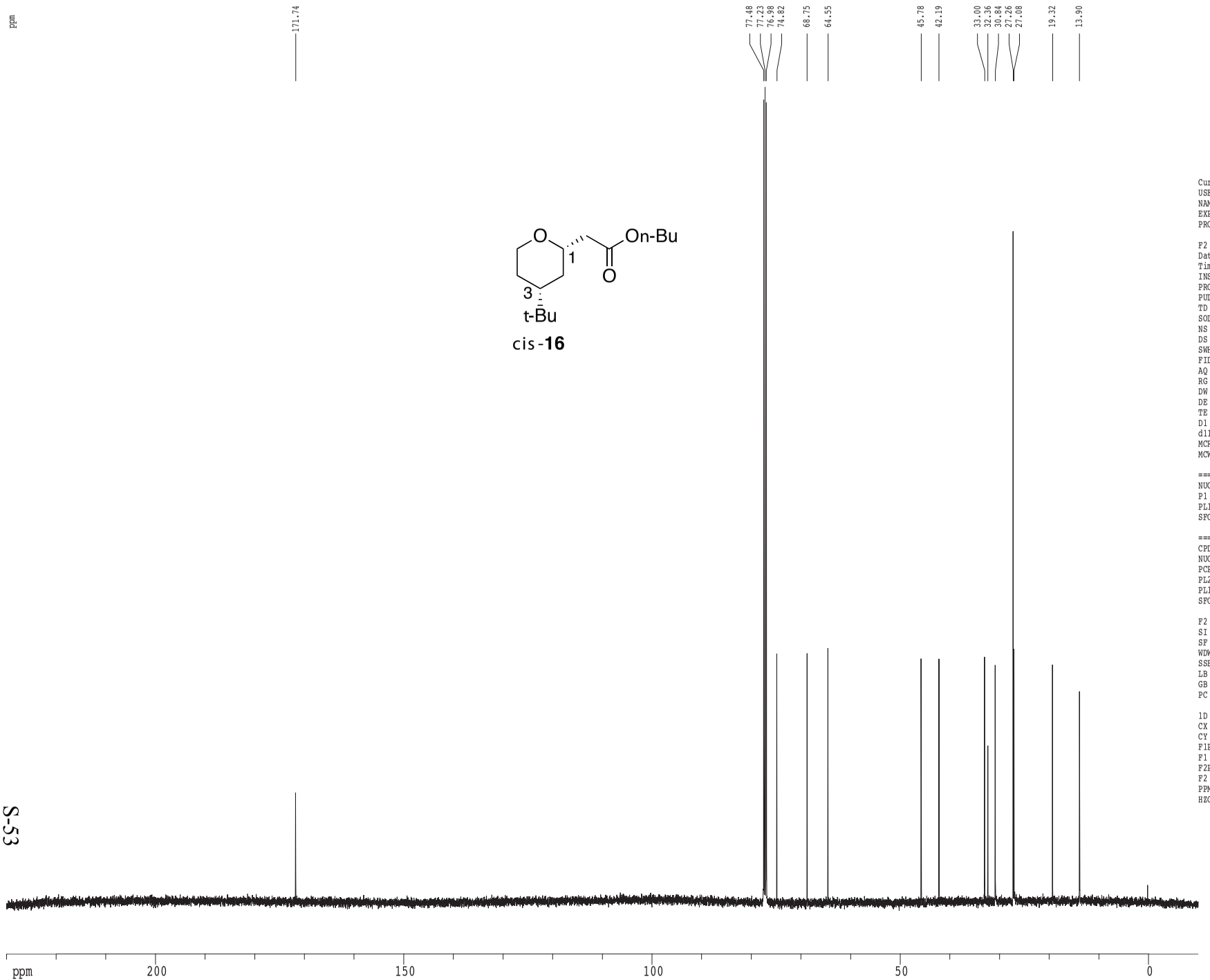
F2 - Acquisition Parameters
Date_ 20071002
Time 23.07
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3T
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0999398 sec
RG 4.5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 8.00 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200266 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 10.500 ppm
F1 5252.31 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.48246 ppm/cm
HZCM 241.33423 Hz/cm

WAS-III-166-26
13C spectrum with 1H decoupling



Current Data Parameters
USER walter
NAME WAS-III-166-26
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20071002
Time 16.32
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65536
SOLVENT CDCl3T
NS 77
DS 0
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0814105 sec
RG 13004
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======= CHANNEL f1 =======
NUC1 13C
P1 15.00 usec
PL1 -1.00 dB
SFO1 125.7942548 MHz

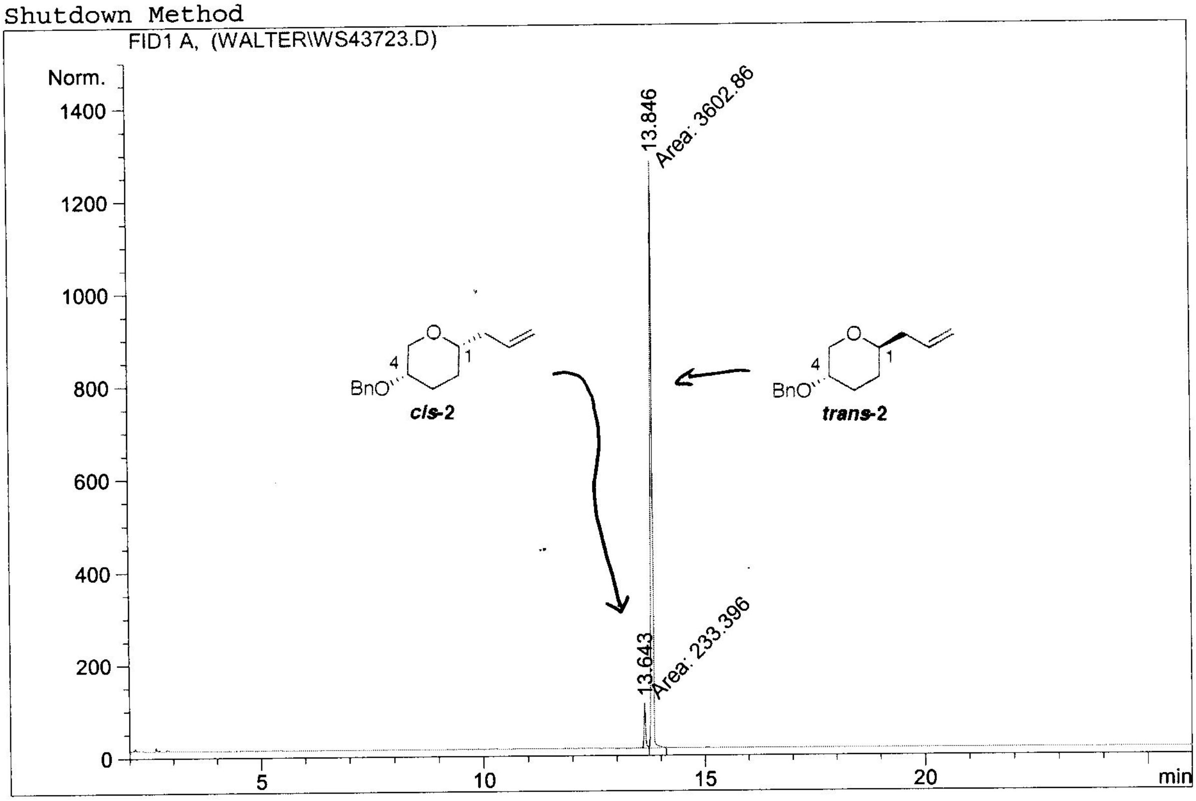
======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 23.54 dB
SFO2 500.2225011 MHz

F2 - Processing parameters
SI 65536
SF 125.7803997 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 230.000 ppm
F1 28929.49 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 10.52632 ppm/cm
HZCM 1324.00427 Hz/cm

Table 1, Entry 1

```
=====================================================================
Injection Date  : 5/2/2008 3:46:51 PM          Seq. Line :    7
Sample Name     : WAS-IV-37-23                   Location : Vial 20
Acq. Operator   : jelena                              Inj :    1
                                               Inj Volume : 1 µl
Different Inj Volume from Sequence !    Actual Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\1\METHODS\WALTER.M
Last changed    : 11/12/2005 3:11:30 PM by Susan
Analysis Method : C:\HPCHEM\1\METHODS\SHUTDOWN.M
Last changed    : 6/10/2008 9:37:46 AM by jelena
                  (modified after loading)
Shutdown Method
```




```
=====================================================================
                      Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000


Signal 1: FID1 A,

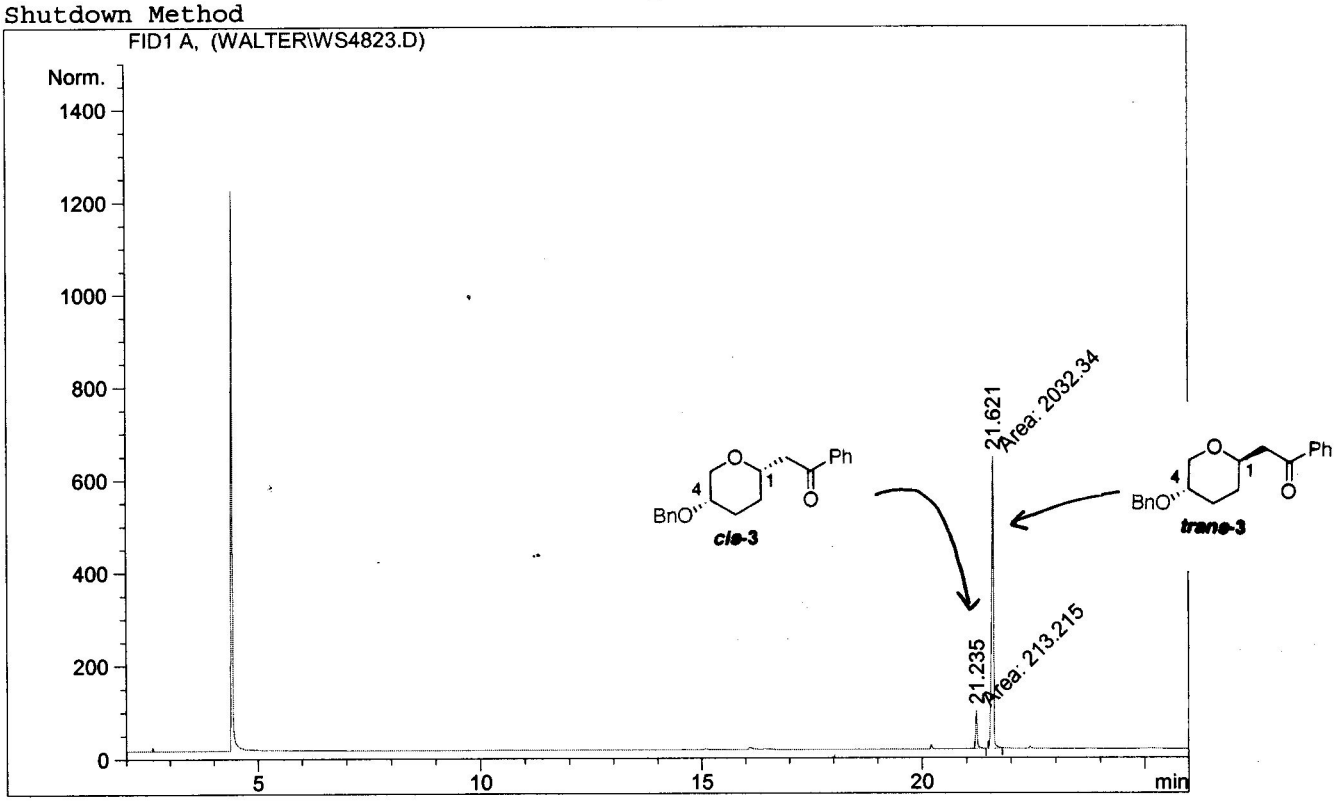
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1  13.643 MM    0.0399  233.39619   97.60922  6.08395
   2  13.846 MM    0.0473 3602.86206 1270.33093 93.91605

Totals :                  3836.25826 1367.94016

 Results obtained with enhanced integrator!
=====================================================================
                       *** End of Report ***
```

Table 1, Entry 2

```
==========================================================================
Injection Date   : 2/14/2008 7:31:59 PM          Seq. Line :   2
Sample Name      : WAS-IV-8-23                    Location : Vial 8
Acq. Operator    : jelena                             Inj :   1
                                                Inj Volume : 1 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 5 µl
Acq. Method      : C:\HPCHEM\1\METHODS\WALTER.M
Last changed     : 11/12/2005 3:11:30 PM by Susan
Analysis Method  : C:\HPCHEM\1\METHODS\SHUTDOWN.M
Last changed     : 6/10/2008 9:38:48 AM by jelena
                   (modified after loading)
Shutdown Method
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000


Signal 1: FID1 A,

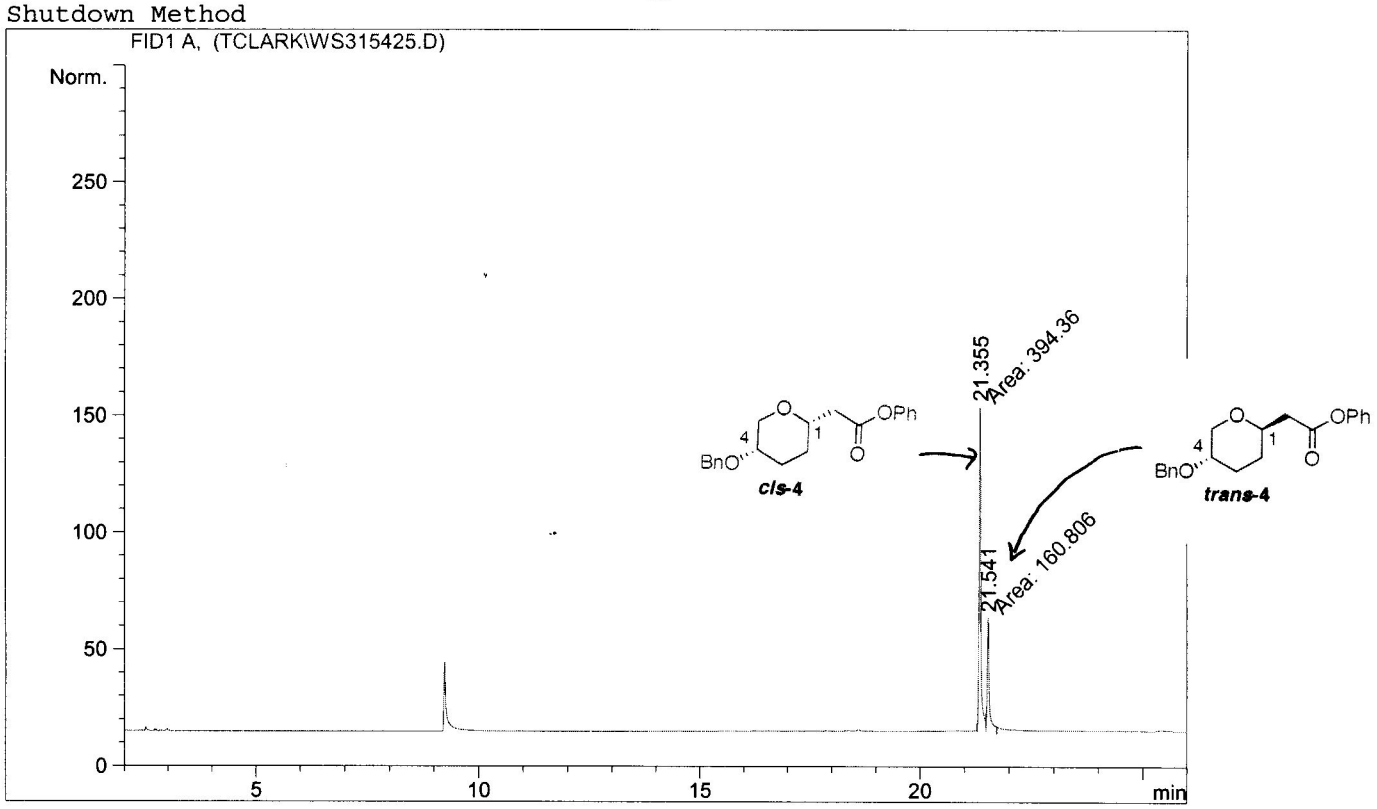
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1  21.235 MM    0.0430  213.21509   82.68630  9.49497
   2  21.621 MM    0.0537 2032.34253  630.24927 90.50503

Totals :                  2245.55762  712.93557

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

Table 1, Entry 3

```
==========================================================================
Injection Date  : 9/11/2007 1:57:12 PM          Seq. Line :   1
Sample Name     : WAS-III-154-25                 Location : Vial 4
Acq. Operator   : jelena                            Inj :   1
                                                Inj Volume : 1 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\1\METHODS\WALTER.M
Last changed    : 11/12/2005 3:11:30 PM by Susan
Analysis Method : C:\HPCHEM\1\METHODS\SHUTDOWN.M
Last changed    : 6/10/2008 9:47:47 AM by jelena
                  (modified after loading)
Shutdown Method
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000


Signal 1: FID1 A,

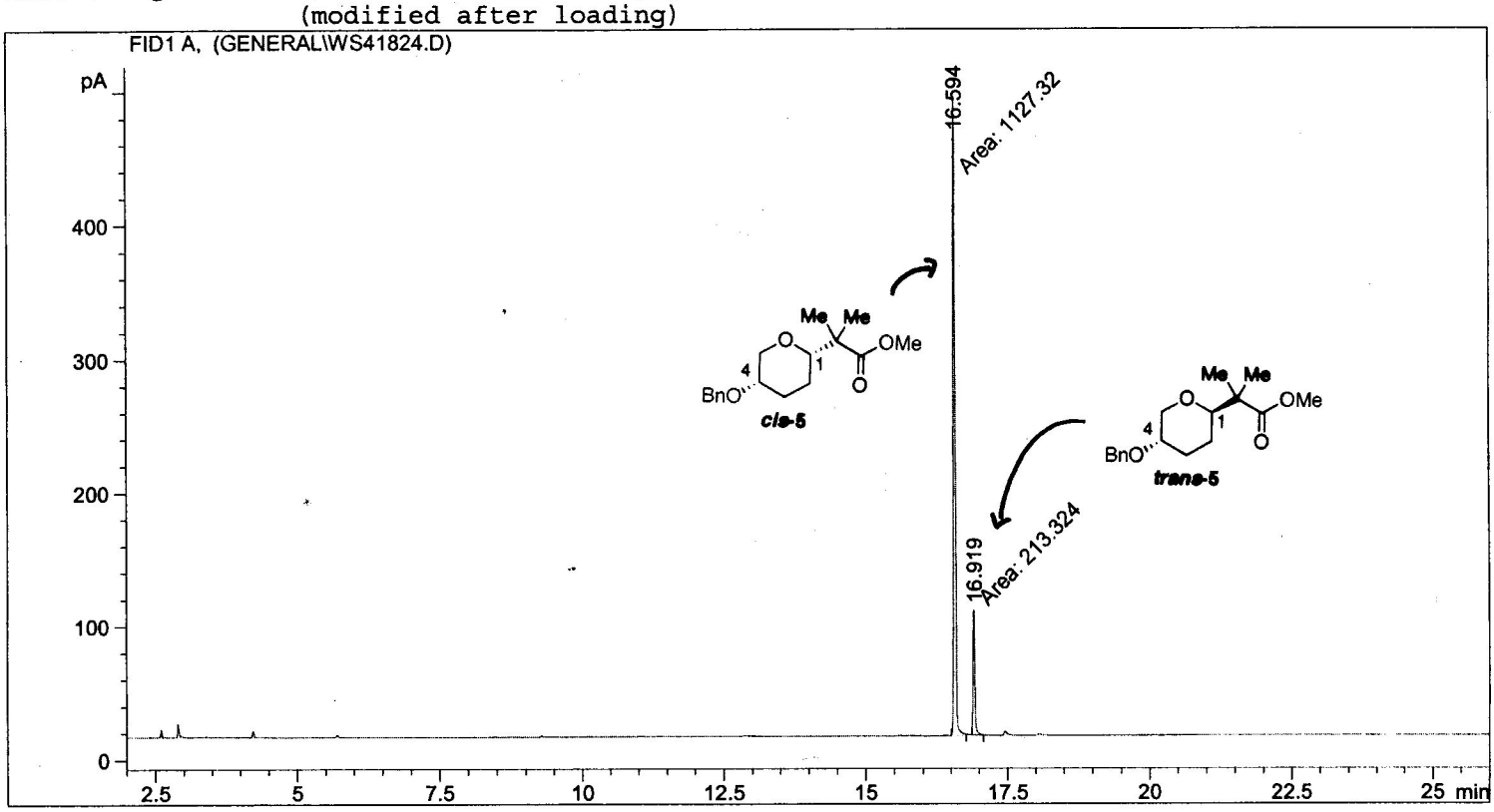
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1  21.355 MM    0.0474  394.36014  138.73720 71.03465
   2  21.541 MM    0.0551  160.80571   48.66586 28.96535

Totals :                   555.16585  187.40305

 Results obtained with enhanced integrator!
==========================================================================
                        *** End of Report ***
```

Table 1, Entry 4

```
Injection Date  : 3/3/2008 9:09:07 PM          Seq. Line :   2
Sample Name     : WAS-IV-18-24                 Location : Vial 2
Acq. Operator   : jelena                          Inj :   1
                                               Inj Volume : 1 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\1\METHODS\WALTER.M
Last changed    : 11/12/2005 3:11:30 PM by Susan
Analysis Method : C:\HPCHEM\1\METHODS\OFF.M
Last changed    : 3/4/2008 9:18:21 AM by jelena
                  (modified after loading)
```




```
                        Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1  16.594 MM    0.0392 1127.31763  478.89899 84.08791
   2  16.919 MM    0.0377  213.32408   94.20467 15.91209

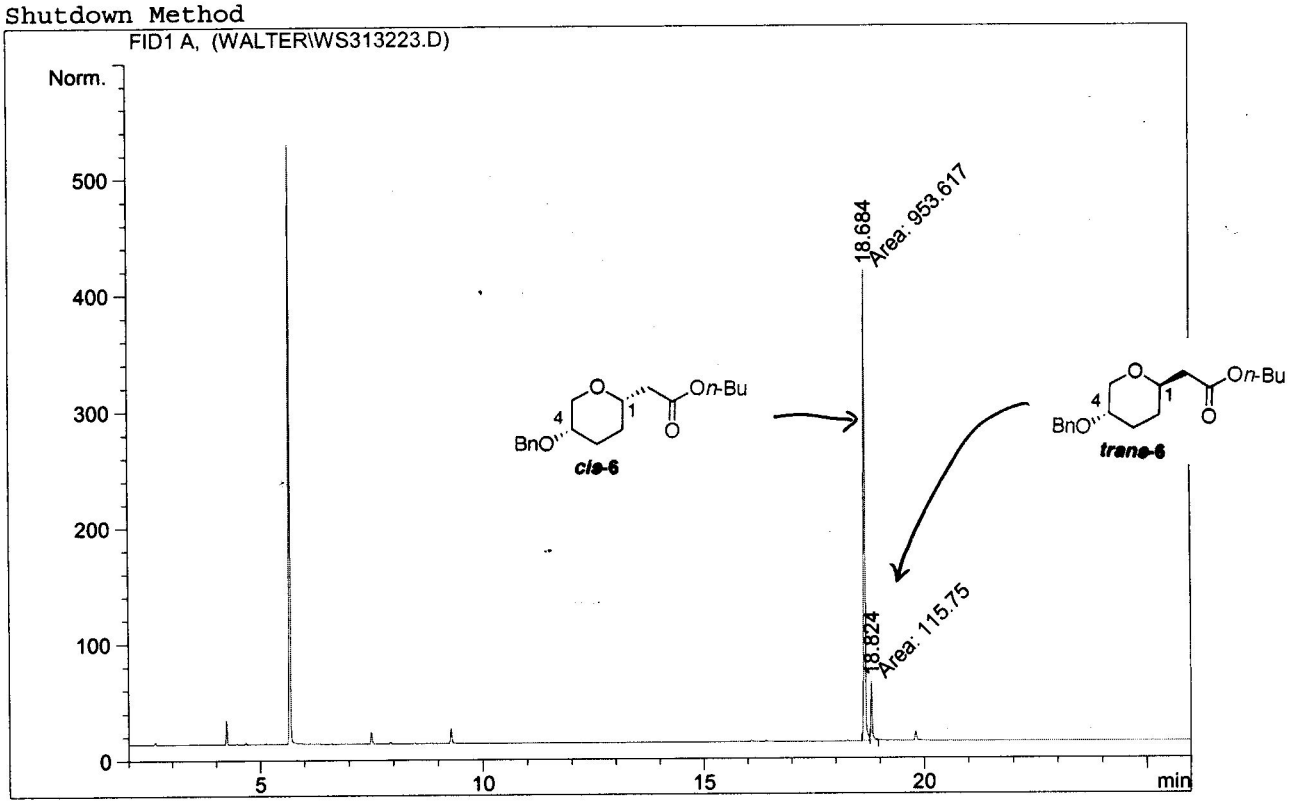
Totals :                  1340.64171  573.10366

 Results obtained with enhanced integrator!

                        *** End of Report ***
```

Table 1, Entry 5

```
==========================================================================
Injection Date  : 7/19/2007 3:19:51 PM          Seq. Line :   3
Sample Name     : WAS-III-132-23                 Location : Vial 17
Acq. Operator   : jelena                            Inj :   1
                                                Inj Volume : 1 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\1\METHODS\WALTER.M
Last changed    : 11/12/2005 3:11:30 PM by Susan
Analysis Method : C:\HPCHEM\1\METHODS\SHUTDOWN.M
Last changed    : 6/10/2008 9:41:43 AM by jelena
                  (modified after loading)
Shutdown Method
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FID1 A,

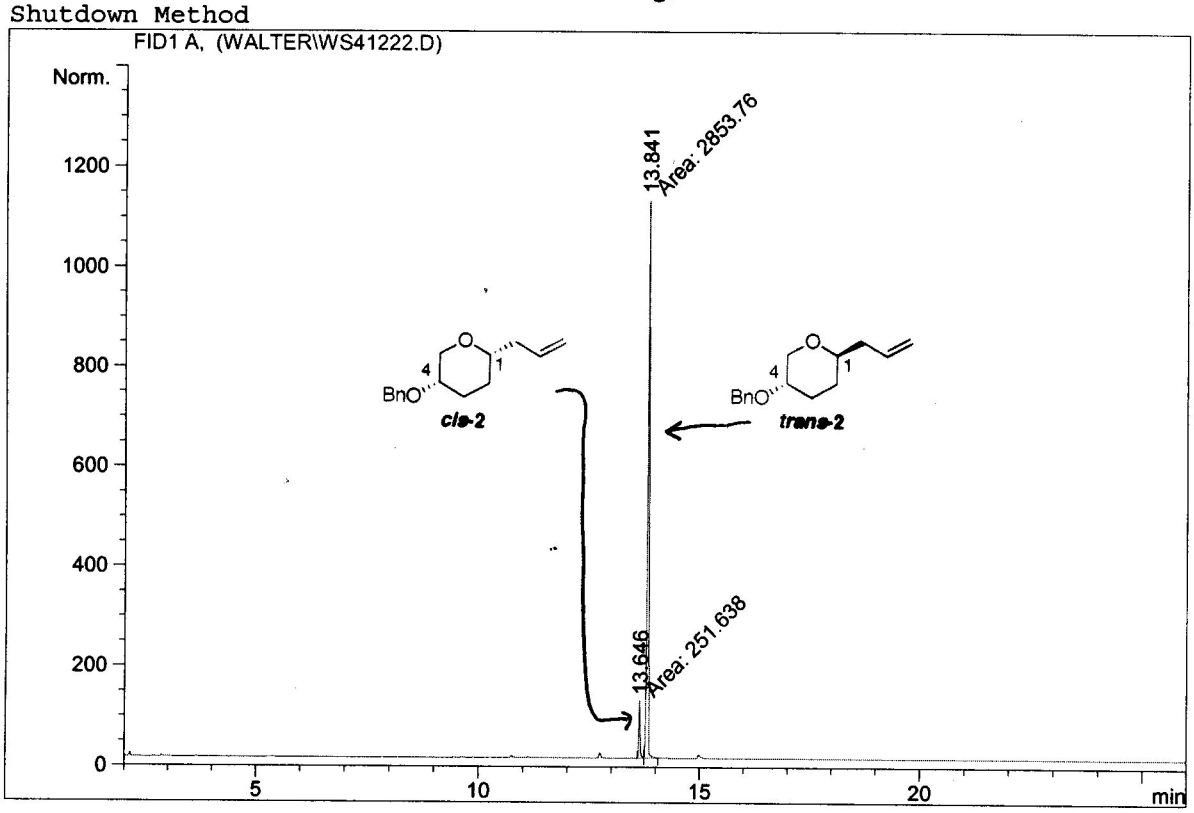
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1  18.684 MM    0.0393  953.61658  404.30756 89.17585
   2  18.824 MM    0.0384  115.74980   50.21415 10.82415

Totals :                  1069.36638  454.52170

 Results obtained with enhanced integrator!
==========================================================================
                        *** End of Report ***
```

Table 2, Entry 1

```
Injection Date   : 2/21/2008 10:22:15 PM          Seq. Line :   1
Sample Name      : WAS-IV-12-22                   Location : Vial 5
Acq. Operator    : jelena                            Inj :   1
                                                 Inj Volume : 1 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 5 µl
Acq. Method      : C:\HPCHEM\1\METHODS\WALTER.M
Last changed     : 11/12/2005 3:11:30 PM by Susan
Analysis Method  : C:\HPCHEM\1\METHODS\SHUTDOWN.M
Last changed     : 6/10/2008 9:29:12 AM by jelena
                   (modified after loading)
Shutdown Method
```




```
                          Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1  13.646 MM    0.0365  251.63829  114.98837  8.10325
   2  13.841 MM    0.0425 2853.76147 1119.56714 91.89675

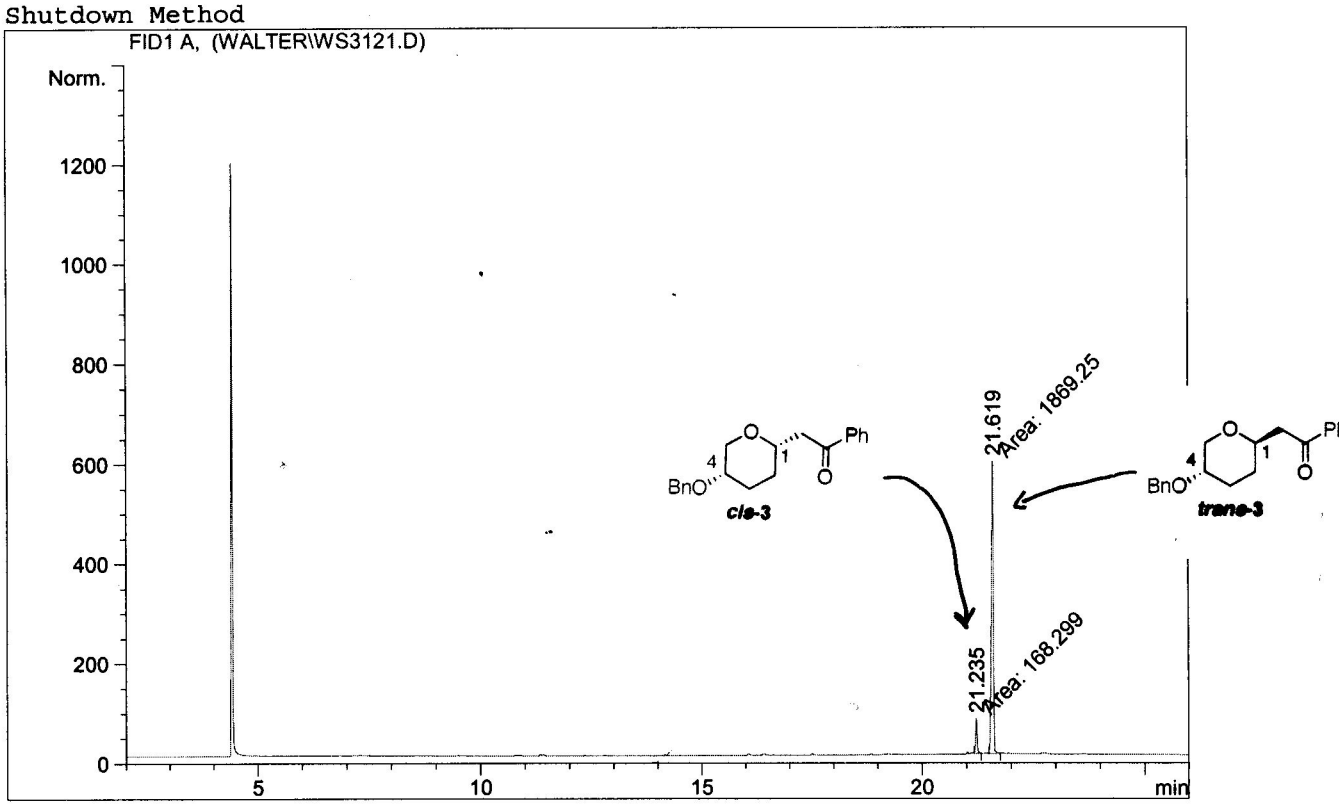
Totals :                  3105.39977 1234.55551

 Results obtained with enhanced integrator!

                          *** End of Report ***
```

Table 2, Entry 2

```
==========================================================================
Injection Date  : 6/22/2007 3:07:27 PM          Seq. Line :   3
Sample Name     : WAS-III-121                    Location : Vial 1
Acq. Operator   : jelena                            Inj :   1
                                                Inj Volume : 1 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\1\METHODS\WALTER.M
Last changed    : 11/12/2005 3:11:30 PM by Susan
Analysis Method : C:\HPCHEM\1\METHODS\SHUTDOWN.M
Last changed    : 6/10/2008 9:31:32 AM by jelena
                  (modified after loading)
Shutdown Method
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000


Signal 1: FID1 A,

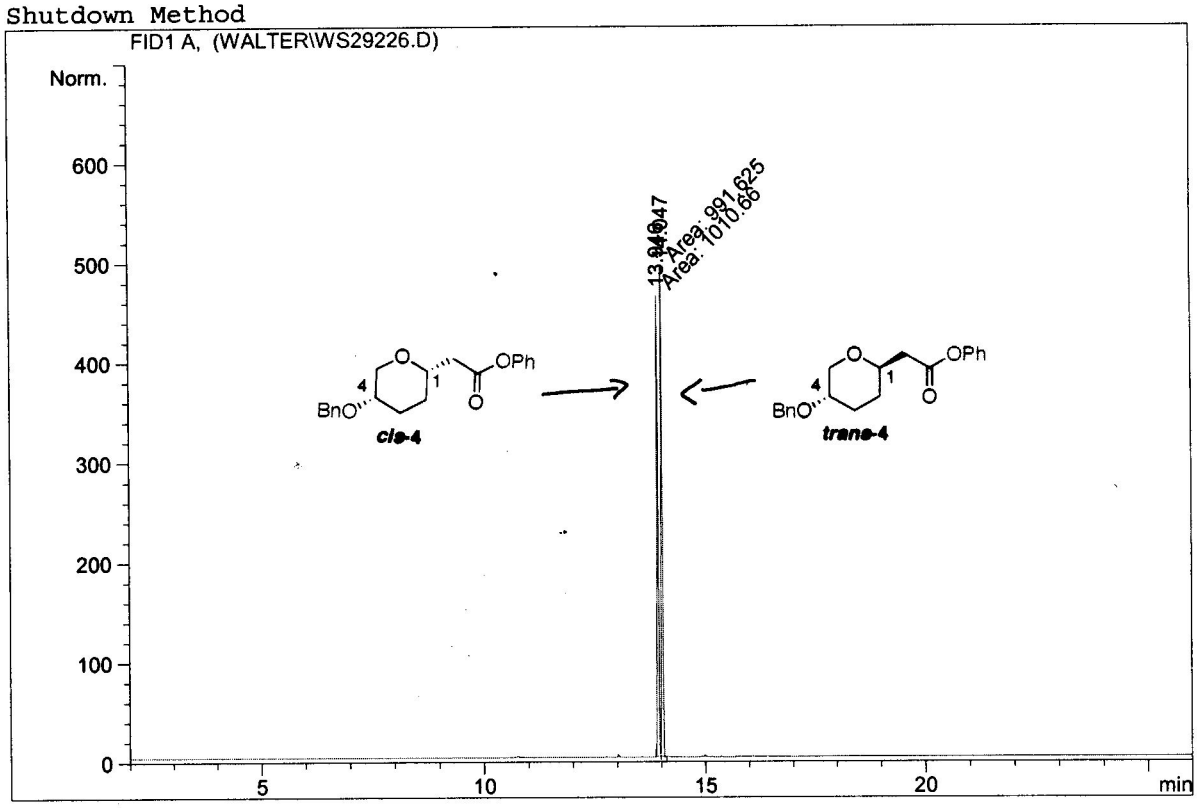
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1  21.235 MM    0.0401  168.29939   70.01180  8.25990
   2  21.619 MM    0.0533 1869.24854  584.89423 91.74010

Totals :                  2037.54793  654.90602

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

Table 2, Entry 3

```
Injection Date  : 11/4/2005 3:55:37 PM          Seq. Line :   5
Sample Name     : WAS-II-92-26                  Location : Vial 2
Acq. Operator   : jelena                           Inj :   1
                                               Inj Volume : 1 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\1\METHODS\WALTER.M
Last changed    : 7/28/2005 3:54:19 PM by jelena
Analysis Method : C:\HPCHEM\1\METHODS\SHUTDOWN.M
Last changed    : 6/10/2008 9:33:41 AM by jelena
                  (modified after loading)
Shutdown Method
```




```
                          Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1  13.946 MM    0.0364 1010.65692  462.99808 50.47527
   2  14.047 MM    0.0337  991.62451  490.82718 49.52473

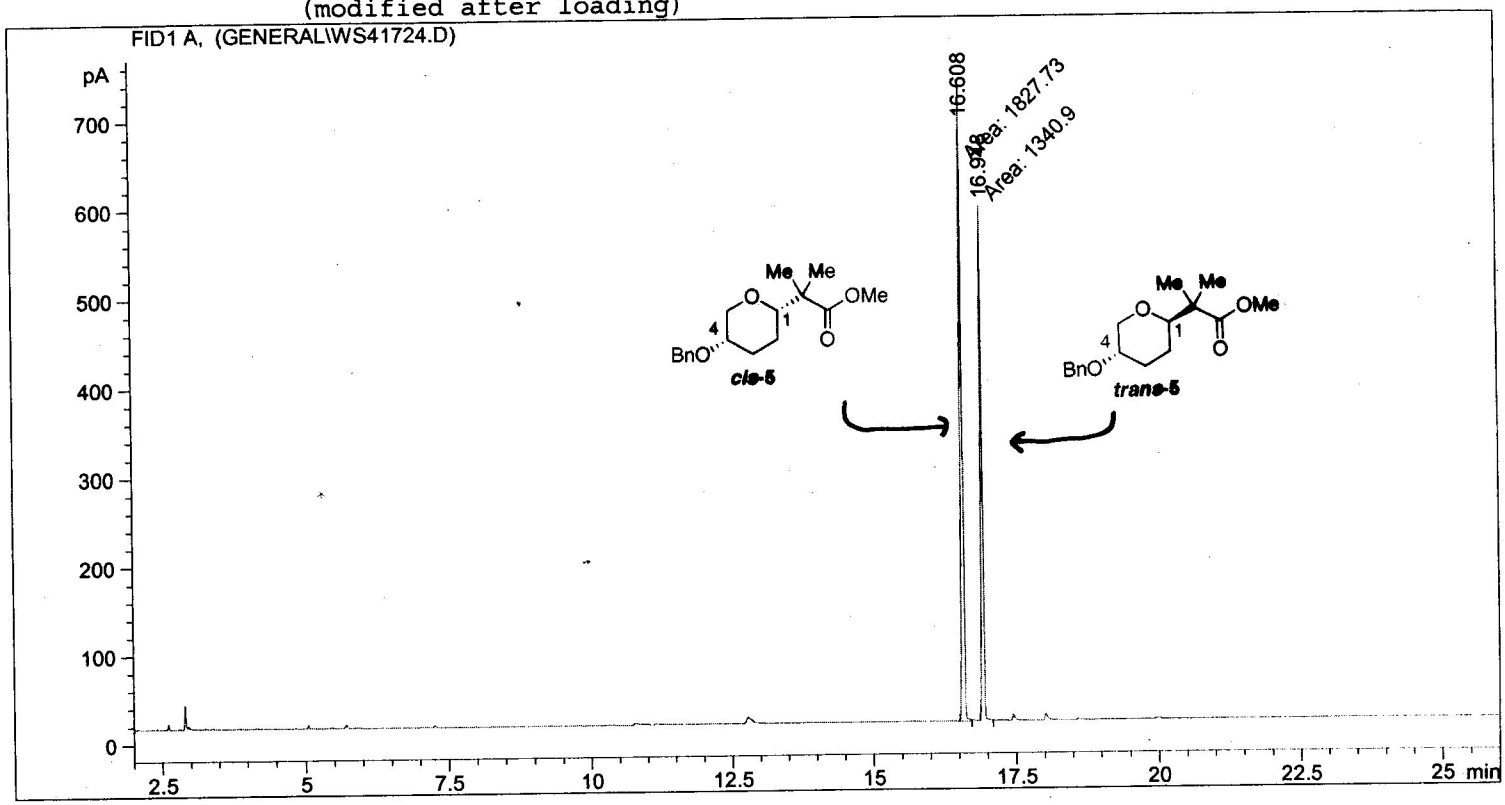
Totals :                  2002.28143  953.82526

 Results obtained with enhanced integrator!

                          *** End of Report ***
```

Table 2, Entry 4

```
Injection Date  : 3/3/2008 8:33:30 PM          Seq. Line :  1
Sample Name     : WAS-IV-17-24                  Location : Vial 1
Acq. Operator   : jelena                           Inj :  1
                                              Inj Volume : 1 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\1\METHODS\WALTER.M
Last changed    : 11/12/2005 3:11:30 PM by Susan
Analysis Method : C:\HPCHEM\1\METHODS\OFF.M
Last changed    : 3/4/2008 9:19:13 AM by jelena
                  (modified after loading)
```




```
                        Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [pA*s]     [pA]       %
----|-------|----|-------|----------|----------|--------|
   1  16.608 MM    0.0424 1827.72974  719.08203 57.68210
   2  16.948 MM    0.0385 1340.89600  580.03223 42.31790

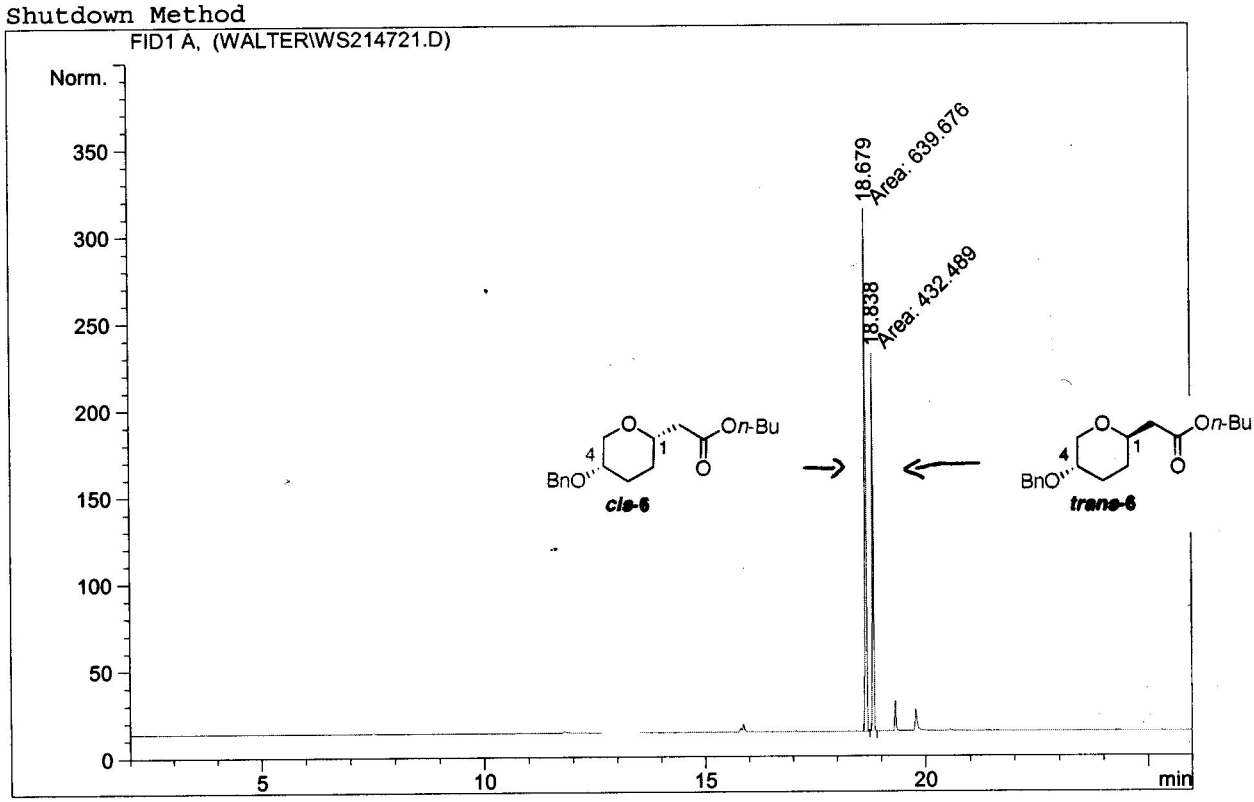
Totals :                  3168.62573 1299.11426

 Results obtained with enhanced integrator!

                        *** End of Report ***
```

```
==========================================================================
Injection Date  : 4/12/2006 1:30:20 PM          Seq. Line :   2
Sample Name     : WAS-II-147-21                  Location : Vial 13
Acq. Operator   : Jelena                            Inj :   1
                                                Inj Volume : 1 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 3 µl
Acq. Method     : C:\HPCHEM\1\METHODS\WALTER.M
Last changed    : 11/12/2005 3:11:30 PM by Susan
Analysis Method : C:\HPCHEM\1\METHODS\SHUTDOWN.M
Last changed    : 6/10/2008 9:36:06 AM by jelena
                  (modified after loading)
Shutdown Method
```

Table 2, Entry 5




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000


Signal 1: FID1 A,

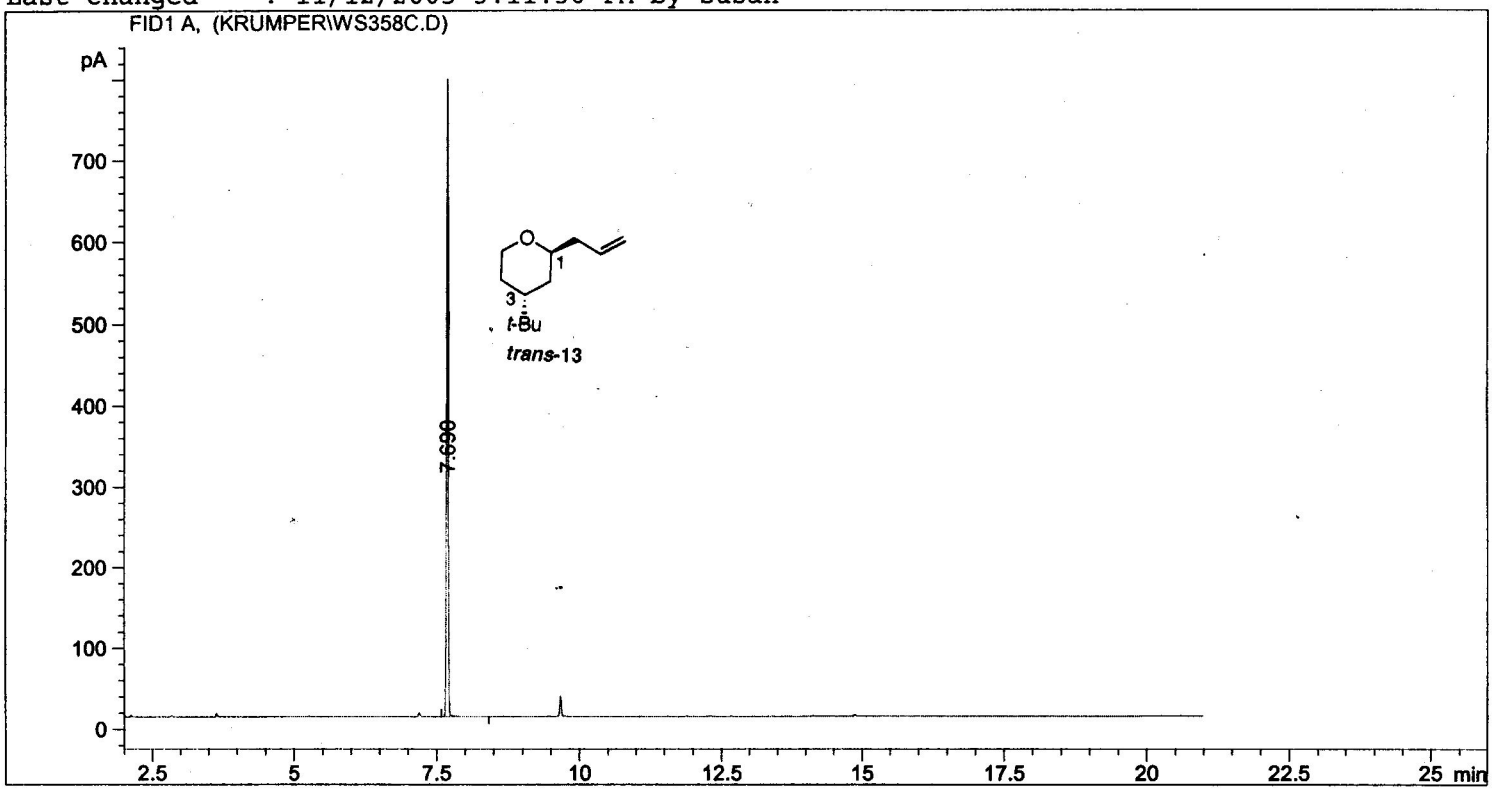
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1  18.679 MM    0.0355  639.67639  300.33771 59.66213
   2  18.838 MM    0.0332  432.48853  217.42862 40.33787

Totals :                  1072.16492  517.76633

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

Table 3, Entry 1

```
==========================================================================
Injection Date  : 2/5/2007 11:13:31 AM          Seq. Line :   1
Sample Name     : WAS-III-58-c                   Location : Vial 1
Acq. Operator   : jennifer                           Inj :   1
                                               Inj Volume : 1 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 5 µl
Sequence File   : C:\HPCHEM\1\SEQUENCE\KRUMPER.S
Method          : C:\HPCHEM\1\METHODS\WALTER.M
Last changed    : 11/12/2005 3:11:30 PM by Susan
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FID1 A,

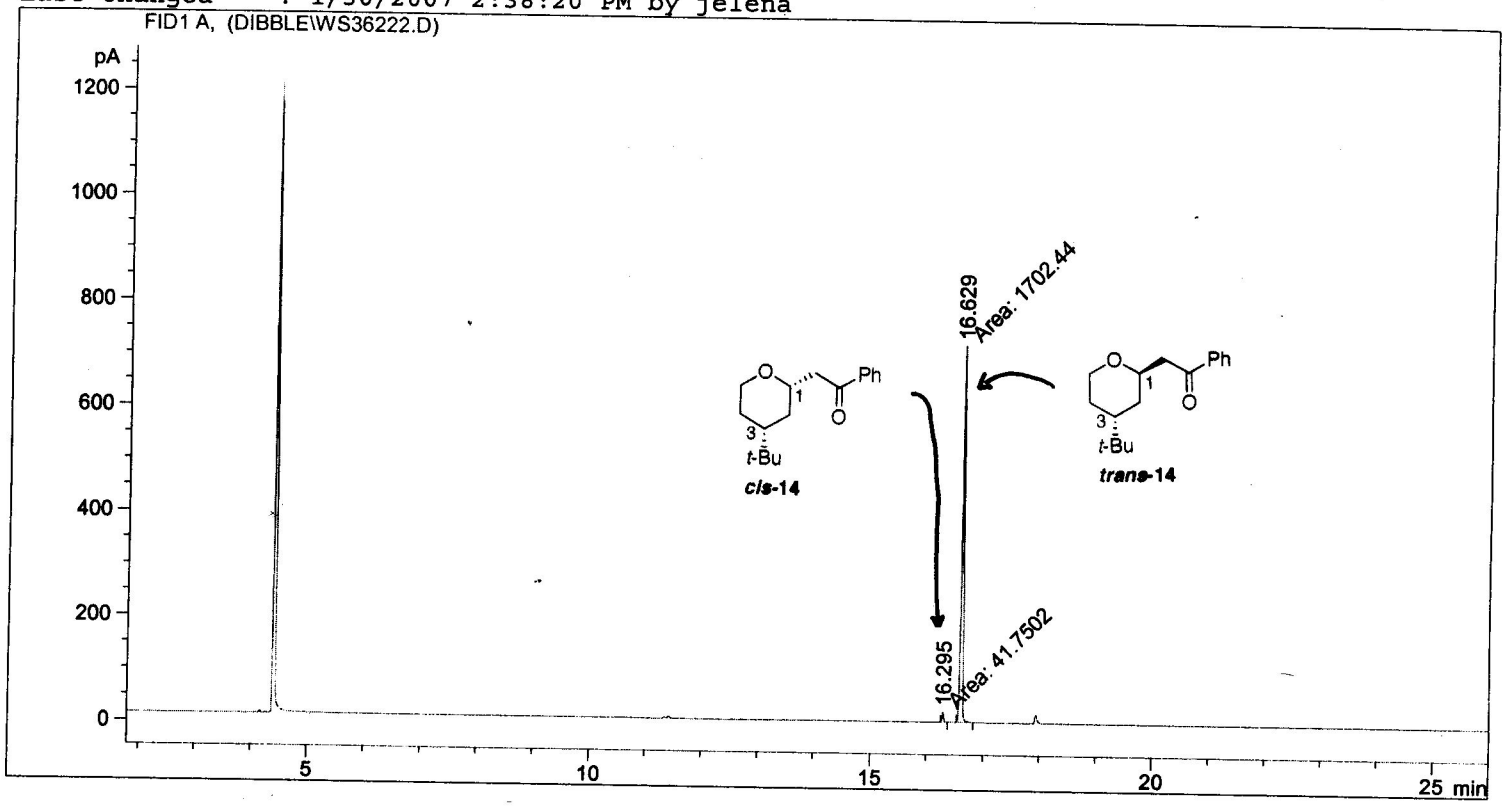
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1   7.690 BP    0.0875 1543.63904  294.10623 1.000e2

Totals :                  1543.63904  294.10623

 Results obtained with enhanced integrator!
==========================================================================
                        *** End of Report ***
```

```
==========================================================================
Injection Date   : 2/8/2007 5:43:13 PM          Seq. Line :   5
Sample Name      : WAS-III-62-22                 Location : Vial 11
Acq. Operator    : jennifer                          Inj :   1
                                                 Inj Volume : 1 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 5 µl
Acq. Method      : C:\HPCHEM\1\METHODS\WALTER.M
Last changed     : 11/12/2005 3:11:30 PM by Susan
Analysis Method  : C:\HPCHEM\1\METHODS\KRUMPER.M
Last changed     : 1/30/2007 2:38:20 PM by jelena
```

Table 3, Entry 2




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1  16.295 MM    0.0352   41.75023   19.78161  2.39368
   2  16.629 MM    0.0397 1702.43750  715.33228 97.60632

Totals :                  1744.18773  735.11388

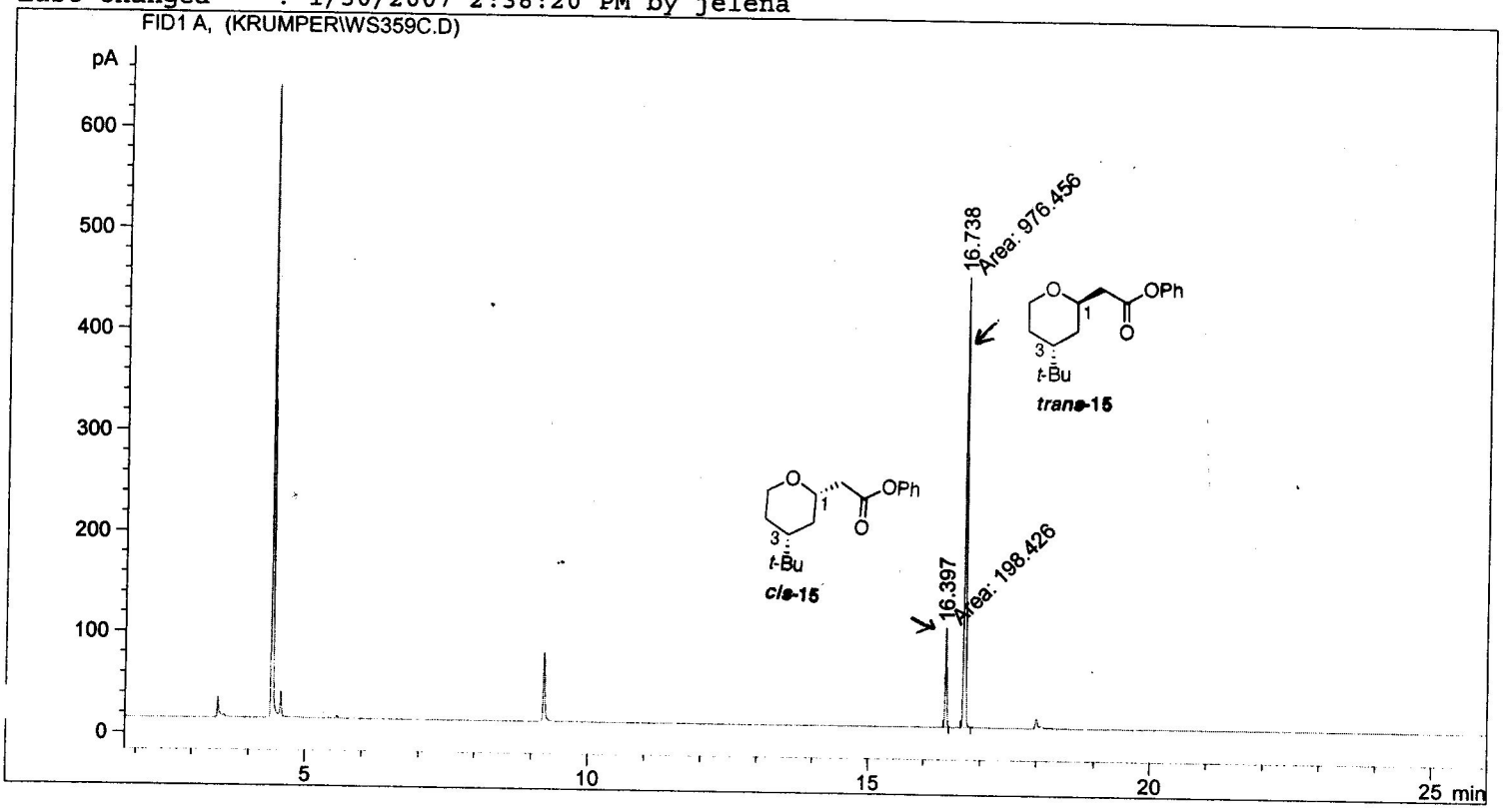
 Results obtained with enhanced integrator!
==========================================================================
                        *** End of Report ***
```

Injection Date : 2/5/2007 11:49:07 AM
Sample Name : WAS-III-59-c
Acq. Operator : jennifer

Seq. Line : 2
Location : Vial 2
Inj : 1
Inj Volume : 1 µl

Table 3, Entry 3

Different Inj Volume from Sequence ! Actual Inj Volume : 5 µl
Acq. Method : C:\HPCHEM\1\METHODS\WALTER.M
Last changed : 11/12/2005 3:11:30 PM by Susan
Analysis Method : C:\HPCHEM\1\METHODS\KRUMPER.M
Last changed : 1/30/2007 2:38:20 PM by jelena



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: FID1 A,

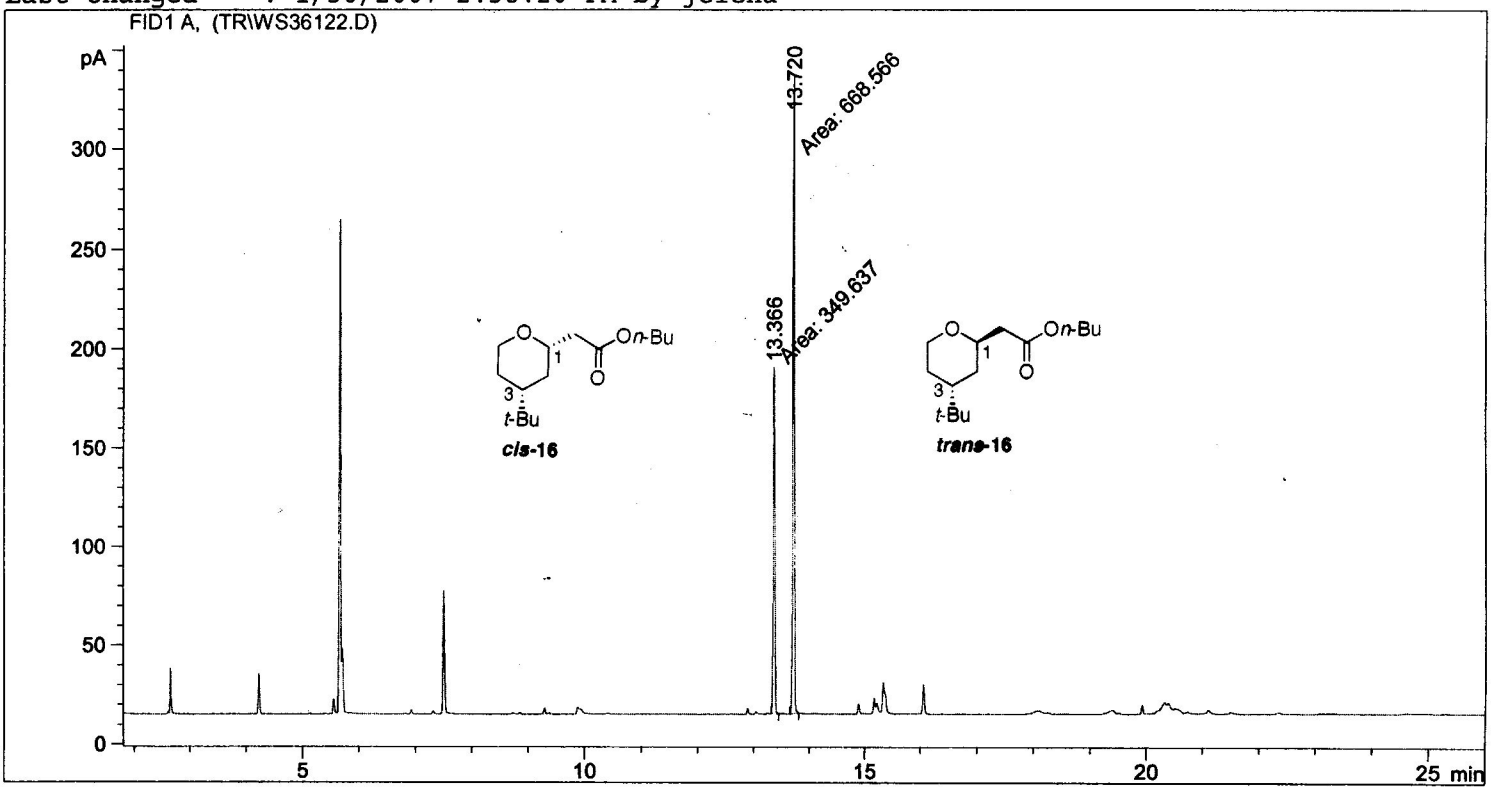
Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	16.397	MM	0.0338	198.42632	97.87888	16.88904
2	16.738	MM	0.0366	976.45593	444.12595	83.11096
Totals :				1174.88225	542.00483	

Results obtained with enhanced integrator!

*** End of Report ***

```
==================================================================
Injection Date  : 2/6/2007 5:33:45 PM          Seq. Line :   2
Sample Name     : WAS-III-61-22                Location : Vial 8
Acq. Operator   : jennifer                        Inj :   1
                                              Inj Volume : 1 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\1\METHODS\WALTER.M
Last changed    : 11/12/2005 3:11:30 PM by Susan
Analysis Method : C:\HPCHEM\1\METHODS\KRUMPER.M
Last changed    : 1/30/2007 2:38:20 PM by jelena
```

Table 3, Entry 4




```
==================================================================
                      Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]    [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1  13.366 MM    0.0333  349.63651  174.95299 34.33861
   2  13.720 MM    0.0348  668.56580  319.88483 65.66139

Totals :                  1018.20230  494.83781

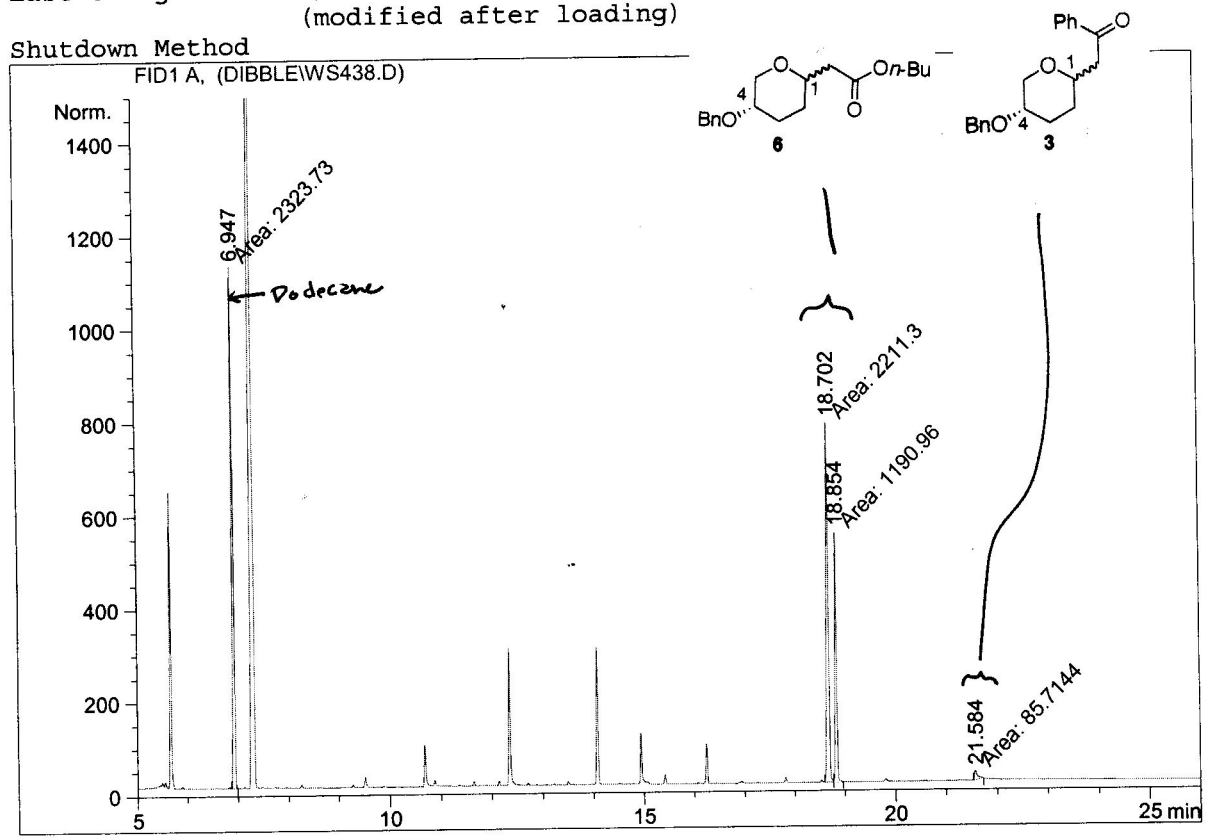
 Results obtained with enhanced integrator!
==================================================================
                      *** End of Report ***
```

Table 4, Entry 1

Injection Date : 5/20/2008 10:44:47 AM
Sample Name : WAS-IV-38
Acq. Operator : jelena
Seq. Line : 1
Location : Vial 2
Inj : 1
Inj Volume : 1 µl
Actual Inj Volume : 5 µl

Different Inj Volume from Sequence !
Acq. Method : C:\HPCHEM\1\METHODS\WALTER.M
Last changed : 11/12/2005 3:11:30 PM by Susan
Analysis Method : C:\HPCHEM\1\METHODS\SHUTDOWN.M
Last changed : 5/20/2008 11:49:48 AM by jelena
(modified after loading)

Shutdown Method



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

D	prod 6	prod 3
2323	3401	86
÷ 1.0	÷ 1.1	÷ 1.25
2323 :	3092 :	69

prod 6 : prod 3 = 98:2

GC yield prod 6 = 67%
prod 3 = 1%

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %	
1	6.947	MM	0.0346	2323.73486	1119.60278	39.98368	
2	18.702	MM	0.0479	2211.30249	769.95667	38.04910	← cis prod 6
3	18.854	MM	0.0370	1190.95605	536.26324	20.49236	← trans prod 6
4	21.584	MM	0.0728	85.71442	19.61160	1.47486	

cis-6 : trans-6
65 : 35

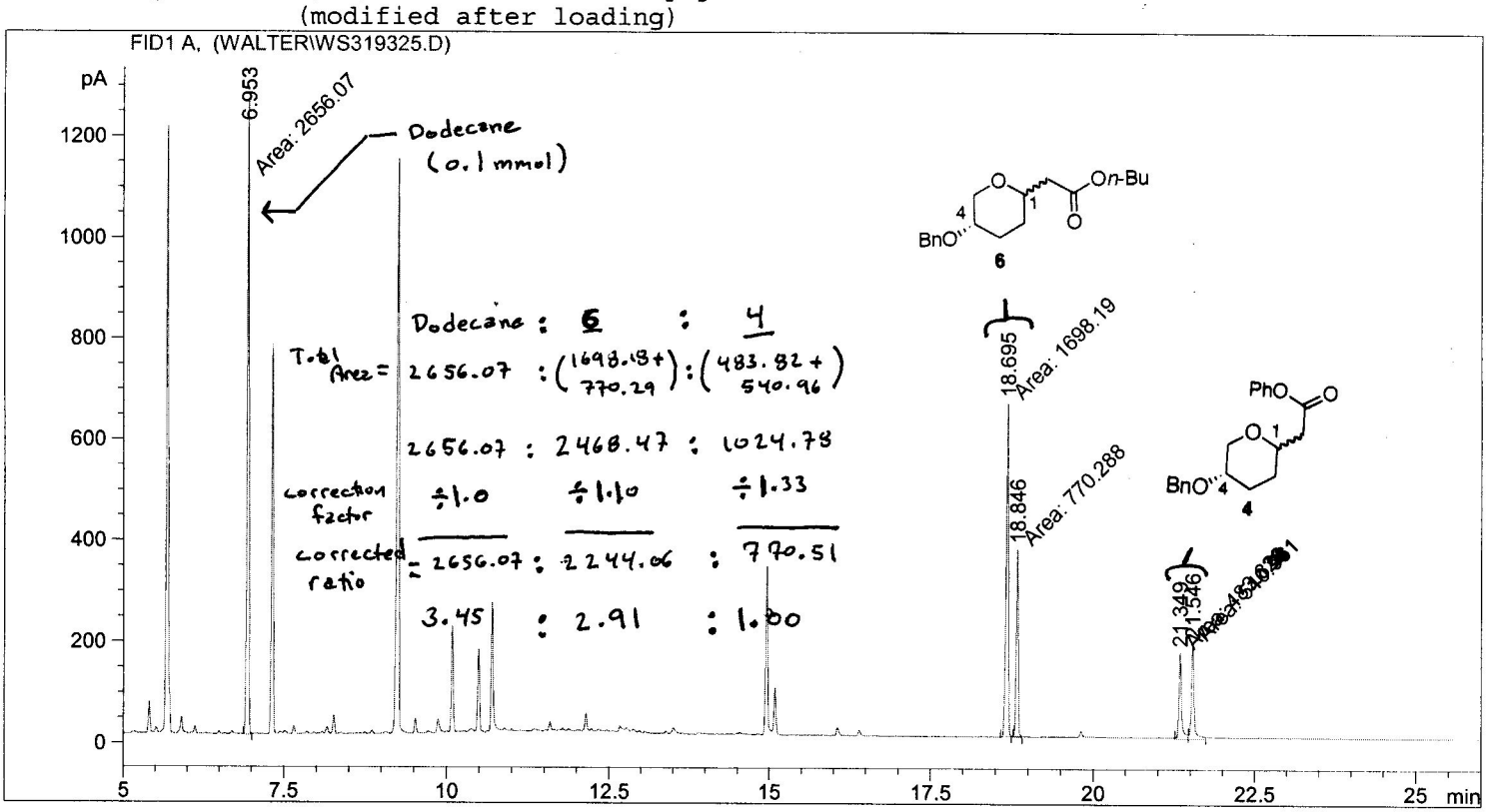
Totals : 5811.70783 2445.43429

Results obtained with enhanced integrator!

*** End of Report ***

Table 4, Entry 2

```
Injection Date    : 1/4/2008 6:25:26 PM          Seq. Line :   3
Sample Name       : WAS-III-193-24                Location : Vial 7
Acq. Operator     : jelena                           Inj :   1
                                                Inj Volume : 1 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 5 µl
Acq. Method       : C:\HPCHEM\1\METHODS\WALTER.M
Last changed      : 11/12/2005 3:11:30 PM by Susan
Analysis Method   : C:\HPCHEM\1\METHODS\BEAVER1.M
Last changed      : 5/14/2008 12:38:03 PM by jelena
                    (modified after loading)
```




```
Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [pA*s]     [pA]       %
----|-------|----|-------|----------|----------|--------|
   1   6.953 MM    0.0352 2656.06934 1258.35022 43.19284
   2  18.695 MM    0.0430 1698.18530  658.24811 27.61579
   3  18.846 MM    0.0347  770.28845  369.60751 12.52639
   4  21.349 MM    0.0476  483.82300  169.45120  7.86790
   5  21.546 MM    0.0483  540.96106  186.53477  8.79708

Totals :                  6149.32715 2642.19182

 Results obtained with enhanced integrator!

*** End of Report ***
```

Dodecane : 5 : 4

corrected ratio 3.45 : 2.91 : 1.00

	5	:	4
corrected ratio	74	:	26

Theor yield was 0.20 mmol
Based on Dodecane (0.10 mmol added)

GC Yield 5 = 42%
GC Yield 4 = 14%

← cis-5
← trans-5
← cis-4
← trans-4

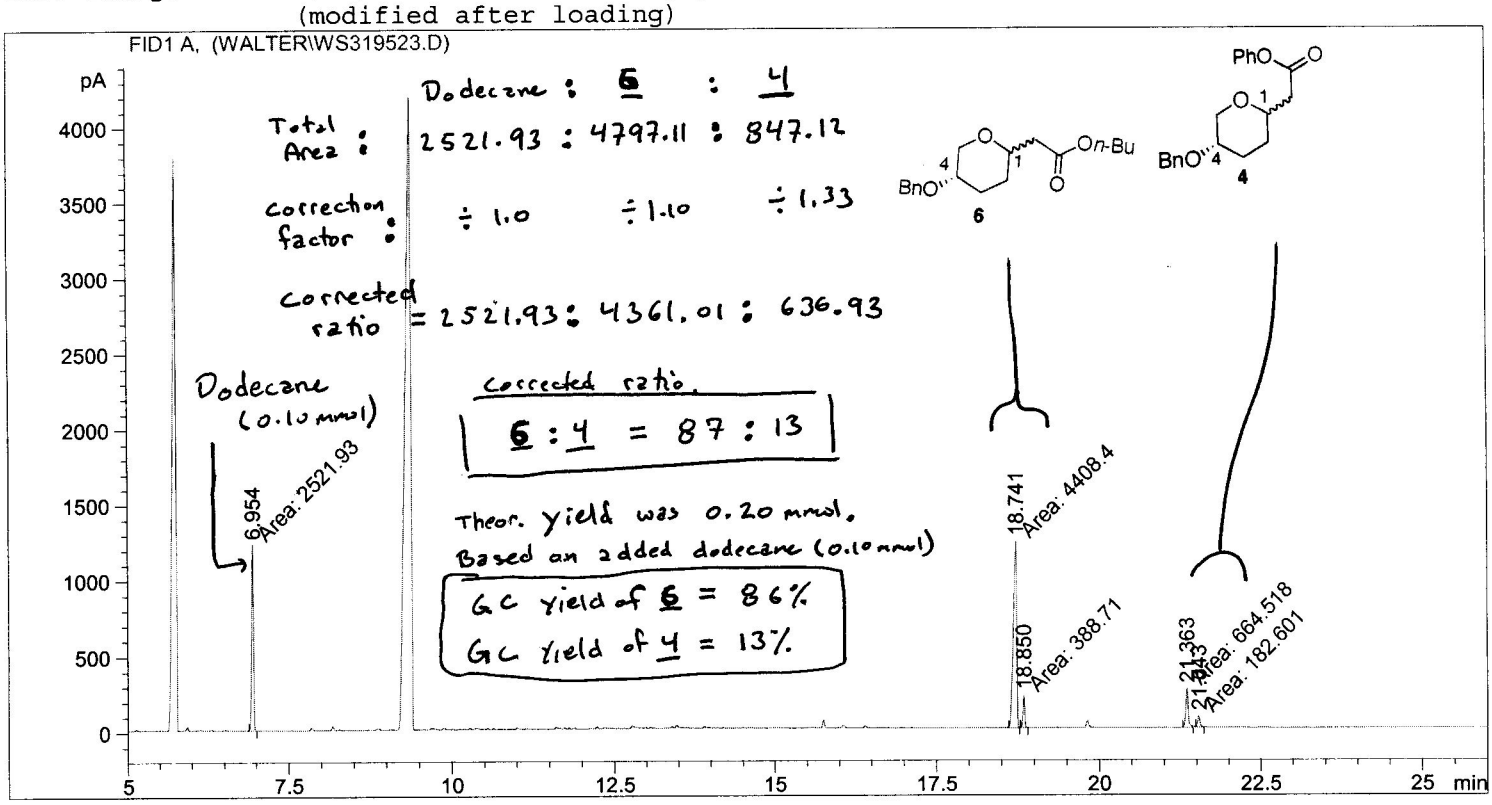
cis-5 : trans-5 = 69 : 31
cis-4 : trans-4 = 47 : 53

==========

Injection Date : 1/9/2008 6:14:17 PM Seq. Line : 1
Sample Name : WAS-III-195-23 Location : Vial 7
Acq. Operator : jelena Inj : 1
Inj Volume : 1 µl
Different Inj Volume from Sequence ! Actual Inj Volume : 5 µl

Table 4, Entry 3

Acq. Method : C:\HPCHEM\1\METHODS\WALTER.M
Last changed : 11/12/2005 3:11:30 PM by Susan
Analysis Method : C:\HPCHEM\1\METHODS\BEAVER1.M
Last changed : 5/14/2008 12:39:43 PM by jelena
(modified after loading)



==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %	
1	6.954	MM	0.0344	2521.93359	1221.22583	30.88273	
2	18.741	MM	0.0599	4408.39844	1226.80090	53.98373	← cis-5
3	18.850	MM	0.0307	388.71021	211.15019	4.76001	← trans-5
4	21.363	MM	0.0431	664.51801	256.71835	8.13746	← cis-4
5	21.543	MM	0.0413	182.60135	73.71474	2.23607	← trans-4
Totals :				8166.16159	2989.61002		

cis-6 : trans-6 = 92 : 8
cis-4 : trans-4 = 78 : 22

Results obtained with enhanced integrator!

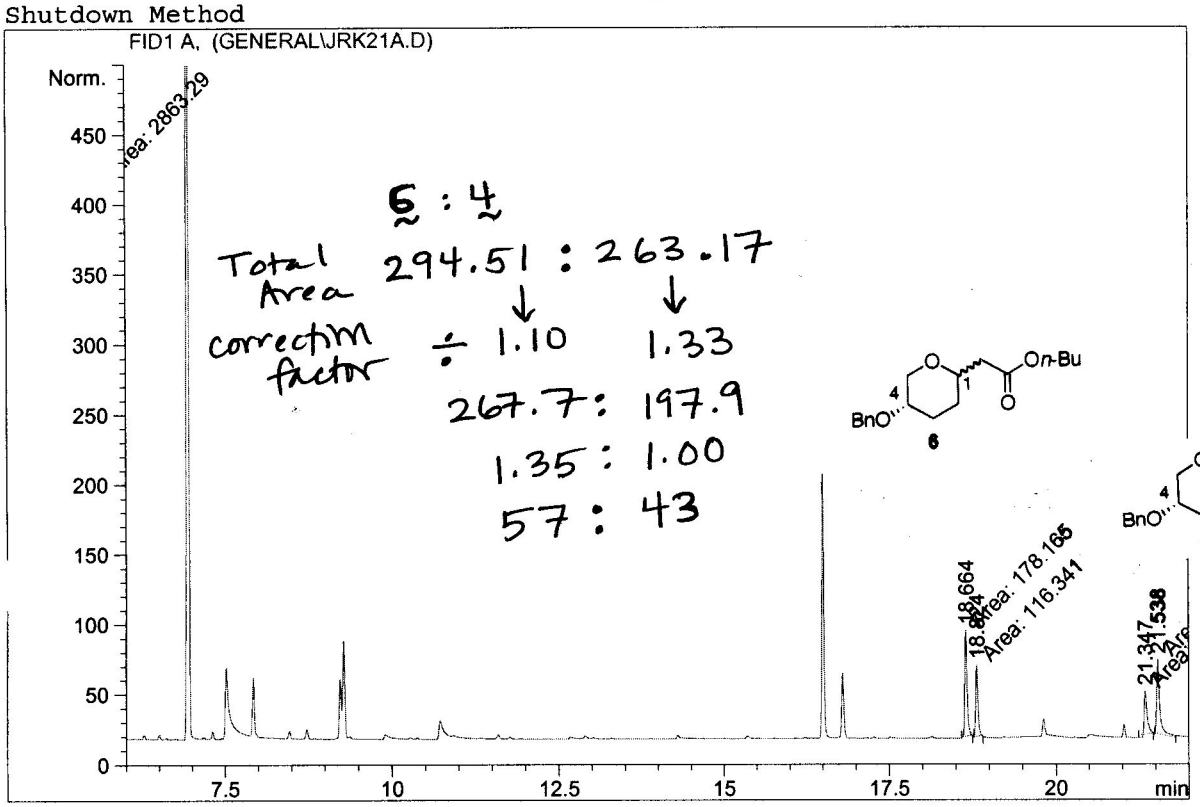
==========

*** End of Report ***

jrk-2-21.a crude GC after workup with dodecane IS

```
==========================================================================
Injection Date  : 2/1/2008 5:55:12 PM          Seq. Line :   1
 ample Name     : jrk-2-21a                    Location : Vial 21
.cq. Operator   : jelena                          Inj :   1
                                              Inj Volume : 1 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\1\METHODS\KRUMPER.M
Last changed    : 9/5/2007 11:13:31 AM by jelena
Analysis Method : C:\HPCHEM\1\METHODS\SHUTDOWN.M
Last changed    : 5/20/2008 12:24:08 PM by jelena
                  (modified after loading)
Shutdown Method
```

eq 1




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]      [min]    [pA*s]       [pA]       %
----|-------|----|-------|----------|----------|--------|
   1   5.698 MM    0.0354 2863.28760 1349.37781 83.69802
   2  18.664 MM    0.0396  178.16505   75.03540  5.20802
   3  18.824 MM    0.0382  116.34081   50.79527  3.40081
   4  21.347 MM    0.0500   91.66496   30.53539  2.67950
   5  21.538 MM    0.0541  171.51556   52.82352  5.01365

Totals :                  3420.97398 1558.56738

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

cis-5 ← 18.664 MM 178.16505 75.03540 5.20802
trans-5 ← 18.824 MM 116.34081 50.79527 3.40081
} cis : trans 60 : 40

cis-4 ← 21.347 MM 91.66496 30.53539 2.67950
trans-4 ← 21.538 MM 171.51556 52.82352 5.01365
} cis : trans ≈ 35 : 65