Remote Control of Diastereoselectivity in Intramolecular Reactions of Chiral Allylsilanes

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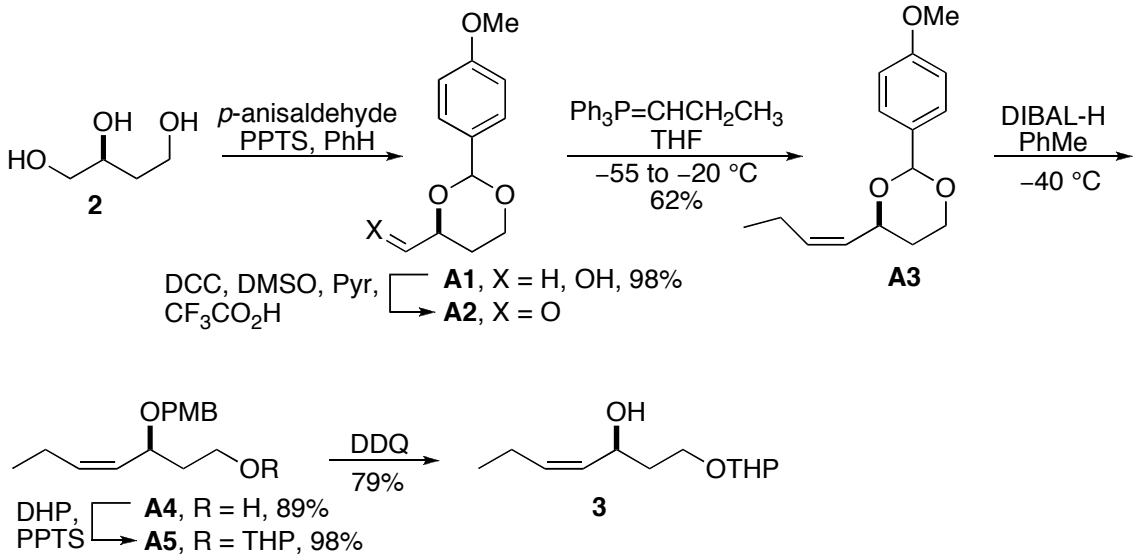
List of Known Compounds

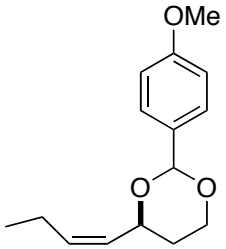
The following compounds are known: (*E*)-5-(trimethylsilyl)pent-3-en-1-ol (**D4**),[1] 1-chloro-2,2,2-trimethyl-1,1-diphenyldisilane,[2], (4*S*)-2-(4'-methoxyphenyl)-1,3-dioxan-4-ylmethanol (**A1**),[3] (4*S*)-2-(4'-methoxyphenyl)-1,3-dioxane-4-carbaldehyde (**A2**),[3] but-3-enyl pivalate (**1.30**),[4] 1-benzyloxy-3-butyne (**6**),[5] 1-benzyloxy-5-hexyne (**B2**),[6] 1-tert-butyldiphenylsilyloxy-3-butyne (**B3**),[7] 4-methoxy-benzyloxy-3-butyne (**B4**),[8] and ethyl 4-(*N-tert*-butoxycarbonylamino)-3-formylbenzoate.[1]

Preparation of Chiral Allylic Alcohols

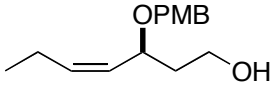
Allyic alcohol **3** was prepared from the *p*-methoxybenzyl acetonide of (*S*)-(–)-1,2,4-butanetriol (**A1**)[3] (Scheme A). Moffatt oxidation[9] of **A1** afforded aldehyde **A2**,[3] which was used without further purification. The olefin moiety was then installed via a Wittig reaction to give a 12.5:1 mixture of Z/E isomers that were separable by chromatography. DIBAL-H reduction of the major Z isomer **A3** followed by protecting group manipulations afforded allylic alcohol **3**.

Scheme A. *Synthesis of allylic alcohol* **3**.



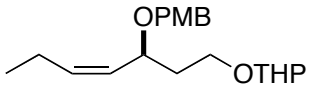


(3*S*,*Z*)-4-But-1-enyl-2-(4'-methoxyphenyl)[1,3]dioxane (A3). *n*-BuLi (1.0 M, 22.7 mL) was added to a solution of propyltriphenylphosphonium bromide (11.6 g, 30.3 mmol) in THF (60 mL) at 0 °C. After stirring for 20 min, the reaction mixture was cooled to –78 °C, and aldehyde **A2** was added as a solution in THF (20 mL). The reaction mixture was allowed to slowly warm to –20 °C, and was stirred for an additional 6 h. After warming to 0 °C, the reaction was quenched with water (50 mL) and extracted with ether (3 × 50 mL). The combined organic extracts were washed with brine, dried (Na_2SO_4), filtered, and concentrated. The residue obtained was triturated with hexanes and filtered. The filtrate was concentrated to give a crude oil containing ca. 12.5:1 (*Z*/*E*) mixture of olefin isomers. The crude product mixture was purified by chromatography (20:1 hexanes:EtOAc) to give 2.4 g (64%) of (*Z*)-**A3** as a colorless oil. $[\alpha]_D$ +61.6 (*c* 1.00, $CDCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.03 (t, *J* = 7.5 Hz, 3H), 1.50 (m, 1H), 1.98 (m, 1H), 2.17 (m, 2H), 3.81 (s, 3H), 4.02 (dt, *J* = 2.4, 12.4Hz, 1H), 4.28 (ddd, *J* = 1.2, 5.0, 11.4 Hz, 1H), 4.69 (m, 1H), 5.49 (dd, *J* = 7.7, 11.0 Hz, 1H), 5.55 (m, 2H), 6.91 (d, *J* = 8.8 Hz, 2H), 7.44 (d, *J* = 8.7 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 14.7, 21.7, 31.4, 55.7, 67.3, 74.0, 101.5, 114.0, 127.8, 129.4, 131.7, 134.4, 160.3; IR (film) 1615 cm^{-1}; MS (CI) *m*/*z* 249 (M^+ + H), 152, 95; HRMS calcd for $C_{15}H_{21}O_3$ (M^+ + H) 249.1491, found 249.1509.



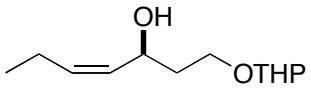
(3*S*,*Z*)-3-(4'-Methoxybenzyloxy)hept-4-en-1-ol (A4). Diisobutylaluminum hydride (37 mL, 1.5 M solution in toluene) was added to a solution of **A3** (2.73 g, 11.0 mmol) in anhydrous toluene (50 mL) at –40 °C. After stirring at –40 °C for 2h, the

reaction was quenched by the slow addition of MeOH (20 mL). The resulting mixture was subsequently stirred in the presence of saturated aqueous potassium sodium tartrate (50 mL) while warming to room temperature for 10 h. The phases thus formed were separated and the aqueous layer was extracted with CH_2Cl_2 (3 × 50 mL). The combined organic layers were dried (Na_2SO_4), filtered, and concentrated. The crude material was purified by chromatography (3:1 hexanes:EtOAc) to afford 2.5 g (91%) of a colorless oil. $[\alpha]_D$ –45.0 (*c* 1.25, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.01 (t, *J* = 7.5 Hz, 3H), 1.69 (m, 1H), 1.89 (m, 1H), 2.08 (m, 2H), 2.64 (br, s, 1H), 3.76 (m, 2H), 3.82 (s, 3H), 4.28 (d, *J* = 11.4 Hz, 1H), 4.40 (dt, *J* = 4.3, 8.6 Hz, 1H), 4.55 (d, *J* = 11.4 Hz, 1H), 5.35 (m, 1H), 5.64 (dt, *J* = 11.0, 7.4 Hz, 1H), 6.89 (dt, *J* = 8.7, 2.0 Hz, 2H), 7.25 (d, *J* = 8.6 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 14.7, 21.5, 38.3, 55.7, 61.4, 70.0, 74.1, 114.2, 129.7, 129.8, 130.8, 135.8, 159.6; IR (film) 3413, 1613, 1514 cm^{-1}; MS (EI) *m/z* 268 (M^+ + NH_4), 137, 121; HRMS calcd for $C_{15}H_{26}NO_3$ (M^+ + NH_4) 268.1913, found 268.1930.



2-[(3*S*,*Z*)-3'-(4''-Methoxybenzyloxy)hept-4'-enyloxy]tetrahydro-2*H*-pyran (A5). DHP (1.0 g, 12.0 mmol) was added to a solution of **A4** (1.88 g, 7.51 mmol) and PPTS (0.09 g, 0.38 mmol) in THF (35 mL) at 0 °C. The reaction mixture was allowed to warm to room temperature and stirred for 16 h. A saturated aqueous $NaHCO_3$ solution (25 mL) was then added, and the aqueous layer was extracted with ether (3 × 30 mL). The combined organic layers were washed with brine, dried (Na_2SO_4), filtered, and concentrated. The crude material was purified by chromatography (10:1 hexanes:EtOAc) to give 2.5 g (98%) of a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) *Mixture of THP ether diastereomers*: δ 1.00 (dt, *J* = 1.4, 7.5 Hz, 3H), 1.50-2.10 (m, 11H), 3.5 (m, 2H), 3.82 (m, 4H), 4.31 (m, 2H), 4.52 (dd, *J* = 4.6, 4.2 Hz, 2H), 5.31 (m, 1H), 5.64 (m, 1H), 6.88 (dd, *J* = 0.4, 8.5 Hz,2H), 7.27 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$) *Mixture of THP ether diastereomers; all peaks listed*: δ 14.8, 20.0, 20.1, 21.4, 21.5, 25.9, 25.9, 31.1, 31.2, 36.3, 36.4, 55.7, 62.6, 62.7, 64.5, 64.5, 69.8, 70.0, 71.1, 71.7, 99.1, 99.5, 114.1, 114.1, 129.7,

129.8, 130.3, 130.3, 131.4, 131.4, 135.6, 135.7, 159.4, 159.4; IR (film) 1613, 1512 cm^{-1}; MS (CI) *m/z* 352 ($M^+ + NH_4$), 335, 121; HRMS calcd for $C_{20}H_{34}NO_4$ ($M^+ + NH_4$) 352.2488, found 352.2499.

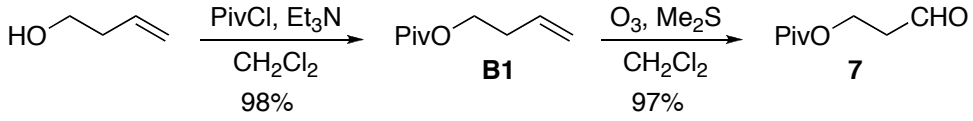


(3*S*,*Z*)-1-(Tetrahydro-2*H*-pyran-2'-yloxy)hept-4-en-3-ol (3). To a mixture of **A5** (1.44 g, 4.30 mmol) in CH_2Cl_2/water (60 mL: 3mL) at room temperature was added DDQ (1.19 g, 5.25 mmol). The reaction mixture was stirred vigorously for 1.5 h, and then a saturated aqueous solution of $NaHCO_3$ (50 mL) was added. The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (3 × 50 mL). The combined organic layers were washed with brine (1 × 50 mL), dried (Na_2SO_4), filtered, and concentrated. Chromatography (5:1 to 2:1 hexanes:EtOAc) gave 0.73 g (79%) of a colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) *Mixture of THP ether diastereomers*: δ 1.01 (dt, *J* = 1.7, 7.5 Hz, 3H), 1.56-1.64 (complex, 4H), 1.74-1.95 (complex, 4H), 2.11 (m, 2H), 2.67 (br, s, 1H), 3.55 (m, 2H), 3.94 (m, 2H), 4.61 (m, 1H), 4.69 (m, 1H), 5.45 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$) *Mixture of THP ether diastereomers; all peaks listed*: δ 14.8, 19.8, 20.0, 21.4, 25.7, 30.9, 31.1, 37.4, 37.4, 62.6, 63.0, 65.8, 66.0, 66.8, 67.4, 99.3, 99.5, 131.9, 131.9, 133.8, 133.9; IR (film) 3431 cm^{-1}; MS (CI) *m/z* 215 (M^+ + H), 197, 85; HRMS calcd for $C_{12}H_{23}O_3$ (M^+ + H) 215.1647, found 215.1639.

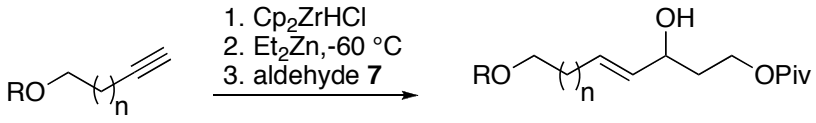
Ether-substituted allylic alcohols were prepared via a zirconium-mediated coupling of aldehyde **7** with benzyloxy or *tert*-butyldiphenylsilyloxy alkynes. The racemic allylic alcohol **8** was subjected to a kinetic resolution under Sharpless conditions[10] to furnish the enantioenriched allylic alcohol (*R*)-**8** in 42% yield and ≥99% er as determined by proton NMR of the Mosher's ester derivative[11] All other ether-substituted allylic alcohols were prepared and used in racemic form.

Scheme B. *Synthesis of ether-substituted allylic alcohols.*

Preparation of the aldehyde coupling component:



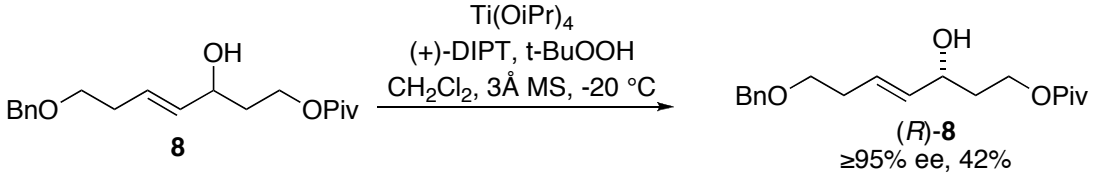
Synthesis of racemic allylic alcohols:

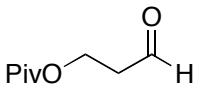


6: R = Bn, n = 1
B2: R = Bn, n = 3
B3: R = TBDPS, n = 1
B4: R = PMB, n = 1

8: R = Bn, n = 1, 63%
B5: R = Bn, n = 3, 58%
B6: R = TBDPS, n = 1, 75%
B7: R = PMB, n = 1, 56%

Generation of enantiopure allylic alcohol:

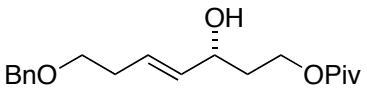




3-Oxopropyl pivalate (7). Ozone was bubbled through a solution of 3-butenyl pivalate (**B1**)[4] (12.87 g, 82.37 mmol) in CH_2Cl_2 (150 mL) at –78 °C until a persistent blue color resulted (ca. 2 h). The reaction was quenched with dimethyl sulfide (65 mL) and allowed to warm to room temperature with stirring for 16 h. The reaction mixture was concentrated under reduced pressure and the resulting residue was dissolved in ether and filtered through a pad of silica gel. The filtrate was concentrated to afford 12.70 g (97%) of the title compound as a colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.21 (s, 9H), 2.78 (td, J = 6.1, 1.5, Hz, 2H), 4.42 (t, J = 6.1 Hz, 2H), 9.81 (t, J = 1.5 Hz, 1H); ^{13}C NMR

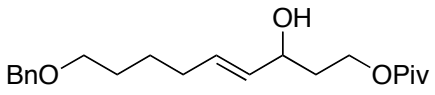
(125.8 MHz, $CDCl_3$) d 27.0, 38.6, 57.9, 178.2, 199.3; IR (neat) 1728, 1483 cm^{-1}; MS (CI) *m/z* 159 (M^+ + H), 85, 57; HRMS calcd for $C_8H_{15}O_3$ (M^+ + H) 159.1021, found 159.1020.

General procedure for the synthesis of ether-substituted allylic alcohols. According the procedure of Wipf and Xu,[12] bis(cyclopentadienyl)zirconium chloride hydride (Schwartz's reagent) (5.0 g, 19.4 mmol) was added to a solution of the alkyne (16.2 mmol) in dry CH_2Cl_2 (60 mL) at 0 °C. The resulting mixture was then warmed to room temperature and stirred until a homogeneous solution formed. The resulting yellow solution was stirred at room temperature for an additional 20 min, and then cooled to –60 °C. Diethylzinc (1.0 M in hexanes, 19.4 mmol) was then added dropwise over 45 min at –60 °C. After the addition was complete, the resulting solution was stirred at –60 °C for an additional 10 min. The reaction flask was immersed in an ice bath, and a solution of aldehyde **7** (3.1 g, 19.4 mmol) in CH_2Cl_2 (10 mL) was added dropwise over 45 min. The resulting solution was stirred at 0 °C for an additional 6 h. The light-yellow reaction mixture was then slowly poured into an ice-cold solution of aq 5% $NaHCO_3$ (200 mL) and stirring was continued at room temperature until gas evolution ceased. The resulting mixture was extracted with Et_2O (3 × 200 mL) and the combined organic extracts were washed with brine (1 × 200 mL), dried (Na_2SO_4), and filtered through a pad of Florisil®. The filtrate was concentrated, and the resulting residue was purified by chromatography to afford the allylic alcohol.



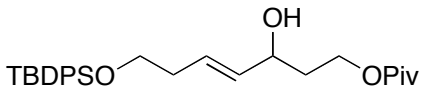
(3*R*,*E*)-7-Benzyloxy-3-hydroxyhept-4-enyl pivalate (8). The racemic allylic alcohol **8** was prepared according to the general procedure from alkyne **6**[5] (3.09 g, 19.3 mmol) and aldehyde **7** (3.66 g, 23.2 mmol). Chromatography (5:1 hexanes:EtOAc) gave 3.9 g (63%) of a colorless oil. The racemic material was subjected to a kinetic resolution using the procedure of Sharpless.[10] Accordingly, a mixture of the allylic alcohol *rac*-**8** (4.06 g, 12.7 mmol), (+)-diisopropyl tartrate (0.89 g, 3.8 mmol), and 3Å powdered

molecular sieves (1.0 g) in CH_2Cl_2 (50.0 mL) was cooled to –20 °C and then treated with Ti(O-*i*-Pr)$_4$ (0.72 g, 2.5 mmol). After stirring for 30 min at –20 °C, a solution of TBHP (5.0 M, 1.9 mL) was added to the reaction mixture and stirring at –20 °C was continued for 12-14 h. After >50% conversion was obtained (monitored by ^{1}H NMR), the reaction was quenched with an aqueous solution (60 mL) of $FeSO_4$ (1.57 g) and citric acid (0.53 g) and stirred at room temperature for 1 h. The phases were separated and the aqueous phase was extracted with CH_2Cl_2 (2 × 75 mL). The combined organic layers were concentrated to approximately 50 mL and then stirred for 30 min with 30% NaOH (100 mL). The phases were separated and extracted as before, and the combined organic layers were washed with brine, dried (Na_2SO_4), filtered, and concentrated. The crude product was purified by chromatography (5:1 hexanes:EtOAc) to afford 1.72 g (42%) of the allylic alcohol (*R*)-**8** in ≥97% er (determined by ^{1}H NMR of the (–)-MTPA chloride-derived ester.)[10] $[\alpha]_D$ –3.18 (*c* 0.98, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.22 (s, 9H), 1.81 (q, *J* = 6.3 Hz, 2H), 2.36 (q, *J* = 6.6 Hz, 2H), 3.52 (t, *J* = 6.7 Hz, 2H), 4.13 (m, 2H), 4.26 (dt, *J* = 11.2, 6.6 Hz, 1H), 4.52 (s, 2H), 5.57 (dd, *J* = 15.5, 6.7 Hz, 1H), 5.70 (dt, *J* = 15.5, 6.6 Hz, 1H), 7.32 (m, 5H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 27.6, 33.0, 36.6, 39.1, 61.7, 70.0, 70.1, 73.3, 128.0, 128.1, 128.8, 128.8, 134.5, 138.7, 179.2; IR (film) 3434, 1724 cm^{-1}; MS (CI) *m/z* 338 ($M^+ + NH_4$), 320 (M^+), 108, 91; HRMS calcd for $C_{19}H_{32}NO_4$ ($M^+ + NH_4$) 338.2331, found 338.2326.

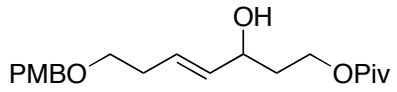


(±)-(*E*)-9-(Benzyloxy)-3-hydroxynon-4-enyl pivalate (B5). Prepared according to the general procedure from 1-benzyloxy-5-hexyne (**B2**)[13] (3.10 g, 16.2 mmol) and aldehyde **7** (3.10 g, 19.4 mmol). The crude product was purified by chromatography (5:1 hexanes:EtOAc) to give 3.30 g (58%) of a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.22 (s, 9H), 1.48 (m, 2H), 1.64 (m, 2H), 1.83 (m, 2H), 2.01 (br, s, 1H), 2.07 (q, *J* = 7.0 Hz, 2H), 3.48 (t, *J* = 6.4 Hz, 2H), 4.15 (m, 2H), 4.27 (m, 1H), 4.52 (s, 2H), 5.49 (dd, *J* = 6.8, 15.4 Hz, 1H), 5.68 (td, *J* = 6.6, 15.3 Hz, 1H), 7.31 (m, 5H); ^{13}C NMR (100.6 MHz,

$CDCl_3$) δ 26.1, 27.6, 29.7, 32.4, 36.7, 39.1, 61.8, 70.3, 70.6, 73.3, 127.9, 128.0, 128.8, 132.5, 132.7, 139.0, 179.2; IR (film) 3442, 1726, 1478, 1450 cm^{-1}; MS (FAB+) *m/z* 349 (M^+ + H), 331, 229, 137; HRMS calcd for $C_{21}H_{36}N0_4$ (M^+ + NH_4) 366.2644, found 366.2634.



(±)-(*E*)-7-(*tert*-Butyldiphenylsilyloxy)-3-hydroxyhept-4-enyl pivalate (B6). Prepared according to the general procedure from alkyne **B3**[7] (5.00 g, 16.2 mmol) and aldehyde **7** (3.34 g, 21.1 mmol). The crude product was purified by chromatography (6:1 hexanes:EtOAc) to give 5.71 g (75%) of a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.07 (s, 10H), 1.22 (s, 9H), 1.83 (q, *J* = 6.3 Hz, 2H), 2.31 (q, *J* = 6.6 Hz, 2H), 3.73 (t, *J* = 6.6 Hz, 2H), 4.14 (m, 2H), 4.28 (td, *J* = 6.6, 11.2 Hz, 1H), 5.54 (dd, *J* = 6.7, 15.4 Hz, 1H), 5.69 (td, *J* = 6.7, 15.5 Hz, 1H), 7.44 (m, 6H), 7.68 (m, 4H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 19.6, 27.2, 27.6, 36.0, 36.6, 39.2, 61.7, 63.8, 70.2, 128.0, 129.2, 130.0, 134.3, 134.4, 136.0, 179.2; IR (film) 3466, 1728, 1710 cm^{-1}; MS (FAB+) *m/z* 486 (M^+ + H), 307, 154; HRMS calcd for $C_{28}H_{44}N0_4Si$ (M^+ + NH_4) 486.3040, found 486.3034.

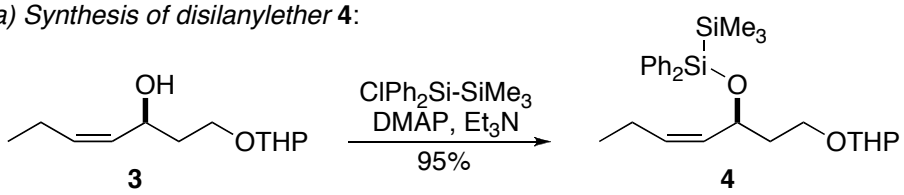


(±)-(*E*)-3-Hydroxy-7-(4-methoxybenzyloxy)hept-4-enyl pivalate (B7). Prepared according to the general procedure from alkyne **B4** (2.82 g, 16.17 mmol) and aldehyde **7** (3.60 g, 22.64 mmol). The crude product was purified by chromatography (5:1 to 1:1 hexanes:EtOAc) to give 3.2 g (56%) of a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.20 (s, 9H), 1.84 (q, *J* = 6.3 Hz, 2H), 2.35 (q, *J* = 6.7 Hz, 2H), 3.49 (t, *J* = 6.8 Hz, 2H), 3.82 (s, 3H), 4.13 (m, 1H), 4.27 (m, 1H), 4.45 (s, 2H), 5.58 (dd, *J* = 15.5, 6.6Hz, 1H),

5.70 (dt, J = 15.5, 6.7 Hz, 1H), 6.89 (d, J = 8.6 Hz, 2H), 7.27 (d, J = 8.7 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 27.2, 32.6, 36.2, 38.7, 55.3, 61.3, 69.3, 69.7, 72.6, 113.8, 128.6, 129.3, 130.4, 134.0, 159.2, 178.8; IR (film) 3410, 1680, 1570, 1480 cm^{-1}; MS (ES+) m/z 373 ($M^+ + NH_4$), 335, 319; HRMS calcd for $C_{20}H_{34}NO_5$ ($M^+ + NH_4$) 368.2437, found 368.2416.

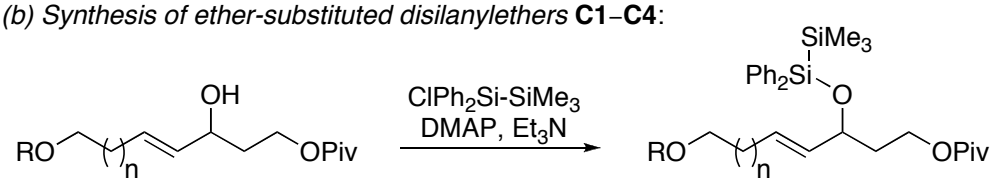
Scheme C. *Synthesis of disilanyl ethers.*

(a) Synthesis of disilanylether **4**:



3 **4**

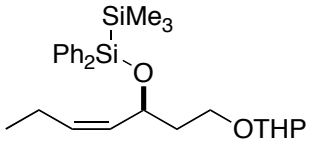
(b) Synthesis of ether-substituted disilanylethers **C1**–**C4**:



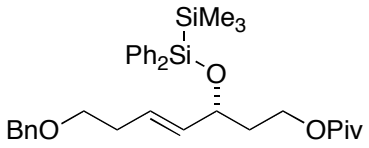
(*R*)-**8**: R = Bn, n = 1
(±)-**B5**: R = Bn, n = 3
(±)-**B6**: R = TBDPS, n = 1
(±)-**B7**: R = PMB, n = 1

(*R*)-**C1**: R = Bn, n = 1, 98%
(±)-**C2**: R = Bn, n = 3, 95%
(±)-**C3**: R = TBDPS, n = 1, 83%
(±)-**C4**: R = PMB, n = 1, 98%

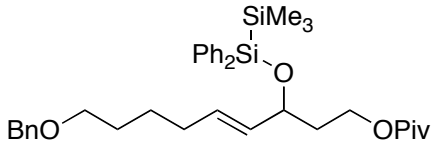
General procedure for the preparation of disilanyl ethers. To a mixture of the allylic alcohol (1.0 equiv), triethylamine (1.5 equiv), and a spatula tip of DMAP in THF was added a solution of 1-chloro-2,2,2-trimethyl-1,1-diphenyl disilane[2] (1.5 equiv) in THF at 0 °C. The reaction mixture was allowed to warm to room temperature and stirred for 16 h. Upon completion of the reaction, saturated aq NH_4Cl was added and the layers were separated. The aqueous layer was extracted with ether (3 × 60 mL) and the combined organic extracts were washed with brine, dried (Na_2SO_4), filtered, and concentrated. The crude material was purified by chromatography to afford the product disilanylether.



(3*S*,*Z*)-Tetrahydropyran-2-yloxy-3-(2',2',2'-trimethyl-1',1'-diphenyl-disilyloxy)hept-4-ene (4). Prepared according to the general procedure for the preparation of disilanyl ethers from allylic alcohol **3** (0.72 g, 3.36 mmol). The crude product was purified by chromatography (25:1 hexanes:EtOAc) to give 1.50 g (95%) of a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) *Mixture of THP ether diastereomers*: δ 0.21 (s, 9H), 0.81 (td, *J* = 6.9, 10.4 Hz, 3H), 1.47-2.03 (m, 10H), 3.46 (m, 2H), 3.82 (m, 2H), 4.48 (td, *J* = 3.7, 44.4 Hz, 1H), 4.72 (m, 1H), 5.28 (qd, *J* = 11.0, 6.9 Hz, 1H), 5.41 (m, 1H), 7.39 (m, 6H), 7.59 (m, 4H); ^{13}C NMR (100.6 MHz, $CDCl_3$) *Mixture of THP ether diastereomers; all peaks listed*: δ –0.8, 14.4, 14.6, 19.8, 20.0, 21.3, 23.1, 25.9, 31.1, 32.0, 38.9, 39.1, 62.4, 62.6, 64.2, 64.3, 67.8, 68.1, 98.9, 99.1, 128.1, 129.7, 132.3, 132.4, 132.7, 132.9, 135.3, 135.4, 137.6; IR (film) 2947, 1428 cm^{-1}; MS (EI) *m/z* 311, 255, 85; HRMS calcd for $C_{27}H_{44}Si_2O_3N$ 486.2860 ($M^+ + NH_4$), found 486.2877.

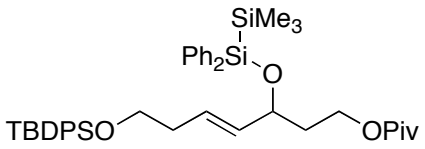


(3*R*,*E*)-7-Benzyloxy-3-(2',2',2'-trimethyl-1',1'-diphenyldisilyloxy)hept-4-enyl pivalate (C1). Prepared according to the general procedure for the preparation of disilanyl ethers from allylic alcohol (*R*)-**8** (4.10 g, 12.8 mmol). Chromatography (25:1 hexanes:EtOAc) afforded 7.33 g (ca. 100%) of a colorless oil. $[\alpha]_D$ +12.4 (*c* 0.98, $CHCl_3$). ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.19 (s, 9H), 1.14 (s, 9H), 1.78 (m, 1H), 1.93 (m, 1H), 2.24 (q, *J* = 6.7 Hz, 2H), 3.35 (m, 2H), 4.10 (t, *J* = 6.5 Hz, 2H), 4.30 (q, *J* = 6.5 Hz, 1H), 4.48, (s, 2H), 5.41 (dt, *J* = 15.4, 6.4 Hz, 1H), 5.48 (dd, *J* = 15.5, 7.0 Hz, 1H), 7.30-7.55 (m, 15H); ^{13}C (100.6 MHz, $CDCl_3$) δ –0.85, 27.6, 33.0, 37.6, 39.0, 61.5, 70.0, 72.6, 73.3, 128.0, 128.1, 128.2, 128.4, 128.8, 129.8, 134.6, 135.3, 135.3, 137.3, 138.8, 178.8; IR (film) 1727 cm^{-1}: MS (FAB+) *m/z* 581.3, 255.1, 154.1; HRMS calcd for $C_{34}H_{50}NO_4Si_2$ ($M^+ + NH_4$) 592.3278, found 592.3257.

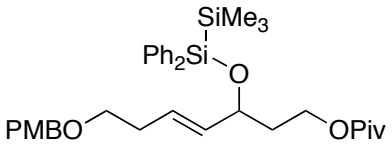


(±)-(*E*)-9-(Benzyloxy)-3-(2',2',2'-trimethyl-1',1'-diphenyldisilyloxy)non-4-enyl pivalate (C2). Prepared according to the general procedure for the preparation of disilanyl ethers from allylic alcohol **B5** (2.29 g, 3.42 mmol). The crude product was purified by chromatography (25:1 hexanes:EtOAc) to give 3.77 g (95%) of a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.20 (s, 9H), 1.15 (s, 9H), 1.34 (m, 2H), 1.56 (m, 2H), 1.78 (m, 1H), 1.94 (m, 3H), 3.45 (t, *J* = 6.5 Hz, 2H), 4.09 (t, *J* = 6.4 Hz, 2H), 4.27 (q, *J* = 6.1 Hz, 1H), 4.52 (s, 2H), 5.39 (m, 2H), 7.36 (m , 10H), 7.55 (m, 5H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ –0.8, 26.0, 27.6, 29.7, 32.3, 37.7, 39.0, 61.6, 70.6, 72.7, 73.3, 127.9, 128.0, 128.1, 128.8, 132.2, 132.8, 135.3, 135.3, 137.4, 139.1, 178.8; IR (film) 1731,1481

cm^{-1}; MS (FAB+) *m/z* 620 ($M^+ + NH_4$), 370, 255; HRMS calcd for $C_{36}H_{54}NO_4Si_2$ ($M^+ + NH_4$) 620.3591, found 620.3617.



(±)-(*E*)-7-(*tert*-Butyldiphenylsilyloxy)-3-(2',2',2'-trimethyl-1,1-diphenyl-disilyloxy)hept-4-enyl pivalate (C3). Prepared according to the general procedure for the preparation of disilanyl ethers from allylic alcohol **B6** (4.0 g, 5.07 mmol). Chromatography (20:1 hexanes:EtOAc) gave 5.13 g (83%) of a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ □□□□□(s, 9H), 1.05 (s, 10H), 1.14 (s, 9H), 1.77 (m, 1H), 1.91 (m, 1H), 2.17 (q, *J* = 6.6 Hz, 2H), 3.51 (q, *J* = 7.0 Hz, 2H), 4.09 (t, *J* = 6.4 Hz, 2H), 4.26 (q, *J* = 6.4 Hz, 1H), 5.42 (m, 2H), 7.33–7.67 (m, 20H); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ –1.3, 15.2, 19.1, 26.7, 27.1, 35.4, 37.2, 38.5, 61.0, 63.3, 65.8, 72.1, 127.5, 127.6, 128.0, 129.3, 129.5, 133.8, 134.0, 134.8, 135.5, 136.8, 178.3; IR (film) 1730, 1427 cm^{-1}; HRMS calcd for $C_{43}H_{62}NO_4Si_3$ ($M^+ + NH_4$) 740.3987, found 740.4001.



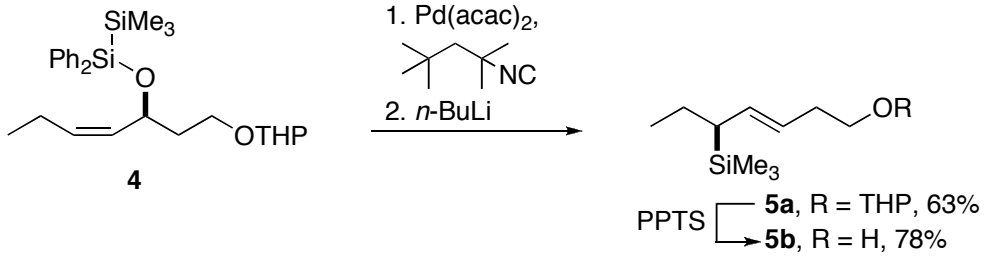
(±)-(*E*)-7-(4'-Methoxybenzyloxy)-3-(2',2',2'-trimethyl-1',1'-diphenyldi-silyloxy)hept-4-enyl pivalate (C4). Prepared according to the general procedure for the preparation of disilanyl ethers from allylic alcohol **B7** (2.32 g, 6.62 mmol). The crude product was purified by chromatography (25:1 hexanes:EtOAc) to give 3.94 g (98%) of a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.19 (s, 9H), 1.14 (s, 9H), 1.29 (t, *J* = 7.0 Hz, 2H), 1.78 (m, 1H), 1.92 (m, 1H), 2.22 (q, *J* = 6.8 Hz, 2H), 3.34 (m, 2H), 3.32 (m, 2H), 3.83 (s, 3H), 4.09 (t, *J* = 6.4 Hz, 2H), 4.28 (q, *J* = 6.4 Hz, 1H), 4.41 (s, 2H), 5.41(dt, *J* = 15.5, 6.2 Hz, 1H), 5.47 (dd, *J* = 15.5, 6.9 Hz, 1H), 6.89 (d, *J* = 8.7 Hz, 2H), 7.26 (d, *J* =

8.6 Hz, 2H), 7.40–7.56 (m, 10 H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ –1.2, 27.2, 32.6, 37.2, 38.6, 55.3, 61.1, 69.4, 72.2, 72.6, 113.8, 127.76, 128.0, 128.1, 129.3, 129.4, 129.5, 134.0, 134.9, 159.2, 178.4; IR (film) 1710, 1440 cm^{-1}; MS (ES+) *m/z* 622 ($M^+ + NH_4$), 389, 374, 373; HRMS calcd for $C_{35}H_{52}NO_5Si_2$ ($M^+ + NH_4$) 622.3384, found 622.3376.

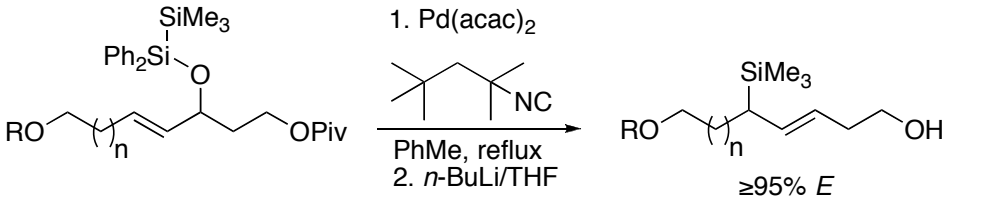
Preparation of Chiral Allylsilanes

Scheme D. *Synthesis of chiral allylsilanes.*

(a) Synthesis of ethyl-substituted allylsilane **5b**:



(b) Synthesis of ether-substituted allylsilanes **9**, **D1**, **D2**:

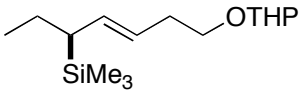


(*R*)-**C1**: R = Bn, n = 1
(±)-**C2**: R = Bn, n = 3
(±)-**C3**: R = TBDPS, n = 1
(±)-**C4**: R = PMB, n = 1

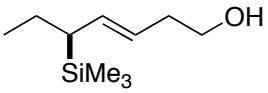
(*S*)-**9**: R = Bn, n = 1, 67%
(±)-**D1**: R = Bn, n = 3, 61%
(±)-**D2**: R = TBDPS, n = 1, 59%
(±)-**D3**: R = PMB, n = 1, 55%

General procedure for the conversion of disilanyl ethers to chiral allylsilanes. According to the procedure of Ito,[14] a mixture of 1,1,3,3-tetramethylbutyl isocyanide (0.41 equiv) and $Pd(acac)_2$ (0.10 equiv) in toluene was stirred at room temperature under Ar for 5 min, resulting in a red-brown solution. A solution of the disilanyl ether (1.0 equiv) in toluene (ca. 5 mL) was then added and the resulting mixture was heated at reflux for 3.5–6 h. After cooling to room temperature, the reaction mixture was filtered through Florisil® and concentrated under reduced pressure. The residue obtained was

dissolved in THF and cooled to 0 °C. The resulting solution was charged with *n*-BuLi (2 equiv for pivalate esters; 1.5 equiv for THP ethers), and stirred at 0 °C for 30 min. The reaction was quenched with water (20 mL) and the layers were separated. The aqueous layer was extracted with ether (3 × 30 mL), and the combined organic extracts were washed with brine, dried (Na_2SO_4), filtered, and concentrated. Chromatography of the crude material afforded the *E*-allylsilane product.

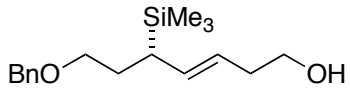


(5*S*,*E*)-1-(Tetrahydropyran-2'-yloxy)-5-trimethylsilylhept-3-ene (5a). Prepared according to the general procedure from disilanyl ether **4** (2.00 g, 4.27 mmol). The crude material was purified by chromatography (30:1 hexanes:EtOAc) to give 0.88 g (76%) of an orange-brown oil. ^{1}H NMR (500 MHz, $CDCl_3$) *Mixture of THP ether diastereomers*: δ –0.03 (s, 9H), 0.91 (t, *J* = 6.9 Hz, 3H), 1.32 (m, 2H), 1.58 (m, 5H), 1.72 (m, 1H), 1.85 (m, 1H), 2.32 (q, *J* = 6.7 Hz, 2H), 3.42 (m, 1H), 3.51 (m, 1H), 3.75 (td, *J* = 7.1, 10.8 Hz, 1H), 3.90 (m, 1H), 4.62 (s, 1H), 5.27 (m, 2H); ^{13}C NMR (125.8 MHz, $CDCl_3$) *Mixture of THP ether diastereomers; all peaks listed*: δ –3.22, 14.1, 19.4, 19.5, 21.8, 21.9, 25.1, 30.6, 33.4, 35.3, 62.0, 62.1, 67.7, 67.8, 98.5, 98.6, 124.1, 124.2, 133.5; IR (film) 2956, 1456 cm^{-1}; MS (CI) *m*/*z* 271 (M^+ + H), 197, 102; HRMS calcd for $C_{15}H_{34}NO_2Si$ (M^+ + NH_4) 288.2359, found 288.2362.

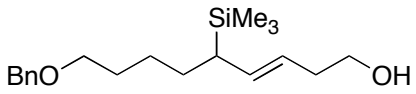


(5*S*, *E*)-5-Trimethylsilylhept-3-en-1-ol (5b). A solution of **5a** (0.55 g, 2.02 mmol) and PPTS (0.10 g, 0.41 mmol) in EtOH (15 mL) was stirred at 55 °C for 4.5 h. After cooling to room temperature, water (5.0 mL) was added to the reaction mixture,

which was extracted with ether (3 × 30 mL). The combined organic extracts were washed with brine, dried (Na_2SO_4), filtered, and concentrated. Chromatography (20:1 to 10:1 hexanes:EtOAc) gave 0.30 g (78%) of a pale yellow oil. $[\alpha]_D$ +7.8 (*c* 0.83, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ –0.02 (s, 9H), 0.91 (t, *J* = 6.3 Hz, 3H), 1.30 (m, 2H), 1.52 (m, 2H), 2.30 (q, *J* = 6.6 Hz, 2H), 3.61 (t, *J* = 6.3 Hz, 2H), 5.20 (td, 6.8, 15.2 Hz, 1H), 5.40 (dd, *J* = 7.2, 15.3 Hz, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ –3.2, 14.2,21.8, 35.5, 36.3, 62.3, 123.4, 135.5; IR (film) 3334, 1451 cm^{-1}; MS (CI) *m/z* 187 (M^+ + H), 147, 90; HRMS calcd for $C_{10}H_{26}NOSi$ ($M^+ + NH_4$) 204.1784, found 204.1779.

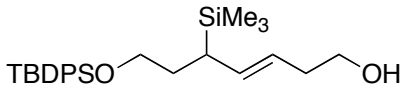


(5*S*,*E*)-7-Benzyloxy-5-trimethylsilylhept-3-en-1-ol (9). Prepared according to the general procedure using disilanyl ether (*R*)-**C1** (1.80 g, 3.18 mmol). Chromatography of the crude product (5:1 hexanes:EtOAc) gave 0.62 g (67%) of an orange-brown oil. $[\alpha]_D$ +28.4 (*c* 1.22, $CHCl_3$). ^1H NMR (500 MHz, $CDCl_3$) δ 0.01 (s, 9H), 1.53 (br s, 1H), 1.64 (m, 2H) 1.84 (dt, *J* = 10.6, 7.7 Hz, 1H), 2.27 (q, *J* = 6.4 Hz, 2H) 3.44 (m, 1H), 3.54 (m, 1H), 3.59 (t, *J* = 6.3 Hz, 2H), 4.52 (AB q, *J* = 12.0, Δν = 31.4 Hz, 2H), 5.22 (dt, *J* = 15.3, 7.0 Hz, 1H), 5.36 (dd, *J* = 15.1, 9.2 Hz, 1H), 7.4 (m, 5H); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ –3.4, 28.7, 29.9, 36.2, 62.2, 69.9, 72.8, 123.8, 127.4, 127.6, 128.3, 134.7, 138.5; IR (film) 3399 cm^{-1}; MS (FAB +) *m/z* 293 (M^+ + H); HRMS calcd for $C_{17}H_{29}SiO_2$ (M^+ + H) 293.1937, found 293.1933.

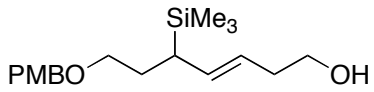


(±)-(*E*)-9-Benzyloxy-5-(trimethylsilyl)non-3-en-1-ol (D1). Prepared according to the general procedure from disilanyl ether **C2** (1.15 g, 1.90 mmol). The crude product was purified by chromatography (6:1 to 3:1 hexanes:EtOAc) to give 0.70 g (61%) of an

orange-brown oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ –0.01 (s, 9H), 1.34 (m, 2H), 1.50 (m, 4H), 1.66 (m, 1H), 2.30 (q, *J* = 6.5 Hz, 2H), 3.48 (m, 2H), 3.61 (t, *J* = 6.3 Hz, 2H), 4.52 (s, 2H), 5.21 (td, *J* = 7.0, 15.3 Hz, 1H), 5.34 (dd, *J* = 9.6, 15.2 Hz, 1H), 7.37 (m, 5H); ^{13}C NMR (125.8 MHz, $CDCl_3$), δ –3.3, 25.8, 28.4, 29.4, 33.2, 36.3, 62.3, 70.3, 72.8, 123.5, 127.4, 127.6, 128.3, 135.4, 138.6; IR (film) 3395, 1454 cm^{-1}; MS (FAB+) *m/z* 321 (M^+ + H), 136; HRMS calcd for $C_{19}H_{36}NO_2Si$ ($M^+ + NH_4$) 338.2515, found 338.2506.



(±)-(*E*)-7-(*tert*-Butyldiphenylsilyloxy)-5-(trimethylsilyl)hept-3-en-1-ol (D2). Prepared according to the general procedure from disilanyl ether **C3** (2.50 g, 3.46 mmol). Chromatography (10:1 to 5:1 hexanes:EtOAc) gave 0.90 g (59%) of an orange-brown oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ –0.01 (s, 9H), 1.08 (s, 9H), 1.52 (m, 1H), 1.63 (td, *J* = 11.5, 2.2 Hz, 1H), 1.76 (m, 1H), 2.22 (q, *J* = 6.4 Hz, 2H), 3.55 (m, 2H), 3.62 (m, 1H), 3.73 (m, 1H), 5.11 (dt, *J* = 15.3, 6.9 Hz, 1H), 5.27 (dd, *J* = 15.3, 9.3 Hz, 1H), 7.42 (m, 6H), 7.69 (m, 4H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ –2.8, 19.6, 27.3, 29.6, 31.9, 36.7, 62.7, 63.7, 124.0, 128.0, 129.9, 134.5, 135.2, 136.0; IR (film) 3356, 1427 cm^{-1}; MS (FAB+) *m/z* 441 (M^+ + H), 307, 154; HRMS calcd for $C_{26}H_{44}NSi_2O_2$ ($M^+ + NH_4$) 458.2911, found 458.2914.

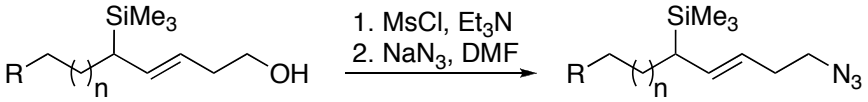


(±)-(*E*)-7-(4'-Methoxybenzyloxy)-5-(trimethylsilyl)hept-3-en-1-ol (D3). Prepared according to the general procedure for the conversion of disilanyl ethers to chiral allylsilanes from **C4** (2.0 g, 3.31 mmol). The crude product was purified by chromatography (5:1 hexanes:EtOAc) to give 0.59 g (55%) of an orange-brown oil. ^{1}H

NMR (400 MHz, $CDCl_3$) δ –0.103 (s, 9H), 1.61 (m, 2H), 1.80 (m, 1H), 2.27 (q, J = 6.4 Hz, 2H), 3.39 (m, 1H), 3.50 (m, 1H), 3.59 (q, J = 6.0 Hz, 2H), 4.45 (AB q, J = 11.5 Hz, Δν = 21.8 Hz), 5.20 (dt, J = 15.3, 6.9 Hz, 1H), 5.35 (dd, J = 15.3, 9.0 Hz, 1H), 6.89 (d, J = 8.7 Hz, 2H), 7.28 (d, J = 8.9 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ –3.3, 28.8, 30.0, 36.3, 55.3, 62.3, 69.8, 72.6, 113.7, 123.8, 129.3, 130.7, 134.9, 159.1; IR (film) 3400, 1610, 1480 cm^{-1}; MS (ES+) m/z 323 (M^+ + H), 233, 215; HRMS calcd for $C_{18}H_{31}O_3Si$ (M^+ + H) 323.2043, found 323.2040.

$TiCl_4$-Promoted Cyclization Reactions

Scheme E. *Synthesis of azido allylsilanes.*

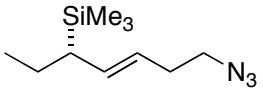


(*S*)-**5b**: R = H, n = 1
(*S*)-**9**: R = OBn, n = 1
(±)-**D1**: R = OBn, n = 3
(±)-**D2**: R = OTBDPS, n = 1

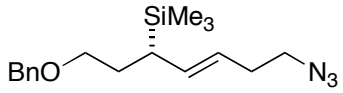
(*S*)-**1**: R = H, n = 1, 92%
(*S*)-**2**: R = OBn, n = 1, 85%
(±)-**E1**: R = OBn, n = 3, 64%
(±)-**E2**: R = OTBDPS, n = 1, 76%

General procedure for the preparation of azido allylsilanes. To a solution of the allylsilyl alcohol (1.0 equiv) and triethylamine (1.5 equiv) in CH_2Cl_2 at 0 °C, was added methanesulfonyl chloride (1.3 equiv). After stirring at 0 °C for 1 h, the reaction was quenched with saturated aqueous NH_4Cl. The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (3 × 20 mL). The combined organic layers were dried (Na_2SO_4), filtered, and concentrated. The crude product was filtered through a pad of silica gel (5:1 hexanes:EtOAc) and the filtrate was concentrated. The mesylate product thus obtained (1.0 equiv) was dissolved in DMF, and sodium azide (4.0 equiv) was added to the resulting solution. The reaction mixture was heated to 110 °C for 2 h. After cooling to room temperature, the reaction mixture was partitioned between water and ether. The layers were separated and the aqueous layer was extracted with ether. The combined

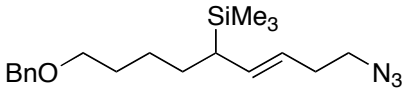
organic layers were washed with brine, dried (Na_2SO_4), filtered, and concentrated. The crude product was purified by chromatography to afford the azido allylsilane.



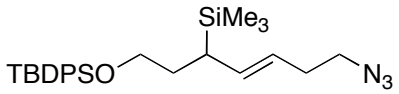
(5*S*, *E*)-1-Azido-5-trimethylsilylhept-3-ene (1). Prepared according to the general procedure from alcohol **5b** (0.09 g, 0.48 mmol). The crude product was purified by chromatography (20:1 hexanes:EtOAc) to give 0.093 g (92%) of a colorless oil. $[\alpha]_D$ –2.64 (*c* 1.25, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ –0.02 (s, 9H), 0.93 (t, *J* = 7.0 Hz, 3H), 1.35 (m, 2H), 1.55 (m, 1H), 2.34 (q, *J* = 6.9 Hz, 2H), 3.27 (dt, *J* = 2.5, 7.0 Hz, 2H), 5.23 (td, *J* = 6.8, 15,2 Hz, 1H), 5.36 (ddd, *J* = 1.1, 8.1, 15.2 Hz, 1H); ^{13}C (100.6 MHz, $CDCl_3$) δ –2.8, 14.7, 22.3, 32.8, 35.9, 52.0, 123.7, 135.6; IR (film) 2094, 1726 cm^{-1}; MS (CI) *m/z* 184 [(M^+ + H) – N_2], 167, 149, 73. It was not possible to obtain a HRMS of the M^+ peak.



(5*S*,*E*)-1-Azido-7-benzyloxy-5-trimethylsilylhept-3-ene (2). Prepared according to the general procedure from alcohol **9** (0.58g, 2.0 mmol). The crude product was purified by chromatography (10:1 hexanes:EtOAc) to give 0.54 g (85%) of a colorless oil. $[\alpha]_D$ +32.4 (*c* 0.21, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.01 (s, 9H), 1.62 (m, 2H), 1.84 (ddd, *J* = 12.8, 7.4, 2.3 Hz, 1H), 2.31 (qd, *J* = 6.4, 0.9 Hz, 2H), 3.24 (t, *J* = 7.0 Hz, 2H), 3.42 (m, 1H), 3.55 (m,1H), 4.51 (AB q, *J* = 11.4 Hz, Δν = 18.5 Hz), 5.22 (dt, *J* = 15.3, 6.8 Hz, 1H), 5.40 (ddd, *J* = 15.2, 8.1, 1.0 Hz, 1H) 7.4 (m, 5H); ^{13}C NMR (100.6 MHZ, $CDCl_3$) δ –2.9, 29.2, 30.1, 32.7, 51.4, 70.4, 73.4, 124.0, 127.9, 128.0, 128.8, 134.9, 139.1; IR (film) 2100, 1678 cm^{-1}; MS (CI) *m/z* 318 (M^+ + H), 91, 73; HRMS calcd for $C_{17}H_{28}SiN_3O$ (M^+ + H) 318.2002, found 318.1984.

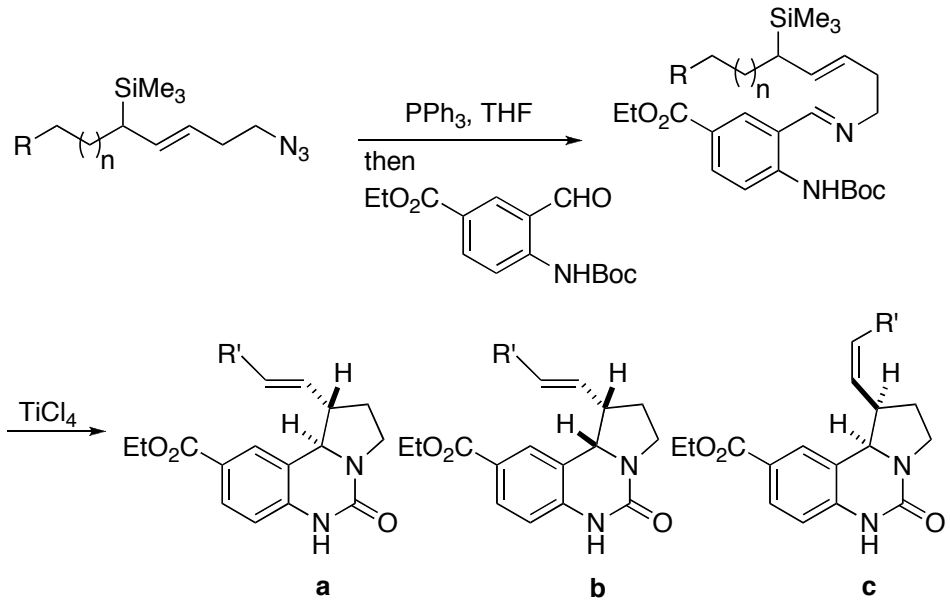


(±)-(*E*)-1-Azido-9-benzyloxy-5-trimethylsilyl-3-nonene (E1). Prepared according to the general procedure from alcohol **D1** (0.23 g, 0.72 mmol). The crude product was purified by chromatography (10:1 hexanes:EtOAc) to give 0.16 g (64%) of a colorless to pale yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ –0.01 (s, 9H), 1.27 (m,1H), 1.35 (m, 1H), 1.45-1.58 (m, 4H), 1.65 (m, 1H), 2.32 (dq, *J* = 0.9, 7.0 Hz, 2H), 3.26 (m, 2H), 3.48 (t, *J* = 6.6 Hz, 2H), 4.52 (s, 2H), 5.23 (td, *J* = 6.8, 15.3 Hz, 1H), 5.36 (dd, *J* = 5.9, 15.2 Hz, 1H), 7.36 (m, 5H); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ –3.3, 25.9, 28.5, 29.5, 32.3, 33.2, 51.5, 70.4, 72.8, 123.1, 127.4, 127.5, 128.2, 134.3, 135.1, 138.6; IR (film) 2096; MS (FAB+) *m/z* 318 [(M^+ + H) – N_2], 226, 136; HRMS calcd for $C_{19}H_{32}N_3SiO$ (M^+ + H) 346.2315, found 346.2315.



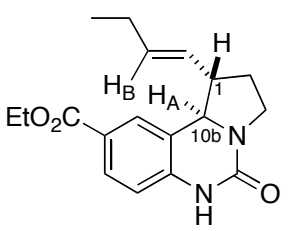
(±)-(*E*)-1-Azido-7-(*tert*-butyldiphenylsilyloxy)-5-trimethylsilylhept-3-en-1-ol (E2). Prepared according to the general procedure from alcohol **D2** (0.14 g, 0.32 mmol). Chromatography (10:1 hexanes:EtOAc) gave 0.11 g (76%) of a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ –0.02 (s, 9H), 1.07 (s, 10H), 1.56 (m, 2H), 1.77 (m, 1H), 2.24 (q, *J* = 6.7 Hz, 2H), 3.17 (td, *J* = 7.3, 1.6 Hz, 2H), 3.59 (m, 1H), 3.71 (m, 1H), 5.10 (dt, *J* = 15.3, 6.8 Hz, 1H), 5.27 (dd, *J* = 15.2, 9.1 Hz, 1H), 7.42 (m, 6H), 7.68 (m, 4H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ –2.9, 19.6, 27.3, 29.6, 31.9, 32.7, 51.8, 63.8, 123.6, 128.0, 129.9, 134.5, 134.9, 136.0; IR (film) 2095, 1429 cm^{-1}; MS (FAB+) *m/z* 466 (M^+ + H), 438 [(M^+ + H) – N_2], 313, 154; HRMS calcd for $C_{26}H_{43}N_4Si_2O$ (M^+ + NH_4) 483.2975, found 483.2970.

Scheme F. *$TiCl_4$-promoted cyclizations of allylsilylimines.*

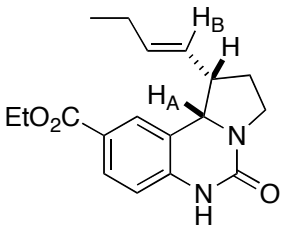


General procedure for $TiCl_4$-promoted cyclizations of imines. A solution of the azido allylsilane (1.0 equiv) and triphenylphosphine (1.0 equiv) in THF was stirred at room temperature for 3 h. Ethyl 4-(*N-tert*-butoxycarbonyl)amino-3-formylbenzoate[1] (1.0 equiv) was then added, and the reaction mixture was heated at reflux for 20 h. After cooling to room temperature, the reaction mixture was concentrated, triturated with ether, filtered through Florisil®, and the filtrate was concentrated. The crude imine thus obtained was dissolved in CH_2Cl_2 (to form a ca. 0.01 M solution) and the resulting solution was cooled to 0 °C in an ice bath. $TiCl_4$ (5.0 equiv) was then added dropwise to the reaction mixture at 0 °C. Upon compete addition of $TiCl_4$, the ice bath was removed and the reaction mixture was stirred at room temperature for 18 h. The reaction was quenched by the slow addition of a 10% aq NaOH solution, and the mixture was extracted with CH_2Cl_2. The combined organic layers were washed with brine, dried (Na_2SO_4), filtered, and concentrated. The residue obtained was subjected to chromatography to afford product. ^1H NMR integration values of the crude reaction mixtures were used for product ratio determinations (normalized to 100%).

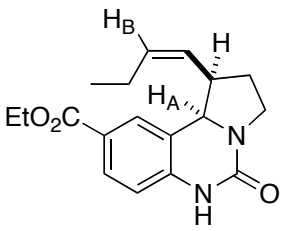
Determination of product stereochemistry and isomer ratios: Chemical shift and coupling constant values for the doublet corresponding to H_A (H10b) of the 1,10b-cis and 1,10b-trans isomers were used for the assignment of relative stereochemistry of the cyclization reaction products. The assignments of doublets corresponding to the cis and trans isomers were made in analogy to previous work and later confirmed through X-ray crystallography (see below).[1] In most cases, the olefin geometry was readily determined by the corresponding coupling constants for the olefinic hydrogens. However, in some examples the corresponding *J* values were obscured in the ^{1}H NMR for the most minor isomer. In these cases, the olefin geometry was assigned by default.



12a (15.0%)
H_A: δ 4.43, *J* = 9.6 Hz
H_B: δ 5.86, dt, *J* = 15.3, 6.4 Hz



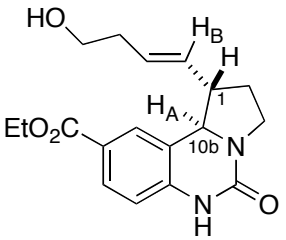
12b (66.4%)
4.84, *J* = 4.6 Hz
5.14, dd, *J* = 8.9, 15.3 Hz



12c (18.6%)
4.87, *J* = 4.5 Hz
5.38, dt, 10.9, 7.3 Hz

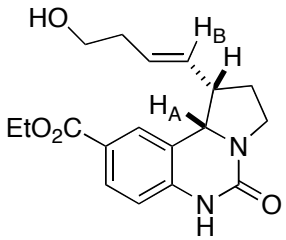
9-Carboethoxy-1-(but-1'-enyl)-2,3,6,10b-hexahydro-1*H*-pyrrolo[1,2-*c*]quinazoline-5-one (12a–c). Prepared according to the general procedure from azido allylsilane **1** (0.07 g, 0.34 mmol). Chromatography (1:3 hexanes:EtOAc) gave 0.09 g (85%) of a yellow solid isolated as a mixture of isomers. IR (KBr) 1709, 1673, 1617 cm^{-1}; MS (CI) *m/z* 315 (M^+ + H), 235, 111; HRMS calcd for $C_{18}H_{23}N_2O_3$ (M^+ + H) 315.1709, found 315.1704. Major isomer, **(1*S*,10b*R*)-(*E*)-12b**: ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.73 (t, *J* = 7.4 Hz, 3H), 1.39 (t, *J* = 7.1 Hz, 3H), 1.81 (m, 2H), 1.93 (dd, *J* = 7.1, 11.8 Hz, 1H), 2.18 (m, 2H), 3.24 (m, 1H), 3.55 (m, 1H), 3.79 (m, 1H), 4.35 (dq, *J* = 1.6, 7.0, Hz, 2H), 4.84 (d, *J* = 4.6 Hz, 1H), 5.14 (dd, *J* = 8.9, 15.3 Hz, 1H), 5.61 (dt, *J* = 15.3, 6.9Hz, 1H), 6.84 (d, *J* = 8.4 Hz, 1H), 7.60 (s, 1H), 7.77 (d, *J* = 8.3, 1H), 9.61 (s, 1H); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 13.7, 14.2, 25.5, 30.1, 43.4, 44.7, 60.5, 61.5, 113.7, 118.1, 123.3, 126.1, 128.7, 129.7, 135.4, 141.8, 153.3, 166.1. **(1*R*,10b*S*)-(*Z*)-12c** (diagnostic peaks only): ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.87 (d, *J* = 4.5 Hz, 1H), 5.38 (dt, *J* = 10.9, 7.3 Hz, 1H).

(1*S*,10b*S*)-(*E*)-12a (diagnostic peaks only): ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.43 (d, *J* = 9.6 Hz, 1H), 5.86 (dt, *J* = 15.3, 6.4 Hz, 1H).



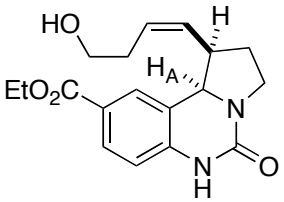
13a (87.3%)

H$_A$: δ 4.45, *J* = 9.7 Hz
H$_B$: δ 5.68, dd, *J* = 15.1, 8.9 Hz



13b (9.8%)

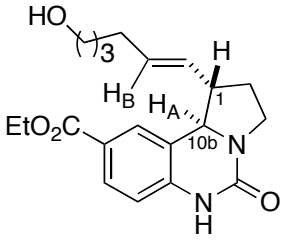
4.81, *J* = 4.6 Hz
5.26, dd, *J* = 9.8, 15.4 Hz



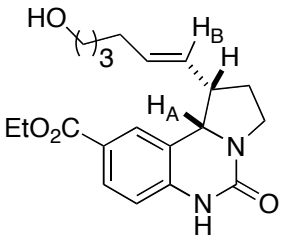
13c (2.9%)

4.85 d, *J* = 4.5 Hz
signal obscured

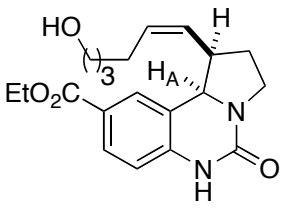
9-Carboethoxy-1-(4'-hydroxybut-1'-enyl)-2,3,6,10b-tetrahydro-1*H*-pyrrolo[1,2-*c*]quinazolin-5-one (13a–c). Prepared according to the general procedure using azido allylsilane **2** (0.10 g, 0.32 mmol). Chromatography (1:3 hexanes:EtOAc) gave 0.085 g (80%) of a yellow solid as a mixture of isomers. The major isomer **(1*S*, 10b*S*)-(*E*)-13a** was isolated by crystallization from the reaction mixture (EtOAc/hexanes, 42%). Major isomer **(1*S*,10b*S*)-(*E*)-13a**: mp 162–164 °C (EtOAc/hexanes); $[\alpha]_D$ –22.0 (*c* 0.45, $CHCl_3$). ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.38 (t, *J* = 7.0 Hz, 3H), 1.90 (quintet, *J* = 11.1 Hz, 1H), 2.21 (m, 1H), 2.4 (q, *J* = 6.1 Hz, 2H), 2.98 (quintet, *J* = 9.5 Hz, 1H), 3.21 (br, s, 1H), 3.66 (m, 2H), 3.83 (t *J* = 5.6 Hz, 2H), 4.36 (q, *J* = 7.1 Hz, 2H), 4.45 (d, *J* = 9.7 Hz, 1H) 5.68 (dd, *J* = 15.1, 8.9 Hz, 1H), 5.86 (dt, *J* = 14.9, 6.7 Hz, 1H), 6.80 (d, *J* = 8.4 Hz, 1H), 7.83 (d, *J* = 8.4 Hz, 1H), 8.28 (s, 1H), 8.74 (br, s, 1H); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 14.3, 30.6, 36.2, 43.5, 50.0, 60.1, 61.0, 61.7, 113.5, 121.1, 123.4, 126.7, 130.0, 131.5, 132.8, 141.3, 152.8, 166.7; IR (KBr) 3216, 1709, 1671, 1615 cm^{-1}; MS (CI) *m/z* 331 (M^+ + H), 232; HRMS calcd for $C_{18}H_{22}N_2O_4$ (M^+ + H) 331.1658, found 331.1662. **(1*S*,10b*R*)-(*E*)-13b** (diagnostic peaks only): ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.81 (d, *J* = 4.6 Hz, 1H), 5.26 (dd, *J* = 9.8, 15.4 Hz, 1H). **(1*R*,10b*S*)-(*Z*)-13c** (diagnostic peaks only): ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.85 (d, *J* = 4.5 Hz, 1H).



15a (20.2%)
H_A: δ 4.43, d, *J* = 9.7 Hz
H_B: δ 5.80, dt, *J* = 15.2, 6.8 Hz

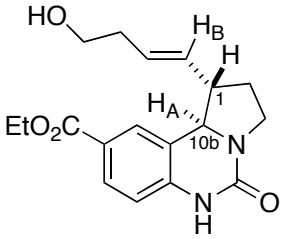


15b (67.9%)
4.83, d, *J* = 4.6 Hz
5.15, dd, *J* = 8.6, 15.4 Hz



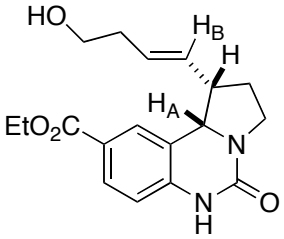
15c (11.9%)
4.86, d, *J* = 4.5 Hz
signal obscured

(±)-9-Carboethoxy-1-(6'-hydroxyhex-1'-enyl)-1,2,3,6,10b-tetrahydro-1*H*-pyrrolo[1,2-*c*]quinazoline-5-one (15a–c). Prepared according to the general procedure from azido allylsilane **E1** (0.30 g, 0.87 mmol). Chromatography (1:3 hexanes:EtOAc) gave 0.23 g (73%) of a yellow foam as a mixture of isomers. IR (film) 3328, 1675, 1614 cm^{-1}; MS (CI) *m/z* 359 (M^+ + H), 232; HRMS calcd for $C_{20}H_{27}N_2O_4$ (M^+ + H) 359.1971, found 359.1971. Major isomer **(1*S*,10b*R*)-(*E*)-15b**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.39 (t, *J* = 7.1 Hz, 3H), 1.63 (m, 2H), 1.98 (m, 4H), 2.20 (m, 3H), 3.46 (t, *J* = 6.1 Hz, 2H), 3.67 (m, 2H), 3.81 (m, 1H), 4.35 (m, 2H), 4.83 (d, *J* = 4.6 Hz, 1H), 5.15 (dd, *J* = 8.6, 15.4 Hz, 1H), 5.54 (dt, *J* = 15.3, 6.9 Hz, 1H), 6.78 (d, *J* = 8.1 Hz, 1H), 7.70 (s, 1H), 7.82 (dd, *J* = 1.6, 8.4 Hz, 1H), 9.02 (s, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 14.8, 25.6, 29.6, 32.1, 32.5, 43.7, 45.3, 61.3, 62.2, 63.1, 114.2, 118.6, 124.0, 128.1, 129.5, 130.3, 133.9, 142.2, 153.6, 166.8. **(1*S*,10b*S*)-(*E*)-15a** (diagnostic peaks only): ^{1}H NMR (400 MHz, $CDCl_3$) 4.43 (d, *J* = 9.7 Hz, 1H), 5.80 (dt, *J* = 15.2, 6.8 Hz, 1H). **(1*R*,10b*S*)-(*Z*)-15c**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.86 (d, *J* = 4.5 Hz, 1H).



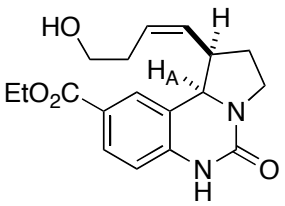
13a (52.8%)

H_A: δ4.46, *J* = 9.8 Hz
H_B: δ5.69, dd, *J* = 9.0, 15.4 Hz



13b (42.3%)

4.84, *J* = 4.7 Hz
5.29, dd, 9.2, 15.4 Hz)



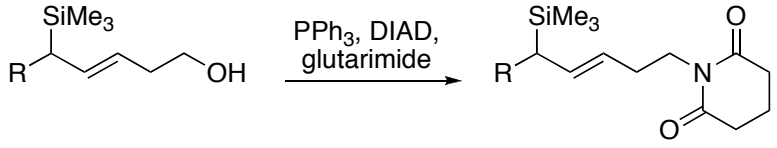
13c (4.9%)

4.89, *J* = 4.5 Hz
signal obscured

(±)-9-Carboethoxy-1-(4'-hydroxy-but-1'-enyl)-2,3,6,10b-tetrahydro-1*H*-pyrrolo[1,2-*c*]quinazolin-5-one (13a–c). Prepared according to the general procedure using azido allylsilane **E2** (0.11 g, 0.24 mmol). Chromatography (1:3 hexanes:EtOAc) gave 0.05 g (64%) of a yellow solid as a mixture of isomers. MS (FAB+) *m/z* 331 (M^+ + H), 232, 154, 136; HRMS calcd for $C_{18}H_{23}N_2O_4$ 331.1658, found 331.1649. Full product characterization for **13a–c** is given above. **(1*S*,10b*S*)-13a** (diagnostic peaks only): ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.46 (d, *J* = 9.8 Hz, 1H), 5.69 (dd, *J* = 9.0, 15.4 Hz, 1H). **(1*S*,10b*R*)-13b** (diagnostic peaks only): ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.84 (d, *J* = 4.7 Hz, 1H), 5.29 (dd, 9.2, 15.4 Hz) 5.63 (m, 1H). **(1*R*,10b*S*)-13c** (diagnostic peaks only): ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.89 (d, *J* = 4.5 Hz, 1H).

TFA-Promoted Cyclization Reactions

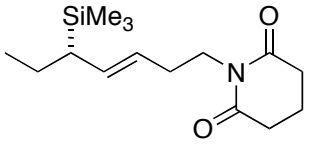
Scheme G. *Synthesis of N-allylsilyl glutarimides.*



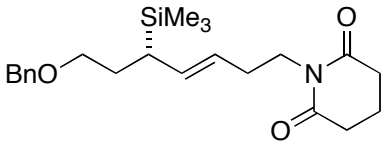
(*S*)-**5b**: R = Et
(*S*)-**9**: R = $(CH_2)_2$OBn
(±)-**D1**: R = $(CH_2)_4$OBn
(±)-**D2**: R = $(CH_2)_2$OTBDPS
D4: R = H
(±)-**D3**: R = $(CH_2)_2$OPMB

(*S*)-**21**: 82%
(*S*)-**23**: 80%
(±)-**25**: 90%
(±)-**27**: 62%
29: 98%
(±)-**G1**: 98%

General procedure for the preparation of *N*-allylsilyl glutarimides.[15] To a solution of the allylsilyl alcohol (1.0 equiv), glutarimide (1.5 equiv), and triphenylphosphine (1.5 equiv) in THF at 0°C was added diisopropyl azodicarboxylate (1.5 equiv). The reaction mixture was warmed to room temperature and stirred for 16 h. Water was then added, and the mixture was extracted with ether. The combined organic extracts were washed with brine, dried ($Na2SO_4$), filtered, and concentrated. The crude material was purified by chromatography to afford product.

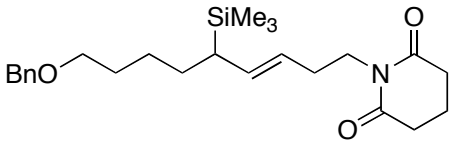


(5'*S*,*E*)-1-[5'-Trimethylsilylhept-3'-enyl]piperidine-2,6-dione (21). Prepared according to the general procedure from alcohol (*S*)-**5b** (0.26 g, 1.40 mmol). Chromatography (5:1 hexanes:EtOAc) gave 0.32 g (82%) of a pale yellow oil. $[\alpha]_D$ +6.2 (*c* 0.90, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ –0.05 (S, 9H), 0.88 (t, *J* = 7.1 Hz, 3H), 1.30 (m, 2H), 1.51 (m, 1H), 1.91 (quintet, *J* = 6.7 Hz, 2H), 2.24 (q, *J* = 7.1 Hz, 2H), 2.64 (t, *J* = 6.5 Hz, 4H), 3.82 (m, 2H), 5.21 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ –2.7, 14.7, 17.6, 22.3, 32.0, 33.3, 35.7, 40.0, 124.5, 134.6, 172.8; IR (film) 1726, 1673 cm^{-1}; MS (CI) *m*/*z* 282 (M^+ + H), 90, 73; HRMS calcd for $C_{15}H_{28}NsiO_2$ (M^+ + H) 282.1889, found 282.1891.

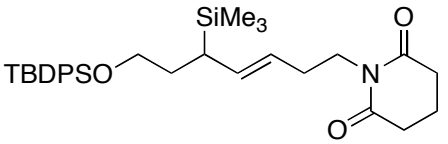


(5'*S*,*E*)-1-[7'-Benzyloxy-5'-trimethylsilylhept-3'-enyl]piperidine-2,6-dione (23). Prepared according to the general procedure from alcohol (*S*)-**9** (0.90 g, 3.0 mmol). Chromatography (6:1 to 3:1 hexanes:EtOAc) gave 0.93 g (80%) of a yellow oil. $[\alpha]_D$ +17.6 (*c* 0.68, $CHCl_3$). ^{1}H NMR (500 MHz, $CDCl_3$) δ –0.03 (s, 9H), 1.57 (m, 2H), 1.80

(m, 1H), 1.91 (quintet, J = 6.7 Hz, 2H), 2.21 (q, J = 7.0, 2H), 2.62 (t, J = 6.6 Hz, 4H), 3.39 (m, 1H), 3.51 (ddd, J = 3.9, 9.0, 17.8 Hz, 1H), 3.77 (t, J = 7.6 Hz, 2H), 4.50 (AB q, J = 11.4 Hz, $\Delta\nu$ = 22.2 Hz, 2H), 5.20 (dt, J = 15.2, 6.7 Hz, 1H), 5.30 (dd, J = 8.7, 15.3 Hz, 1H), 7.33 (m, 5H); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ –3.3, 17.1, 28.7, 29.4, 31.4, 32.8, 39.5, 70.0, 72.8, 124.3, 127.4, 127.5, 128.2, 133.4, 138.6, 172.3; IR (film) 1724, 1675 cm^{-1}; MS (CI) m/z 388 (M^+ + H), 298, 91; HRMS calcd for $C_{22}H_{34}NO_3Si$ 388.2308 (M^+ + H), found 388.2286.

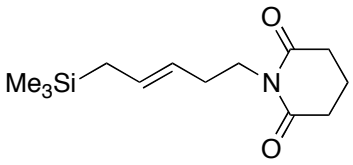


(±)-(*E*)-1-[9'-Benzyloxy-5'-trimethylsilylnon-3'-enyl]piperidine-2,6-dione (25). Prepared according to the general procedure from alcohol **D1** (0.24 g, 0.75 mmol). The crude product was purified by chromatography (6:1 to 3:1 hexanes:EtOAc) to give 0.28 g (90%) of a colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ –0.05 (s, 9H), 1.28 (m, 2H), 1.43 (m, 4H), 1.55 (m,1H), 1.64 (m, 1H), 1.89 (m, 2H), 2.25 (q, J = 6.9 Hz, 2H), 2.62 (t, J = 6.4 Hz, 4H), 3.46 (t, J = 6.5 Hz, 2H), 3.80 (m, 2H), 4.51 (s, 2H), 5.18 (dt, J = 15.2, 6.7 Hz, 1H), 5.25 (dd, J = 8.6, 15.3 Hz, 1H), 7.34 (m, 5H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ –2.8, 17.6, 22.1, 26.3, 28.5, 29.0, 30.0, 32.0, 33.3, 40.0, 70.8, 73.2, 124.4, 128.0, 128.7, 134.6, 139.1, 172.8 IR (film) 1727, 1675 cm^{-1}; MS (CI) m/z 416 (M^+ + H), 266, 91; HRMS calcd for $C_{24}H_{38}NO_3Si$ (M^+ + H) 416.2621, found 416.2622.

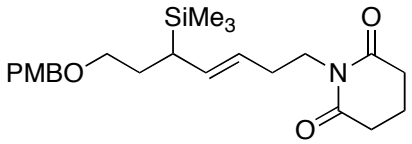


(±)-(*E*)-1-[7'-(*tert*-Butyldiphenylsilyloxy)-5'-trimethylsilylhept-3'-enyl]piperidine-2,6-dione (27). Prepared according to the general procedure from alcohol **D2** (0.14 g, 0.36 mmol). Chromatography (3:1 hexanes:EtOAc) afforded 0.12 g (62%) of a

pale yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ –0.05 (s, 9H), 1.06 (s, 10H), 1.52 (m, 2H), 1.73 (m, 1H), 1.85 (quintet, *J* = 6.4 Hz, 2H), 2.18 (q, 7.2 Hz, 2H), 2.58 (t, *J* = 6.5 Hz, 4H), 3.59 (m, 1H), 3.73 (m, 3H), 5.12 (dt, *J* = 15.3, 6.7 Hz, 1H), 5.20 (dd, *J* = 8.7, 15.3 Hz, 1H), 7.41 (m, 6H), 7.68 (m, 4H); ^{13}C NMR (125.7 MHz, $CDCl_3$) δ –3.4, 17.1, 19.1, 21.7, 26.8, 28.8, 31.4, 32.8, 39.4, 63.3, 124.1, 127.5, 129.4, 133.5, 134.3, 135.5, 172.3; IR (film) 1727, 1678 cm^{-1}; MS (FAB+) *m/z* 536 (M^+ + H), 307, 154; HRMS calcd for $C_{31}H_{49}N_2Si_2O_3$ (M^+ + NH_4) 553.3282, found 553.3293.



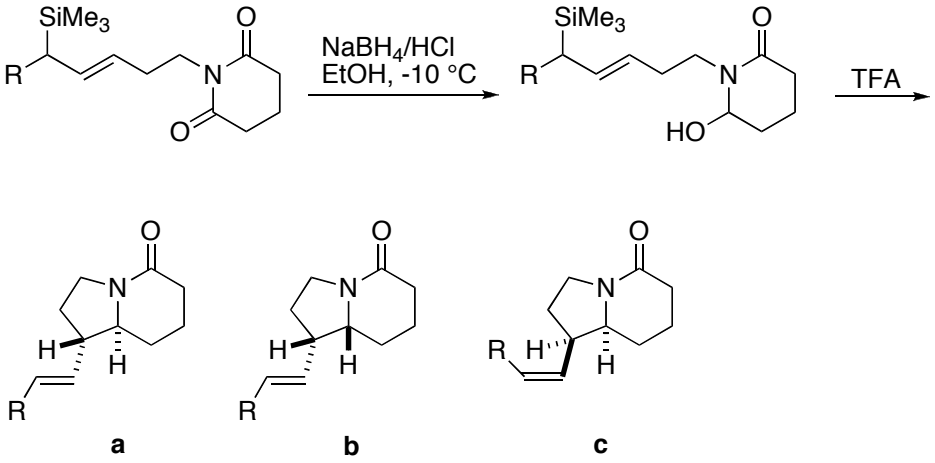
(*E*)-1-[5'-Trimethylsilylpent-3'-enyl]piperidine-2,6-dione (29). Prepared according to the general procedure using known alcohol **D4** (0.10 g, 0.63 mmol).[1] The crude product was purified by chromatography (5:1 to 1:1 hexanes:EtOAc) to give 0.15 g (94%) of a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ –0.02 (s, 9H), 1.31 (d, *J* = 8.0 Hz, 2H), 1.92 (quintet, *J* = 6.5 Hz, 2H), 2.20 (q, *J* = 7.7 Hz, 2H), 2.64 (t, *J* = 6.5 Hz, 4H), 3.78 (t, *J* = 7.5 Hz, 2H), 5.21 (dt, *J* = 15.1, 7.1 Hz, 1H), 5.44 (td, *J* = 8.0, 15.1 Hz, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ –1.6, 17.6, 23.1, 31.8, 33.3, 39.9, 125.0, 129.3, 172.8; IR (film) 1724, 1673 cm^{-1}; MS (CI) *m/z* 254 (M^+ + H) 199, 170, 73; HRMS calcd for $C_{13}H_{23}SiNO_2$ (M^+ + H) 254.1576, found 254.1573.



(±)-(*E*)-1-(7'-(4''-Methoxybenzyloxy)-5'-trimethylsilylhept-3'-enyl)piperidine-2,6-dione (G1). Prepared according to the general procedure from **D3** (0.40 g, 1.24 mmol). The crude product was purified by chromatography (3:1 to 2:1 hexanes:EtOAc) to give 0.51 g (98%) of a pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ

–0.05 (s, 9H), 1.53 (m, 2H), 1.76 (m, 1H), 1.90 (quintet, J = 6.6 Hz, 2H), 2.19 (q, J = 6.7 Hz, 2H), 2.62 (t, J = 6.5 Hz, 4H), 3.35 (m, 1H), 3.46 (m, 1H), 3.75 (t, J = 7.5 Hz, 2H), 3.80 (s, 3H), 4.41 (AB q J = 11.5 Hz, Δν =17.9 Hz, 2H), 5.18 (dt, J = 15.2, 6.5 Hz, 1H), 5.27 (dd J = 15.3, 8.6 Hz, 1H), 6.88 (m, 2H), 7.26 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ –3.28, 17.2, 28.7, 29.5, 31.5, 32.9, 39.6, 55.3, 70.0, 72.5, 113.7, 124.4, 129.2, 130.8, 133.5, 159.1, 172.4; IR (film) 1695, 1640, 1475 cm^{-1}; MS (ES+) m/z 418 (M^+ + H), 409, 354, 313, 227; HRMS calcd for $C_{23}H_{36}NO_4Si$ (M^+ + H) 418.2414, found 418.2431.

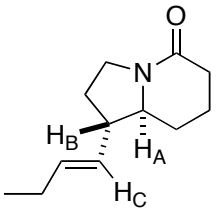
Scheme H. *TFA-promoted cyclizations of hydroxy lactams.*



General procedure for TFA-promoted cyclization reactions. According to the procedure of Speckamp.[15, 16] $NaBH_4$ (4.0 equiv) was added to a solution of the *N*-allylsilyl glutarimide in 100% EtOH at –10 °C. At 15 min intervals, 2–3 drops of 2N HCl in 100% EtOH were added while maintaining the temperature at –10 °C. After 4 to 6 h (monitored by TLC), the reaction was quenched with water, and the mixture was extracted with CH_2Cl_2 (3 × 20 mL). The combined organic extracts were dried (Na_2SO_4), filtered, and concentrated. The crude product was passed through a short column of silica gel (EtOAc) to afford a pale yellow oil. The hydroxy lactam thus obtained was dissolved in CH_2Cl_2 (to give a 0.01 to 0.005 M solution), and the resulting solution was cooled to 0 °C or –55 °C.

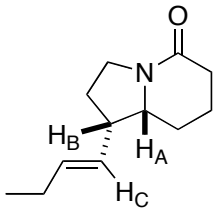
CF_3CO_2H (4.0 equiv) was then added dropwise to the reaction mixture and stirring was continued at the indicated temperature for 2h (at 0 °C) or 24 h (at –55 °C). The reaction was quenched with saturated aq $NaHCO_3$, and the aqueous layer was extracted with CH_2Cl_2. The combined organic layers were washed with brine, dried (Na_2SO_4), filtered, and concentrated. Chromatography afforded lactam product as a mixture of isomers. ^{1}H NMR integration values of the crude reaction mixtures were used for product ratio determinations (normalized to 100%).

Determination of product stereochemistry and isomer ratios: The assignment of relative configuration for the lactam products was based on the ^{1}H NMR chemical shifts and coupling patterns of the 1H and 8aH peaks. Chemical shift values for these hydrogens were established by HMQC and ^{1}H–^{1}H COSY NMR. The chemical shift of the C8a hydrogen was highly dependent on the relative configuration of the C1 hydrogen in a manner consistent with reports by Speckamp for bicyclic lactams of similar structure.[15, 17] The C8a hydrogen absorption of the cis diastereomer occurred between 3.54 and 3.57 ppm, while that of the trans isomer occurred between 3.04 and 3.06 ppm. The cis diastereomer also displayed two C1 hydrogen absorptions centered around 3.15 and 2.73 ppm that were established as arising from Z and E olefin isomers, respectively. Diastereomeric and olefin geometric ratios were determined by ^{1}H NMR integration values.



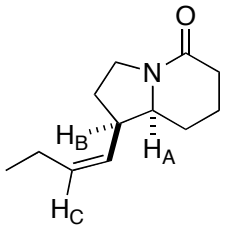
22a (7.9%)

H_A: δ 3.04, td, *J* = 10.6, 3.4 Hz
H_C: δ 5.27, dd, *J* = 7.9, 15.3 Hz



22b (28.2%)

H_B: δ 2.75, quint, *J* = 5.5 Hz
H_C: δ 5.19, dd, *J* = 5.7, 15.3 Hz

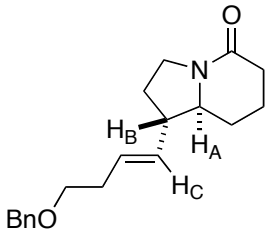


22c (63.9%)

H_A: δ 3.54, dt, *J* = 11.5, 4.4 Hz
H_B: δ 3.11, quint, *J* = 5.5 Hz
H_C: δ 5.10, t, *J* = 1.5, 10.7 Hz

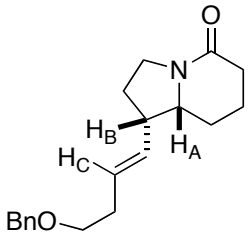
1-(But-1'-enyl)hexahydroindolizin-5-one (22a–c). Prepared according to the general procedure from (*S*)-**21** (0.11 g, 0.39 mmol); cyclization was done at –55 °C. Chromatography (EtOAc) gave 0.070 g (92%) of a colorless oil as a mixture of isomers. IR (film) 1632, 1462 cm^{-1}; MS (CI) *m/z* 194 (M^+ + H), 111, 55; HRMS calcd for $C_{12}H_{20}NO$ (M^+ + H) 194.1545, found 194.1552. Major isomer **(1*R*,8a*S*)-(*Z*)-22c**: ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.98 (t, *J* = 7.5 Hz, 3H), 1.35 (m, 1H), 1.64-1.81 (m, 3H), 1.92-2.09 (complex, 4H), 2.24 (m, 1H), 2.40 (dd, *J* = 6.1, 17.8 Hz, 1H), 3.11 (quintet, *J* = 5.5 Hz, 1H), 3.49 (m, 1H), 3.54 (dt, *J* = 11.5, 4.4 Hz, 1H), 3.63 (ddd, *J* = 8.5, 8.0, 8.1 Hz, 1H), 5.10 (tt, *J* = 1.5, 10.7 Hz, 1H), 5.51 (td, *J* = 7.6, 10.3 Hz, 1H); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 14.2, 20.8, 25.2, 29.2, 31.1, 39.7, 43.2, 61.7, 126.0, 133.4, 169.4. **(1*S*,8a*R*)-(*E*)-22b** (diagnostic peaks only): ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.75 (quintet, *J* = 5.5 Hz, 1H), 5.19 (dd, *J* = 5.7, 15.3 Hz, 1H), 5.53 (dt, *J* = 15.0, 6.3 Hz, 1H); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 13.7, 20.7, 25.5, 28.9, 31.1,43.4, 45.4, 61.8, 126.2, 134.3.

(1*S*,8a*S*)-(*E*)-22a (diagnostic peaks only): ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.04 (td, *J* = 10.6, 3.4 Hz, 1H), 5.27 (dd, *J* = 7.9, 15.3 Hz, 1H), 5.61 (dt, *J* = 5.8, 15.3 Hz, 1H); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 13.6, 20.9, 25.4, 29.4, 44.1, 49.7, 63.4, 127.3, 134.9.



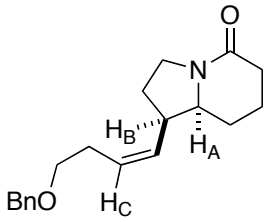
24a (80.3%)

H_A: δ 3.05, td, *J* = 10.6, 3.2 Hz
H_C: δ 5.39, ddd, *J* = 1.1, 8.0, 15.4 Hz



24b (13.1%)

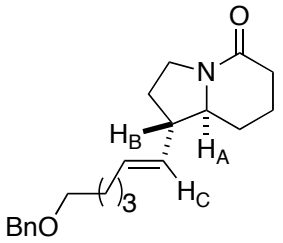
H_B: δ 2.78, quint, *J* = 5.5 Hz
H_C: δ 5.31, td, *J* = 9.5, 15.3 Hz



24c (6.6%)

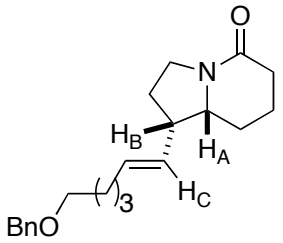
H_B: δ 3.13, quint, *J* = 5.5 Hz
H_C: δ 5.26, td, *J* = 10.6, 1.2 Hz

1-(4'-Benzyloxy-but-1'-enyl)hexahydroindolizin-5-one (24a–c). Prepared according to the general procedure using **23** (0.25 g, 0.65 mmol); cyclization was done at –55 °C. Chromatography (EtOAc) gave 0.17 g (87%) of a colorless oil as a mixture of isomers. IR (film) 1726, 1644 cm^{-1}; MS (CI) *m/z* 300 (M^+ + H), 111, 91; HRMS calcd for $C_{19}H_{26}NO_2$ (M^+ + H) 300.1964, found 300.1951. Major isomer **(1*S*,8a*S*)-(*E*)-24a**: ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.17 (dq, *J* = 1.9, 11.0 Hz, 1H), 1.64 (m, 2H), 1.95 (m, 1H), 2.06 (m, 2H), 2.26 (m, 2H), 2.38 (q, *J* = 6.7 Hz, 2H), 2.45 (dd, *J* = 6.6, 18.0 Hz, 1H), 3.05 (td, *J* = 10.6, 3.2 Hz, 1H), 3.53 (t, *J* = 7.4 Hz, 4H), 4.53 (s, 2H), 5.39 (ddd, *J* = 1.1, 8.0, 15.4 Hz, 1H), 5.62 (td, *J* = 6.8, 15.4 Hz, 1H), 7.34 (m, 5H); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 20.9, 27.4, 29.3, 31.0, 33.0, 44.1, 49.8, 63.3, 69.7, 72.8, 127.5, 127.5, 128.3, 129.5, 130.4, 138.3, 169.1. **(1*R*,8a*S*)-(*Z*)-24c** (diagnostic peaks only): ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.35 (m, 1H), 3.13 (quintet, *J* = 5.5 Hz, 1H), 5.26 (td, *J* = 10.6, 1.2 Hz, 1H); **(1*S*,8a*R*)-(*E*)-24b** (diagnostic peaks only): ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.78 (quintet, *J* = 5.5 Hz, 1H), 5.31 (dd, *J* = 9.5, 15.3 Hz, 1H).



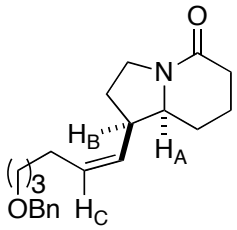
26a (19.3%)

H_A: δ 3.04, td, *J* = 10.7, 3.4 Hz
H_C: δ 5.29, dd, *J* = 7.9, 15.1 Hz



26b (25.3%)

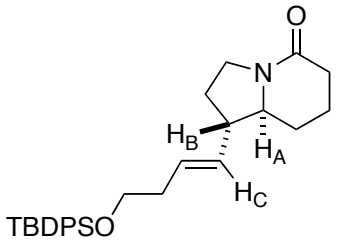
H_B: δ 2.76, quint, *J* = 5.7 Hz
H_C: δ 5.22, dd, *J* = 5.6, 15.3 Hz



26c (55.4%)

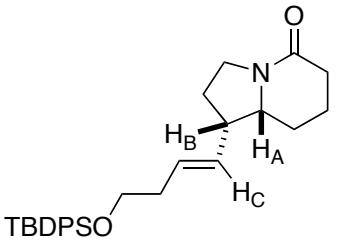
H_B: δ 3.09, quint, *J* = 5.3 Hz
H_C: δ 5.16, t, *J* = 10.8 Hz

(±)-1-(6'-Benzyloxyhex-1'-enyl)hexahydroindolizin-5-one (26a–c). Prepared according to the general procedure from **25** (0.10, 0.24 mmol); cyclization was done at 0 °C. Chromatography (20:1 EtOAc:MeOH) gave 0.070 g (82%) of a colorless oil as a mixture of isomers. IR (film) 1639, 1453 cm^{-1}; MS (FAB+) *m/z* 328 (M^+ + H), 236, 154; HRMS calcd for $C_{21}H_{30}NO_2$ (M^+ + H) 328. 2277, found 328.2261. Major isomer **(1*R**,8a*S**)-(*Z*)-26c**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.32 (m, 2H), 1.45 (quintet, *J* = 7.7 Hz, 2H), 1.65 (m, 4H), 1.76 (m, 1H), 1.94 (m, 2H), 2.03 (m, 3H), 2.24 (m, 1H), 2.41 (m, 1H), 3.09 (quintet, *J* = 5.3 Hz, 1H), 3.47 (t, *J* = 6.3 Hz, 2H), 3.63 (m, 1H), 4.52 (s, 2H), 5.16 (t, *J* = 10.8 Hz, 1H), 5.47 (dt, *J* = 10.4, 6.2 Hz, 1H), 7.33 (m, 5H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 21.3, 25.7, 26.7, 27.7, 29.6, 31.5, 40.2, 43.7, 50.2, 62.2, 70.6, 73.3, 127.4, 127.9, 128.7, 132.0, 132.9, 139.0, 169.9. **(1*S**,8a*R**)-(*E*)-26b** (diagnostic peaks only): ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.76 (quintet, *J* = 5.7 Hz, 1H), 5.22 (dd, *J* = 5.6, 15.3 Hz, 1H). **(1*S**,8a*S**)-(*E*)-26a** (diagnostic peaks only): ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.04 (td, *J* = 10.7, 3.4 Hz, 1H), 5.29 (dd, *J* = 7.9, 15.1 Hz, 1H), 5.58 (dt, *J* = 15.3, 6.7 Hz, 1H).



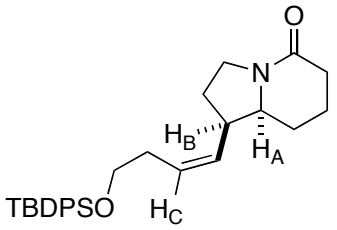
28a (39.2%)

H_A: δ 3.05, td, *J* = 10.7, 3.3 Hz
H_C: δ 5.36, dd, *J* = 7.9, 15.4 Hz



28b (17.4%)

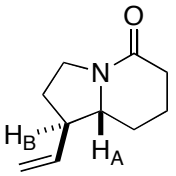
H_B: δ 2.77, quint, *J* = 5.6 Hz
H_C: δ 5.27, dd, *J* = 9.6, 15.3 Hz



28c (43.4%)

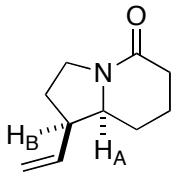
H_B: δ 2.96, quint, *J* = 5.5 Hz
H_C: δ 5.21, t, *J* = 10.8 Hz

(±)-1-[4'-(*tert*-Butyldiphenylsilyloxy)but-1'-enyl]hexahydroindolizin-5-one (28a–c). Prepared according to the general procedure from **27** (0.09 g, 0.15 mmol); cyclization was done at 0 °C. Chromatography (EtOAc) gave 0.05 g (74%) of a pale yellow oil as a mixture of isomers. IR (film) 1641, 1111 cm^{-1}; MS (FAB+) *m*/*z* 448 (M^+ + H), 390, 154, 136; HRMS calcd for $C_{28}H_{38}NsiO_2$ (M^+ + H) 448.2672, found 448.2653. Major isomer **(1*R**,8a*S**)-(*Z*)-28c**: ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.06 (s, 9H), 1.28 (m, 1H), 1.63 (m, 4H), 1.87–2.45 (m, 5H), 2.96 (quintet, *J* = 5.5 Hz, 1H), 3.45–3.74 (m, 5H), 5.21 (t, *J* = 10.8 Hz, 1H), 5.54 (dt, 10.2, 7.7 Hz, 1H), 7.41 (m, 6H), 7.69 (m, 4H); ^{13}C NMR (125.7 MHz, $CDCl_3$) δ 19.1, 20.8, 25.2, 26.7, 27.5, 29.1, 31.0, 35.9, 39.8, 43.2, 49.8, 61.6, 63.4, 127.5, 129.5, 133.8, 135.5, 169.4. **(1*S**,8a*R**)-(*E*)-28b** (diagnostic peaks only): ^{1}H NMR (500MHz, $CDCl_3$) δ 2.77 (quintet, *J* = 5.6 Hz, 1H), 5.27 (dd, *J* = 9.6, 15.3 Hz, 1H); ^{13}C NMR (127.5 MHz, $CDCl_3$) δ 19.1, 20.9, 28.9, 43.4, 63.6, 127.7, 135.5. **(1*S**,8a*S**)-(*E*)-28a** (diagnostic peaks only): ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.05 (td, *J* = 10.7, 3.3 Hz, 1H), 5.36 (dd, *J* = 7.9, 15.4 Hz, 1H), 5.61 (dt, *J* = 15.2, 6.9 Hz, 1H); ^{13}C NMR (127.5 MHz, $CDCl_3$) δ 19.2, 20.9, 29.3, 44.2, 61.8, 63.4, 127.5, 129.5, 133.7, 135.5, 169.2.



30a (10.3%)

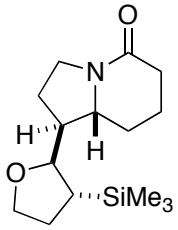
H_A: *signal obscured*
H_B: δ 3.08, td, *J* = 10.5, 3.2 Hz



30b (89.7%)

H_A: δ 3.56, dt, *J* = 9.0, 4.9 Hz
H_B: δ 2.81, quint, *J* = 5.7 Hz

(±)-1-Vinylhexahydroindolizin-5-one (30a,b). Prepared according to the general procedure from **29** (0.23 g, 0.91 mmol); cyclization was done at 0 °C. Chromatography (20:1 EtOAc:MeOH) gave 0.11 g (71%) of a pale yellow oil as a mixture of isomers. IR (film) 1637, 1465, 1447, 1410 cm^{-1}; MS (CI) m/z 166 (M^+ + 1), 150, 111; HRMS calcd for $C_{10}H_{16}NO$ (M^+ + H) 166.1232, found 166.1230. Major isomer **(1*R**,8a*S**)-30b**: ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.35 (m, 1H), 1.69 (m, 1H), 1.83 (m, 2H), 1.93-2.03 (complex, 2H), 2.27 (m, 1H), 2.42 (dd, *J* = 4.8, 14.3 Hz, 1H), 2.81 (quintet, *J* = 5.7, 1H), 3.50 (t, *J* = 8.3 Hz, 1H), 3.56 (dt, *J* = 9.0, 4.9 Hz, 1H), 3.65 (q, *J* = 8.3 Hz, 1H), 5.10 (dd, *J* = 1.5, 11.5 Hz, 2H), 5.63 (m, 1H); ^{13}C NMR (125.8 MHz, $CDCl_3$) d 20.9, 25.5, 28.5, 31.3, 43.3, 46.6, 61.6, 116.5, 135.9, 169.4. **(1*R**,8a*R**)-30a** (diagnostic peak only): ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.08 (td, *J* = 10.5, 3.2 Hz, 1H).

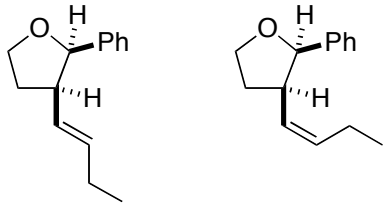


(1*S,8a*R**2'*S**,3'*R**)-1-(3'-Trimethylsilyltetrahydrofuran-2'-yl)hexahydroindolizin-5(1*H*)-one (37).** Prepared according to the general procedure from **G1** (0.28 g, 0.68 mmol); cyclization was done at 0 °C. The crude product was purified by chromatography (EtOAc) to give 0.03 g (20%) of a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.05 (s, 9H), 1.19 (m, 1H), 1.26 (m, 1H), 1.54 (m, 1H), 1.64–1.78 (complex, 2H), 1.90 (m, 3H), 2.08 (m, 1H), 2.33 (m, 2H), 2.42 (m, 2H), 3.35 (m, 1H), 3.46 (m, 1H),

3.61 (m, 1H), 3.70 (q, J = 7.3 Hz, 1H), 3.76 (dd, J = 7.7, 4.8 Hz, 1H), 3.81 (m, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ –2.4, 21.1, 26.8, 28.9, 29.8, 30.2, 30.9, 44.1, 50.7, 62.4, 67.4, 83.0, 169.4; IR (film) 1640, 1460 cm^{-1}; MS (ES+) m/z 282 (M^+ + H); HRMS calcd for $C_{15}H_{28}NO_2Si$ 282.1889 (M^+ + H), found 282.1914.

TMSOTf-Promoted Oxenium Cyclization Reactions

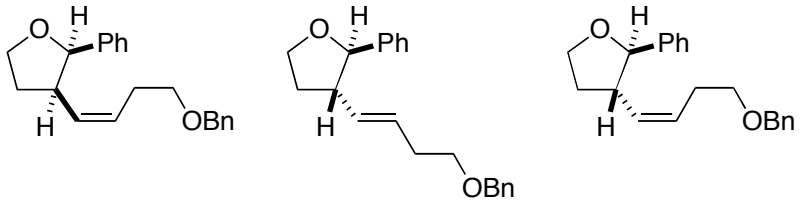
General procedure for oxenium ion cyclization reactions. According to the procedure of Ito,[18] TMSOTf (0.83 mmol) was added to a solution of the allylsilyl alcohol (0.75 mmol) and aldehyde (0.83 mmol) in CH_2Cl_2 at –78 °C. After stirring at –78 °C for 2.5 h, a 10% aq NaOH solution (10 mL) was added to the reaction mixture, which was subsequently warmed to room temperature. The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (3 × 20 mL). The combined organic layers were washed with brine, dried (Na_2SO_4), filtered, and concentrated. The crude reaction mixture was purified by chromatography to afford the corresponding products.



(***E***)-33a (***Z***)-33b

(±)-3-But-1-enyl-2-phenyl tetrahydrofuran (33a,b). Prepared according to the general procedure using allylsilane (±)-**1** (0.080 g, 0.44 mmol) and benzaldehyde (0.050 g, 0.49 mmol). The crude product was purified by chromatography (20:1 hexanes:ether) to give 0.061 g (67%) of a colorless oil as a 2:1 mixture of olefin isomers based on ^{1}H NMR integration. IR (film) 1604, 1493, 1451 cm^{-1}; MS (CI) m/z 203 (M^+ + H), 159, 96, 81; HRMS calcd for $C_{14}H_{19}O$ (M^+ + H) 203.1436, found 203.1426. Major isomer **(2*R**,3*R**)-(*E*)-33a**: ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.80 (t, J = 7.5 Hz, 3H), 1.85 (quintet, J = 6.7 Hz, 2H), 1.93 (m, 1H), 2.16 (m, 1H), 3.08 (quintet, J = 7.5 Hz, 1 H), 4.00 (q, J =

8.1 Hz, 1H), 4.30 (m, 1H), 4.83 (dd, *J* = 9.0, 15.5 Hz, 1H), 5.04 (d, *J* = 7.2 Hz, 1H), 5.42 (td, *J* = 6.4, 15.3 Hz, 1H), 7.27 (m, 5H); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 13.5, 25.3, 32.2, 47.2, 68.0, 83.5, 126.5, 126.7, 127.6, 128.1, 133.2, 140.6. **(2*R**,3*R**)-(*Z*)-1.70b** (diagnostic peaks only): ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.43 (m, 1H), 5.25 (dt, *J* = 10.8, 7.3 Hz, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 14.7, 21.2, 33.4, 42.0, 68.4, 83.7, 126.9, 127.2, 128.1, 128.4, 132.8, 141.0.



(*Z*)-34a **(*E*)-34b** **(*Z*)-34c**

(±)-3-(4'-Benzyloxybut-1'-enyl)-2-phenyl tetrahydrofuran (34a–c). Prepared according to the general procedure from allylsilane (±)-**2** (0.22 g, 0.75 mmol) and benzaldehyde (0.09 g, 0.83 mmol). The crude product was purified by chromatography (20:1 to 5:1 hexanes:ether) to give 0.13 g (56%) of a colorless oil as a 3:1 (E/Z) mixture of olefin isomers, and 1:12 overall cis:trans (i.e., **a**:(**b**+**c**) diastereomeric ratio based on ^{1}H NMR integration. IR (film) 1603, 1494, 1453 cm^{-1}; MS (CI) *m/z* 309 (M^+ + H), 217, 104; HRMS calcd for $C_{21}H_{25}O_2$ (M^+ + H) 309.1855, found 309.1847. Major isomer **(2*R**,3*S**)-(*E*)-34b**: ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.97 (m, 1H), 2.28 (m, 1H), 2.36 (q, *J* = 6.8 Hz, 2H), 2.68 (quintet, *J* = 8.2 Hz, 1H), 3.51 (t, *J* = 6.8 Hz, 2H), 4.10 (td, *J* = 8.4, 4.3 Hz, 1H), 4.14 (q, *J* = 8.3 Hz, 1H), 4.51 (d, *J* = 8.4 Hz, 1H), 4.54 (s, 2H), 5.46 (td, *J* = 6.7, 15.4 Hz, 1H), 5.56 (dd, *J* = 7.9, 15.4 Hz, 1H), 7.33 (m, 10 H); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 33.0, 33.7, 47.3, 51.7, 68.1, 69.9, 72.8, 125.9, 127.2, 127.4, 127.6, 127.6, 128.1, 128.3, 131.2, 138.4, 141.7. **(2*R**,3*S**)-(*Z*)-34c** (diagnostic peaks only): ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.11 (quintet, *J* = 8.1 Hz, 1H), 3.30 (t, *J* = 6.9 Hz, 2H), 4.00 (q, *J* = 8.1 Hz, 1H), 4.30 (td, *J* = 7.0, 2.7 Hz, 1H), 4.46 (s, 2H), 5.04 (d, *J* = 7.2 Hz, 1H); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 32.1, 32.7, 68.0, 69.9, 72.7, 126.4, 126.7, 128.3, 128.6,

131.3138.4. **(2*R**,3*R**)-(*Z*)-34a** (diagnostic peaks only): ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.31 (dt, *J* = 11.2, 3.4 Hz, 1H).

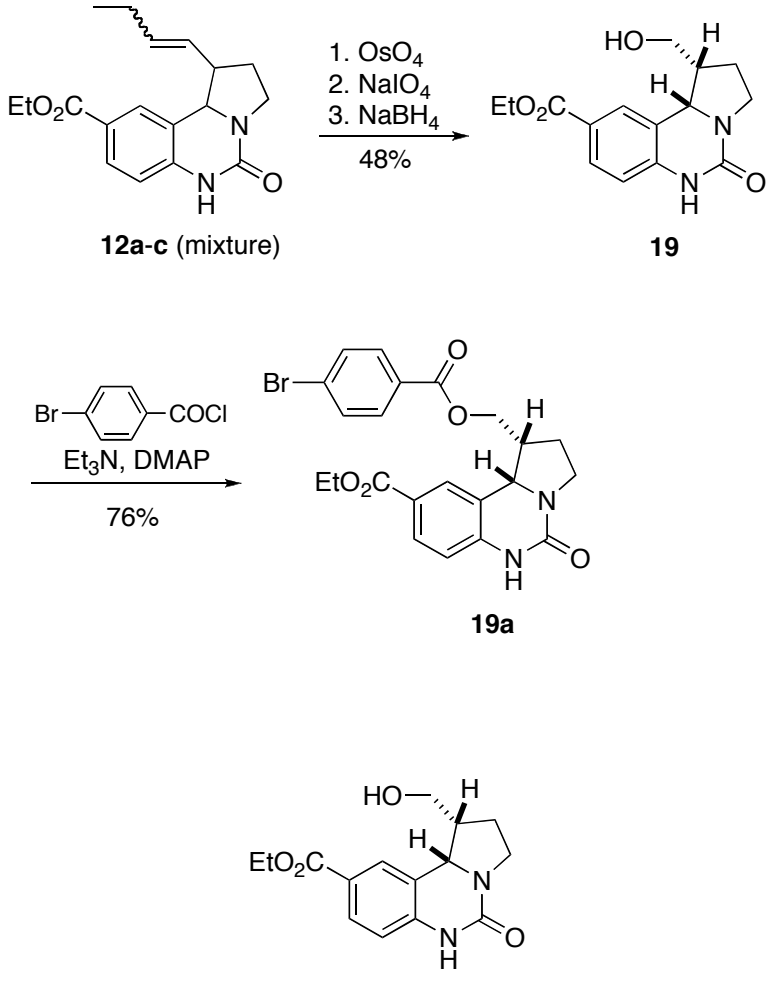
Determination of Absolute Configurations

The absolute configuration of the major isomer obtained in the cyclization reactions employing the enantioenriched azido allylsilanes (*S*)-**1.10** and (*S*)-**1.11** were determined by heavy atom anomalous dispersion X-ray analysis of the corresponding *p*-bromobenzoyl derivatives. In general, preparation of the latter was accomplished by oxidative cleavage of the side chain olefin followed by reduction to the corresponding alcohol and esterification. X-Ray data is given in the Appendix.

General procedure for oxidative cleavage/reduction reactions. OsO_4 (5 mg/mL solution in water, 0.05 equiv) was added to a solution of the olefin (1.0 equiv) and NMO (50% w/v solution in water, 1.5 equiv) in acetone/water/*t*-BuOH (5:5:1, 11 mL total volume), and the resulting mixture was stirred at room temperature for 24 h. Saturated aq $NaHSO_3$ was then added, and the mixture was stirred for 20 min at room temperature. The reaction mixture was extracted several times (EtOAc), and the combined organic extracts were dried (Na_2SO_4), filtered, and concentrated. The resulting residue was passed through a pad of silica gel (10:1 CH_2Cl_2:MeOH) to give the corresponding diol. The diol thus obtained (1.0 equiv) was dissolved in 10% aq THF (ca. 10 mL) and cooled to 0 °C. $NaIO_4$ (1.3 equiv) was then added and the mixture was stirred at 0 °C for 1 h. The reaction mixture was diluted with water (ca. 5 mL) and extracted with EtOAc. The combined organic extracts were dried (Na_2SO_4), filtered through Celite, and concentrated. The resulting residue was dissolved in MeOH (ca. 10 mL) and cooled to 0 °C. $NaBH_4$ (5.0 equiv) was then added, the ice bath was removed, and stirring at room temperature was continued for 2 h. The reaction was quenched with water and extracted with CH_2Cl_2. The combined organic extracts were dried (Na_2SO_4), filtered, and concentrated. Chromatography afforded the corresponding alcohol.

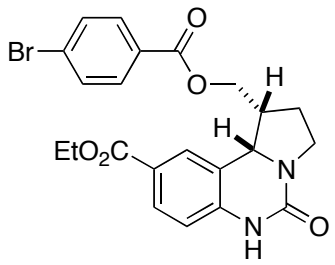
General procedure for the preparation of *p*-bromobenzoyl esters. To a solution of the alcohol (1.0 equiv), triethylamine (3.0 equiv), and a spatula tip amount of DMAP in CH_2Cl_2 (ca. 5.0 mL) at 0 °C, was added 4-bromobenzoyl chloride (1.2 equiv). The reaction mixture was subsequently allowed to warm to room temperature and stirred for 16 h. A saturated aqueous solution of NH_4Cl (10 mL) was then added to the reaction mixture and the aqueous layer was extracted with CH_2Cl_2 (3 × 20 mL). The combined organic extracts were washed with brine (1 × 20 mL), dried (Na_2SO_4), filtered, and concentrated. Chromatography gave the corresponding *p*-bromobenzoyl ester.

Scheme H. *Preparation of p-bromobenzoyl ester* **19a**.

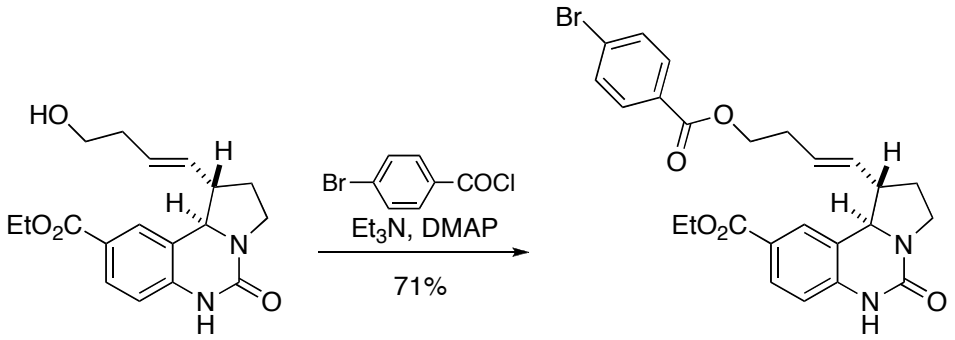


(1*R*,10a*S*)-9-Carboethoxy-1-hydroxymethyl-2,3,6,10b-tetrahydro-1*H*-pyrrolo[1,2-*c*]quinazolin-5-one (19). Prepared according to the general procedure for

the oxidative cleavage/reduction reaction sequence using the purified product mixture containing **12a–c** (0.18 g, 0.58 mmol). Chromatography (20:1 EtOAc:MeOH) gave 0.081 g (48%) of **19** (isolated as a single isomer) as a white foam. $[\alpha]_D$ –54.0 (*c* 0.05, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.40 (t, *J* = 7.1 Hz, 3H), 2.06 (m, 1H), 2.27 (ddd, *J* = 2.2, 8.5, 12.8 Hz, 1H), 2.81 (m, 1H), 3.33 (m, 1H), 3.50 (dt, *J* = 1.2, 10.6 Hz, 1H), 3.69 (m, 1H), 3.87 (m, 1H), 4.36 (t, *J* = 7.6 Hz, 2H), 4.93 (d, *J* = 4.9 Hz, 1H), 6.81 (d, 8.4 Hz, 1H), 7.78 (s, 1H), 7.84 (d, *J* = 8.4 Hz, 1H), 8.62 (br, s, 1H); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 14.1, 14.3, 24.7, 43.2, 43.3, 60.5, 60.9, 113.9, 117.0, 123.7, 128.0, 130.2, 141.2, 152.4, 166.0; IR (film) 3320, 1669, 1614 cm^{-1}; MS (CI) *m/z* 291 (M^+ + H), 122, 105; HRMS calcd for $C_{15}H_{19}N_2O_4$ (M^+ + H) 291.1345, found 291.1326.



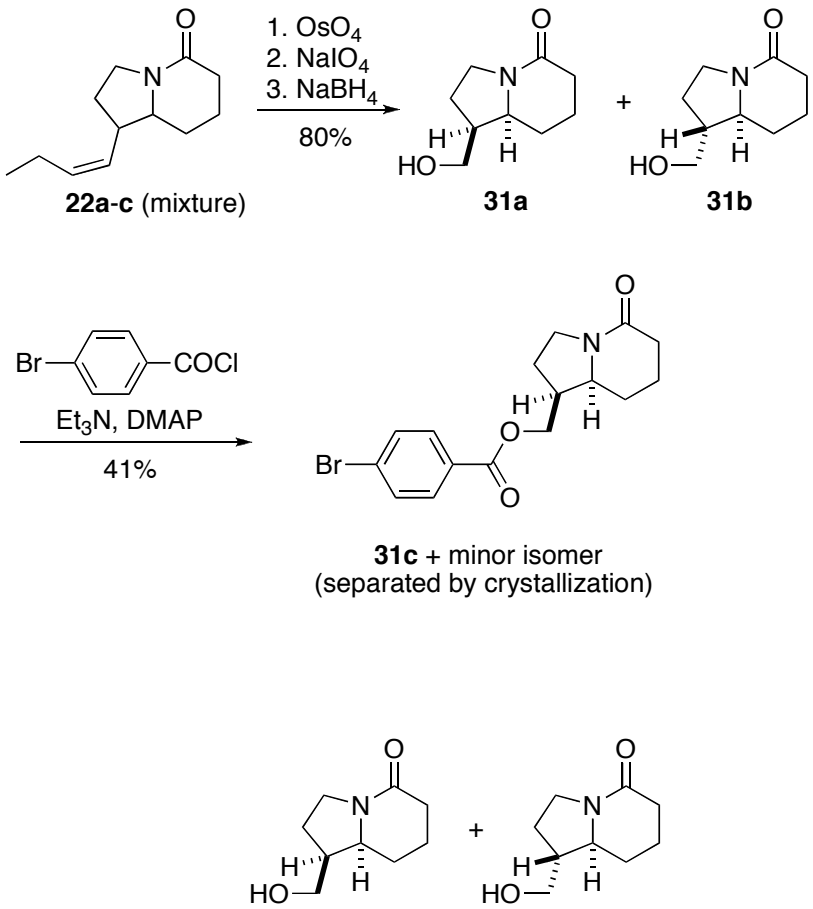
(1*R*,10a*S*)-1-(4'-Bromobenzoyloxymethyl)-9-carboethoxy-2,3,6,10b-tetrahydro-1*H*-pyrrolo[1,2-*c*]quinazolin-5-one (19a). Prepared according to the general procedure for preparation of *p*-bromobenzoyl esters using **19** (0.06 g, 0.21 mmol). Chromatography (1:2 hexanes:EtOAc) gave 0.070 g (76%) of a white solid that was crystallized from EtOAc/hexanes. Mp 186-188 °C; $[\alpha]_D$ –95.6 (*c* 0.25, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.37 (t, *J* = 7.1 Hz, 3H), 2.20 (m, 2H), 3.15 (m, 1H), 3.55 (dt, *J* = 2.7, 10.3 Hz, 1H), 3.96 (q, *J* = 8.9 Hz, 1H), 4.13 (m, 1H), 4.32 (m, 3H), 5.05 (d, *J* = 4.8 Hz, 1H), 6.83 (d, *J* = 8.2 Hz, 1H), 7.47 (d, *J* = 8.5 Hz, 2H), 7.74 (d, *J* = 8.5 Hz, 2H), 7.83 (d, *J* = 8.1 Hz, 2H) 9.11 (s, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 14.8, 25.9, 41.2, 43.6, 61.1, 61.3, 64.1, 114.6, 116.8, 124.5, 128.6, 128.7, 128.8, 130.9, 131.5, 132.0, 141.6, 152.8, 166.0, 166.3; IR (film) 1715, 1670, 1618, 1457; MS (CI) m/z 473 (M^+ + H), 475 (M^+ + 3), 272, 185; HRMS calcd for $C_{22}H_{22}N_2O_5Br$ (M^+ + H) 473.0712, found 473.0718. A single crystal was subjected to X-ray analysis.



13a **20**

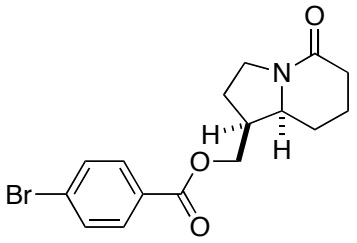
(1*S*,10b*R*)-(*E*)-9-Carboethoxy-1-[4'-(4''-bromobenzoyloxy)but-1'-enyl)]-2,3,6,10b-tetrahydro-1*H*-pyrrolo[1,2-*c*]quinazolin-5-one (20). Prepared according to the general procedure for preparation of *p*-bromobenzoyl esters from **13a** (0.070 g, 0.20 mmol). Chromatography (1:1 hexanes:EtOAc to EtOAc) gave 0.069 g (71%) of a pale yellow solid that was crystallized from EtOAc/hexanes. Mp 166-168 °C; $[\alpha]_D$ –27.2 (*c* 0.25, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.36 (t, *J* = 7.0 Hz, 3H), 1.88 (quintet, *J* = 10.2 Hz, 1H), 2.20 (m, 1H), 2.65 (q, *J* = 6.7 Hz, 2H), 3.01 (quintet, *J* = 8.9 Hz, 1H), 3.67 (m, 2H), 4.31 (q, *J* = 7.1 Hz, 2H), 4.46 (m, 3H), 5.75 (dd, *J* = 8.5, 15.5 Hz, 1H), 5.89 (td, *J* = 6.8, 15.4 Hz, 1H), 6.80 (d, *J* = 8.3 Hz, 1H), 7.54 (d, *J* = 8.4 Hz, 2H), 7.88 (t, *J* = 6.8 Hz, 3H), 8.11 (s, 1H), 8.42 (br, s, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 14.8, 31.3, 32.5, 44.0, 49.6, 60.9, 61.2, 64.5, 114.0, 121.5, 124.4, 126.7, 128.5, 129.3, 129.5, 130.7, 131.5, 132.1, 133.5, 141.8, 153.3, 166.2, 166.4; IR (KBr) 1714, 1673, 1619, 1591 cm^{-1}; MS (CI) 513 (M^+ + H), 515 (M^+ + 3), 232; HRMS calcd for $C_{25}H_{25}N_2O_5Br$ (M^+) 512.0947, found 512.0938. A single crystal was subjected to X-ray analysis.

Scheme I. *Preparation of p-bromobenzoyl ester* **31c**.



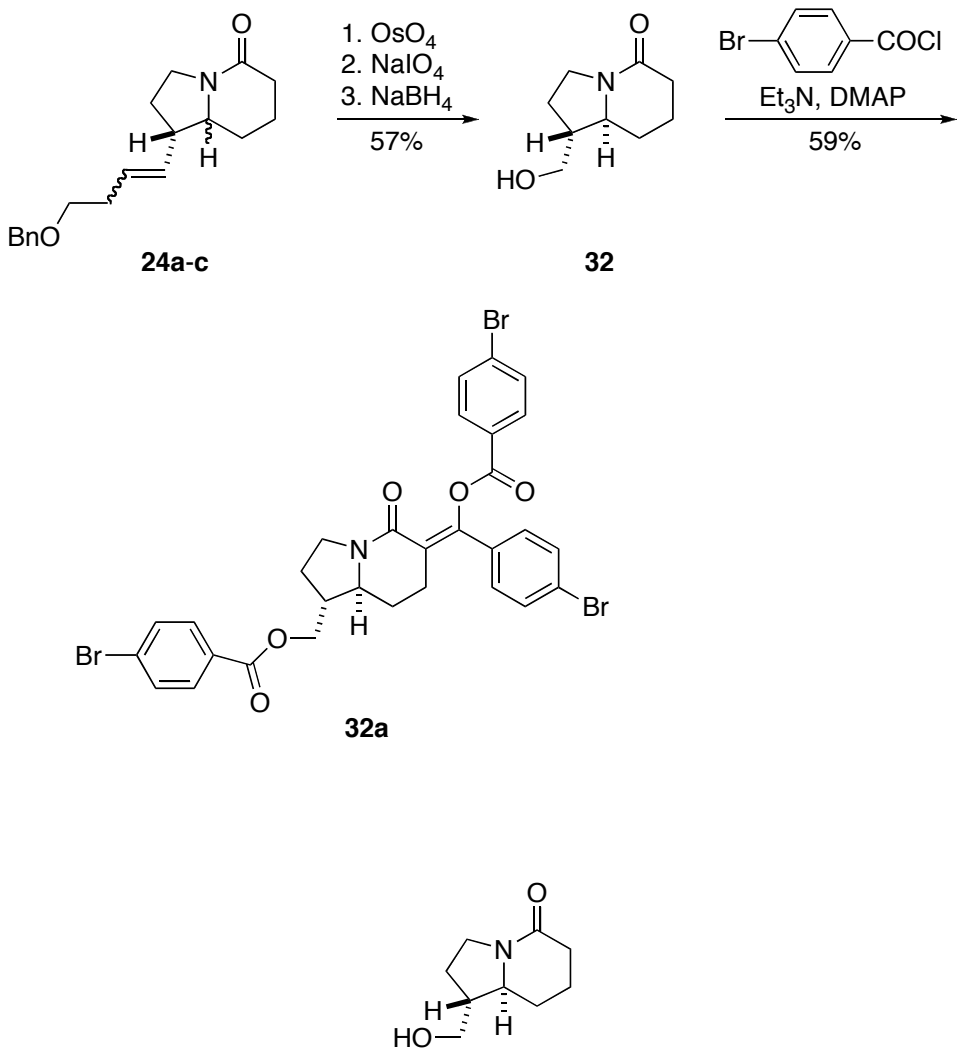
(1*S*,8a*S*)- and (1*R*,8a*S*)-1-Hydroxymethylhexahydroindolizin-5-one (31a,b). Prepared according to the general procedure for the oxidative cleavage/reduction reaction sequence using the purified product mixture containing **22a–c** (0.08 g, 0.41 mmol). Chromatography (20:1 CH_2Cl_2:MeOH) gave 0.061 g (80% over 3 steps) of a colorless oil as a mixture of isomers. IR (film) 3380, 1608, 1470 cm^{-1}; MS (CI) *m/z* 170 (M^+ + H), 111, 82; HRMS calcd for $C_9H_{16}NO_2$ (M^+ + H) 170.1181, found 170.1188. Major isomer **(1*S*,8a*S*)-31a**: ^1H NMR (400 MHz, $CDCl_3$) δ 1.48 (dq, *J* = 3.1, 11.9 Hz, 1H), 1.69 (m, 1H), 1.91 (m, 1H), 1.97 (m, 3H), 2.27 (m, 1H), 2.42 (m, 2H), 2.49 (br, s, 1H), 3.45 (m, 2H), 3.55-3.74 (m, 3H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 21.3, 24.8, 28.5, 31.0, 43.4, 43.6, 60.8, 61.0, 169.4. Minor isomer **(1*R*,8a*S*)-31b** (diagnostic peaks only): ^1H NMR

(400 MHz, $CDCl_3$) δ 1.28 (m, 1H), 3.24 (dt, *J* = 3.5, 10.3 Hz, 1H); ^{13}CNMR (100.6 MHz, $CDCl_3$) δ 20.9, 25.4, 44.0, 48.2, 61.5, 63.1.



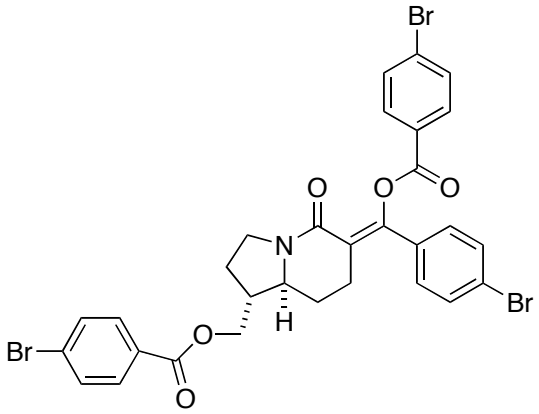
(1*S*,8a*S*)-1-(4-Bromobenzoyloxymethyl)hexahydroindolizin-5-one (31c). Prepared according to the general procedure for preparation of *p*-bromobenzoyl esters using a mixture of **31a** and **31b** (0.060 g, 0.37 mmol). Chromatography (20:1CH_2Cl_2 MeOH) gave 0.12 g (92%) of a white solid that was crystallized (EtOAc/hexanes) to afford 0.070 g (56%) of **31c** as a single isomer. Mp 85-87 °C; $[\alpha]_D$ –2.7 (*c* 0.47, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.47 (dq, *J* = 2.9, 12.8 Hz, 1H), 1.69 (m, 1H), 2.02 (m, 4H), 2.26 (m, 1H), 2.46 (dd, *J* = 6.1, 17.9 Hz, 1H), 2.68 (quintet, *J* = 6.3 Hz, 1H), 3.52 (dt, *J* = 1.8, 9.1 Hz, 1H), 3.70 (m, 2H), 4.18 (dd, *J* = 6.6, 11.3 Hz, 1H), 4.35 (dd, *J* = 6.5, 11.3 Hz, 1H), 7.60 (d, *J* = 8.5 Hz, 2H), 7.86 (d, *J* = 8.5 Hz, 2H); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 21.3, 25.0, 25.4, 31.1, 40.6, 43.4, 60.5, 63.9, 128.3, 128.6, 131.0, 131.8, 165.6, 169.1; IR (film) 1719, 1621, 1590 cm^{-1}; MS (CI) m/z 352 (M^+ + 1), 354 (M^+ + 3), 151, 82; HRMS calcd for $C_{16}H_{19}NO_3Br$ (M^+ + H) 352.0548, found 352.0548. A single crystal was subjected to X-ray analysis.

Scheme J. *Preparation of p-bromobenzoyl ester* **32a**.



(1*R*,8a*S*)-1-Hydroxymethylhexahydroindolizin-5-one (32). Prepared according to the general procedure for the oxidative/reduction reaction sequence using the purified product mixture containing **24a–c** (0.22 g, 0.72 mmol). Chromatography (20:1 CH_2Cl_2:MeOH) gave 0.11 g (86%) of a white solid that was crystallized (EtOAc/hexanes) to afford 0.068 g (57%) of **32** as a single isomer. Mp 114-116 °C; $[\alpha]_D$ –26.4 (*c* 0.55, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.32, (m, 1H), 1.67 (m, 3H), 1.98 (m, 2H), 2.04 (m, 1H), 2.30 (m, 2H), 2.47 (dd, *J* = 6.6, 18.0 Hz, 1H), 3.25 (dt, *J* = 3.4, 10.5 Hz, 1H), 3.51 (td, *J* = 1.5, 11.7 Hz, 1H), 3.66 (m, 1H), 3.77 (dd, *J* = 1.4, 5.4 Hz, 2H); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 21.0, 25.4, 28.5, 31.0, 44.0, 48.2, 61.5, 63.2, 169.1; IR (film)

3396, 1614 cm^{-1}; MS (FAB+) *m/z* 170 (M^+ + H) 110; HRMS calcd for $C_9H_{16}NO_2$ (M^+ + H) 170.1181, found 170.1181.

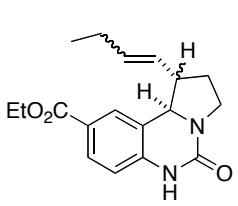
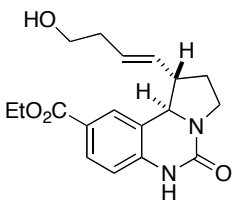
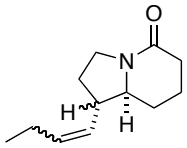
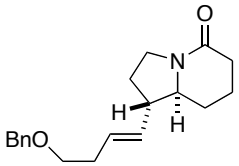
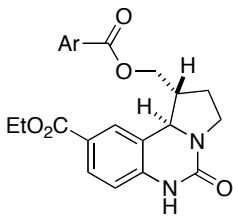
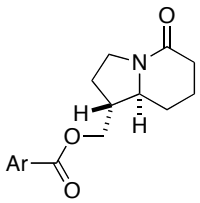
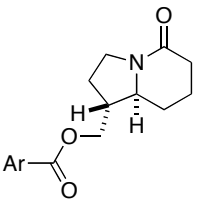


(1*R*,8a*S*)-(*Z*)-6-[4'-Bromobenzoyloxy(4''-bromophenyl)methylene]-1-(4'-bromobenzoyloxymethyl)octahydroindolizin-5-one (32a). Prepared according to the general procedure for preparation of *p*-bromobenzoyl esters from **32** (0.05 g, 0.30 mmol) with a modification of the workup: upon completion of the reaction, the reaction mixture was diluted with EtOAc (10 mL) and washed sequentially with cold 1M HCl (2 × 10 mL), followed by saturated aqueous $NaHCO_3$ (2 × 10 mL) and brine (1 × 10 mL). The organic layer was dried (Na_2SO_4), filtered, and concentrated. Chromatography (10:1 to 1:1 hexanes:EtOAc) gave 0.14 g (64%) of a white solid that was crystallized (EtOAc/hexanes) to afford 0.080 g (40%) of **32a**. Mp 186-187 °C; $[\alpha]_D$ –49.4 (*c* 0.35, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.55 (dq, *J* = 4.2, 13.3 Hz, 1H), 1.74 (quintet, *J* = 9.9 Hz, 1H), 2.16 (quintet, *J* = 6.4 Hz, 1H), 2.26 (m, 2H), 2.60 (dt, *J* = 4.3, 13.6 Hz, 1H), 2.79 (td, *J* = 3.5, 15.7 Hz, 1H), 3.45 (dt, *J* = 3.1, 10.8 Hz, 1H), 3.58 (m, 1H), 3.67 (m, 1H), 4.36 (dd, *J* = 6.7, 11.4 Hz, 1H), 4.46 (dd, *J* = 5.1, 11.4 Hz, 1H), 7.37 (d, *J* = 8.3 Hz, 2H), 7.55 (d, *J* = 8.3 Hz, 2H), 7.62 (dd, *J* = 4.1, 8.3 Hz, 4H), 7.88 (d, *J* = 8.3 Hz, 2H), 8.00 (d, *J* = 8.3 Hz, 2H); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 26.0, 27.4, 28.5, 44.5, 45.2, 61.7, 65.0, 118.4, 123.7, 128.4, 128.4, 128.5, 128.6, 130.3, 131.0, 131.6, 131.7, 131.7, 131.8, 134.1, 149.6, 161.4, 164.1, 165.5; IR (film) 1721, 1650, 1590 cm^{-1}; MS (FAB+) *m/z* 716 (M^+ + H), 718 (M^+ + 3), 720 (M^+ + 5), 722 (M^+ + 7), 307, 154; HRMS calcd for $C_{30}H_{25}NO_5Br_3$ (M^+ + H) 715.9283, found 715.9296. A single crystal was subjected to X-ray analysis.

Determination of Enantiomeric Purities

The enantiomeric ratios of the major products obtained in the cyclization reactions employing the enantiomerically enriched allylsilanes (*S*)-**1** and (*S*)-**2** were determined by chiral HPLC. In all cases, with the exception of **13a** (which was amenable to analysis without derivatization), the *p*-bromobenzoyl esters were required to facilitate separation of the component enantiomers in the racemic standards. All compound derivatizations for the purpose of HPLC analysis were carried out as described above with the exception that crystallization was avoided throughout to prevent alteration of the enantiomeric purity obtained in the original products.

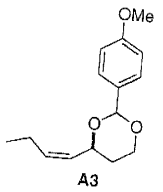
Table A. Summary of er determination.

Parent Compound	 **12a,b**	 **13a**	 **22a,b**	 **24a**
Derivative Analyzed[a]		N/A		
chiral column[b]	Chiralcel OD-H	Chrirobiotic T	Chiralcel OD-H	Chiralcel OD-H
eluent[c]	5-10% IPA/hexanes gradient over 60 min	30% EtOH/hexanes	5-10% IPA/hexanes gradient over 60 min	5-10% IPA/hexanes gradient over 60 min
RT[d] (major isomer)	33.4 min	18.2 min	37.9 min	37.5 min
RT (minor isomer)	40.4 min	20.9 min	34.7 min	35.4 min
er	97.1:2.9 (*minor diastereomer)*	99:1	93.3:6.7 *(minor diastereomer)*	97.0:3.0

[a]Ar = *p*-bromophenyl; [b]Diacel Chemical; [c]IPA = 2-propanol, EtOH = ethanol; [d]RT = retention time

References

1. Frank, K. E.; Aubé, J. *J. Org. Chem.* **2000**, *65*, 655-666.
2. Tamao, K.; Kawachi, A.; Nakagawa, Y.; Ito, Y. *J. Organomet. Chem.* **1994**, *473*, 29-34.
3. Breuilles, P.; Uguen, D. *Tetrahedron Lett.* **1998**, *39*, 3145-3148.
4. Vettel, S.; Vaupel, A.; Knochel, P. *J. Org. Chem.* **1996**, *61*, 7473-7481.
5. Johnson, W. S.; Wiedhaup, K.; Brady, S. F.; Olson, G. L. *J. Am. Chem. Soc.* **1974**, *96*, 3979-3984.
6. Burgstahler, A. W.; Weigel, L. W.; Sanders, M. E.; Shaefer, C. G.; Bell, W. J.; Vuturo, S. B. *J. Org. Chem.* **1977**, *42*, 566-568.
7. Sinha, S. C.; Sinha, S. C.; Keinan, E. *J. Org. Chem.* **1999**, *64*, 7067-7073.
8. Takimoto, M.; Shimizu, K.; Mori, M. *Org. Lett.* **2001**, *3*, 3345-3347.
9. Moffatt, J. G. *Org. Synth.* **1973**, *Coll. Vol. 5*, 242-244.
10. Gao, Y.; Hanson, R. M.; Klunder, J. M.; Ko, S. Y.; Masamune, H.; Sharpless, K. B. *J. Am. Chem. Soc.* **1987**, *109*, 5765-5780.
11. Dale, J. A.; Dull, D. L.; Mosher, H. S. *J. Org. Chem.* **1969**, *34*, 2543-2549.
12. Wipf, P.; Xu, W. *Org. Synth.* **1997**, *74*, 205-211.
13. Zhou, J.-C.; Cia, J.; Soloway, A. H.; Barth, R. F.; Adams, D. M.; Ji, W.; Tjarks, W. *J. Med. Chem.* **1999**, *42*, 7067-7073.
14. Suginome, M.; Matsumoto, A.; Ito, Y. *J. Am. Chem. Soc.* **1996**, *118*, 3061-3062.
15. Hiemstra, H.; Sno, M. H. A. M.; Vijn, R. J.; Speckamp, W. N. *J. Org. Chem.* **1985**, *50*, 4014-4020.
16. Hubert, J. C.; Wijnberg, J. B. P. A.; Speckamp, W. N. *Tetrahedron* **1975**, *31*, 1437-1441.
17. Schoemaker, H. E.; Dijkink, J.; Speckamp, W. N. *Tetrahedron* **1978**, *34*, 163-172.
18. Suginome, M.; Iwanami, T.; Ito, Y. *J. Org. Chem.* **1998**, *63*, 6096-6097.



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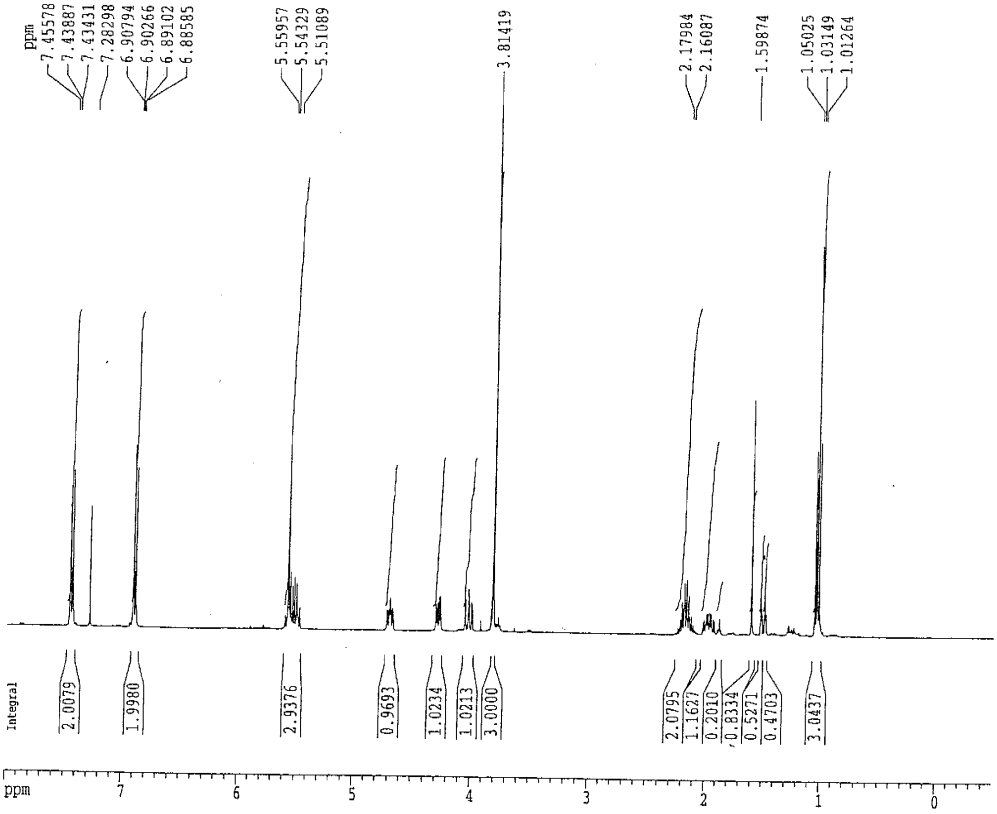

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DW	104.400 usec
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F2 - Processing parameters

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PC	1.00

1D NMR plot parameters

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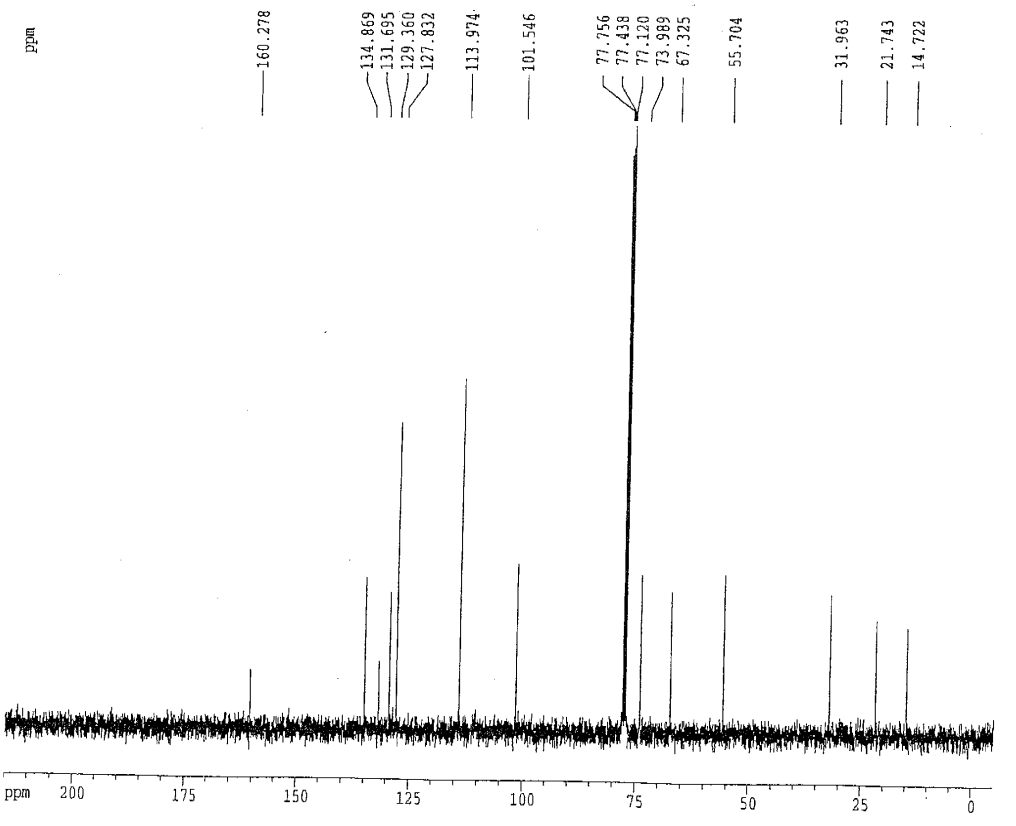

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========== CHANNEL f2 ==========

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PCPD2	100.00 usec
PL2	0.00 dB
PL12	18.00 dB
PL13	18.00 dB
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F2 - Processing parameters

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1D NMR plot parameters

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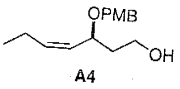

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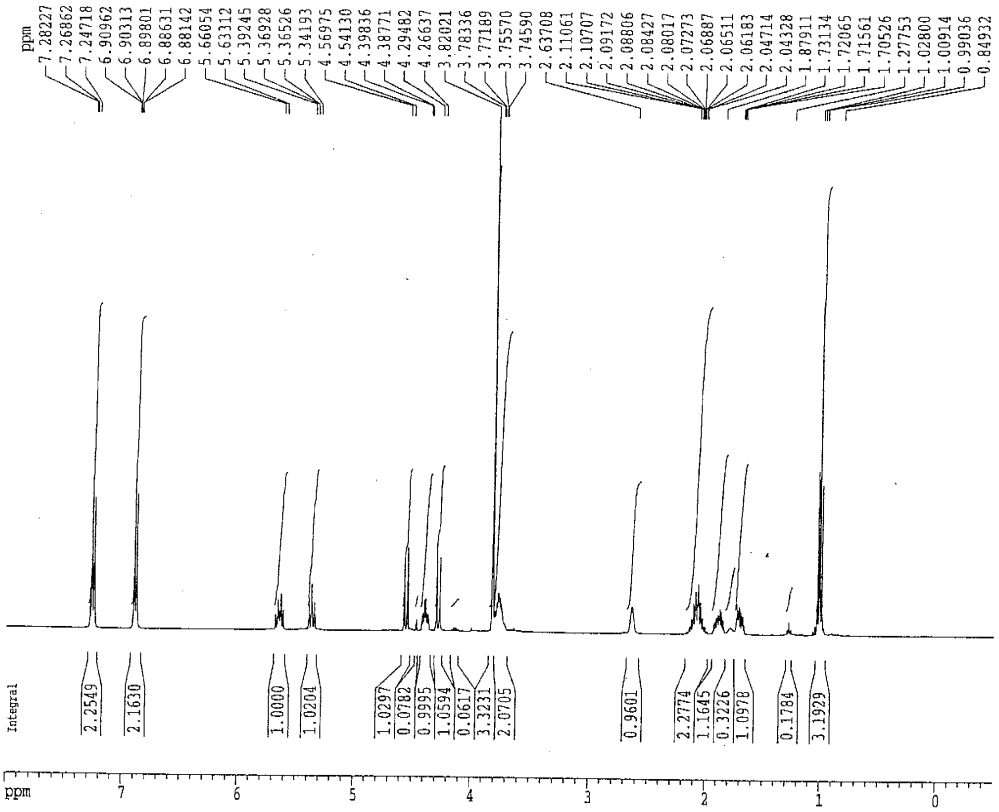


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1D NMR plot parameters
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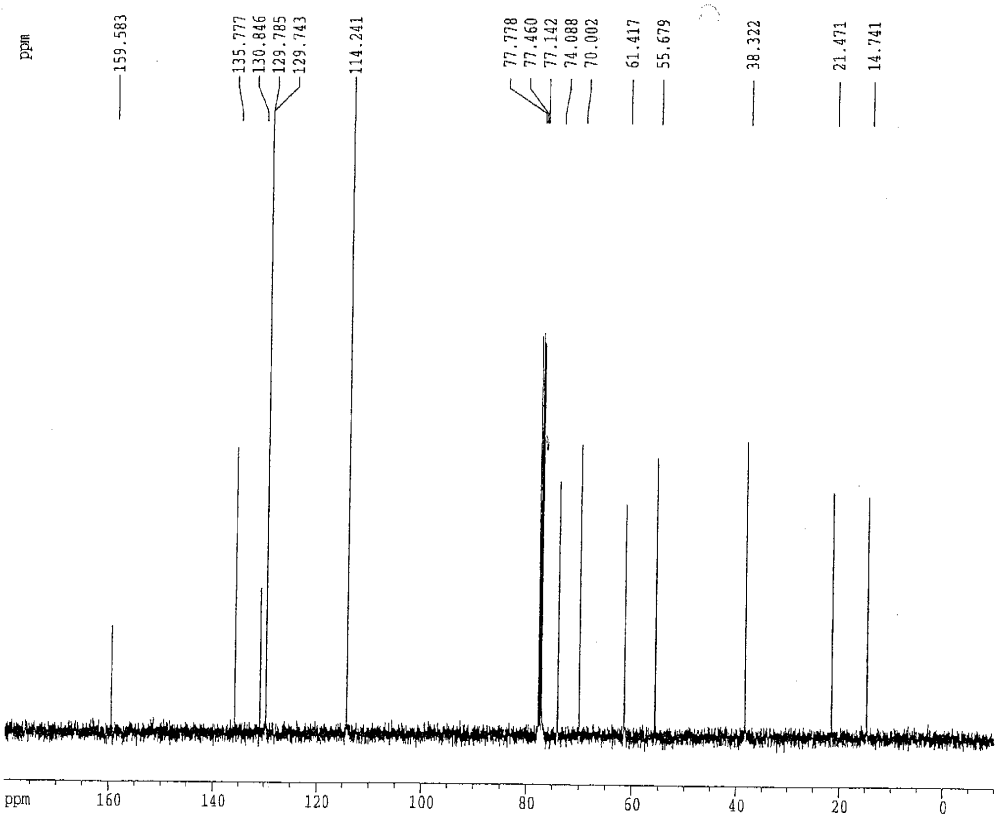


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PROCNO                      1

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DE                     4.50 usec
TE                   300.0 K
D1                0.05000000 sec
d11               0.03000000 sec
d12               0.00002000 sec

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NUC2                      1H
PCPD2                100.00 usec
PL2                     0.00 dB
PL12                   18.00 dB
PL13                   18.00 dB
SFO2              400.1316005 MHz

F2 - Processing parameters
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SF                100.6127290 MHz
WDW                         EM
SSB                          0
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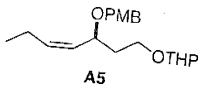
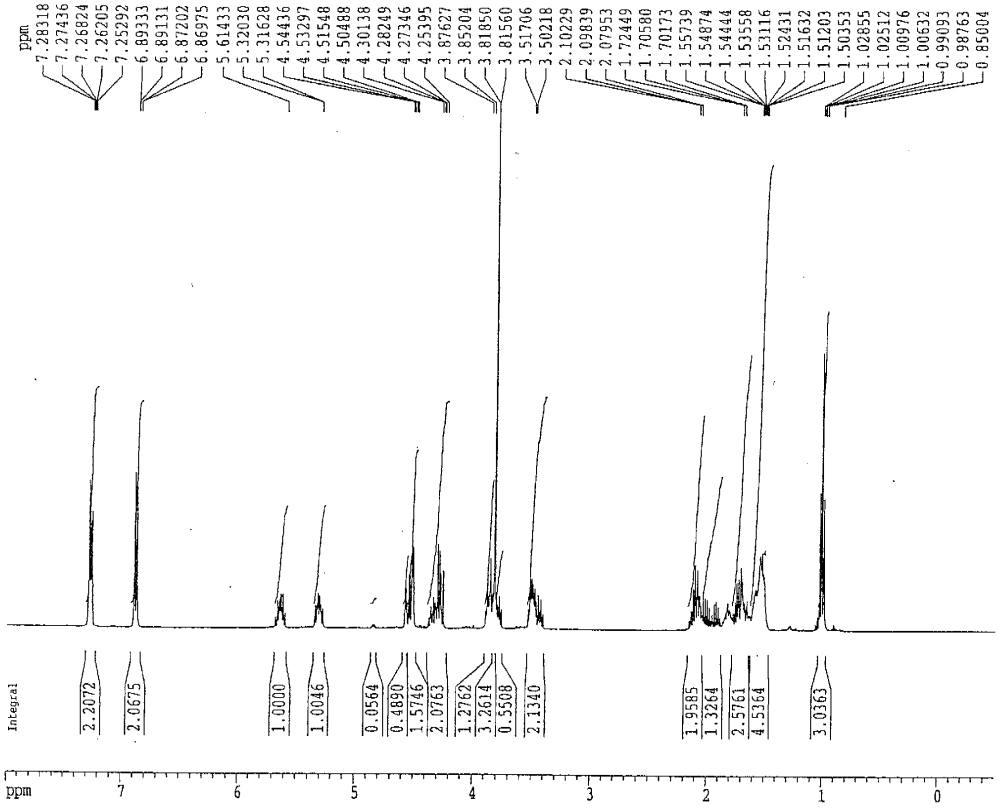
GB                           0
PC                        1.40

1D NMR plot parameters
CX                       20.00 cm
F1P                     180.000 ppm
F1                    18110.29 Hz
F2P                     -10.036 ppm
F2                    -1009.74 Hz
PPMCM                  9.50179 ppm/cm
HZCM                 956.00128 Hz/cm
```



A5



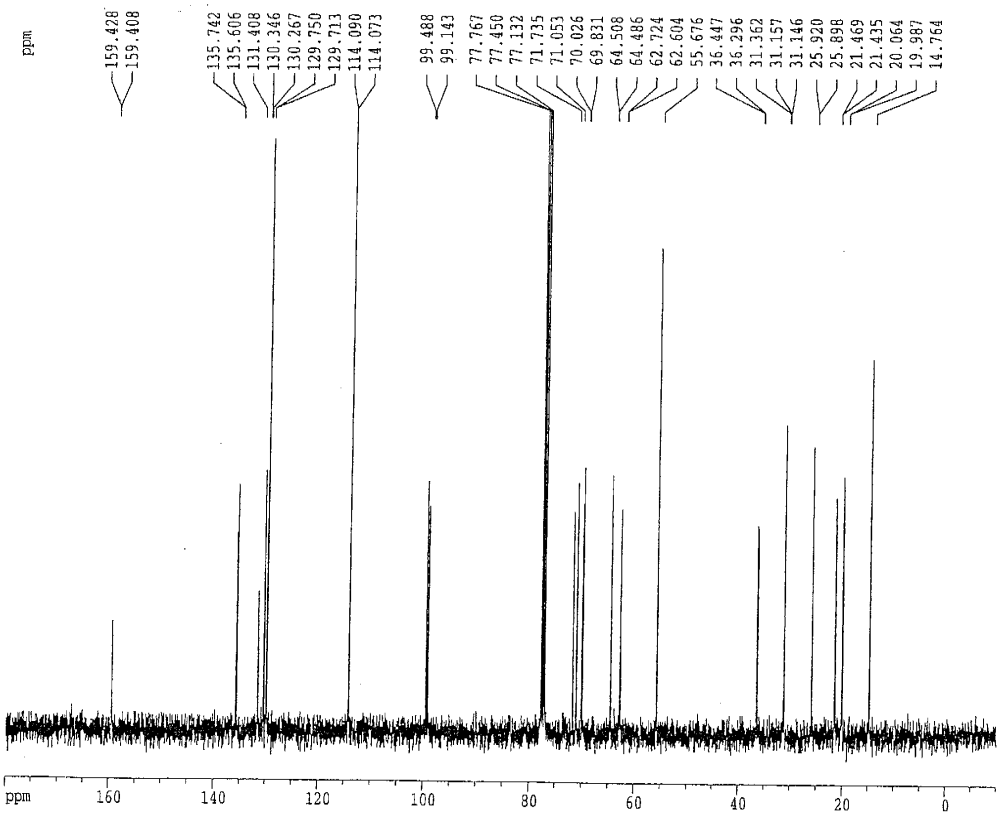

```
Current Data Parameters
NAME                  wj2-134
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_               20011126
Time                   10.25
INSTRUM                drx400
PROBHD      5 mm Multinu
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                4789.272 Hz
FIDRES             0.146157 Hz
AQ                 3.4210291 sec
RG                       64
DW                  104.400 usec
DE                    4.50 usec
TE                   300.0 K
D1                1.00000000 sec

============ CHANNEL f1 ============
NUC1                      1H
P1                      7.70 usec
PL1                    -6.00 dB
SFO1              400.1320007 MHz

F2 - Processing parameters
SI                        16384
SF                400.1300000 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00

1D NMR plot parameters
CX                        20.00 cm
F1P                      8.000 ppm
F1                    3201.04 Hz
F2P                     -0.500 ppm
F2                    -200.07 Hz
PPMCM                  0.42500 ppm/cm
HZCM                 170.05525 Hz/cm
```




```
Current Data Parameters
NAME                  wj2-134
EXPNO                       2
PROCNO                      1

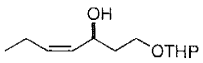
F2 - Acquisition Parameters
Date_               20011126
Time                   10.31
INSTRUM                drx400
PROBHD      5 mm Multinu
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      249
DS                        2
SWH               23148.148 Hz
FIDRES             0.353213 Hz
AQ                 1.4156276 sec
RG                     1024
DW                  21.600 usec
DE                    4.50 usec
TE                   300.0 K
D1                0.05000000 sec
d11               0.03000000 sec
d12               0.00002000 sec

============ CHANNEL f1 ============
NUC1                      13C
P1                     12.30 usec
PL1                     2.00 dB
SFO1              100.6232933 MHz

============ CHANNEL f2 ============
CPDPRG2                waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                     0.00 dB
PL12                   18.00 dB
PL13                   18.00 dB
SFO2              400.1316005 MHz

F2 - Processing parameters
SI                        32768
SF                100.6127290 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40

1D NMR plot parameters
CX                        20.00 cm
F1P                     180.000 ppm
F1                    18110.29 Hz
F2P                     -10.036 ppm
F2                    -1009.73 Hz
PPMCM                  9.50179 ppm/cm
HZCM                 956.00128 Hz/cm
```



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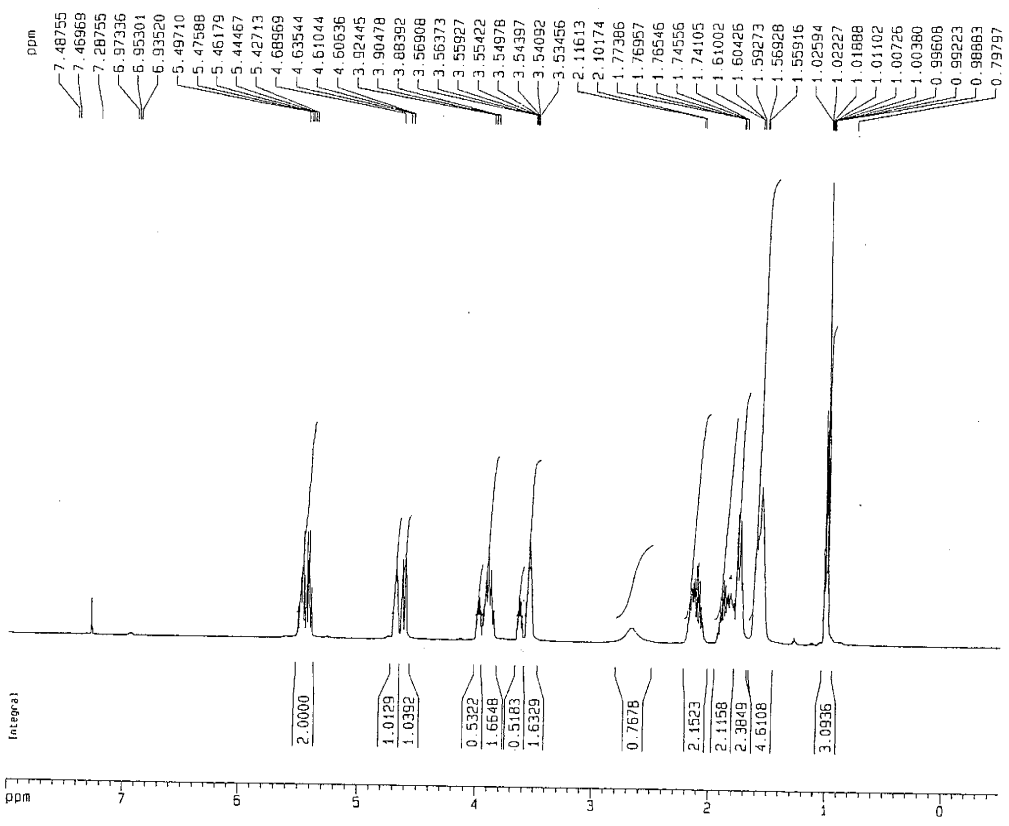


Current Data Parameters
NAME wj2-251
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020417
Time 20.56
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7264309 sec
RG 203.2
DW 83.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.03000000 sec

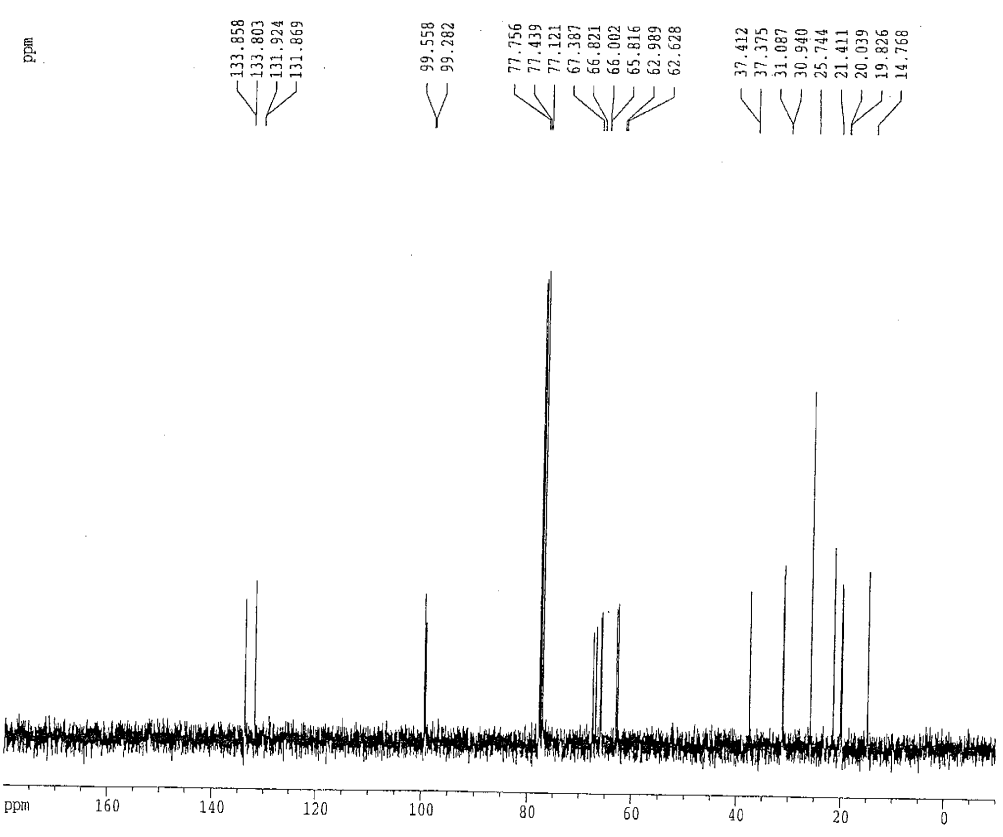
======== CHANNEL f1 ========
NUC1 1H
P1 7.70 usec
PL1 -4.00 dB
SFO1 500.1320005 MHz

F1 - Acquisition parameters
ND0 2
TD 256
SFO1 500.1325 MHz
FIDRES 23.475060 Hz
SW 12.016 ppm

F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1300000 MHz
WDW QSINE
SSB 0
LB 0.30 Hz
GB 0

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 8.000 ppm
F1 4001.04 Hz
F2P -0.500 ppm
F2 -250.07 Hz
PPMCM 0.42990 ppm/cm
HZCM 212.55525 Hz/cm



Current Data Parameters
NAME wj2-214
EXPNO 2
PROCNO 1

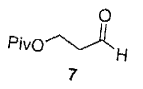
F2 - Acquisition Parameters
Date_ 20020304
Time 13.03
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 100
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 3649.1
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

========== CHANNEL f1 ==========
NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.6232933 MHz

========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDW EM
SSB 0.
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 180.000 ppm
F1 18110.29 Hz
F2P -10.036 ppm
F2 -1009.74 Hz
PPMCM 9.50179 ppm/cm
HZCM 956.00128 Hz/cm



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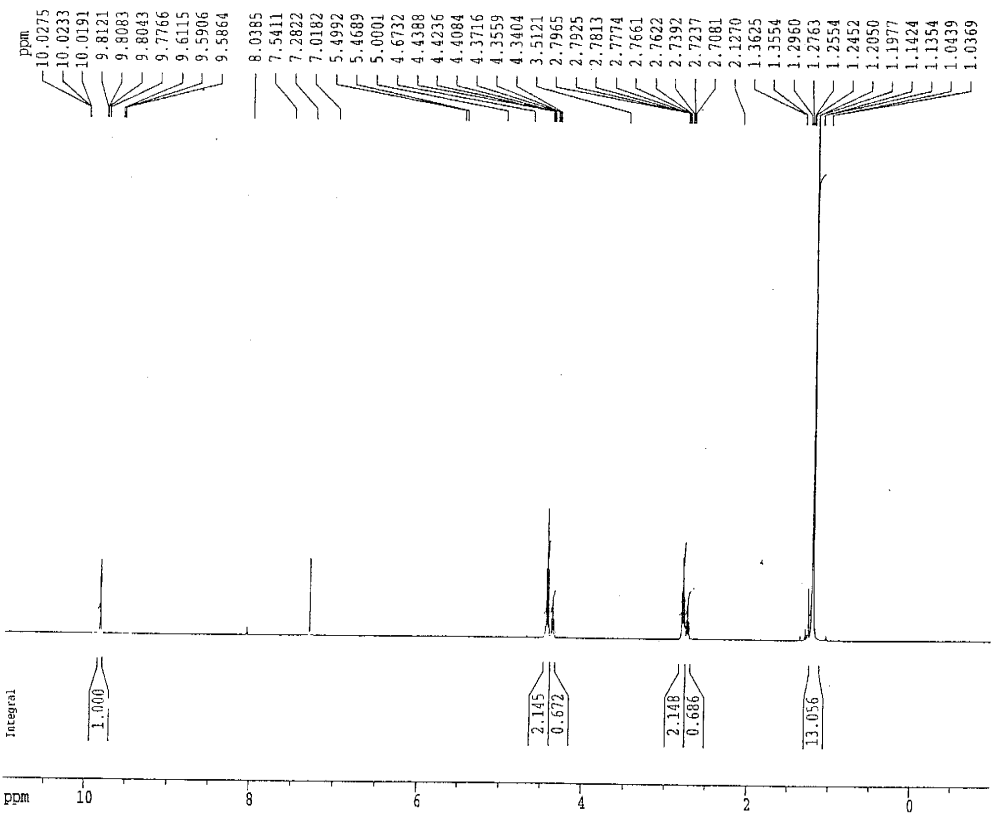


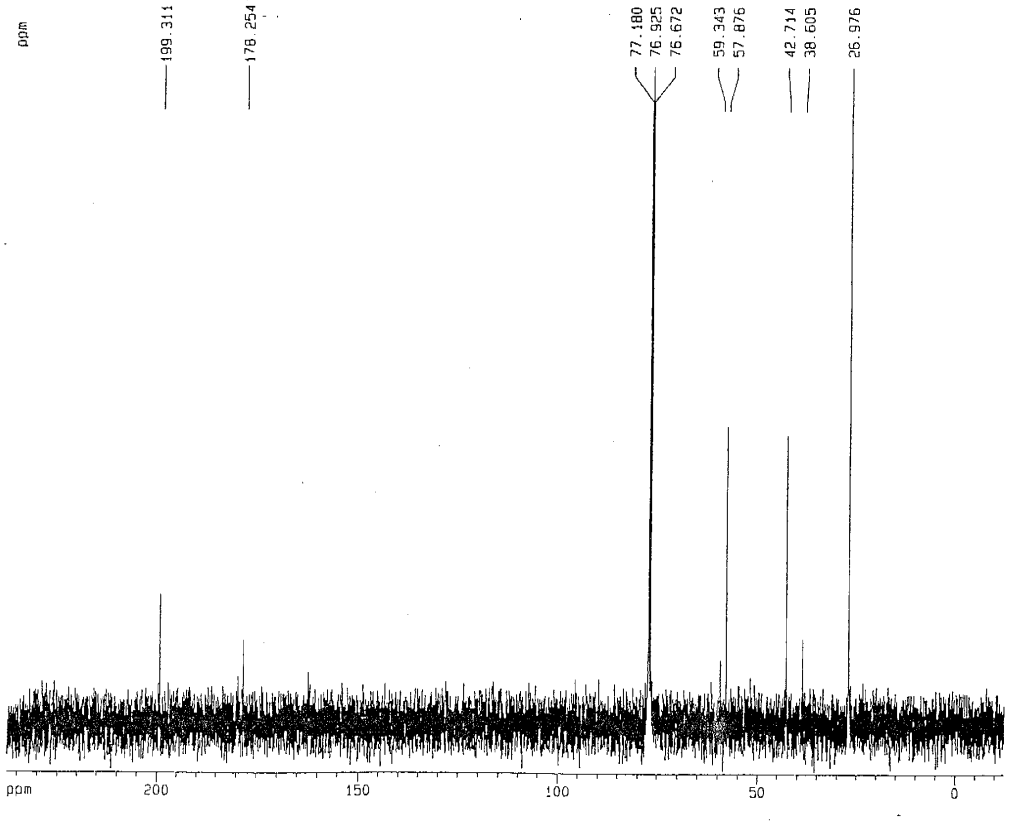

```
Current Data Parameters
NAME                  wj2-145
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20011206
Time                  20.44
INSTRUM              drx400
PROBHD     5 mm Multinu
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               4789.272 Hz
FIDRES            0.146157 Hz
AQ                3.4210291 sec
RG                    161.3
DW                  104.400 usec
DE                    4.50 usec
TE                   300.0 K
D1                1.00000000 sec

============ CHANNEL f1 ============
NUC1                    1H
P1                    7.70 usec
PL1                  -6.00 dB
SFO1           400.1320007 MHz

F2 - Processing parameters
SI                   16384
SF             400.1300000 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                   20.00 cm
F1P                  10.985 ppm
F1                 4395.28 Hz
F2P                  -0.985 ppm
F2                 -393.99 Hz
PPMCM                0.59846 ppm/cm
HZCM               239.46359 Hz/cm
```




```
Current Data Parameters
NAME                  wj2-199
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20020222
Time                  10.57
INSTRUM               spect
PROBHD   5 mm BBO BB-1H
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     313
DS                       4
SWH              31446.541 Hz
FIDRES            0.479896 Hz
AQ                1.0420883 sec
RG                  7298.2
DW                  15.900 usec
DE                    6.00 usec
TE                   300.0 K
D1                0.10000000 sec
d11               0.03000000 sec
d12               0.00002000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                    6.50 usec
PL1                   5.00 dB
SFO1           125.7719472 MHz

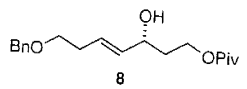
======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                95.00 usec
PL2                  -4.00 dB
PL12                 19.00 dB
PL13                 30.00 dB
SFO2           500.1325000 MHz

F1 - Acquisition parameters
ND0                      2
TD                     256
SFO1           500.1325 MHz
FIDRES         23.475080 Hz
SW                 12.016 ppm

F2 - Processing parameters
SI                   65536
SF             125.7578000 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40

F1 - Processing parameters
SI                    1024
MC2                     QF
SF             500.1300000 MHz
WDW                     nd
SSB                      0
LB                    0.30 Hz
GB                       0

1D NMR plot parameters
CX                   20.00 cm
CY                    8.00 cm
F1P                237.517 ppm
F1               29869.63 Hz
F2P                -12.539 ppm
F2               -1576.91 Hz
PPMCM             12.50262 ppm/cm
HZCM              1572.32703 Hz/cm
```



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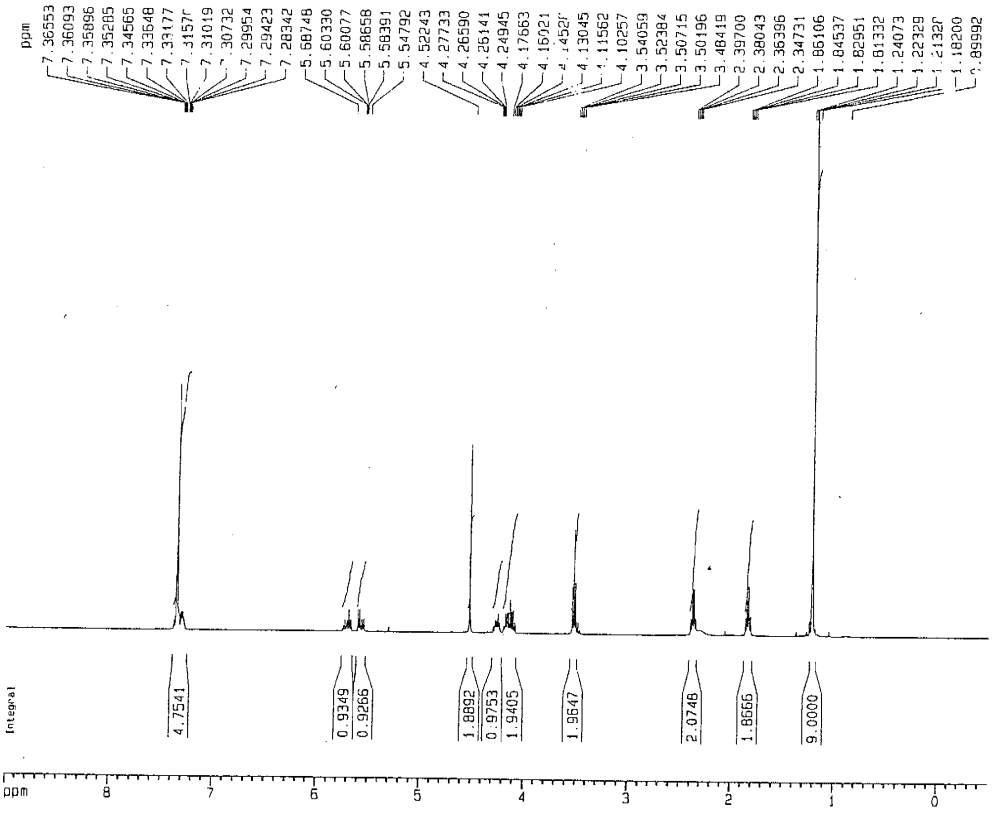
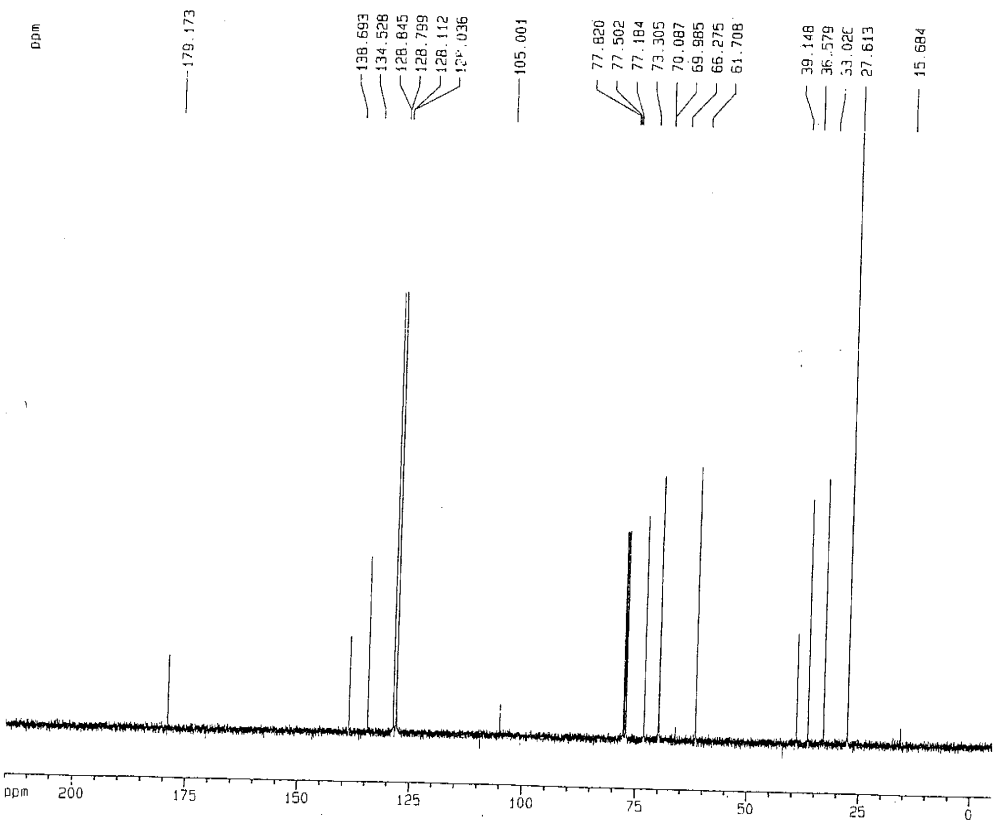


Current Data Parameters
NAME wji-145
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 1000515
Time 12.23
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 64
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.0000[illegible] sec
P1 7.70 usec
DE 4.50 usec
SFO1 400.1320007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.47500 ppm/cm
HZCM 190.06175 Hz/cm

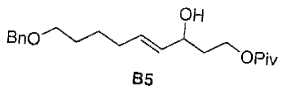


Current Data Parameters
NAME wji-145c
EXPNO 1
PROCNO 1

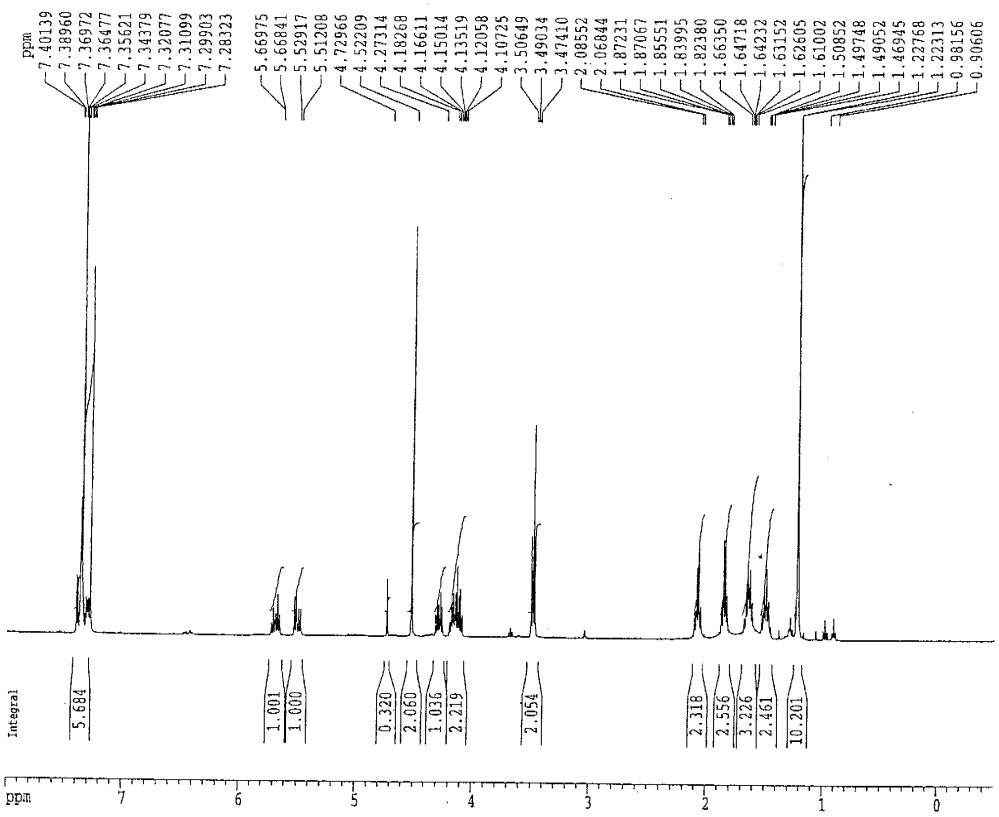
F2 - Acquisition Parameters
Date_ 1000515
Time 12.12
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 239
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 2048
DW 21.600 usec
DE 4.50 usec
TE 300 K
d11 0.0300[illegible] sec
d12 0.00[illegible] sec
PL13 1[illegible] dB
D1 0.05000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 400.1316005 MHz
NUC2 1H
PL2 -6.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SFO1 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 215.000 ppm
F1 21631.74 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCM 11.00000 ppm/cm
HZCM 1106.73999 Hz/cm



B5



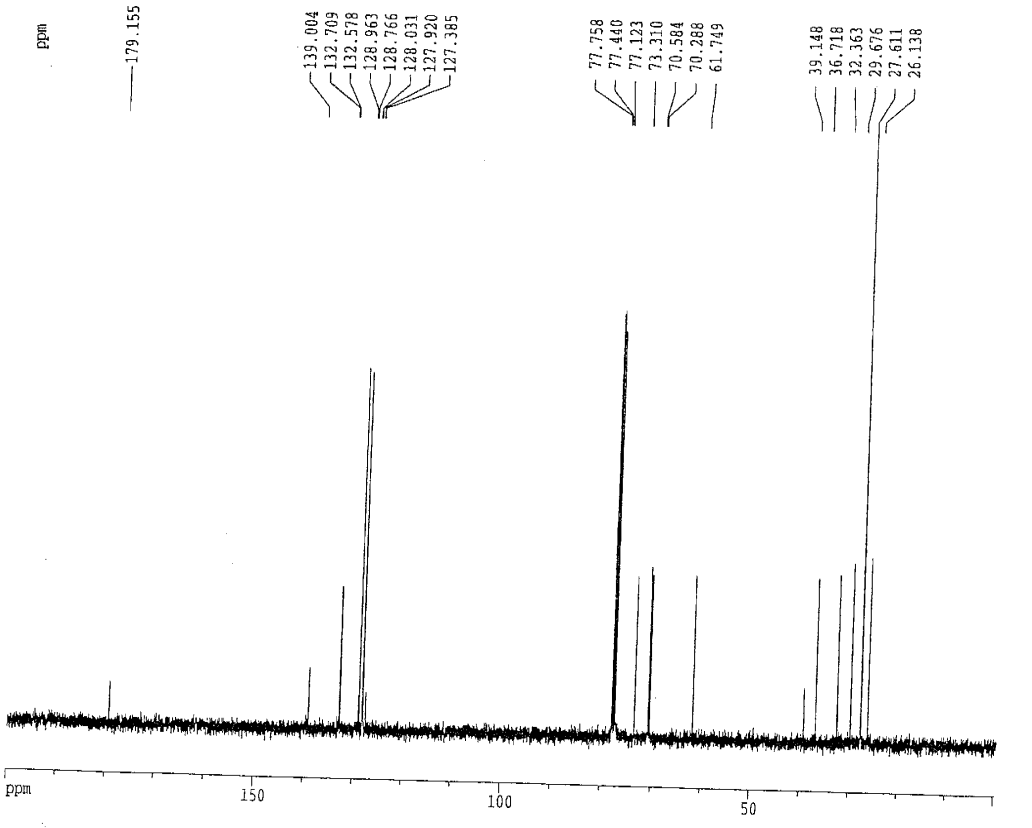
Current Data Parameters
NAME wj4-41
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030709
Time 12.22
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 256
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.70 usec
PL1 -5.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 8.000 ppm
F1 3201.04 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.42500 ppm/cm
HZCM 170.05525 Hz/cm



Current Data Parameters
NAME wj4-41
EXPNO 2
PROCNO 1

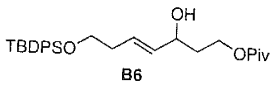
F2 - Acquisition Parameters
Date_ 20030709
Time 12.29
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 175
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 32768
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.6232933 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -0.500 ppm
F2 -50.31 Hz
PPMCM 10.02500 ppm/cm
HZCM 1008.64264 Hz/cm



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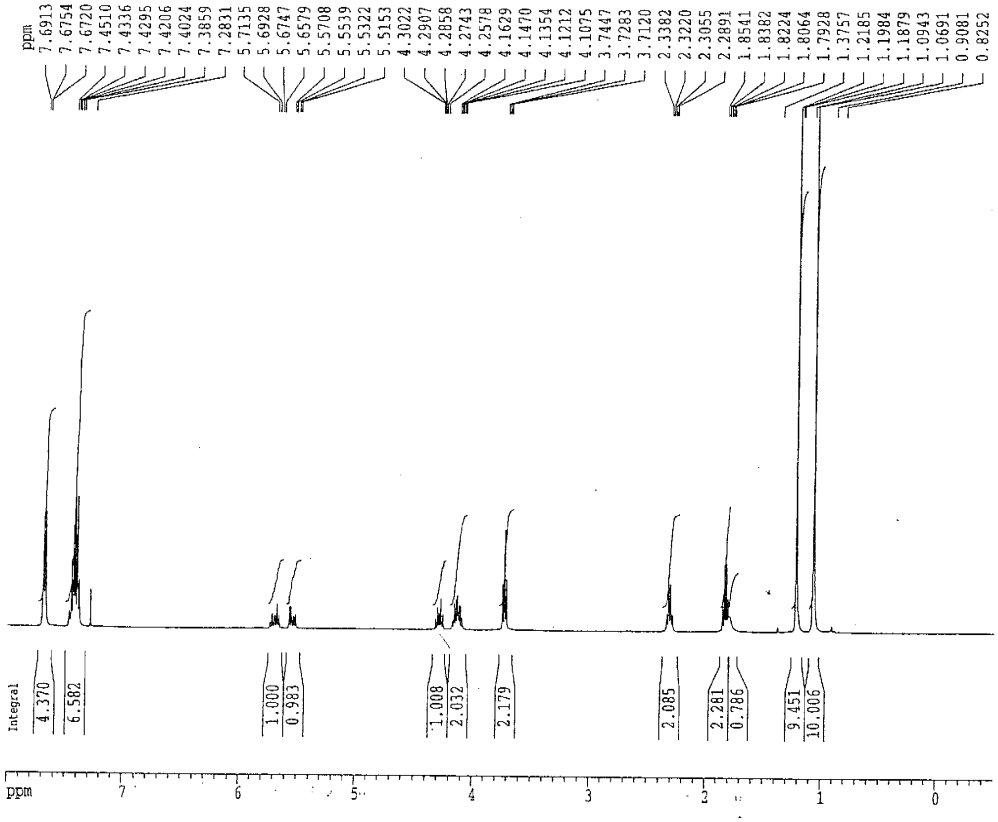


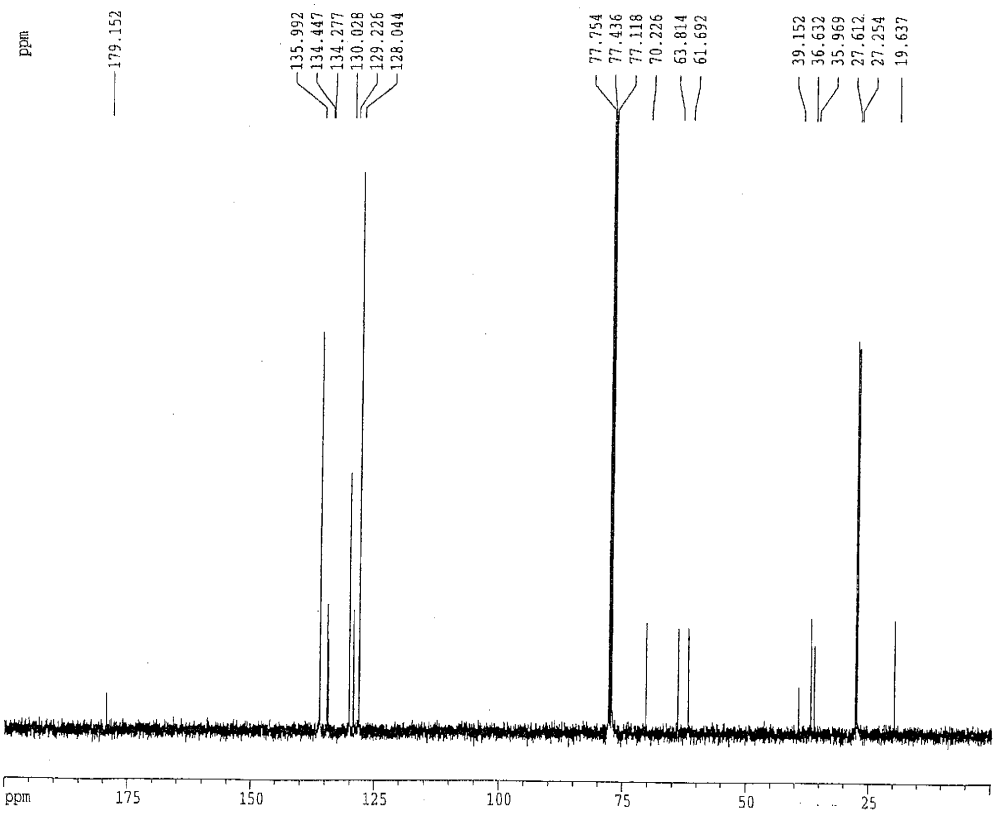
Current Data Parameters
NAME wj4-191,13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040404
Time 15.17
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 128
DW 104.400 usec
DE 5.50 usec
TE 1809.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 8.000 ppm
F1 3201.04 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.42500 ppm/cm
HZCM 170.05525 Hz/cm



Current Data Parameters
NAME wj4-191,13
EXPNO 2
PROCNO 1

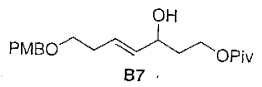
F2 - Acquisition Parameters
Date_ 20040404
Time 15.22
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 548
DS 4
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 12768
DW 21.600 usec
DE 5.50 usec
TE 1809.2 K
D1 0.15000001 sec
d11 0.01000000 sec
DELTA 0.05000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.6232933 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 65536
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -0.500 ppm
F2 -50.31 Hz
PPMCM 10.02500 ppm/cm
HZCM 1008.64264 Hz/cm



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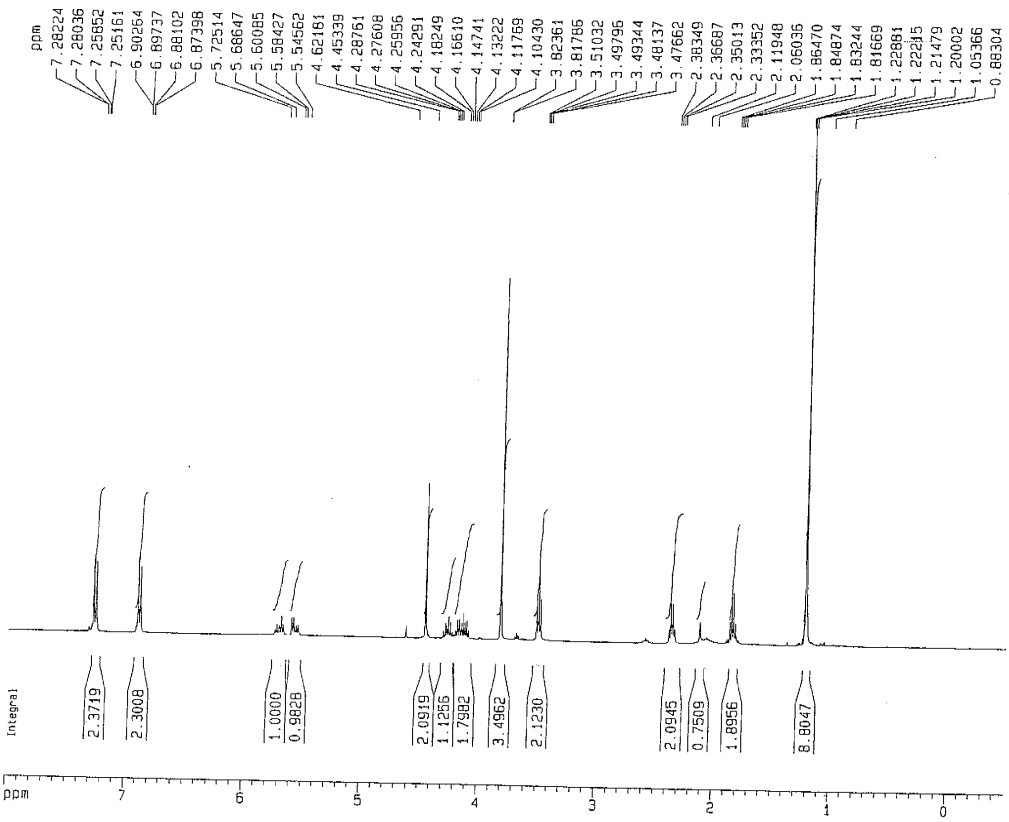


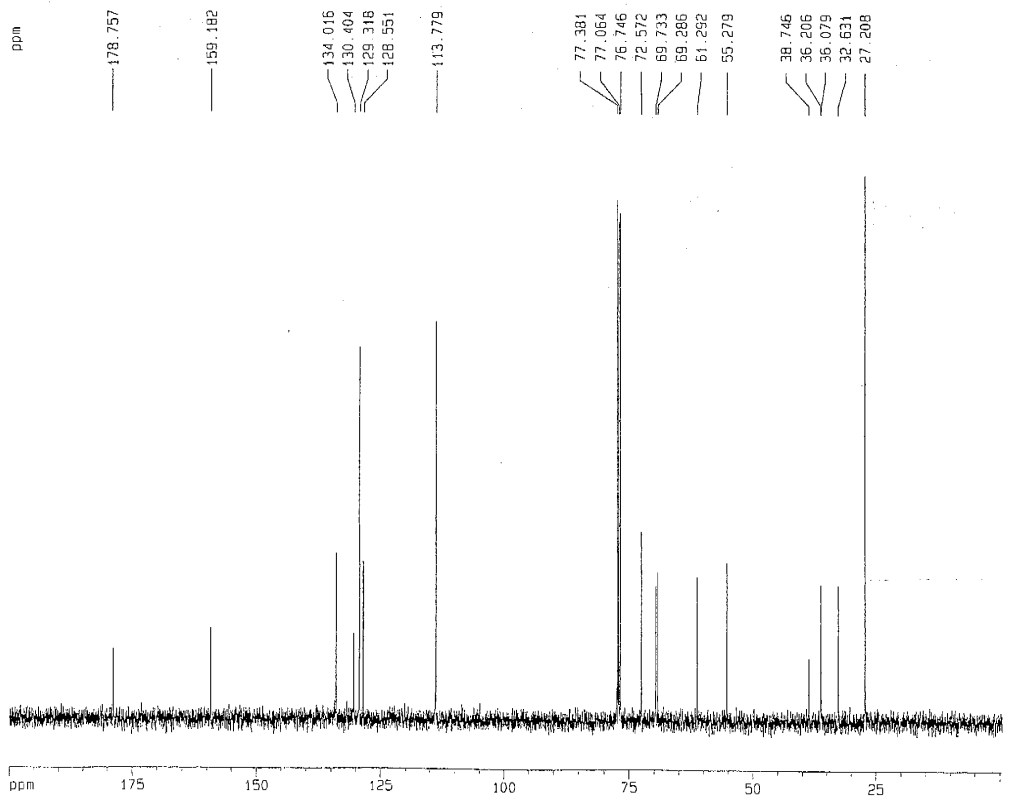
Current Data Parameters
NAME wj5-146
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050320
Time 16.12
INSTRUM spect
PROBHD 5 mm QNP 1H/15
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 114
DW 104.400 usec
DE 6.00 usec
TE 295.6 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.05 usec
PL1 -3.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 8.000 ppm
F1 3201.04 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.42500 ppm/cm
HZCM 170.05525 Hz/cm



Current Data Parameters
NAME wj5-166
EXPNO 2
PROCNO 1

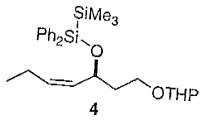
F2 - Acquisition Parameters
Date_ 20050421
Time 21.03
INSTRUM spect
PROBHD 5 mm QNP 1H/15
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 108
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2048
DW 20.850 usec
DE 6.00 usec
TE 295.6 K
D1 0.15000001 sec
d11 0.03000000 sec
DELTA 0.05000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 7.20 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz

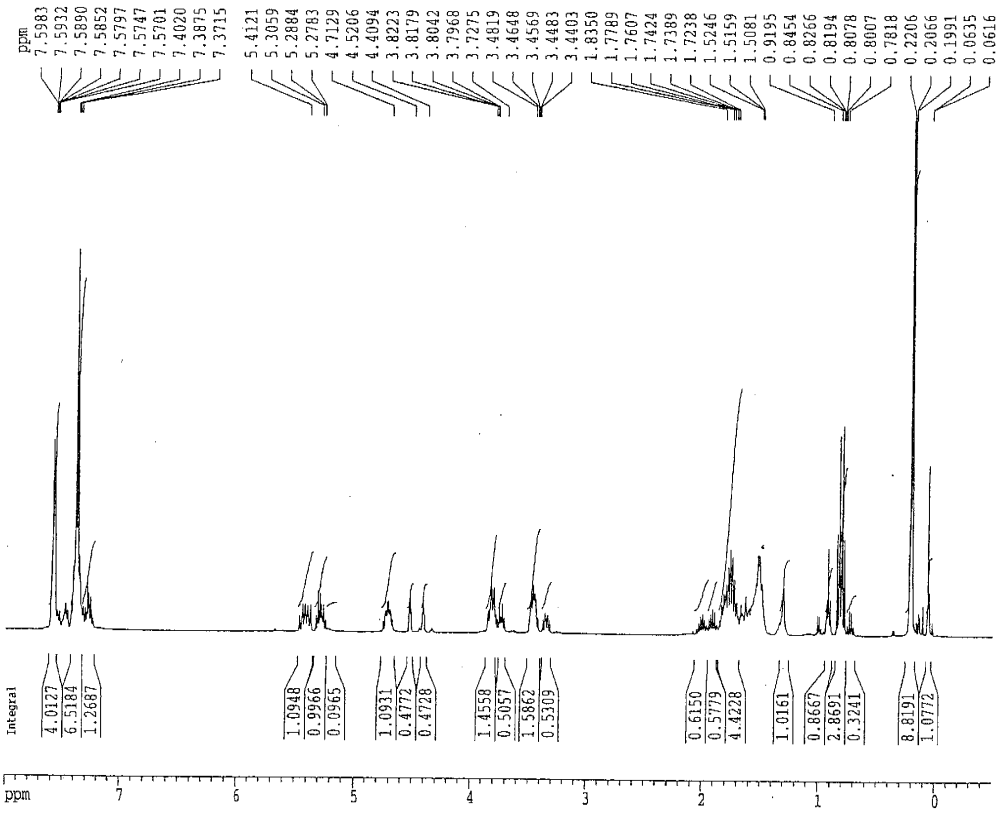
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 98.40 usec
PL2 -3.00 dB
PL12 19.95 dB
PL13 120.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -0.500 ppm
F2 -50.31 Hz
PPMCM 10.02500 ppm/cm
HZCM 1008.64307 Hz/cm



4



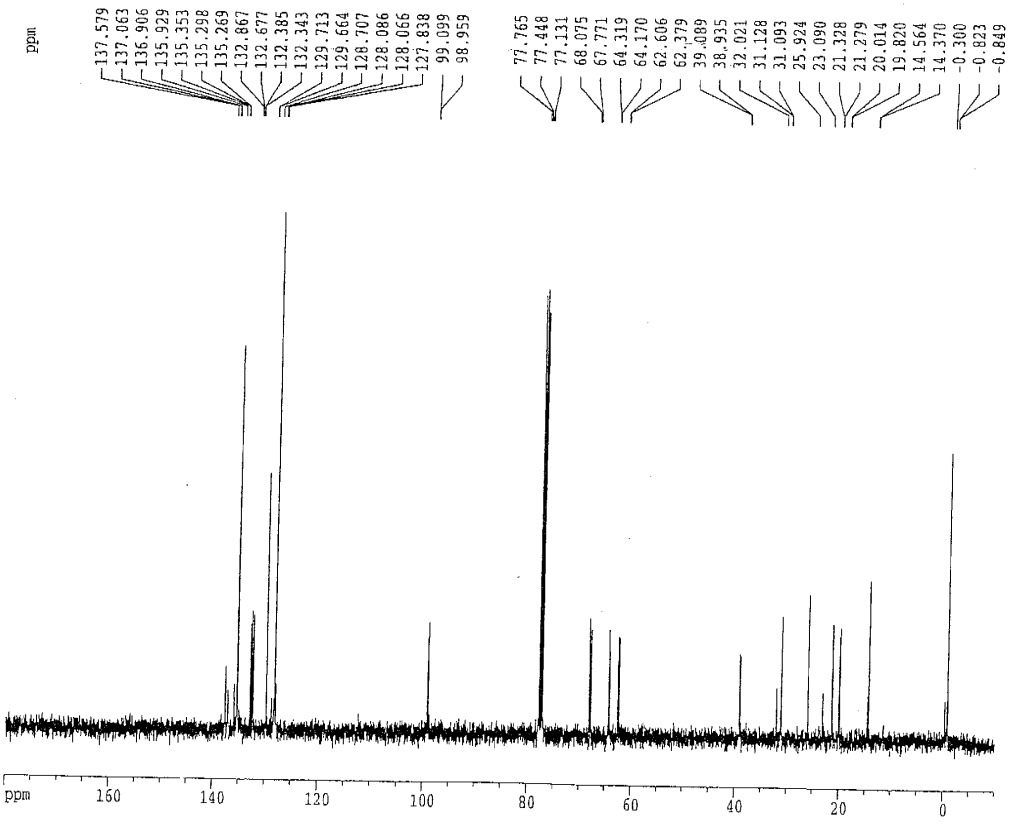
Current Data Parameters
NAME wj2-220
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020313
Time 14.28
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 64
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

========== CHANNEL f1 ==========
NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 8.000 ppm
F1 3201.04 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.42500 ppm/cm
HZCM 170.05525 Hz/cm



Current Data Parameters
NAME wj2-220
EXPNO 2
PROCNO 1

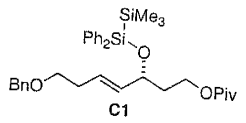
F2 - Acquisition Parameters
Date_ 20020313
Time 14.32
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 180
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 8192
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

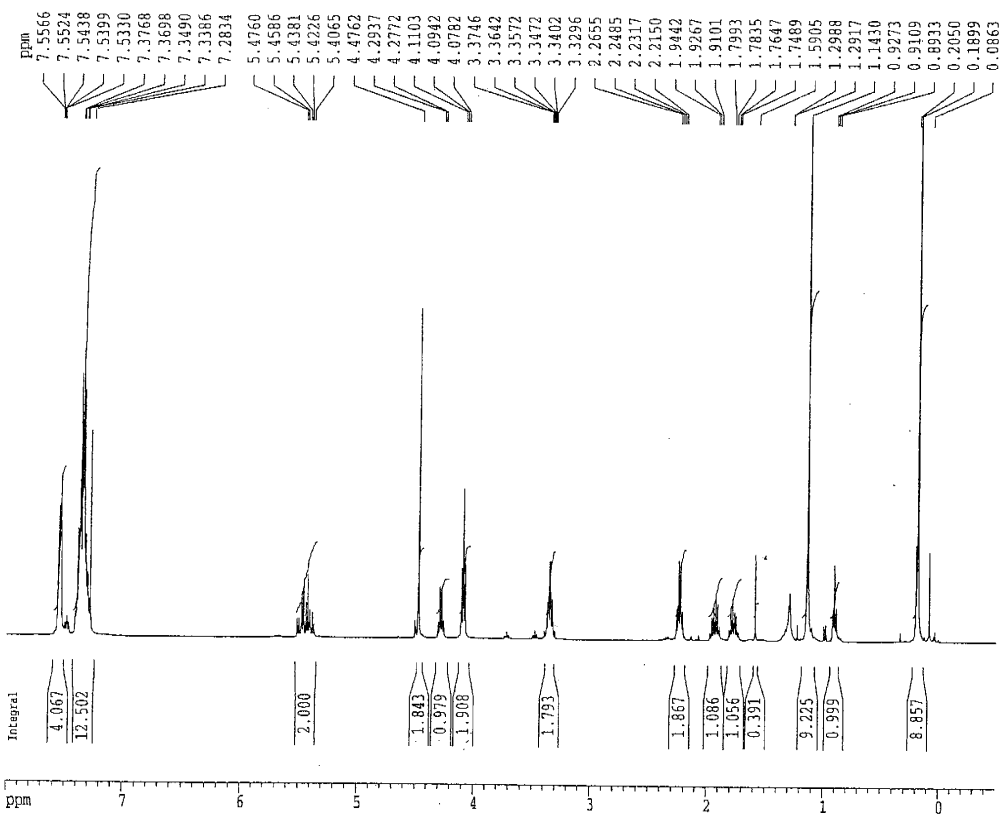
========== CHANNEL f1 ==========
NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.6232933 MHz

========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 180.000 ppm
F1 18110.29 Hz
F2P -10.036 ppm
F2 -1009.74 Hz
PPMCM 9.50179 ppm/cm
HZCM 956.00140 Hz/cm





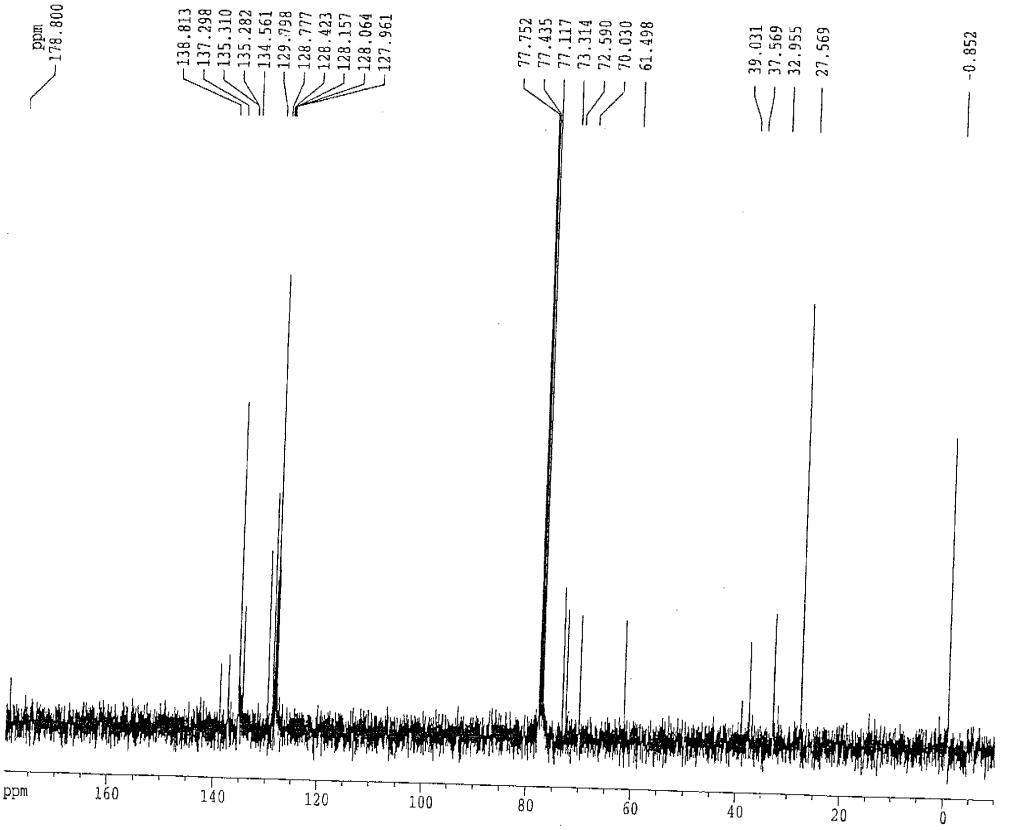
Current Data Parameters
NAME wj2-188
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020211
Time 15.35
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 128
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

========== CHANNEL f1 ==========
NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 8.000 ppm
F1 3201.04 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.42500 ppm/cm
HZCM 170.05525 Hz/cm



Current Data Parameters
NAME wj2-188
EXPNO 2
PROCNO 1

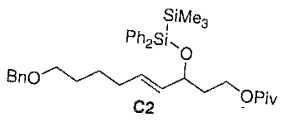
F2 - Acquisition Parameters
Date_ 20020211
Time 15.38
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 326
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 2048
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

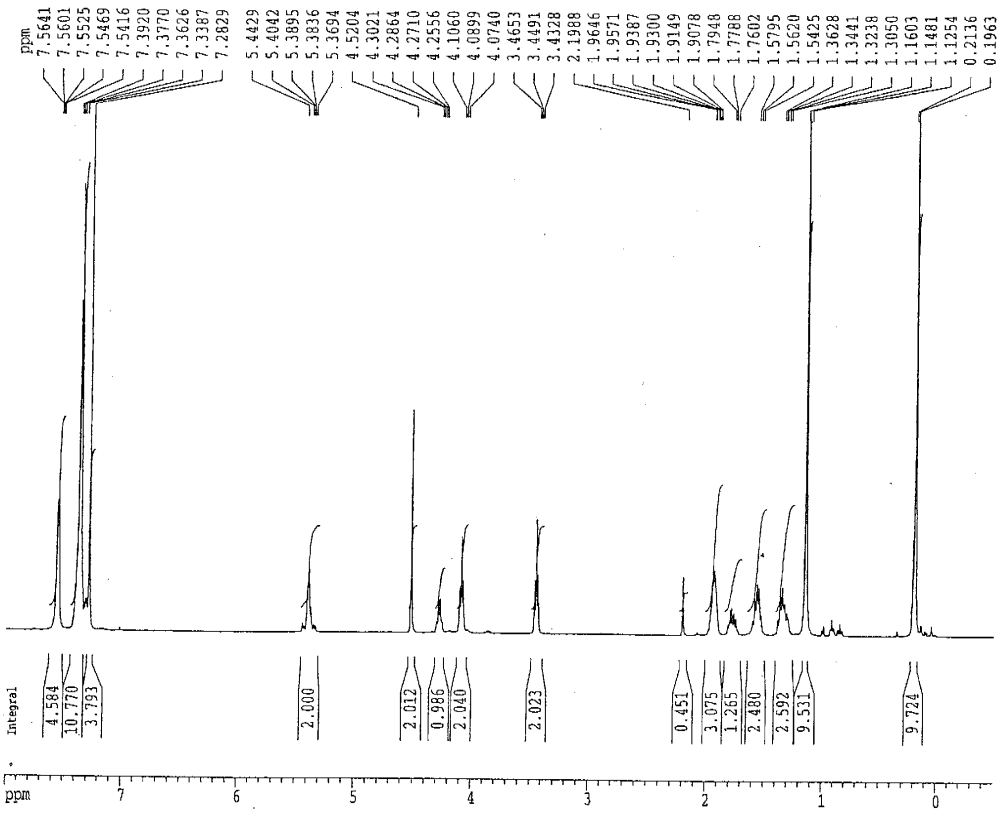
========== CHANNEL f1 ==========
NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.5213933 MHz

========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 180.000 ppm
F1 18110.29 Hz
F2P -10.036 ppm
F2 -1009.73 Hz
PPMCM 9.50179 ppm/cm
HZCM 956.00128 Hz/cm





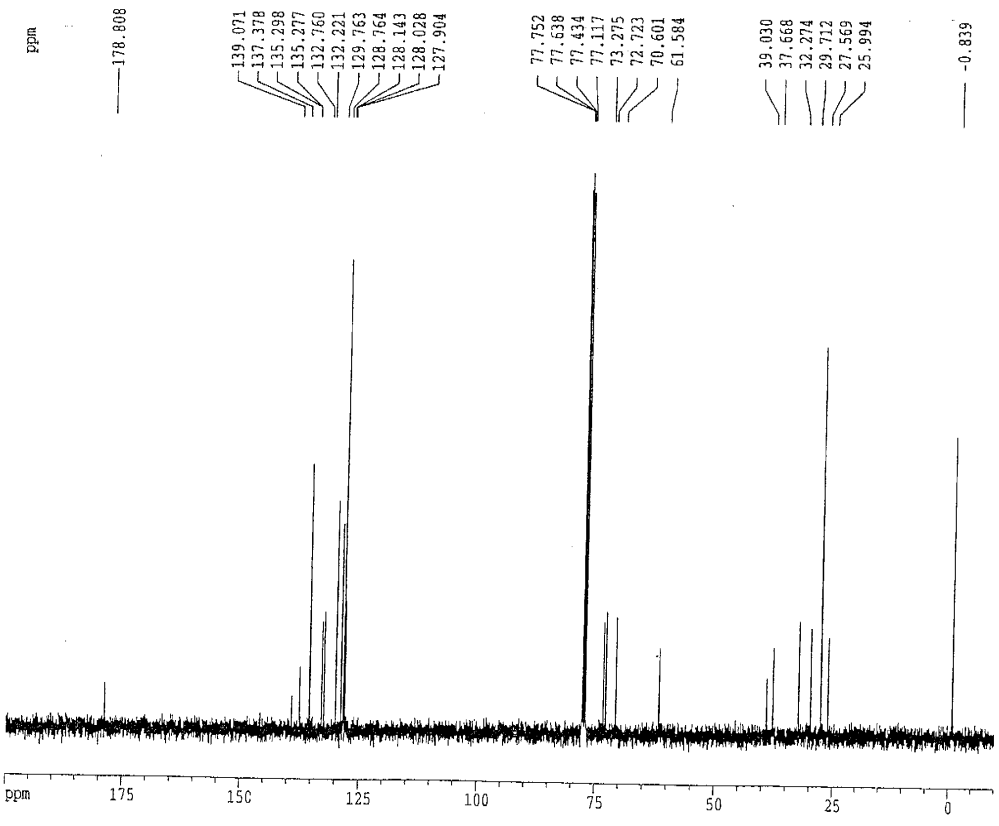
Current Data Parameters
NAME wj3-266
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030416
Time 11.29
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 90.5
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 8.000 ppm
F1 3201.04 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.42500 ppm/cm
HZCM 170.05525 Hz/cm



Current Data Parameters
NAME wj3-266
EXPNO 2
PROCNO 1

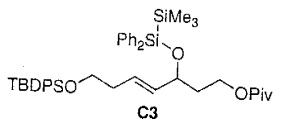
F2 - Acquisition Parameters
Date_ 20030416
Time 11.33
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 338
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 32768
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.6232933 MHz

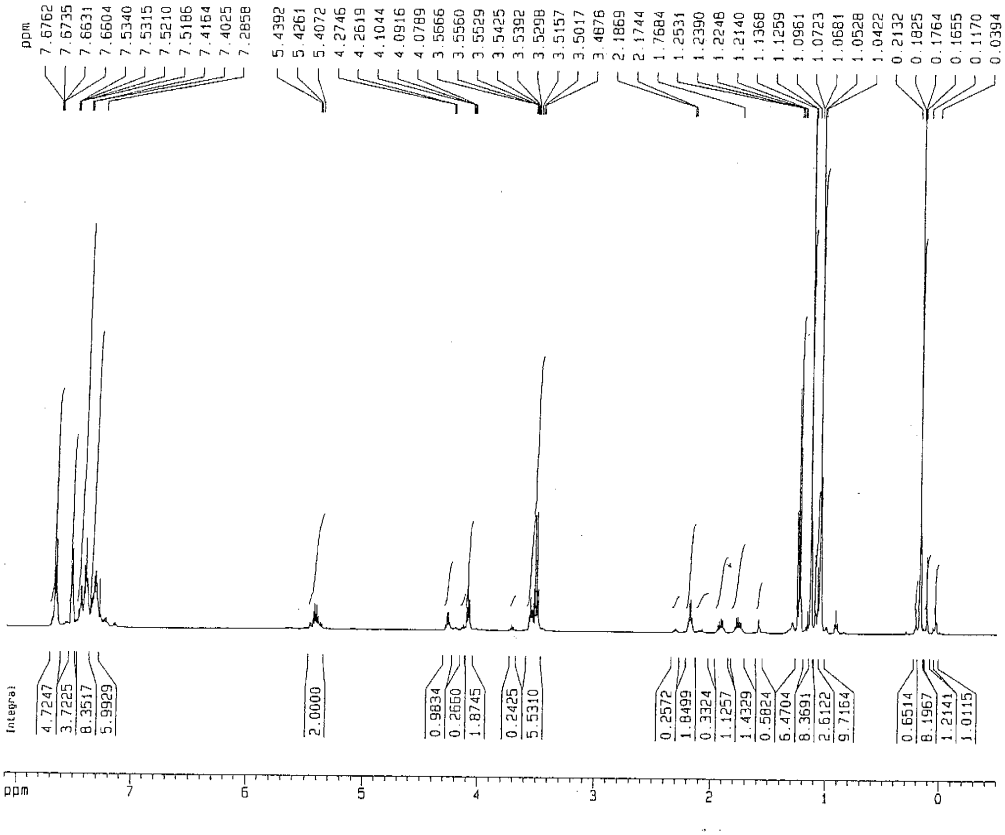
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -10.000 ppm
F2 -1006.11 Hz
PPMCM 10.50000 ppm/cm
HZCM 1056.43372 Hz/cm



C3




```
Current Data Parameters
NAME                  wj4-198.19-20
EXPNO                            1
PROCNO                           1

F2 - Acquisition Parameters
Date_                     20040405
Time_                        19.06
INSTRUM                      spect
PROBHD     5 mm BBO BB-1H
PULPROG                      zg30
TD                          32768
SOLVENT                     CDCl3
NS                             16
DS                              2
SWH                    6009.615 Hz
FIDRES                 0.183399 Hz
AQ                     2.7264309 sec
RG                         203.2
DW                     83.200 usec
DE                      6.00 usec
TE                        296.2 K
D1                   0.03000000 sec
MCREST               0.00000000 sec
MCWRK                0.01500000 sec

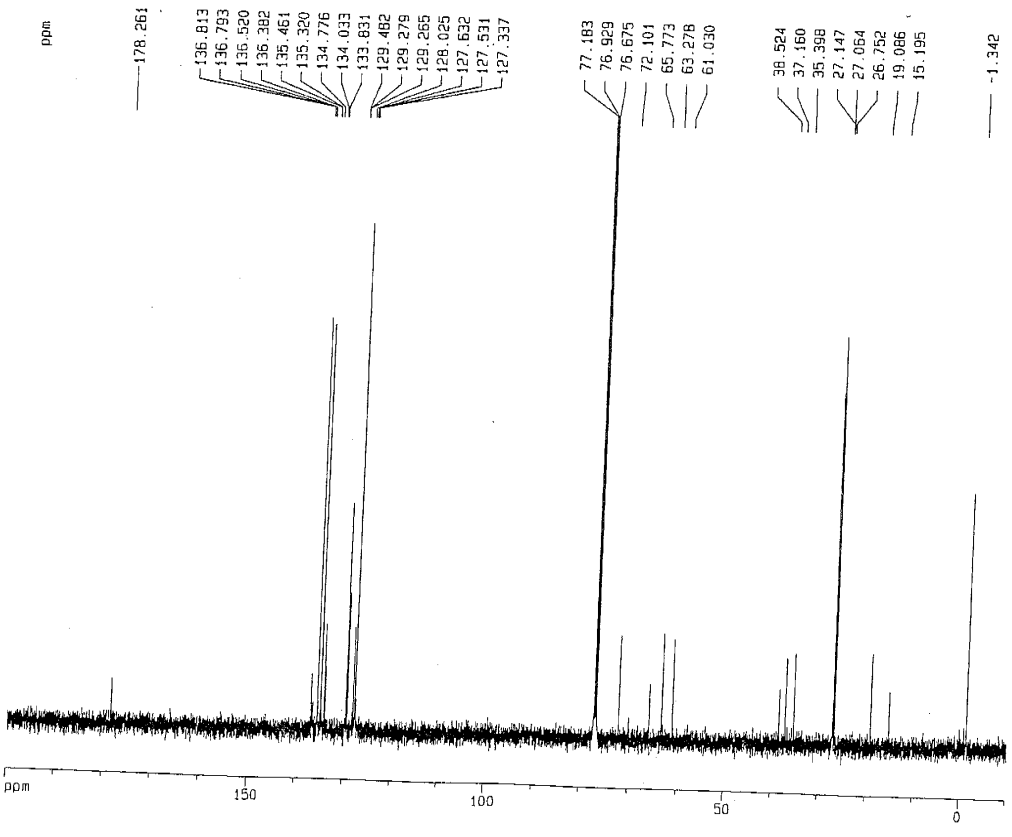
======== CHANNEL f1 ========
NUC1                          1H
P1                        7.70 usec
PL1                      -4.00 dB
SFO1              500.1320005 MHz

F1 - Acquisition parameters
NDO                            2
TD                           256
SFO1                500.1325 MHz
FIDRES              23.475050 Hz
SW                     12.016 ppm
FnMODE                 undefined

F2 - Processing parameters
SI                          32768
SF                  500.1300000 MHz
WDW                            EM
SSB                             0
LB                       0.30 Hz
GB                              0
PC                           1.00

F1 - Processing parameters
SI                           1024
MC2                            QF
SF                  500.1300000 MHz
WDW                         QSINE
SSB                             0
LB                       0.30 Hz
GB                              0

1D NMR plot parameters
CX                          20.00 cm
CY                           0.00 cm
F1P                       8.100 ppm
F1                      4051.05 Hz
F2P                      -0.500 ppm
F2                     -250.07 Hz
PPMCM                 0.43000 ppm/cm
HZCM                215.05591 Hz/cm
```




```
Current Data Parameters
NAME                  wj4-196-1
EXPNO                            2
PROCNO                           1

F2 - Acquisition Parameters
Date_                     20040824
Time                         14.50
INSTRUM                      spect
PROBHD     5 mm BBO BB-1H
PULPROG                   zgpg30
TD                          65536
SOLVENT                     CDCl3
NS                            644
DS                              4
SWH                   31446.541 Hz
FIDRES                0.479836 Hz
AQ                    1.0420724 sec
RG                            256
DW                     15.900 usec
DE                      6.00 usec
TE                        298.0 K
D1                   0.15000001 sec
D11                  0.03000000 sec
DELTA                0.05000000 sec
MCREST               0.00000000 sec
MCWRK                0.01500000 sec

======== CHANNEL f1 ========
NUC1                          13C
P1                        6.50 usec
PL1                       0.00 dB
SFO1              125.7697360 MHz

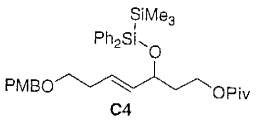
======== CHANNEL f2 ========
CPDPRG2                   waltz16
NUC2                          1H
PCPD2                     95.00 usec
PL2                      -4.00 dB
PL12                     19.00 dB
PL13                     30.00 dB
SFO2              500.1325000 MHz

F1 - Acquisition parameters
NDO                            2
TD                           256
SFO1                500.1325 MHz
FIDRES              23.475060 Hz
SW                     12.016 ppm
FnMODE                 undefined

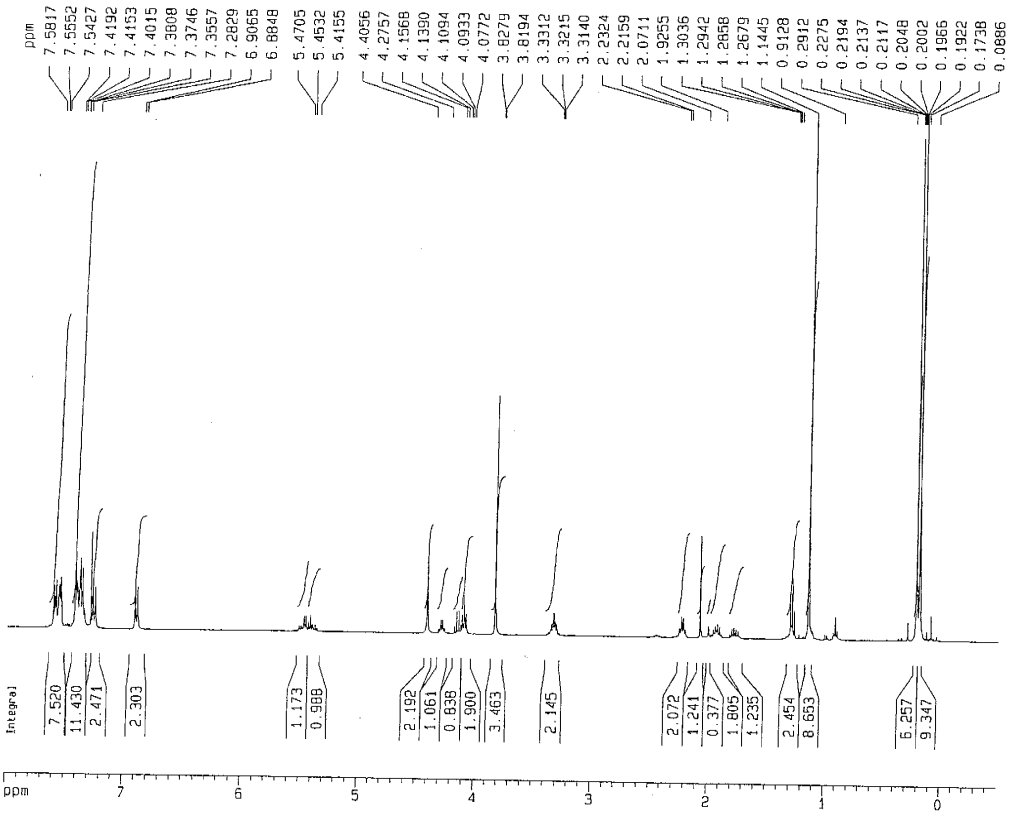
F2 - Processing parameters
SI                          65536
SF                  125.7578009 MHz
WDW                            EM
SSB                             0
LB                       1.00 Hz
GB                              0
PC                           1.40

F1 - Processing parameters
SI                           1024
MC2                            QF
SF                  500.1300000 MHz
WDW                            no
SSB                             0
LB                       0.30 Hz
GB                              0

1D NMR plot parameters
CX                          20.00 cm
CY                           0.00 cm
F1P                       200.000 ppm
F1                      25151.56 Hz
F2P                     -10.000 ppm
F2                     -1257.58 Hz
PPMCM                 10.50000 ppm/cm
HZCM                1320.45679 Hz/cm
```



C4



Current Data Parameters

NAME	wj5-147, 22-37
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050321
Time	18.44
INSTRUM	spect
PROBHD	5 mm QNP 1H/15
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	4789.272 Hz
FIDRES	0.146157 Hz
AQ	3.4210291 sec
RG	181
DW	104.400 usec
DE	6.00 usec
TE	295.6 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

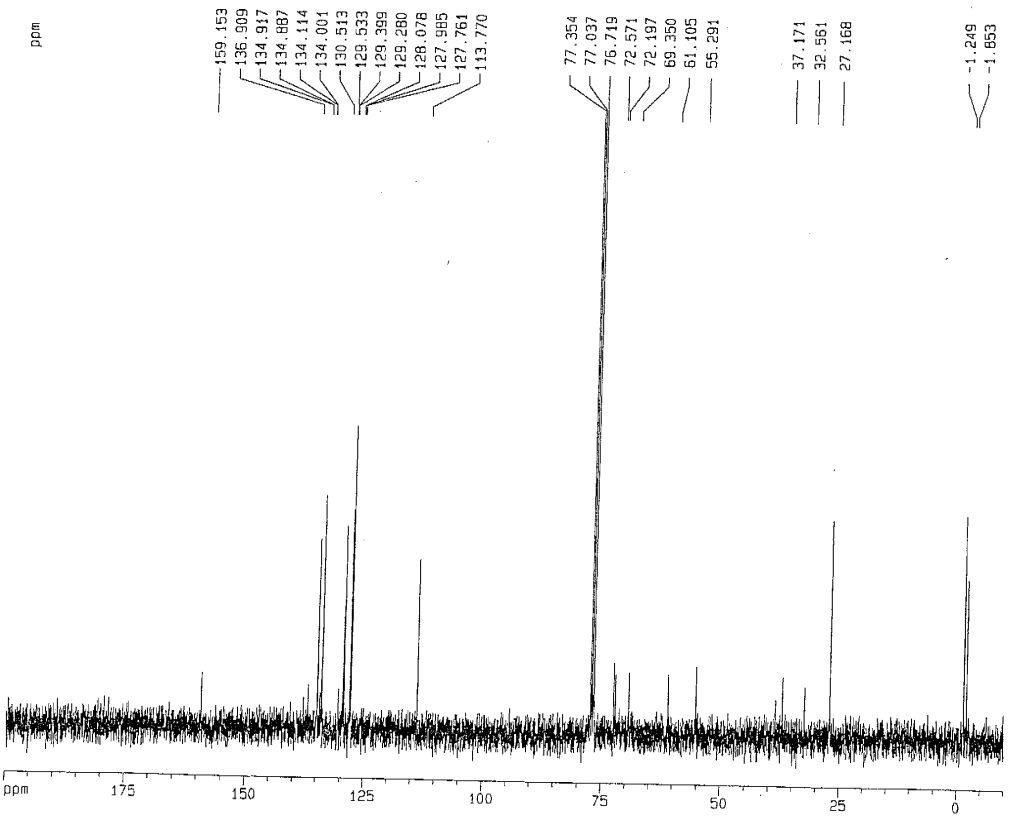
NUC1	1H
P1	7.05 usec
PL1	-3.00 dB
SFO1	400.1320007 MHz

F2 - Processing parameters

SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
CY	0.00 cm
F1P	8.000 ppm
F1	3201.04 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.42500 ppm/cm
HZCM	170.05525 Hz/cm



Current Data Parameters

NAME	wj5-147, 22-37
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20050321
Time	18.50
INSTRUM	spect
PROBHD	5 mm QNP 1H/15
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	498
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	2048
DW	20.850 usec
DE	6.00 usec
TE	295.8 K
D1	0.15000001 sec
d11	0.03000000 sec
DELTA	0.09000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	7.20 usec
PL1	-2.00 dB
SFO1	100.6228298 MHz

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

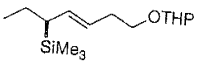
CPDPRG2	waltz16
NUC2	1H
PCPD2	98.40 usec
PL2	-3.00 dB
PL12	15.96 dB
PL13	120.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

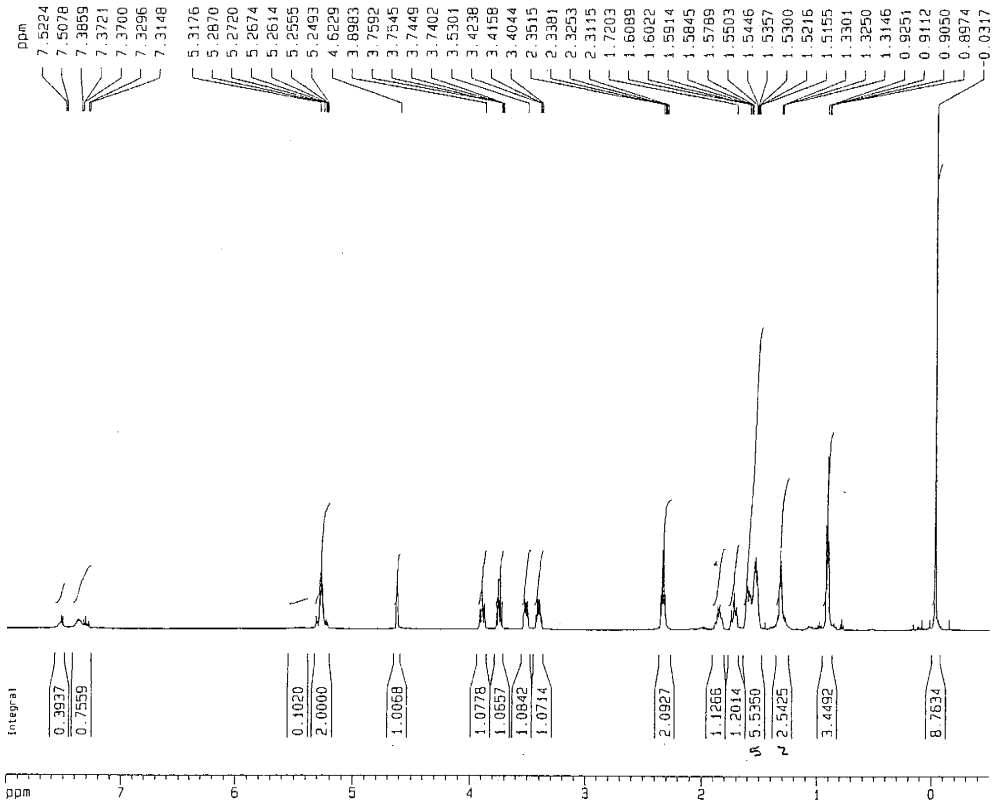
SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	0.00 cm
F1P	200.000 ppm
F1	20122.55 Hz
F2P	-10.000 ppm
F2	-1006.13 Hz
PPMCM	10.50000 ppm/cm
HZCM	1056.43396 Hz/cm



5a




```
Current Data Parameters
NAME                  wj2-229
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20020317
Time                   14.18
INSTRUM                spect
PROBHD     5 mm BBO BB-1H
PULPROG                zg30
TD                     32768
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                6009.615 Hz
FIDRES             0.183399 Hz
AQ                 2.7264309 sec
RG                        32
DW                   83.200 usec
DE                    6.00 usec
TE                   300.0 K
D1                 0.03000000 sec

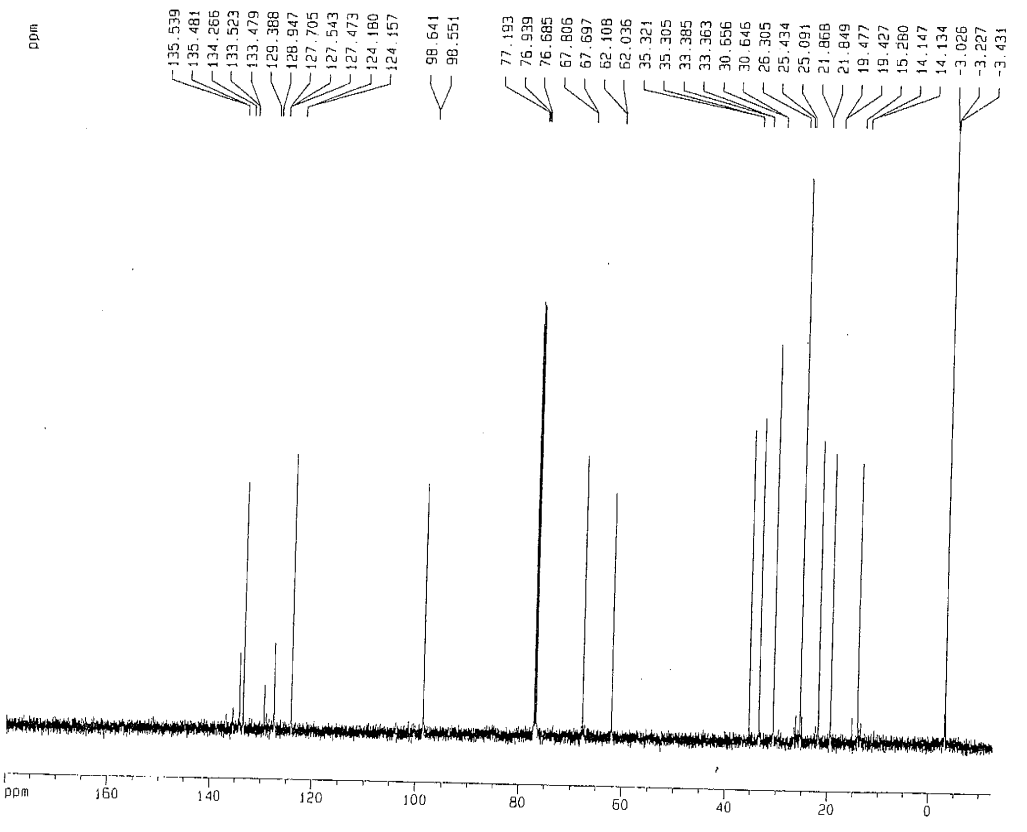
======== CHANNEL f1 ========
NUC1                       1H
P1                       7.70 usec
PL1                     -4.00 dB
SFO1           500.1320005 MHz

F1 - Acquisition parameters
NDO                        2
TD                       256
SFO1           500.1325 MHz
FIDRES         23.475060 Hz
SW                  12.016 ppm

F2 - Processing parameters
SI                     32768
SF             500.1300000 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00

F1 - Processing parameters
SI                      1024
MC2                       QF
SF             500.1300000 MHz
WDW                    QSINE
SSB                        0
LB                      0.30 Hz
GB                         0

1D NMR plot parameters
CX                     20.00 cm
CY                      0.00 cm
F1P                     8.000 ppm
F1                   4001.04 Hz
F2P                    -0.500 ppm
F2                   -250.07 Hz
PPMCM                0.42500 ppm/cm
HZCM               212.76525 Hz/cm
```




```
Current Data Parameters
NAME                  wj2-229
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20020317
Time                   14.22
INSTRUM                spect
PROBHD     5 mm BBO BB-1H
PULPROG                zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       192
DS                         4
SWH               31446.541 Hz
FIDRES             0.479836 Hz
AQ                 1.0420883 sec
RG                    5160.6
DW                   15.900 usec
DE                    6.00 usec
TE                   300.0 K
D1                 0.10000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

======== CHANNEL f1 ========
NUC1                       13C
P1                       6.50 usec
PL1                      5.00 dB
SFO1           125.7719472 MHz

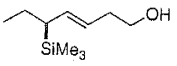
======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   95.00 usec
PL2                     -4.00 dB
PL12                    19.00 dB
PL13                    30.00 dB
SFO2           500.1325000 MHz

F1 - Acquisition parameters
NDO                        2
TD                       256
SFO1           500.1325 MHz
FIDRES         23.475060 Hz
SW                  12.016 ppm

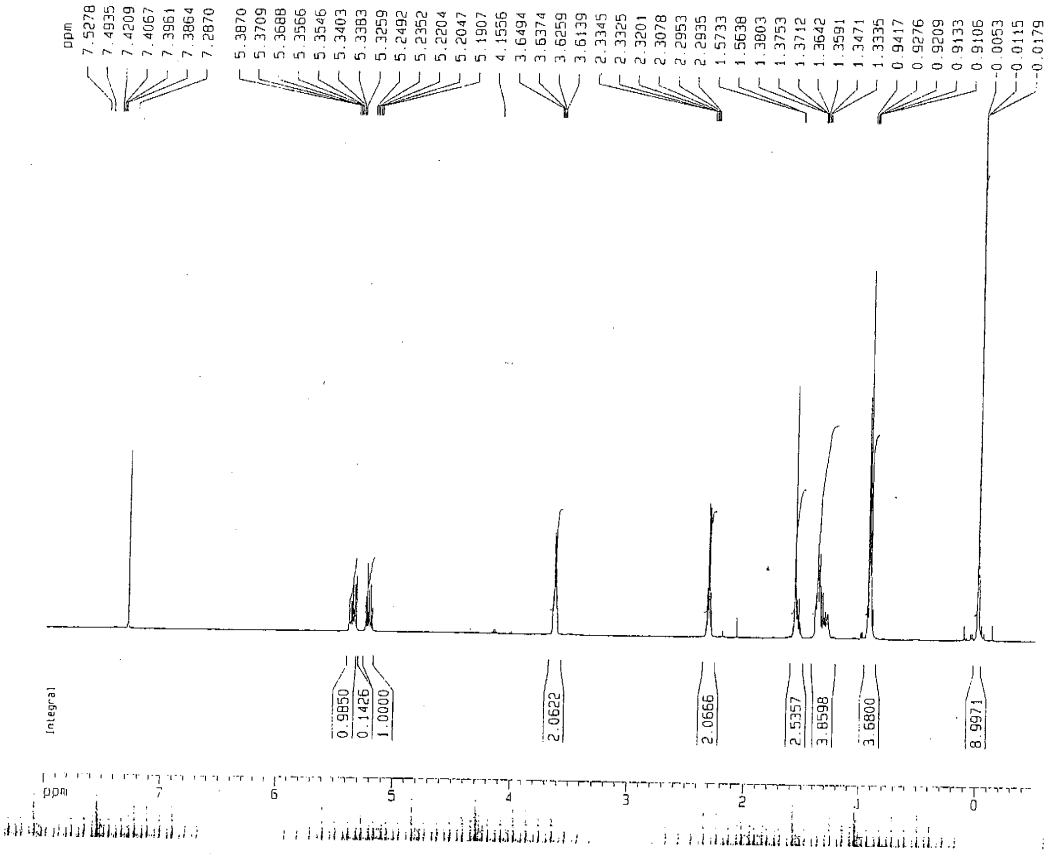
F2 - Processing parameters
SI                     65536
SF             125.7578008 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40

F1 - Processing parameters
SI                      1024
MC2                       QF
SF             500.1300000 MHz
WDW                       no
SSB                        0
LB                      0.30 Hz
GB                         0

1D NMR plot parameters
CX                     20.00 cm
CY                      0.00 cm
F1P                   180.000 ppm
F1                  22636.40 Hz
F2P                   -12.539 ppm
F2                  -1578.91 Hz
PPMCM                9.86696 ppm/cm
HZCM               1240.56577 Hz/cm
```



5b




```
Current Data Parameters
NAME                  wj3-254
EXPNO                       3
PROCNO                      1

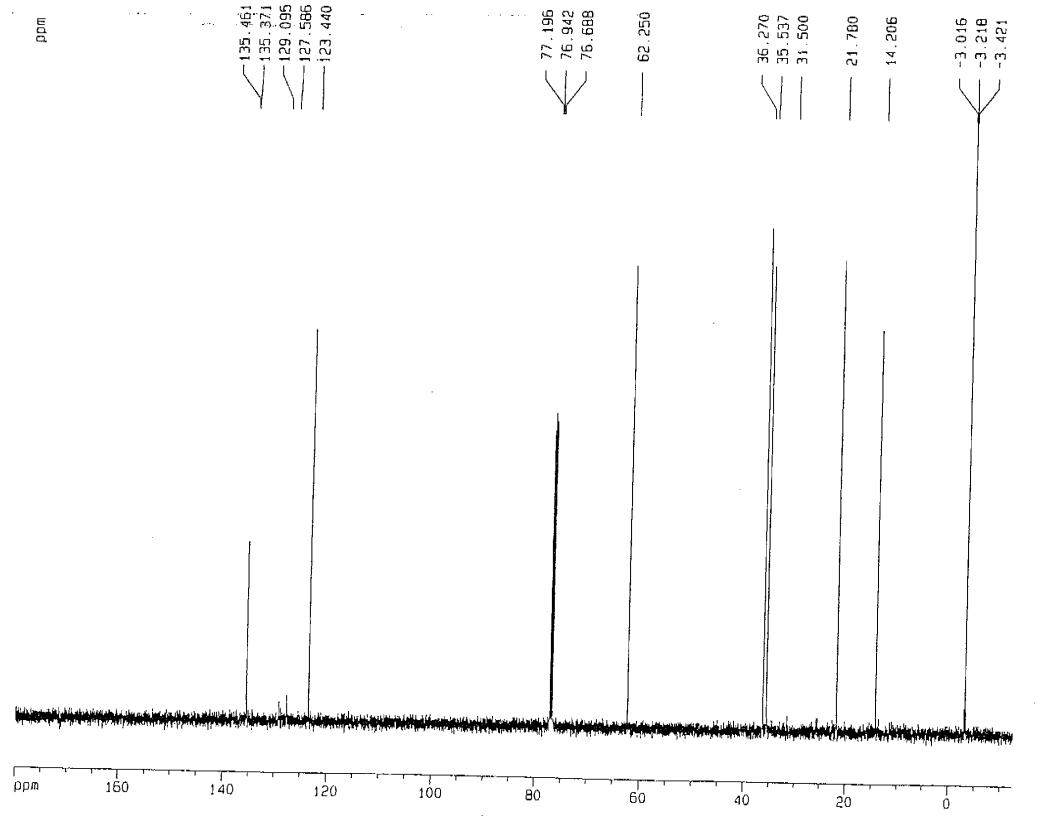
F2 - Acquisition Parameters
Date_              20030405
Time                   16.59
INSTRUM                spect
PROBHD    5 mm BBO BB-1H
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                6009.615 Hz
FIDRES             0.183399 Hz
AQ                 2.7264309 sec
RG                      512
DW                 83.200 usec
DE                  6.00 usec
TE                  300.0 K
D1                 0.03000000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     7.70 usec
PL1                   -4.00 dB
SFO1           500.1320005 MHz

F2 - Processing parameters
SI                    32768
SF             500.1300000 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00

F1 - Processing parameters
SI                     1024
MC2                      QF
SF             500.1300000 MHz
WDW                  QSINE
SSB                       0
LB                     0.30 Hz
GB                        0

1D NMR plot parameters
CX                    20.00 cm
CY                     0.00 cm
F1P                  8.000 ppm
F1                 4001.04 Hz
F2P                -0.500 ppm
F2                 -250.07 Hz
PPMCM              0.42500 ppm/cm
HZCM             212.55525 Hz/cm
```




```
Current Data Parameters
NAME                  wj2-232
EXPNO                       2
PROCNO                      1

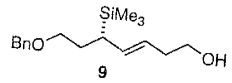
F2 - Acquisition Parameters
Date_              20020319
Time                   10.23
INSTRUM                spect
PROBHD    5 mm BBO BB-1H
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      105
DS                        4
SWH               31446.541 Hz
FIDRES             0.479836 Hz
AQ                 1.0420883 sec
RG                    2500.3
DW                 15.900 usec
DE                  6.00 usec
TE                  300.0 K
D1                 0.10000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                     6.50 usec
PL1                    5.00 dB
SFO1           125.7719472 MHz

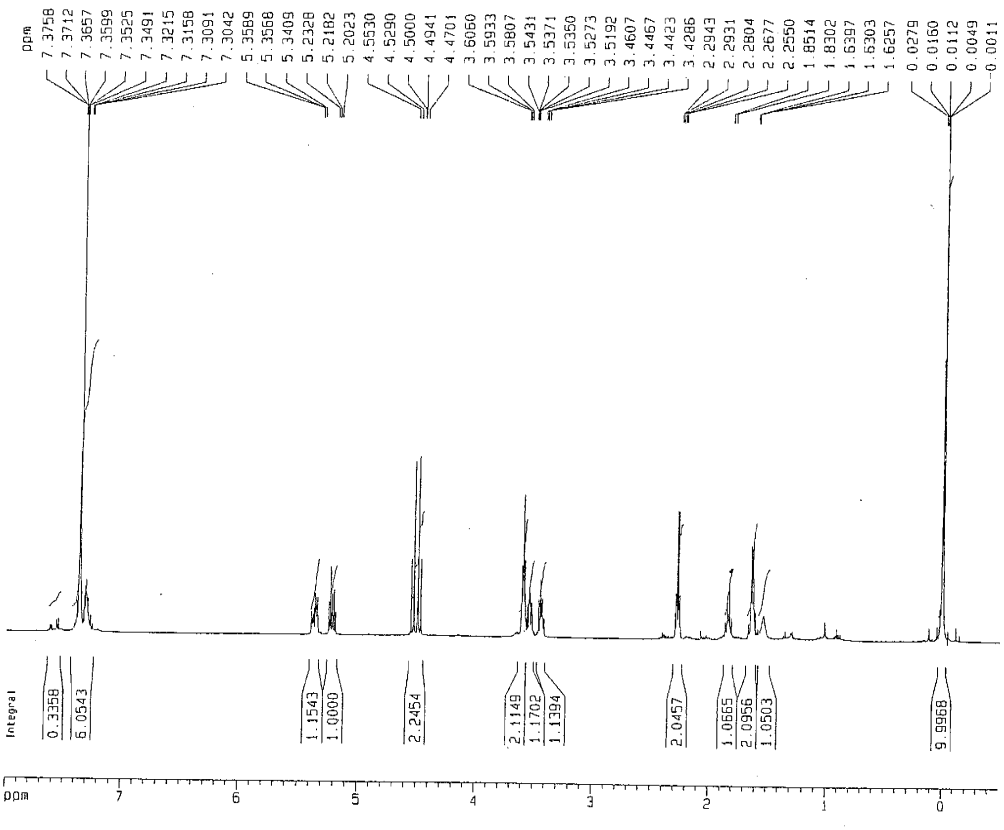
======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                 95.00 usec
PL2                   -4.00 dB
PL12                  19.00 dB
PL13                  30.00 dB
SFO2           500.1325000 MHz

F2 - Processing parameters
SI                    65536
SF             125.7578008 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40

1D NMR plot parameters
CX                    20.00 cm
CY                     0.00 cm
F1P                 180.000 ppm
F1                 22636.41 Hz
F2P                -12.539 ppm
F2                 -1576.91 Hz
PPMCM              9.62696 ppm/cm
HZCM             1210.66589 Hz/cm
```



9




```
Current Data Parameters
NAME                  wj2-227
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20020314
Time                   17.39
INSTRUM                spect
PROBHD     5 mm BBO BB-1H
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                6009.615 Hz
FIDRES            0.183399 Hz
AQ                2.7264309 sec
RG                       57
DW                  83.200 usec
DE                   6.00 usec
TE                  300.0 K
D1                0.03000000 sec

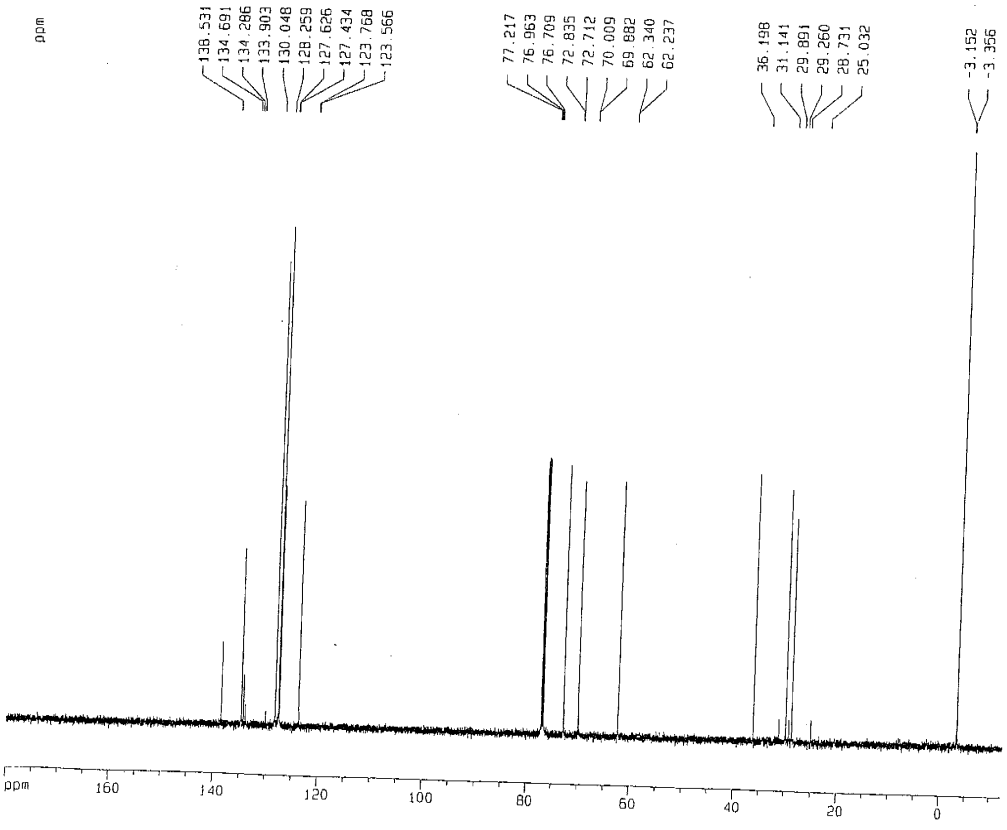
======== CHANNEL f1 ========
NUC1                      1H
P1                     7.70 usec
PL1                   -4.00 dB
SFO1          500.1320005 MHz

F1 - Acquisition parameters
NOD                       2
TD                      256
SFO1          500.1325 MHz
FIDRES        23.475060 Hz
SW                 12.016 ppm

F2 - Processing parameters
SI                    32768
SF          500.1300000 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00

F1 - Processing parameters
SI                     1024
MC2                      QF
SF          500.1300000 MHz
WDW                   QSINE
SSB                       0
LB                     0.30 Hz
GB                        0

1D NMR plot parameters
CX                    20.00 cm
CY                     0.00 cm
F1P                 8.000 ppm
F1                 4001.04 Hz
F2P                -0.500 ppm
F2                 -250.07 Hz
PPMCM              0.42500 ppm/cm
HZCM              212.55525 Hz/cm
```




```
Current Data Parameters
NAME                  wj2-227
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20020314
Time                   17.43
INSTRUM                spect
PROBHD     5 mm BBO BB-1H
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      232
DS                        4
SWH               31446.541 Hz
FIDRES            0.479836 Hz
AQ                1.0420683 sec
RG                     2048
DW                  15.900 usec
DE                   6.00 usec
TE                  300.0 K
D1                0.10000000 sec
D11               0.03000000 sec
D12               0.00002000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                     6.50 usec
PL1                    5.00 dB
SFO1          125.7719472 MHz

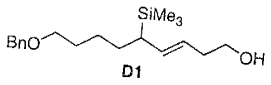
======== CHANNEL f2 ========
CPDPRG2               waltz16
NUC2                      1H
PCPD2                 95.00 usec
PL2                   -4.00 dB
PL12                  19.00 dB
PL13                  30.00 dB
SFO2          500.1325000 MHz

F1 - Acquisition parameters
NOD                       2
TD                      256
SFO1          500.1325 MHz
FIDRES        23.475060 Hz
SW                 12.016 ppm

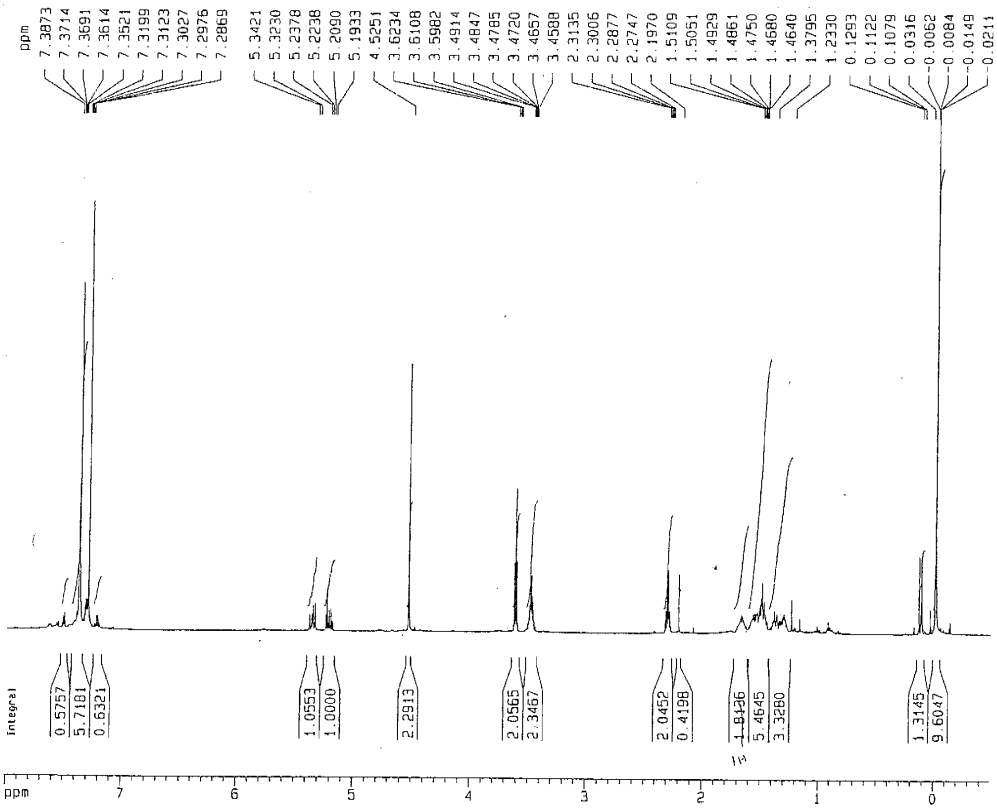
F2 - Processing parameters
SI                    65536
SF          125.7578009 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40

F1 - Processing parameters
SI                     1024
MC2                      QF
SF          500.1300000 MHz
WDW                      no
SSB                       0
LB                     0.30 Hz
GB                        0

1D NMR plot parameters
CX                    20.00 cm
CY                     0.00 cm
F1P                 180.000 ppm
F1                 22636.40 Hz
F2P                -12.539 ppm
F2                 -1576.91 Hz
PPMCM              9.62696 ppm/cm
HZCM              1210.66577 Hz/cm
```



D1




```
Current Data Parameters
NAME                  wj3-268
EXPNO                       1
PROCNO                      1

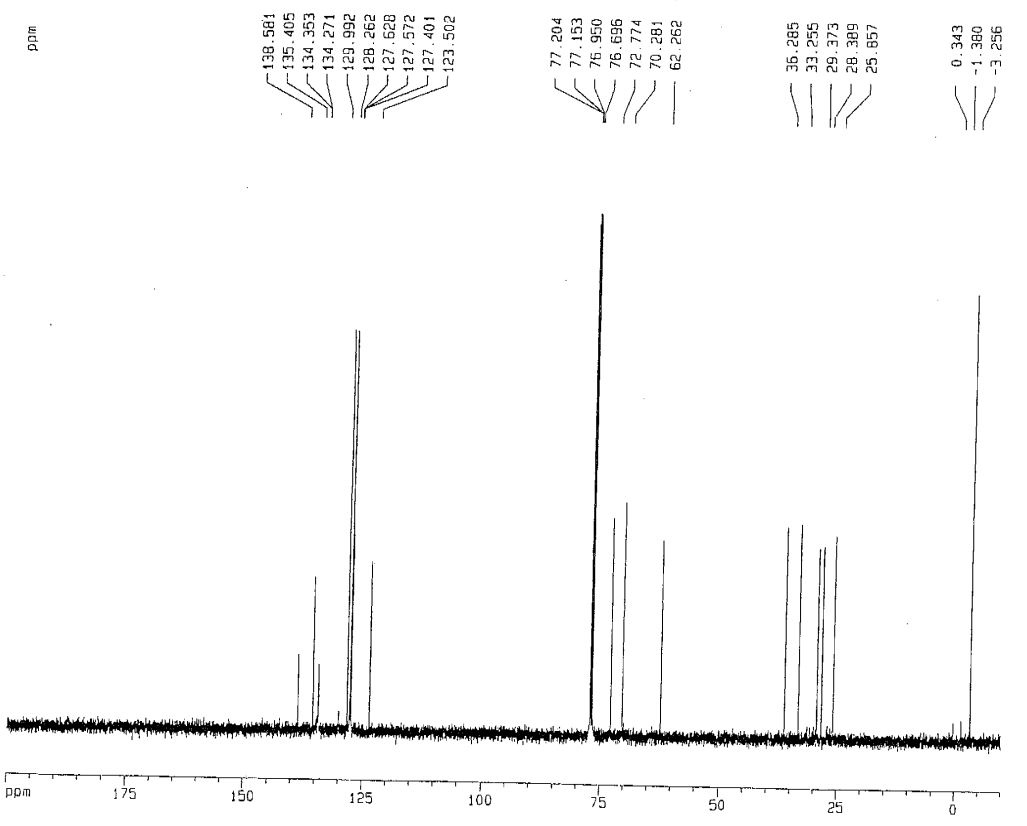
F2 - Acquisition Parameters
Date_              20030418
Time                   19.06
INSTRUM                spect
PROBHD     5 mm BBO BB-1H
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                6009.615 Hz
FIDRES             0.183399 Hz
AQ                 2.7264309 sec
RG                     101.6
DW                  83.200 usec
DE                   6.00 usec
TE                   300.0 K
D1                0.03000000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     7.70 usec
PL1                   -4.00 dB
SFO1           500.1320005 MHz

F2 - Processing parameters
SI                        32768
SF                500.1300000 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00

F1 - Processing parameters
SI                         1024
MC2                          QF
SF                500.1300000 MHz
WDW                          no
SSB                           0
LB                         0.30 Hz
GB                            0

1D NMR plot parameters
CX                        20.00 cm
CY                         0.00 cm
F1P                    8.000 ppm
F1                  4001.04 Hz
F2P                   -0.500 ppm
F2                   -250.07 Hz
PPMCM                 0.42500 ppm/cm
HZCM                212.55525 Hz/cm
```




```
Current Data Parameters
NAME                  wj3-269
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20030418
Time                   19.11
INSTRUM                spect
PROBHD     5 mm BBO BB-1H
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      338
DS                        4
SWH               31446.541 Hz
FIDRES             0.479836 Hz
AQ                 1.0420883 sec
RG                     2048
DW                  15.900 usec
DE                   6.00 usec
TE                   300.0 K
D1                0.10000000 sec
D11               0.03000000 sec
D12               0.00002000 sec

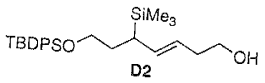
======== CHANNEL f1 ========
NUC1                      13C
P1                     6.50 usec
PL1                    5.00 dB
SFO1           125.7719472 MHz

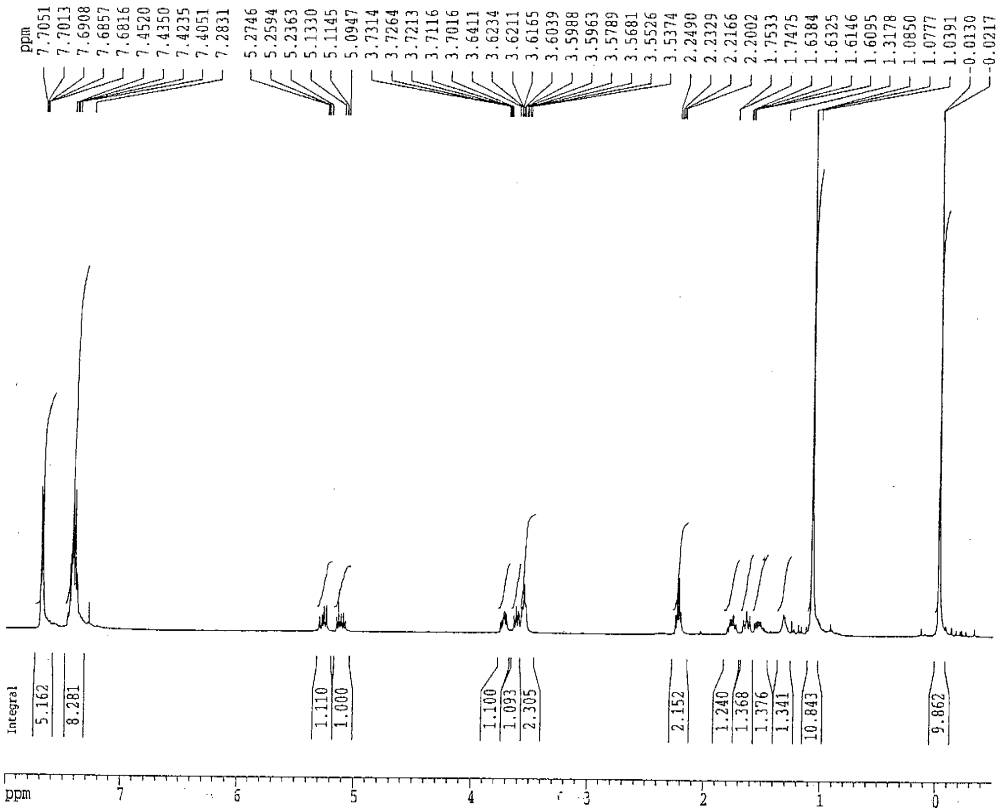
======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                 95.00 usec
PL2                   -4.00 dB
PL12                  19.00 dB
PL13                  30.00 dB
SFO2           500.1325000 MHz

F2 - Processing parameters
SI                        65536
SF                125.7570008 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40

F1 - Processing parameters
SI                         1024
MC2                          QF
SF                500.1300000 MHz
WDW                          no
SSB                           0
LB                         0.30 Hz
GB                            0

1D NMR plot parameters
CX                        20.00 cm
CY                         0.00 cm
F1P                  200.000 ppm
F1                  25151.56 Hz
F2P                  -10.000 ppm
F2                  -1257.58 Hz
PPMCM                10.50000 ppm/cm
HZCM              1320.45691 Hz/cm
```





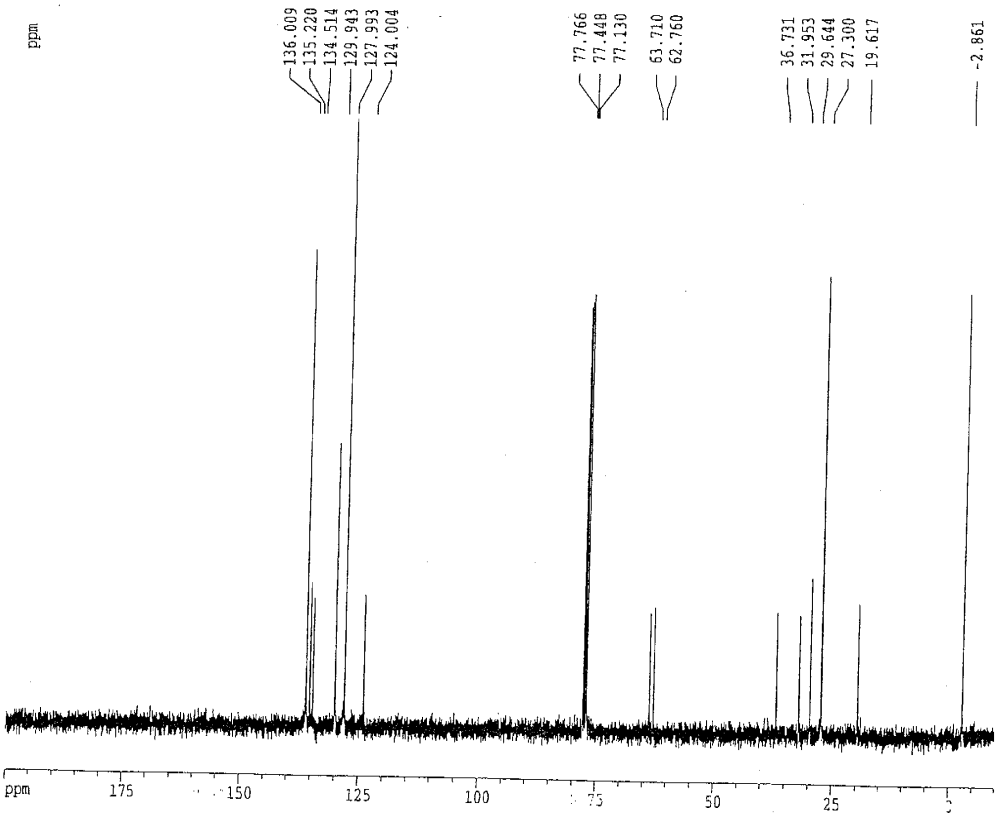
Current Data Parameters
NAME wj4-197
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040408
Time 14.01
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 90.5
DW 104.400 usec
DE 5.50 usec
TE 1809.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 8.000 ppm
F1 3201.04 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.42500 ppm/cm
HZCM 170.05525 Hz/cm



Current Data Parameters
NAME wj4-197
EXPNO 2
PROCNO 1

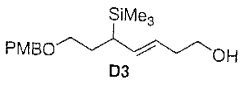
F2 - Acquisition Parameters
Date_ 20040408
Time 14.04
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 180
DS 4
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 32768
DW 21.600 usec
DE 5.50 usec
TE 1809.2 K
D1 0.15000001 sec
d11 0.03000000 sec
DELTA 0.05000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

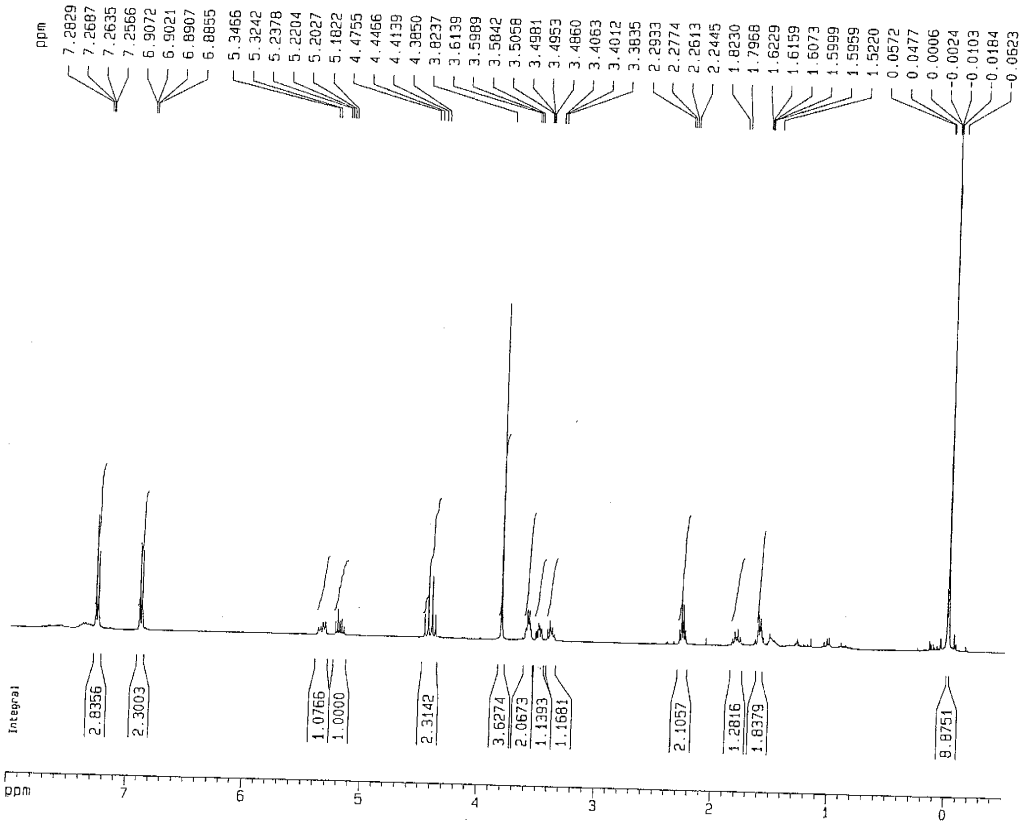
======== CHANNEL f1 ========
NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.6232933 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 65536
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 10.50000 ppm/cm
HZCM 1056.41372 Hz/cm





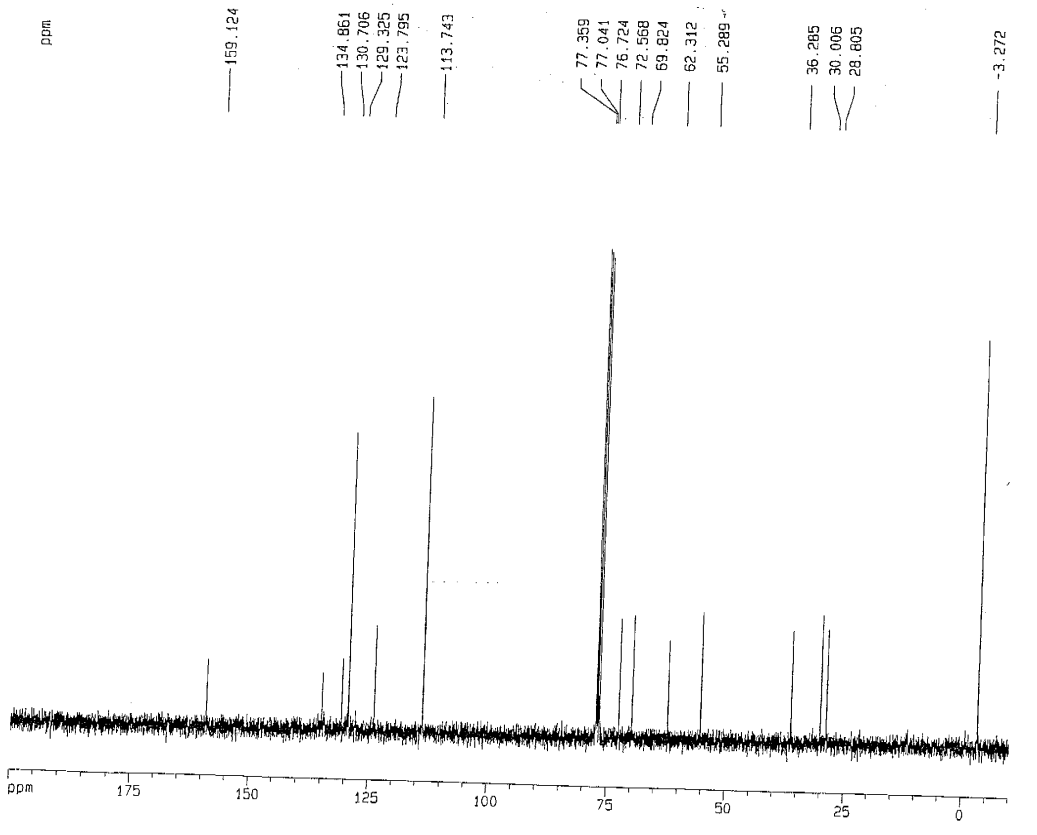
Current Data Parameters
NAME wj5-148
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050322
Time 18.30
INSTRUM spect
PROBHD 5 mm QNP 1H/15
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 161.3
DW 104.400 usec
DE 6.00 usec
TE 296.1 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.05 usec
PL1 -3.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 8.000 ppm
F1 3201.04 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.42500 ppm/cm
HZCM 170.05525 Hz/cm



Current Data Parameters
NAME wj5-148
EXPNO 2
PROCNO 1

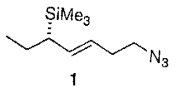
F2 - Acquisition Parameters
Date_ 20050322
Time 18.34
INSTRUM spect
PROBHD 5 mm QNP 1H/15
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 263
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2048
DW 20.850 usec
DE 6.00 usec
TE 296.2 K
D1 0.15000001 sec
d11 0.03000000 sec
DELTA 0.05000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 7.20 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz

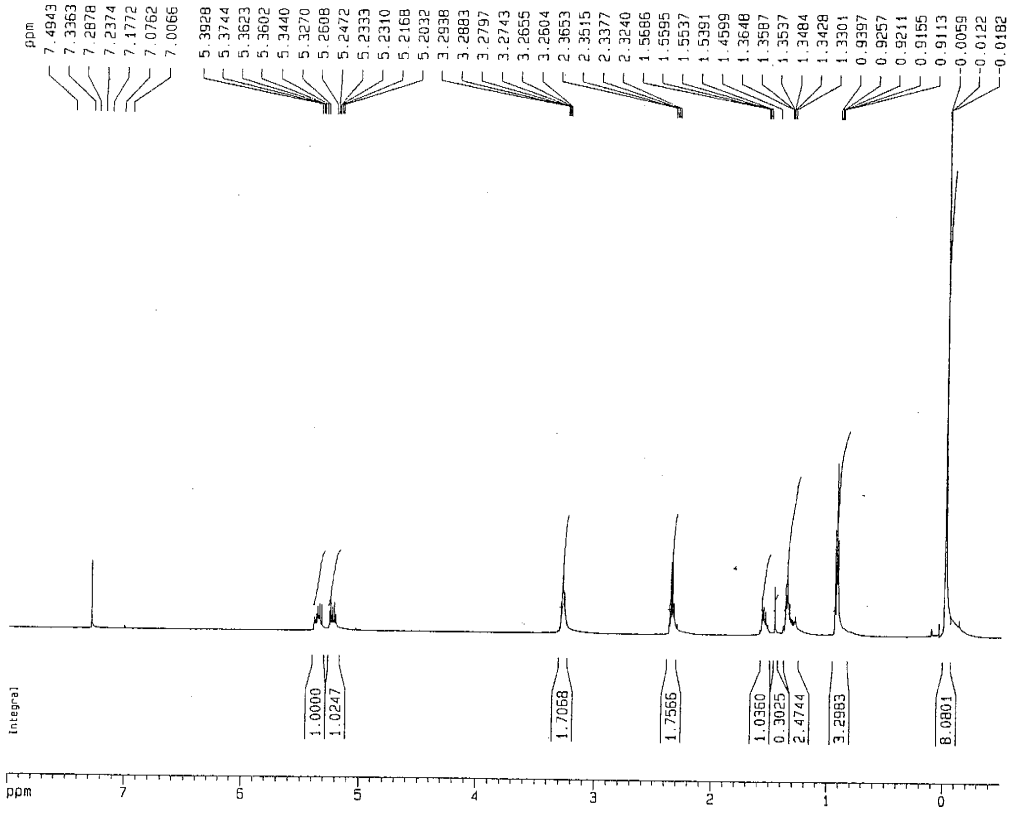
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 98.40 usec
PL2 -3.00 dB
PL12 19.96 dB
PL13 120.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 10.50000 ppm/cm
HZCM 1056.43408 Hz/cm



1




```
Current Data Parameters
NAME                  wj2-153
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20011213
Time                   16.44
INSTRUM                spect
PROBHD   5 mm TXI IN-13
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                6009.615 Hz
FIDRES            0.183399 Hz
AQ                2.7264309 sec
RG                    203.2
DW                 83.200 usec
DE                  6.00 usec
TE                  300.0 K
D1                0.03000000 sec

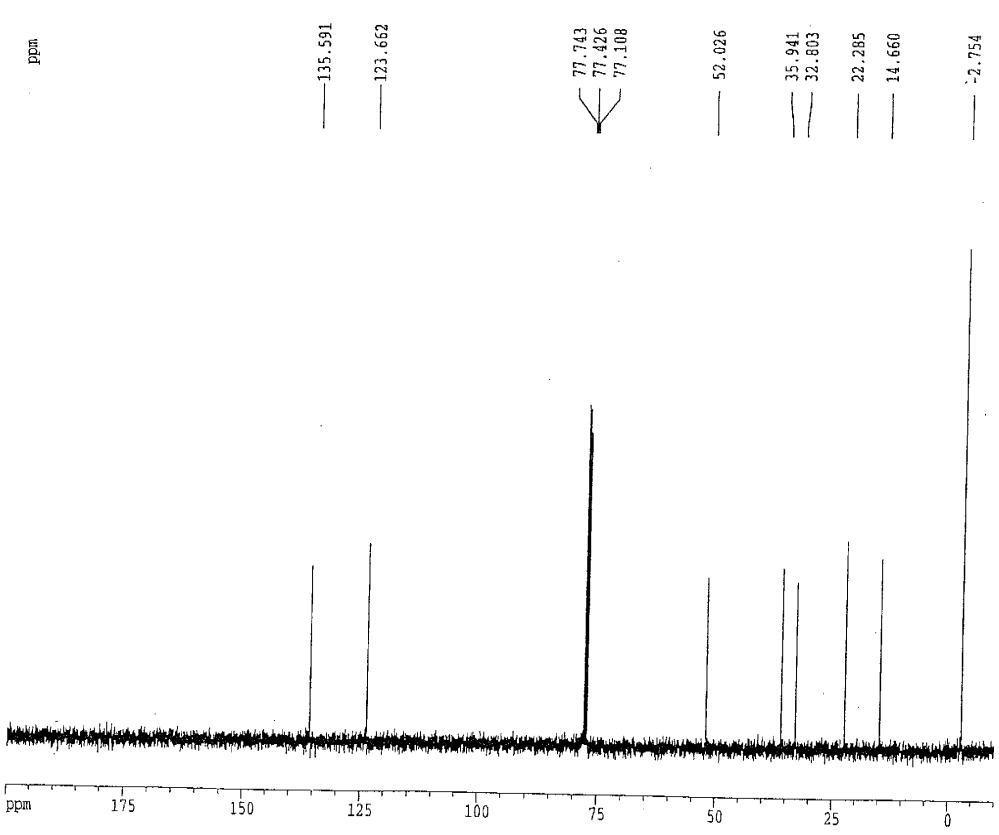
======== CHANNEL f1 ========
NUC1                      1H
P1                    7.70 usec
PL1                  -4.00 dB
SFO1           500.1320005 MHz

F1 - Acquisition parameters
NDO                       2
TD                      256
SFO1           500.1325 MHz
FIDRES         23.475060 Hz
SW                 12.016 ppm

F2 - Processing parameters
SI                    32768
SF           500.1300000 MHz
WDW                      EM
SSB                       0
LB                   0.30 Hz
GB                        0
PC                     1.00

F1 - Processing parameters
SI                     1024
MC2                      QF
SF           500.1300000 MHz
WDW                   QSINE
SSB                       0
LB                   0.30 Hz
GB                        0

1D NMR plot parameters
CX                   20.00 cm
CY                    0.00 cm
F1P                8.000 ppm
F1                 4001.04 Hz
F2P               -0.500 ppm
F2                -250.07 Hz
PPMCM          0.42500 ppm/cm
HZCM           212.55525 Hz/cm
```




```
Current Data Parameters
NAME                  wj3-24,c
EXPNO                       2
PROCNO                      1

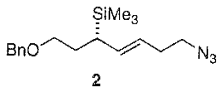
F2 - Acquisition Parameters
Date_              20030506
Time                   15.08
INSTRUM                drx400
PROBHD   5 mm Multinucl
PULPROG               zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      120
DS                        2
SWH               23148.148 Hz
FIDRES            0.353213 Hz
AQ                1.4156276 sec
RG                    32768
DW                 21.600 usec
DE                  4.50 usec
TE                  300.0 K
D1                0.05000000 sec
d11               0.03000000 sec
d12               0.00002000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                   12.30 usec
PL1                   2.00 dB
SFO1           100.6232933 MHz

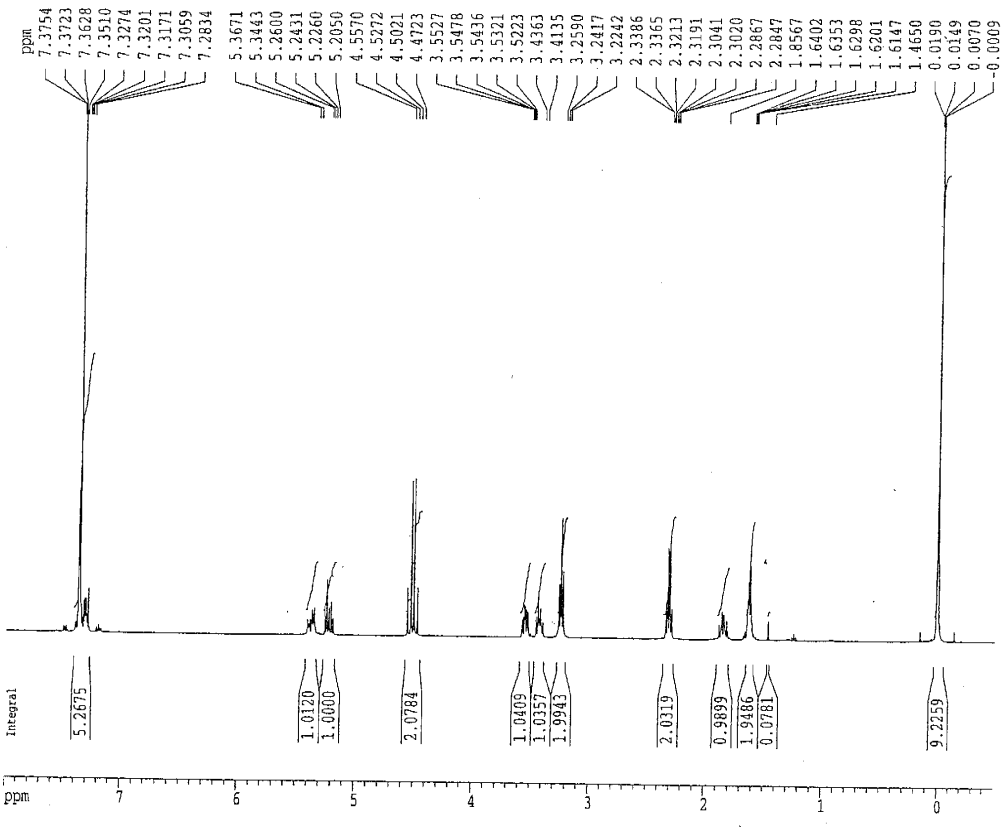
======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                   0.00 dB
PL12                 18.00 dB
PL13                 18.00 dB
SFO2           400.1316005 MHz

F2 - Processing parameters
SI                    32768
SF           100.6127290 MHz
WDW                      EM
SSB                       0
LB                   1.00 Hz
GB                        0
PC                     1.40

1D NMR plot parameters
CX                   20.00 cm
CY                    0.00 cm
F1P                200.000 ppm
F1                 20122.55 Hz
F2P                -10.000 ppm
F2                -1006.13 Hz
PPMCM          10.50000 ppm/cm
HZCM           1056.43372 Hz/cm
```



2



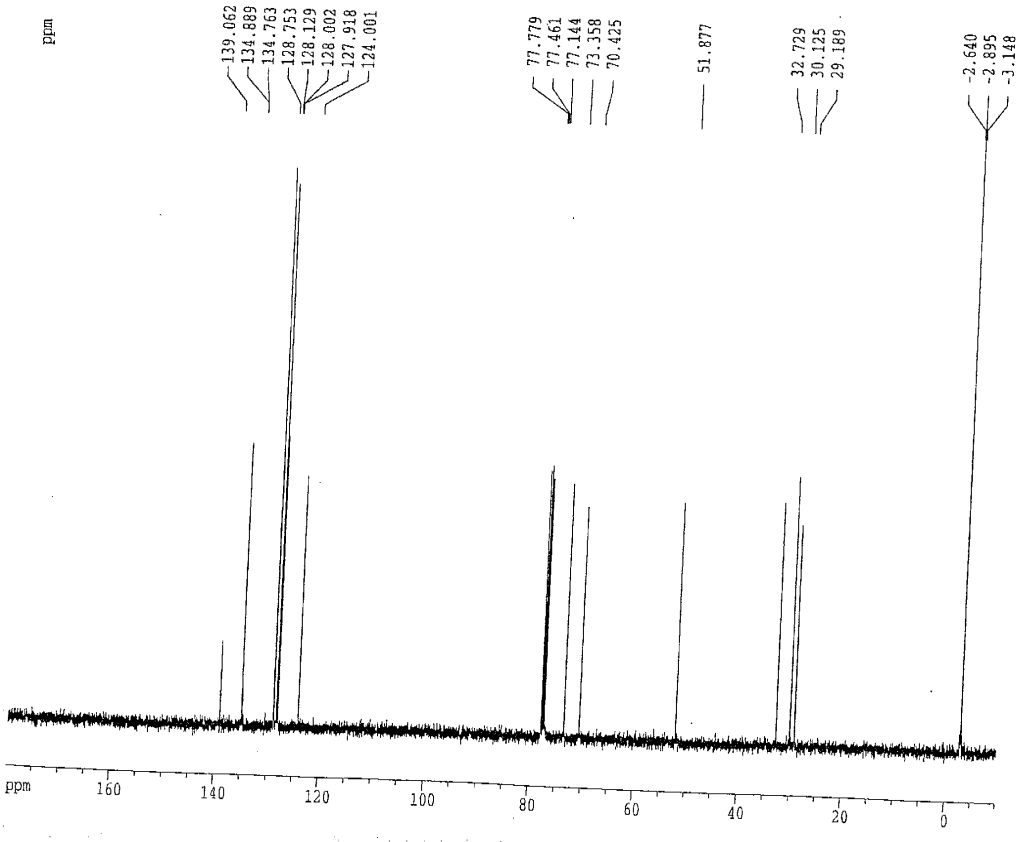

```
Current Data Parameters
NAME                  wj2-200
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_               20020218
Time                   11.08
INSTRUM                drx400
PROBHD     5 mm Multinu
PULPROG                zg30
TD                     32768
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                4789.272 Hz
FIDRES            0.146157 Hz
AQ                 3.4210291 sec
RG                        64
DW                 104.400 usec
DE                   4.50 usec
TE                   300.0 K
D1                1.00000000 sec

============ CHANNEL f1 ============
NUC1                      1H
P1                     7.70 usec
PL1                   -6.00 dB
SFO1          400.1320007 MHz

F2 - Processing parameters
SI                        16384
SF                400.1300000 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00

1D NMR plot parameters
CX                     20.00 cm
F1P                    8.000 ppm
F1                   3201.04 Hz
F2P                   -0.500 ppm
F2                   -200.07 Hz
PPMCM                0.42500 ppm/cm
HZCM               170.05525 Hz/cm
```




```
Current Data Parameters
NAME                  wj2-200
EXPNO                       2
PROCNO                      1

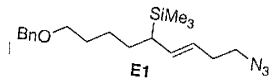
F2 - Acquisition Parameters
Date_               20020218
Time                   11.12
INSTRUM                drx400
PROBHD     5 mm Multinu
PULPROG             zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       155
DS                         2
SWH               23148.148 Hz
FIDRES            0.353213 Hz
AQ                 1.4156276 sec
RG                      1024
DW                 21.600 usec
DE                   4.50 usec
TE                   300.0 K
D1                0.05000000 sec
d11               0.03000000 sec
d12               0.00002000 sec

============ CHANNEL f1 ============
NUC1                      13C
P1                    12.30 usec
PL1                    2.00 dB
SFO1          100.6232933 MHz

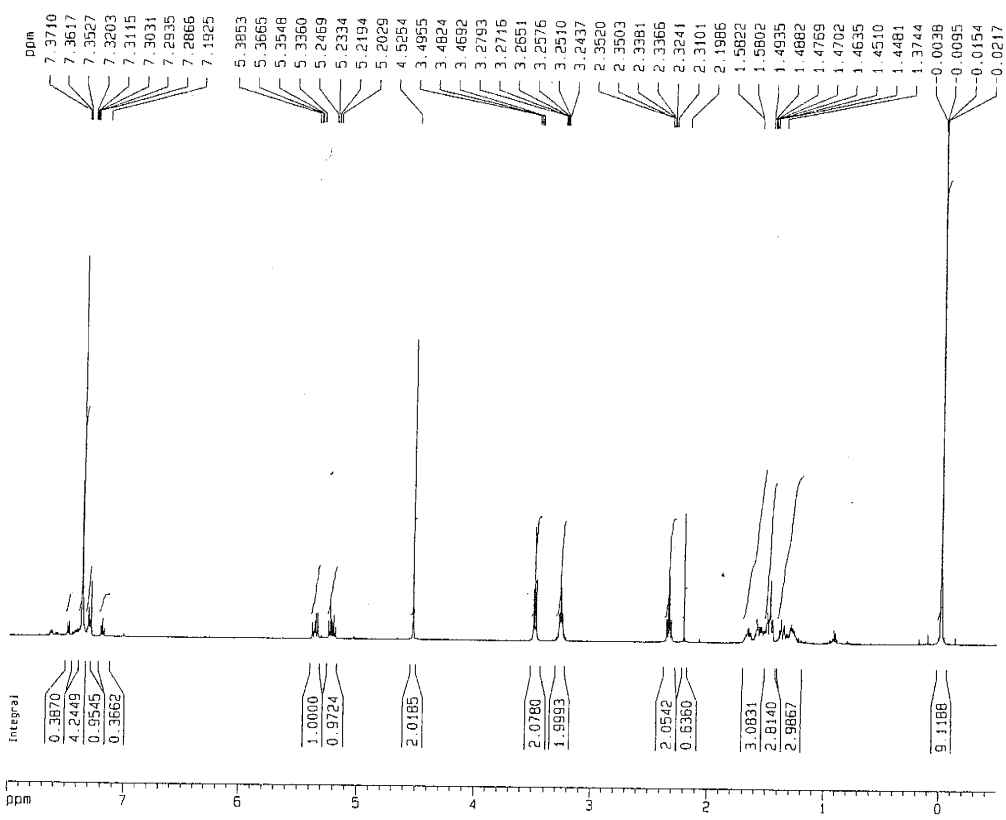
============ CHANNEL f2 ============
CPDPRG2                waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                    0.00 dB
PL12                  18.00 dB
PL13                  18.00 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                        32768
SF                100.6127290 MHz
WDW                       EM
SSB                        0.
LB                      1.00 Hz
GB                         0
PC                      1.40

1D NMR plot parameters
CX                     20.00 cm
F1P                  180.000 ppm
F1                 18110.29 Hz
F2P                  -10.036 ppm
F2                 -1009.71 Hz
PPMCM                9.50179 ppm/cm
HZCM               956.00128 Hz/cm
```



E1




```
Current Data Parameters
NAME                  wj3-278
EXPNO                       1
PROCNO                      1

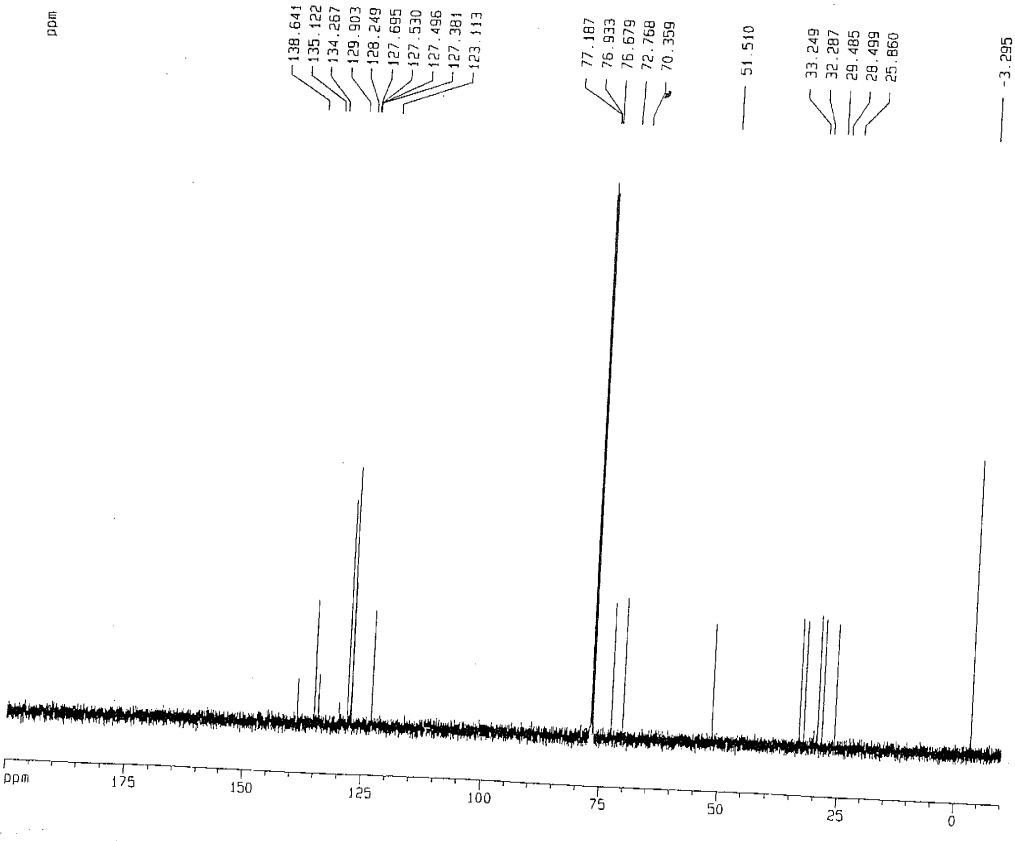
F2 - Acquisition Parameters
Date_              20030427
Time                   16.38
INSTRUM                spect
PROBHD  5 mm BBO BB-1H
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                6009.615 Hz
FIDRES             0.183399 Hz
AQ                 2.7264309 sec
RG                      181
DW                   83.200 usec
DE                    6.00 usec
TE                   300.0 K
D1                 0.03000000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     7.70 usec
PL1                   -4.00 dB
SFO1           500.1320005 MHz

F2 - Processing parameters
SI                        32768
SF                 500.1300000 MHz
WDW                        EM
SSB                         0
LB                      0.30 Hz
GB                          0
PC                       1.00

F1 - Processing parameters
SI                         1024
MC2                          QF
SF                 500.1300000 MHz
WDW                       QSINE
SSB                         0
LB                      0.30 Hz
GB                          0

1D NMR plot parameters
CX                     20.00 cm
CY                      0.00 cm
F1P                  8.000 ppm
F1                4001.04 Hz
F2P                 -0.500 ppm
F2                 -250.07 Hz
PPMCM               0.42500 ppm/cm
HZCM              212.55525 Hz/cm
```




```
Current Data Parameters
NAME                  wj3-278
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20030427
Time                   16.42
INSTRUM                spect
PROBHD  5 mm BBO BB-1H
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      216
DS                        4
SWH               31446.541 Hz
FIDRES             0.479836 Hz
AQ                 1.0420683 sec
RG                     2048
DW                   15.900 usec
DE                    6.00 usec
TE                   300.0 K
D1                 0.10000000 sec
D11                0.03000000 sec
D12                0.00002000 sec

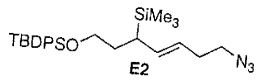
======== CHANNEL f1 ========
NUC1                      13C
P1                    6.50 usec
PL1                   5.00 dB
SFO1           125.7719472 MHz

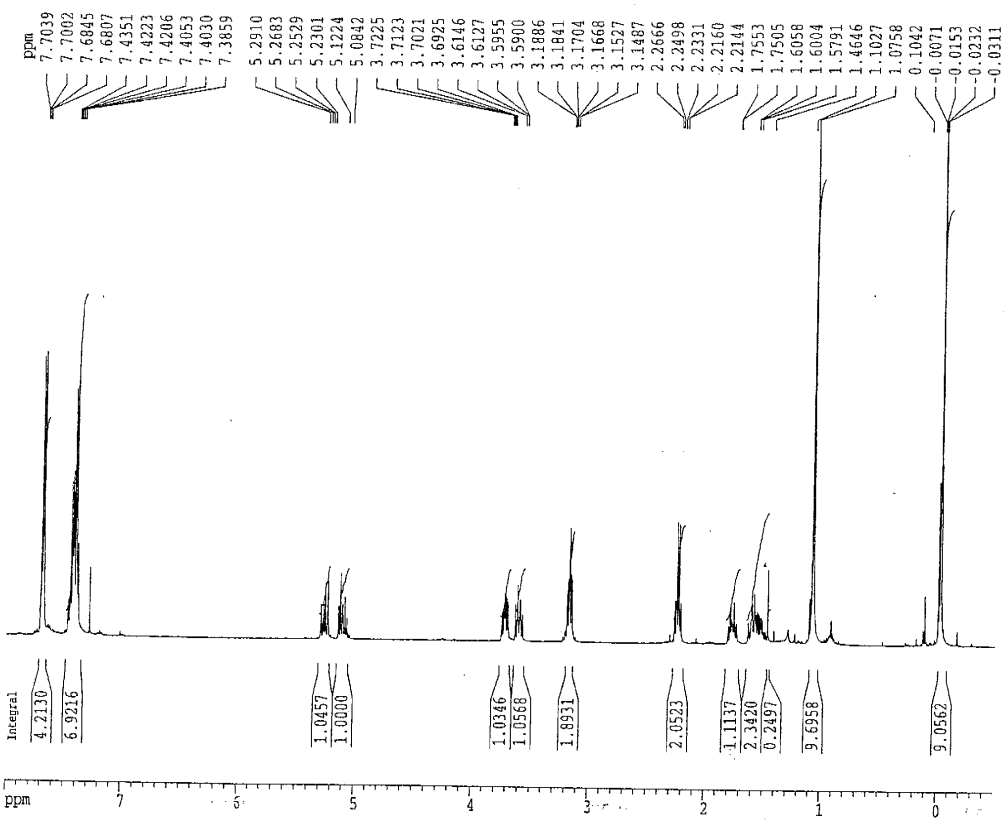
======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                 95.00 usec
PL2                   -4.00 dB
PL12                  19.00 dB
PL13                  30.00 dB
SFO2           500.1325000 MHz

F2 - Processing parameters
SI                        65536
SF                 125.7578006 MHz
WDW                        EM
SSB                         0
LB                      1.00 Hz
GB                          0
PC                       1.40

F1 - Processing parameters
SI                         1024
MC2                          QF
SF                 500.1300000 MHz
WDW                         no
SSB                         0
LB                      0.30 Hz
GB                          0

1D NMR plot parameters
CX                     20.00 cm
CY                      0.00 cm
F1P                  200.000 ppm
F1                25151.58 Hz
F2P                 -10.000 ppm
F2                 -1257.58 Hz
PPMCM               10.50000 ppm/cm
HZCM              1320.45691 Hz/cm
```





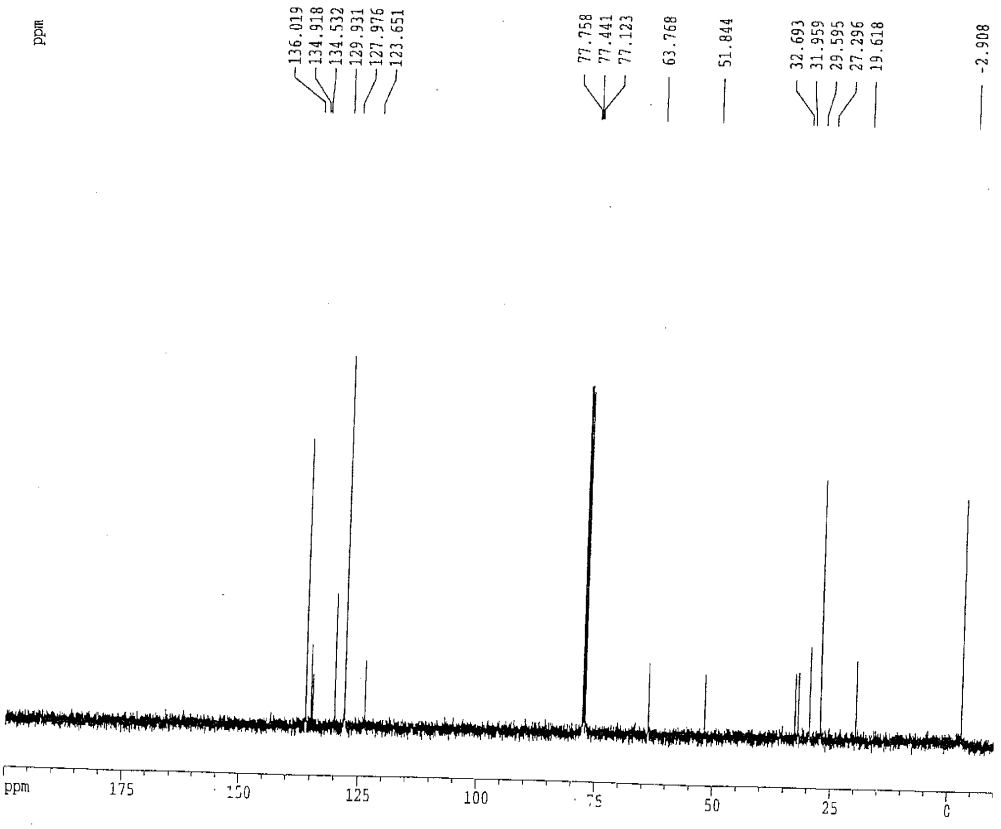
Current Data Parameters
NAME wj4-202
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040412
Time 13.05
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 101.6
DW 104.400 usec
DE 5.50 usec
TE 1809.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 8.000 ppm
F1 3201.04 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.42500 ppm/cm
HZCM 170.05525 Hz/cm



Current Data Parameters
NAME wj4-202
EXPNO 2
PROCNO 1

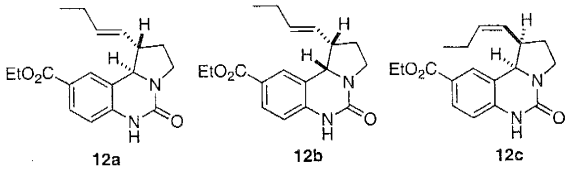
F2 - Acquisition Parameters
Date_ 20040412
Time 11.08
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 140
DS 4
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 32768
DW 21.600 usec
DE 5.50 usec
TE 1809.2 K
D1 0.15000001 sec
d11 0.03000000 sec
DELTA 0.05000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

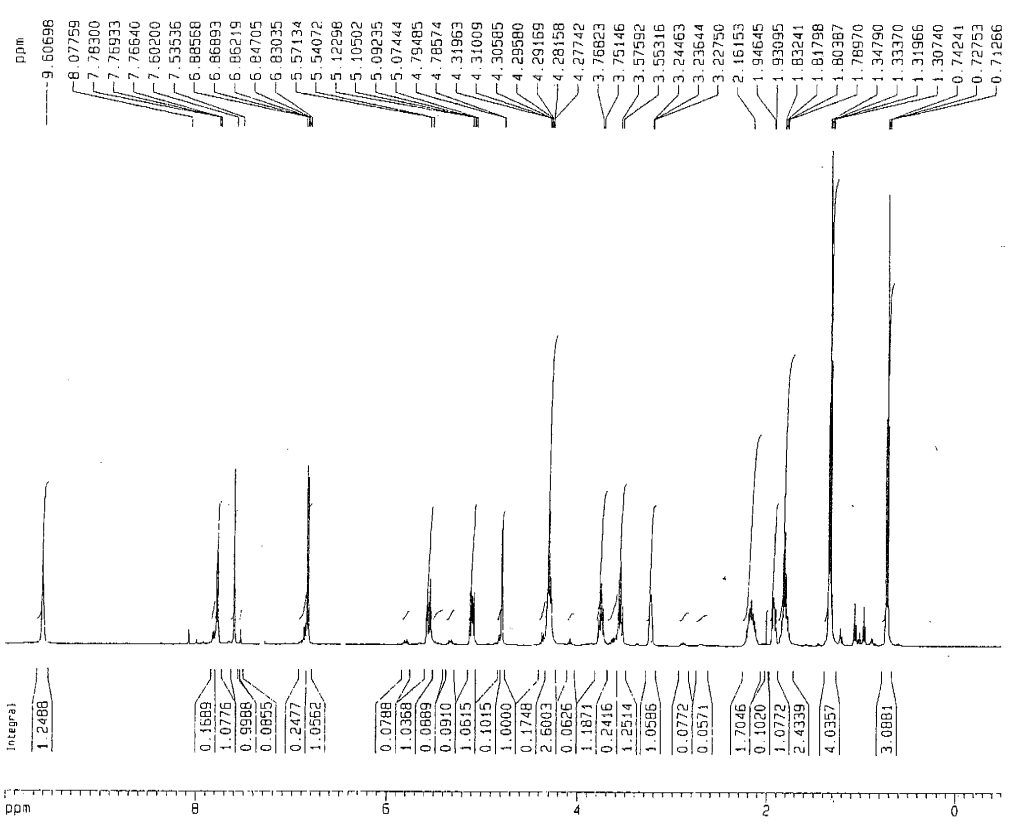
======== CHANNEL f1 ========
NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.6232933 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 65536
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 10.50000 ppm/cm
HZCM 1056.43372 Hz/cm





Current Data Parameters
NAME wj3-197
EXPNO 1
PROCNO 1

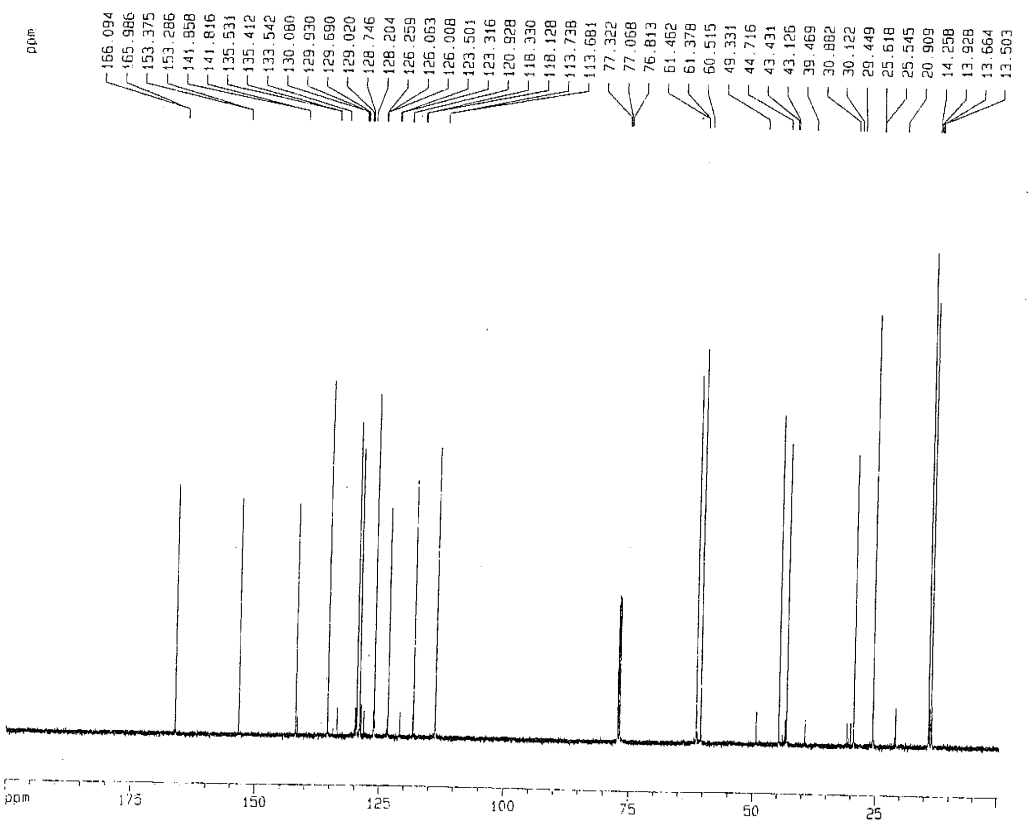
F2 - Acquisition Parameters
Date_ 20030115
Time 13.24
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7264309 sec
RG 32
DW 83.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.03000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.70 usec
PL1 -4.00 dB
SFO1 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1300000 MHz
WDW QSINE
SSB 0
LB 0.30 Hz
GB 0

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 10.000 ppm
F1 5001.30 Hz
F2P -0.500 ppm
F2 -250.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 262.56824 Hz/cm



Current Data Parameters
NAME wj3-197
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030115
Time 13.30
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 262
DS 4
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420883 sec
RG 14596.5
DW 15.900 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

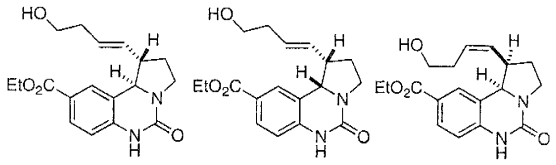
======== CHANNEL f1 ========
NUC1 13C
P1 6.50 usec
PL1 5.00 dB
SFO1 125.7719472 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 -4.00 dB
PL12 19.00 dB
PL13 36.00 dB
SFO2 500.1325000 MHz

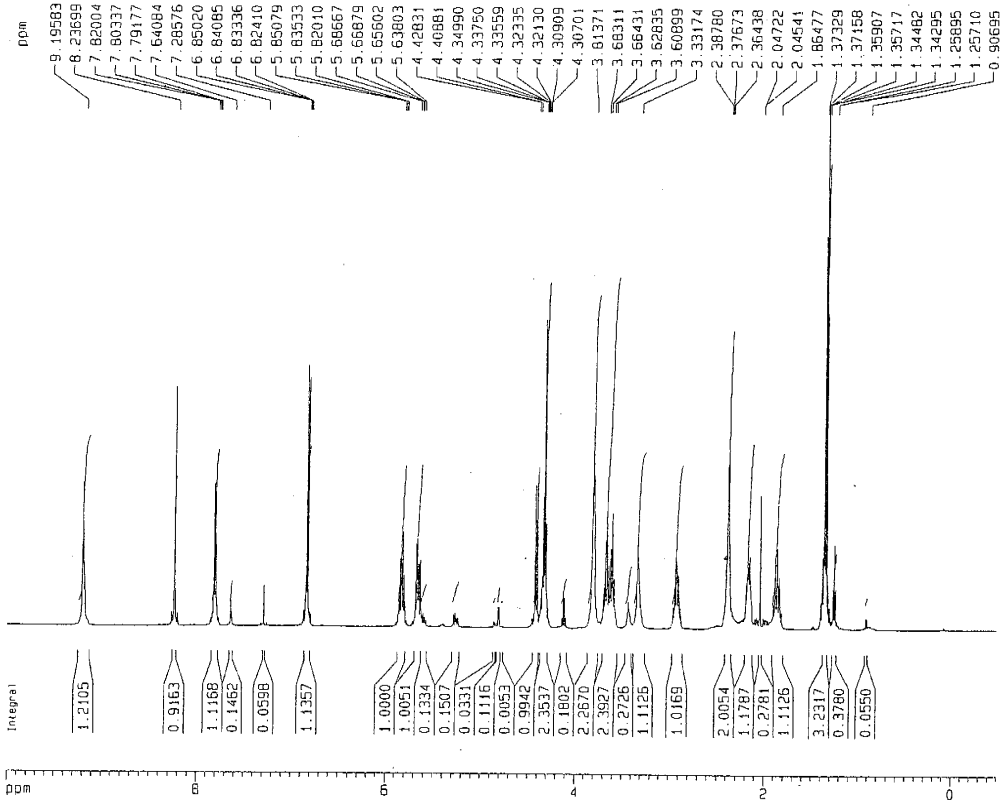
F2 - Processing parameters
SI 65536
SF 125.7578008 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

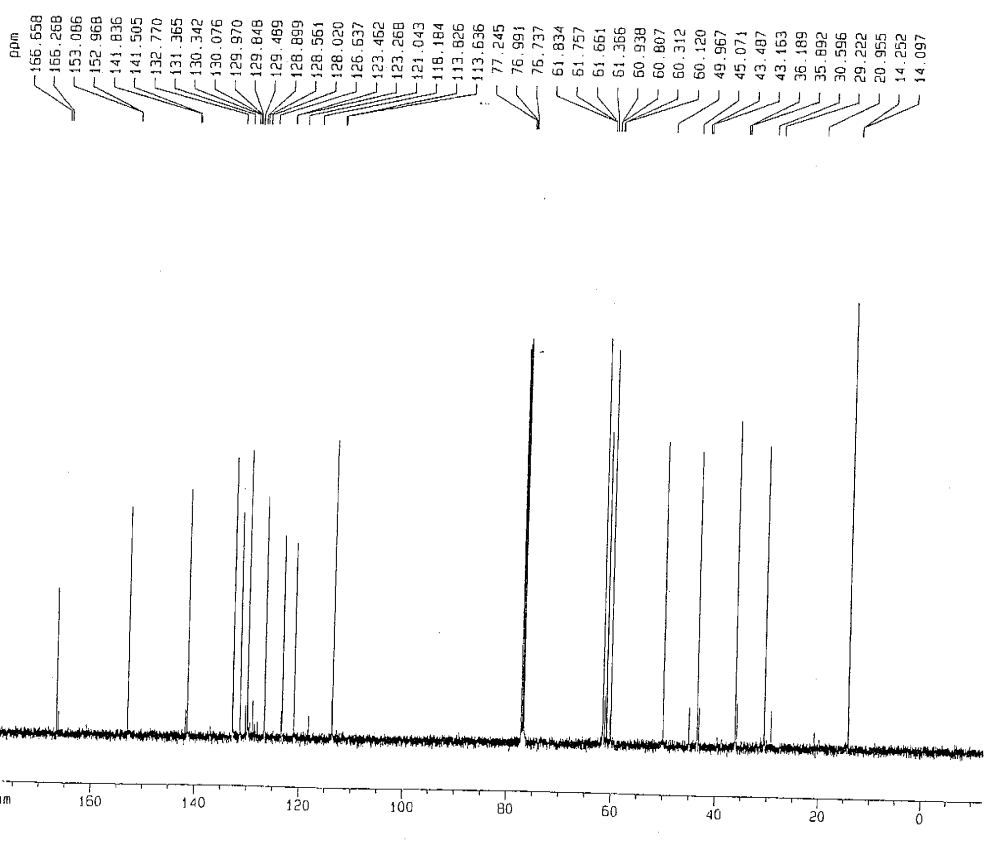
F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1300000 MHz
WDW no
SSB 0
LB 0.30 Hz
GB 0

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 200.000 ppm
F1 25151.56 Hz
F2P -9.500 ppm
F2 -62.68 Hz
PPMCM 12.02500 ppm/cm
HZCM 1260.78192 Hz/cm



13a 13b 13c





EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20010821
Time 10.08
INSTRUM spect
PROBHD 5 mm BBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 97
DW 83.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.03000000 sec

========== CHANNEL f1 ==========
NUC1 1H
P1 7.70 usec
PL1 -4.00 dB
SFO1 500.1320005 MHz

F1 - Acquisition parameters
NDD 2
TD 256
SFO1 500.1325 MHz
FIDRES 23.475060 Hz
SW 12.016 ppm

F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1300000 MHz
WDW no
SSB 0
LB 0.30 Hz
GB 0

1D NMR plot parameters
CX 20.00 cm
F1P 10.006 ppm
F1 5005.33 Hz
F2P -0.900 ppm
F2 -250.07 Hz
PPMCM 0.52540 ppm/cm
HZCM 262.76956 Hz/cm

Current Data Parameters
NAME w12-68
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20010821
Time 10.14
INSTRUM spect
PROBHD 5 mm BBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 387
DS 4
SWH 31446.541 Hz
FIDRES 0.479838 Hz
AQ 1.0420724 sec
RG 2580.3
DW 15.900 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

========== CHANNEL f1 ==========
NUC1 13C
P1 6.50 usec
PL1 5.00 dB
SFO1 125.7719472 MHz

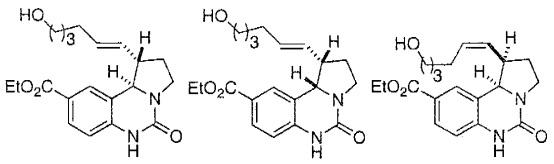
========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 17.50 dB
PL12 19.00 dB
PL13 20.00 dB
SFO2 500.1325000 MHz

F1 - Acquisition parameters
NDD 2
TD 256
SFO1 500.1325 MHz
FIDRES 23.475060 Hz
SW 12.016 ppm

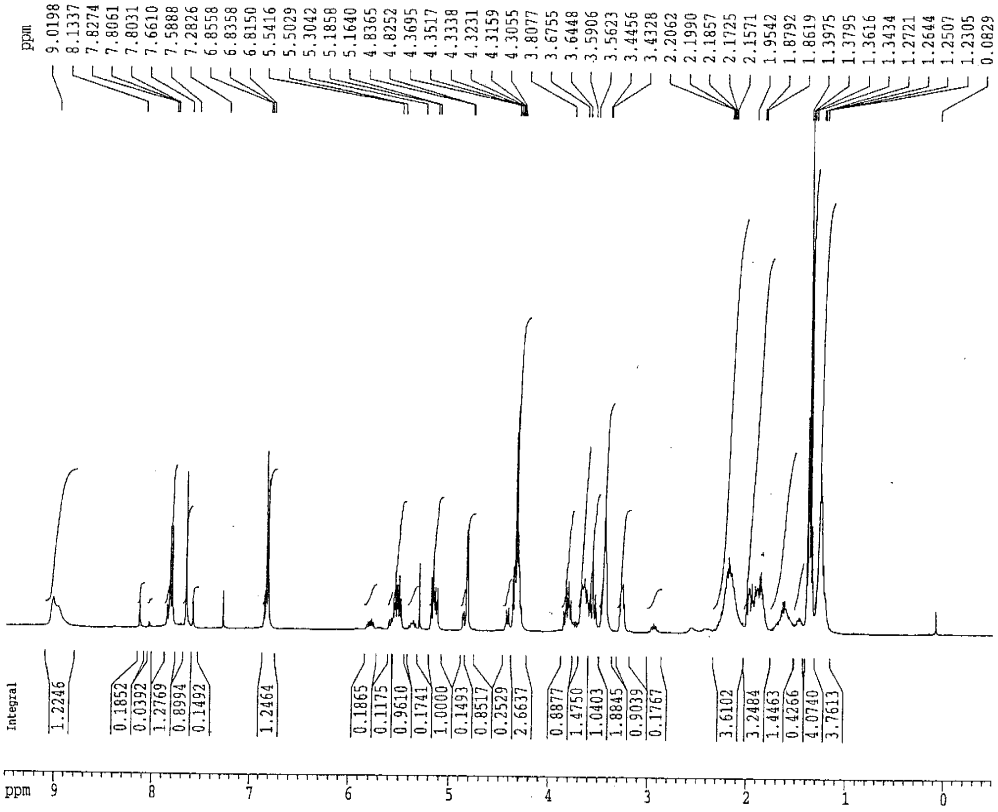
F2 - Processing parameters
SI 65536
SF 125.7578008 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1300000 MHz
WDW no
SSB 0
LB 0.30 Hz
GB 0

1D NMR plot parameters
CX 20.00 cm
F1P 180.000 ppm
F1 22536.40 Hz
F2P -12.539 ppm
F2 1576.91 Hz
PPMCM 9.62696 ppm/cm
HZCM 1210.66522 Hz/cm



15a 15b 15c



NAME	wj4-56
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20030729
Time	14.39
INSTRUM	drx400
PROBHD	5 mm Multinucl
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	4789.272 Hz
FIDRES	0.146157 Hz
AQ	3.4210291 sec
RG	64
DW	104.400 usec
DE	4.50 usec
TE	300.0 K
D1	1.00000000 sec

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

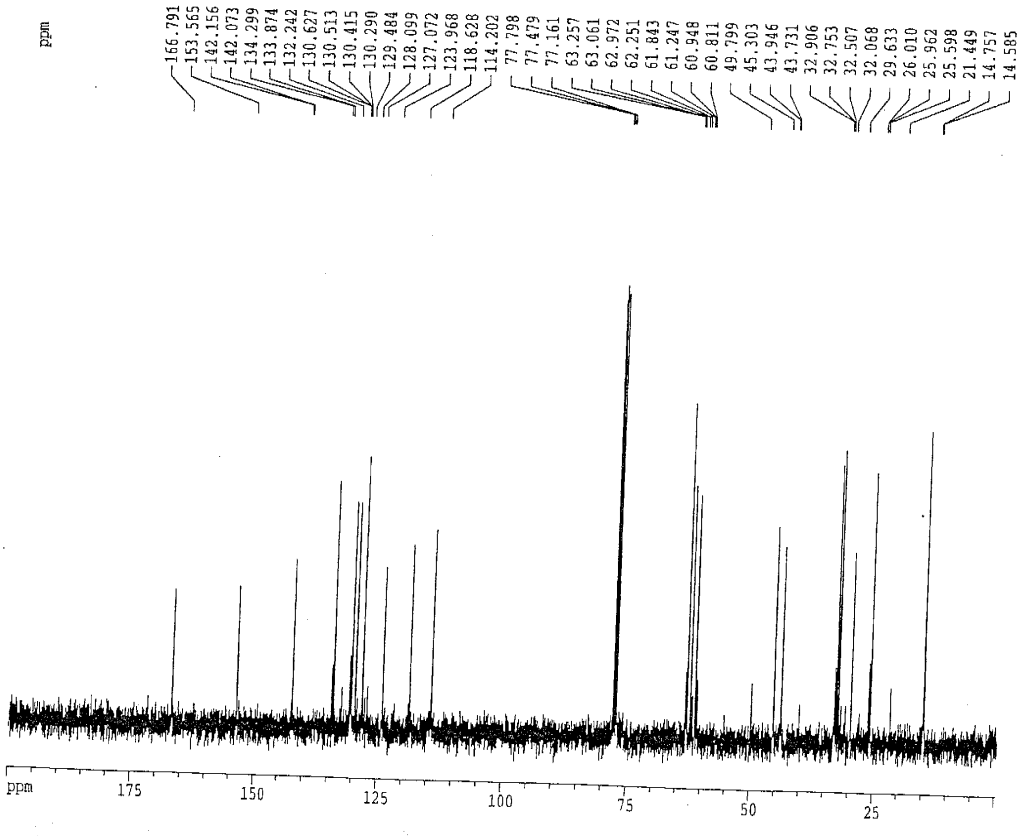
NUC1	1H
P1	7.70 usec
PL1	-6.00 dB
SFO1	400.1320007 MHz

F2 - Processing parameters

SI	16384
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
CY	0.00 cm
F1P	9.500 ppm
F1	3801.24 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.50000 ppm/cm
HZCM	200.06500 Hz/cm



Current Data Parameters

NAME	wj4-56,6-24
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20030729
Time	16.27
INSTRUM	drx400
PROBHD	5 mm Multinucl
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	182
DS	2
SWH	23148.148 Hz
FIDRES	0.353213 Hz
AQ	1.4156276 sec
RG	32768
DW	21.600 usec
DE	4.50 usec
TE	300.0 K
D1	0.05000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

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

NUC1	13C
P1	12.30 usec
PL1	2.00 dB
SFO1	100.6232933 MHz

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

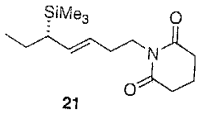
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	0.00 dB
PL12	18.00 dB
PL13	18.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

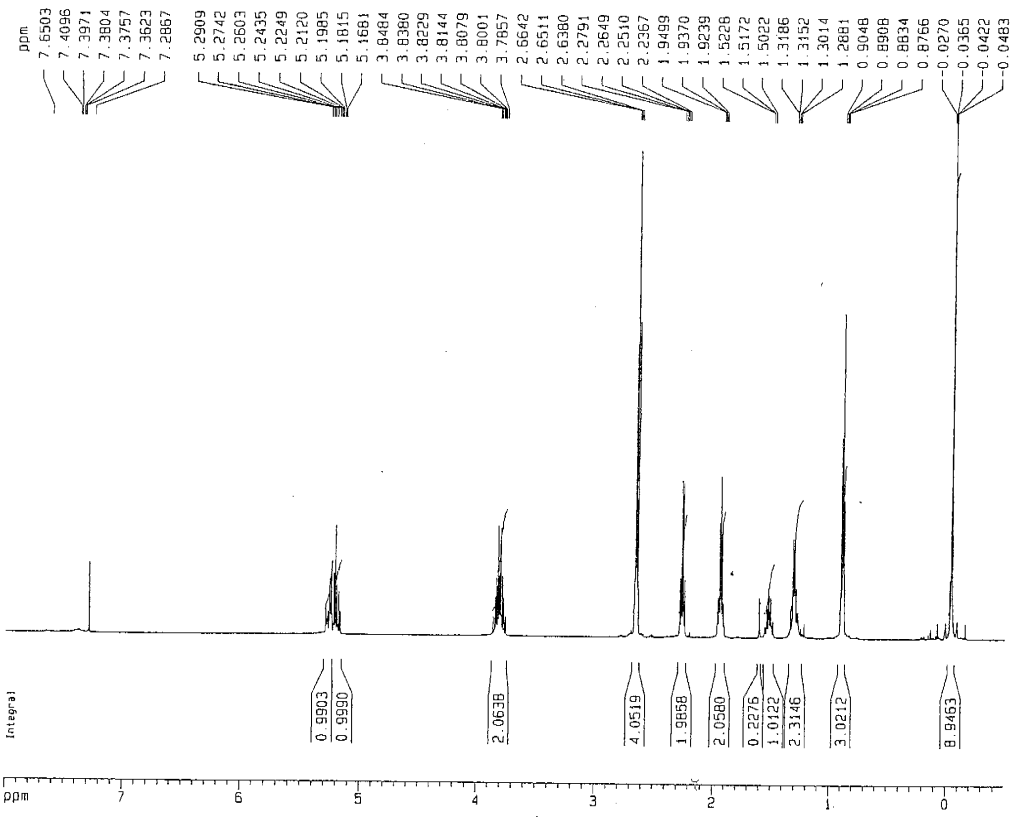
SI	32768
SF	100.6127290 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	0.00 cm
F1P	200.000 ppm
F1	20122.55 Hz
F2P	-0.500 ppm
F2	-50.31 Hz
PPMCM	10.02500 ppm/cm
HZCM	1008.64264 Hz/cm



21




```
Current Data Parameters
NAME                  wj3-185
EXPNO                       1
PROCNO                      1

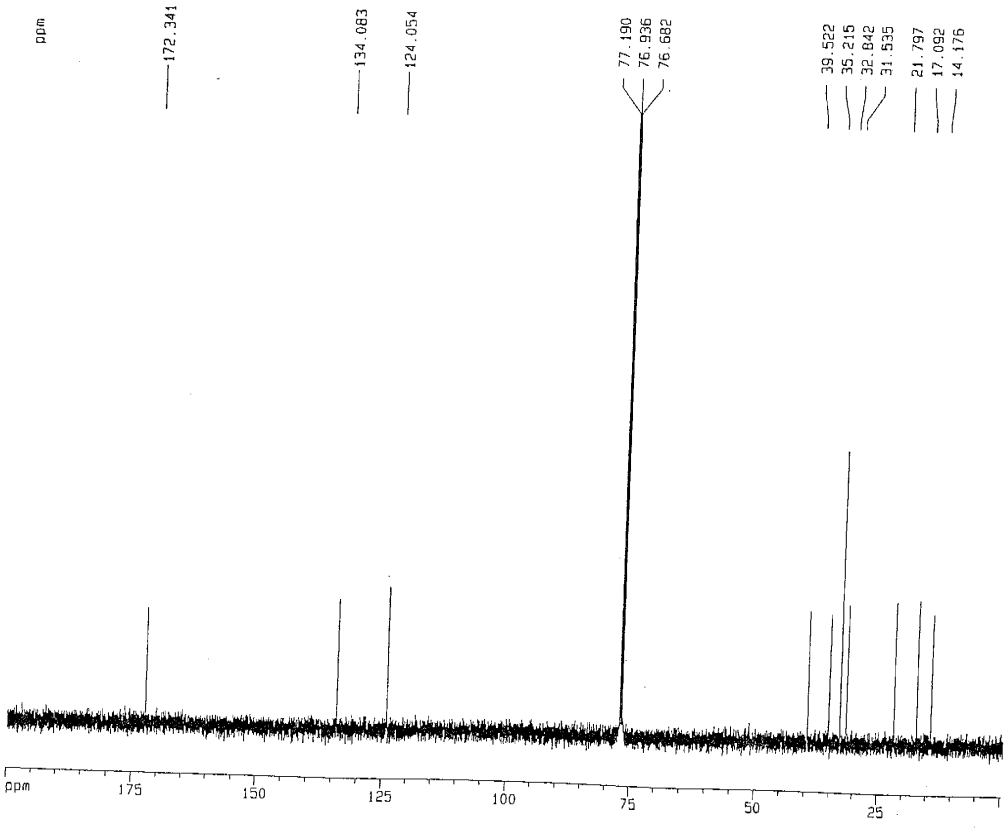
F2 - Acquisition Parameters
Date_              20030120
Time                   12.43
INSTRUM                spect
PROBHD     5 mm BBO BB-1H
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                6009.615 Hz
FIDRES            0.183399 Hz
AQ                2.7264309 sec
RG                      256
DW                 83.200 usec
DE                  6.00 usec
TE                  300.0 K
D1                0.03000000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     7.70 usec
PL1                   -4.00 dB
SFO1           500.1320005 MHz

F2 - Processing parameters
SI                    32768
SF                500.1300000 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00

F1 - Processing parameters
SI                      1024
MC2                       QF
SF                500.1300000 MHz
WDW                     QSINE
SSB                        0
LB                      0.30 Hz
GB                         0

1D NMR plot parameters
CX                     20.00 cm
CY                      0.00 cm
F1P                  8.000 ppm
F1                 4001.04 Hz
F2P                 -0.500 ppm
F2                -250.07 Hz
PPMCM               0.42500 ppm/cm
HZCM              212.55525 Hz/cm
```




```
Current Data Parameters
NAME                  wj3-185
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20030120
Time                   12.48
INSTRUM                spect
PROBHD     5 mm BBO BB-1H
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      294
DS                        4
SWH               31446.541 Hz
FIDRES            0.479836 Hz
AQ                1.0420883 sec
RG                     16384
DW                 15.900 usec
DE                  6.00 usec
TE                  300.0 K
D1                0.10000000 sec
D11               0.03000000 sec
D12               0.00002000 sec

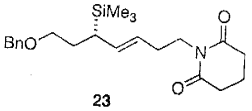
======== CHANNEL f1 ========
NUC1                      13C
P1                     8.50 usec
PL1                    5.00 dB
SFO1           125.7719472 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  95.00 usec
PL2                    -4.00 dB
PL12                   19.00 dB
PL13                   30.00 dB
SFO2           500.1325000 MHz

F2 - Processing parameters
SI                    65536
SF               125.7578008 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40

F1 - Processing parameters
SI                      1024
MC2                       QF
SF                500.1300000 MHz
WDW                       no
SSB                        0
LB                      0.30 Hz
GB                         0

1D NMR plot parameters
CX                     20.00 cm
CY                      0.00 cm
F1P                  200.000 ppm
F1                 25151.56 Hz
F2P                 -0.500 ppm
F2                -62.88 Hz
PPMCM              10.02500 ppm/cm
HZCM              1260.72192 Hz/cm
```



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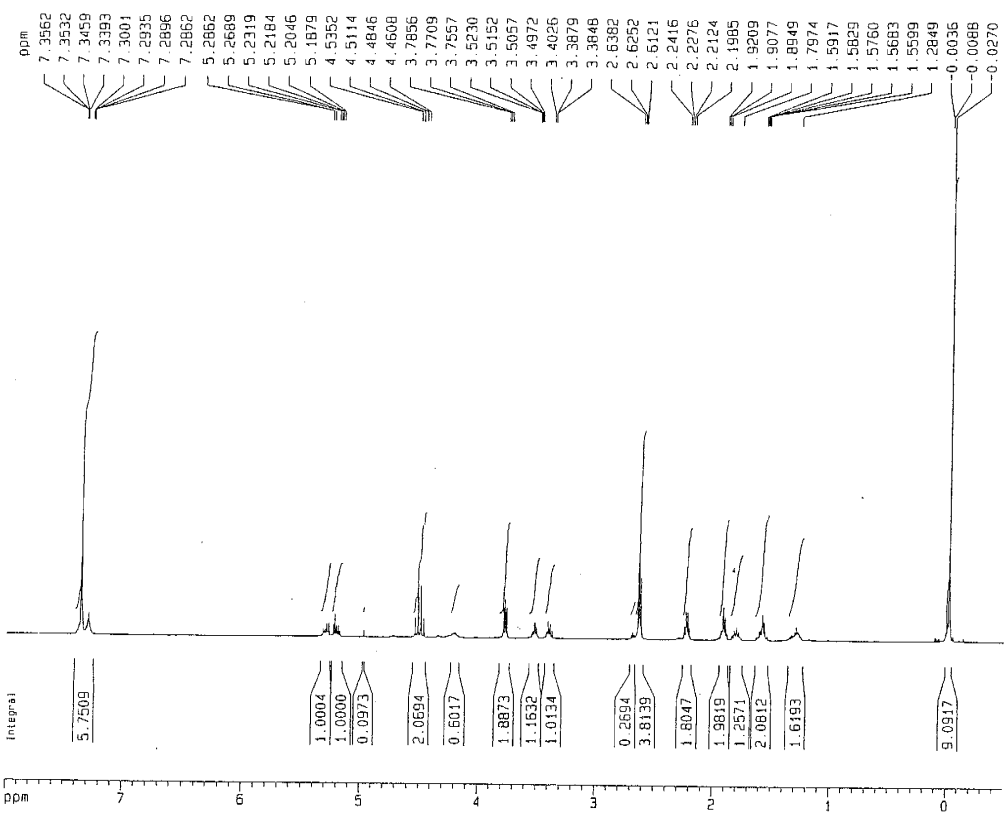



```
Current Data Parameters
NAME                  wj2-74
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20010902
Time                  14.47
INSTRUM                spect
PROBHD     5 mm BBO BB-
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                6009.615 Hz
FIDRES            0.183398 Hz
AQ                2.7263477 sec
RG                       57
DW                  83.200 usec
DE                   6.00 usec
TE                  300.0 K
D1                0.03000000 sec

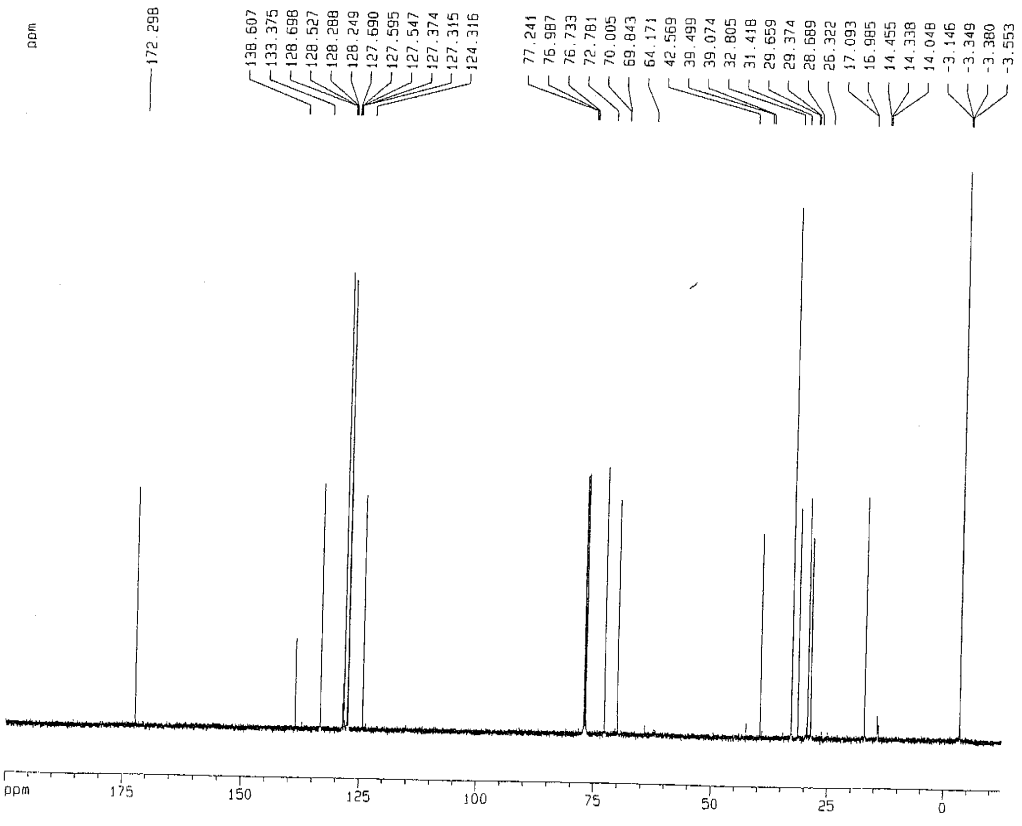
======== CHANNEL f1 ========
NUC1                      1H
P1                    7.70 usec
PL1                  -4.00 dB
SFO1           500.1320005 MHz

F1 - Acquisition parameters
ND0                       2
TD                      256
SFO1           500.1325 MHz
FIDRES           23.475060 Hz
SW                 12.016 ppm

F2 - Processing parameters
SI                    32768
SF           500.1300000 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00

F1 - Processing parameters
SI                     1024
MC2                      QF
SF           500.1300000 MHz
WDW                      no
SSB                       0
LB                     0.30 Hz
GB                        0

1D NMR plot parameters
CX                    20.00 cm
F1P                 8.000 ppm
F1                 4001.04 Hz
F2P                -0.500 ppm
F2                 -250.07 Hz
PPMCM              0.42500 ppm/cm
HZCM              212.55525 Hz/cm
```




```
Current Data Parameters
NAME                  wj2-74
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_              20010902
Time                  14.58
INSTRUM                spect
PROBHD     5 mm BBO BB-
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      486
DS                        4
SWH               31446.541 Hz
FIDRES            0.479836 Hz
AQ                1.0420724 sec
RG                     8192
DW                  15.900 usec
DE                   6.00 usec
TE                  300.0 K
D1                0.10000000 sec
D11               0.03000000 sec
D12               0.00002000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                    6.50 usec
PL1                   5.00 dB
SFO1          125.7719472 MHz

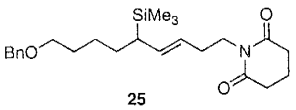
======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                 85.00 usec
PL2                   17.50 dB
PL12                  19.00 dB
PL13                  30.00 dB
SFO2          500.1325000 MHz

F1 - Acquisition parameters
ND0                       2
TD                      256
SFO1           500.1325 MHz
FIDRES           23.475080 Hz
SW                 12.016 ppm

F2 - Processing parameters
SI                    65536
SF           125.7578008 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40

F1 - Processing parameters
SI                     1024
MC2                      QF
SF           500.1300000 MHz
WDW                      no
SSB                       0
LB                     0.30 Hz
GB                        0

1D NMR plot parameters
CX                    20.00 cm
F1P                 200.000 ppm
F1                 25151.56 Hz
F2P                -12.539 ppm
F2                 -1578.91 Hz
PPMCM              10.62666 ppm/cm
HZCM              1338.42358 Hz/cm
```



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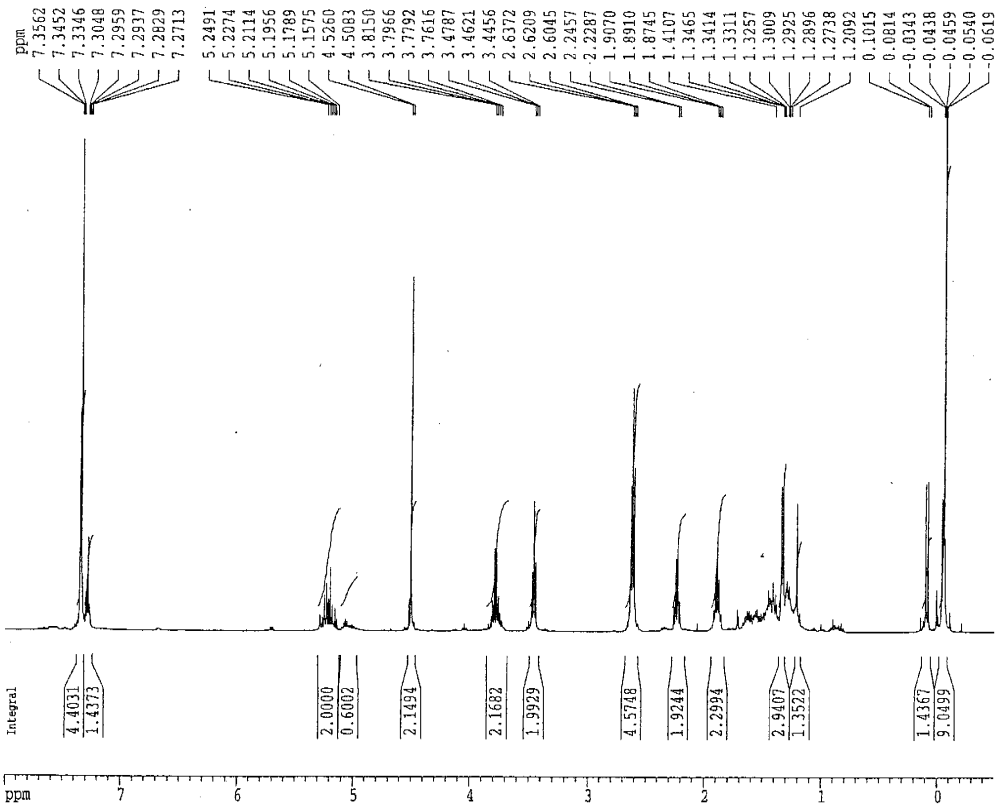


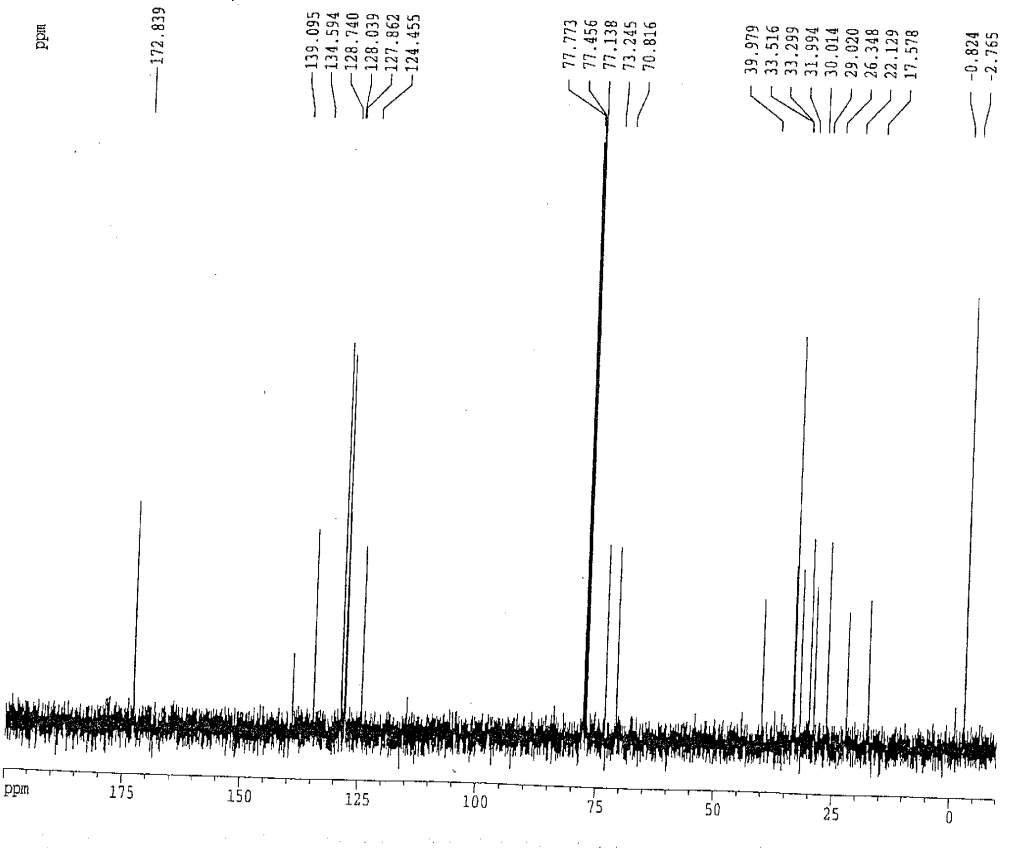
Current Data Parameters
NAME wj3-286
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030505
Time 13.56
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 64
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 8.000 ppm
F1 3201.04 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.42500 ppm/cm
HZCM 170.05525 Hz/cm



Current Data Parameters
NAME wj3-286
EXPNO 2
PROCNO 1

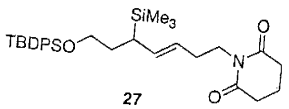
F2 - Acquisition Parameters
Date_ 20030505
Time 13.59
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 90
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 32768
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.6232933 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 10.50000 ppm/cm
HZCM 1056.43372 Hz/cm



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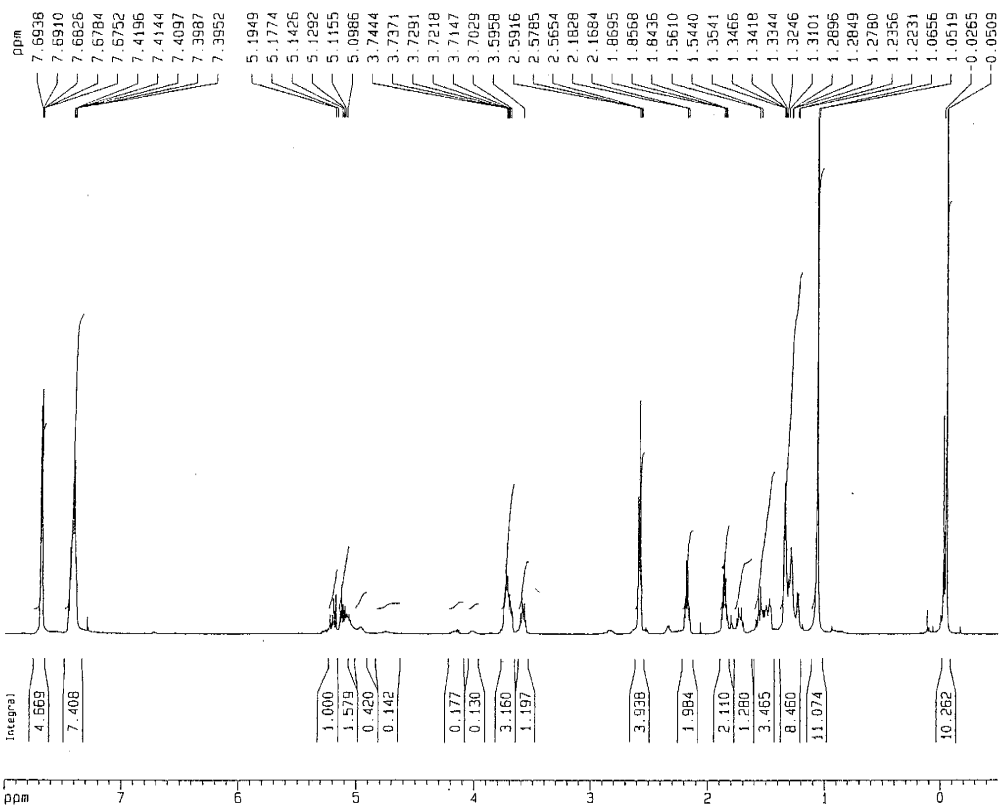



```
NAME                 wj4-200
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_             20040415
Time                 10.32
INSTRUM                spect
PROBHD  5 mm BBO BB-1H
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                6009.615 Hz
FIDRES             0.183399 Hz
AQ                 2.7264309 sec
RG                       32
DW                   83.200 usec
DE                    6.00 usec
TE                   296.2 K
D1                 0.03000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

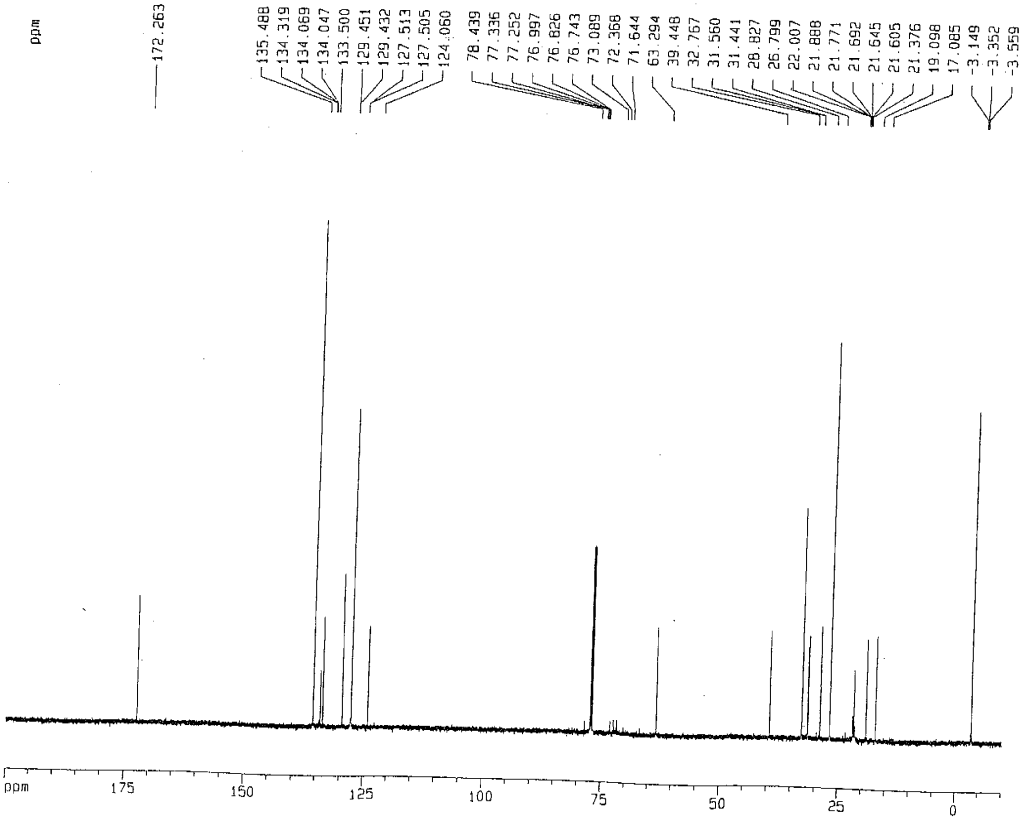
======== CHANNEL f1 ========
NUC1                     1H
P1                    7.70 usec
PL1                  -4.00 dB
SFO1          500.1320005 MHz

F1 - Acquisition parameters
NDO                       2
TD                      256
SFO1          500.1325 MHz
FIDRES            23.475060 Hz
SW                  12.016 ppm
FnMODE              undefined

F2 - Processing parameters
SI                    32768
SF            500.1300000 MHz
WDW                      EM
SSB                       0
LB                   0.30 Hz
GB                        0
PC                     1.00

F1 - Processing parameters
SI                     1024
MC2                      QF
SF            500.1300000 MHz
WDW                    QSINE
SSB                       0
LB                   0.30 Hz
GB                        0

1D NMR plot parameters
CX                   20.00 cm
CY                    0.00 cm
F1P                   8.000 ppm
F1                 4001.04 Hz
F2P                  -0.500 ppm
F2                 -250.07 Hz
PPMCM              0.42500 ppm/cm
HZCM             212.95525 Hz/cm
```




```
Current Data Parameters
NAME                 wj4-200
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_             20040415
Time                 10.36
INSTRUM                spect
PROBHD  5 mm BBO BB-1H
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      198
DS                        4
SWH               31446.541 Hz
FIDRES             0.479836 Hz
AQ                 1.0420883 sec
RG                     2048
DW                   15.900 usec
DE                    6.00 usec
TE                   296.2 K
D1                 0.15000001 sec
D11                0.03000000 sec
DELTA              0.05000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                    6.30 usec
PL1                   3.00 dB
SFO1          125.7697300 MHz

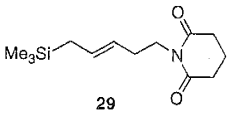
======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2                95.00 usec
PL2                   -4.00 dB
PL12                  19.00 dB
PL13                  30.00 dB
SFO2          500.1325000 MHz

F1 - Acquisition parameters
NDO                       2
TD                      256
SFO1          500.1325 MHz
FIDRES            23.475060 Hz
SW                  12.016 ppm
FnMODE              undefined

F2 - Processing parameters
SI                    65536
SF            125.7578008 MHz
WDW                      EM
SSB                       0
LB                   1.00 Hz
GB                        0
PC                     1.40

F1 - Processing parameters
SI                     1024
MC2                      QF
SF            500.1300000 MHz
WDW                      no
SSB                       0
LB                   0.30 Hz
GB                        0

1D NMR plot parameters
CX                   20.00 cm
CY                    0.00 cm
F1P                 200.000 ppm
F1               25151.56 Hz
F2P                 -15.000 ppm
F2               -1257.58 Hz
PPMCM              10.50000 ppm/cm
HZCM             1320.45691 Hz/cm
```



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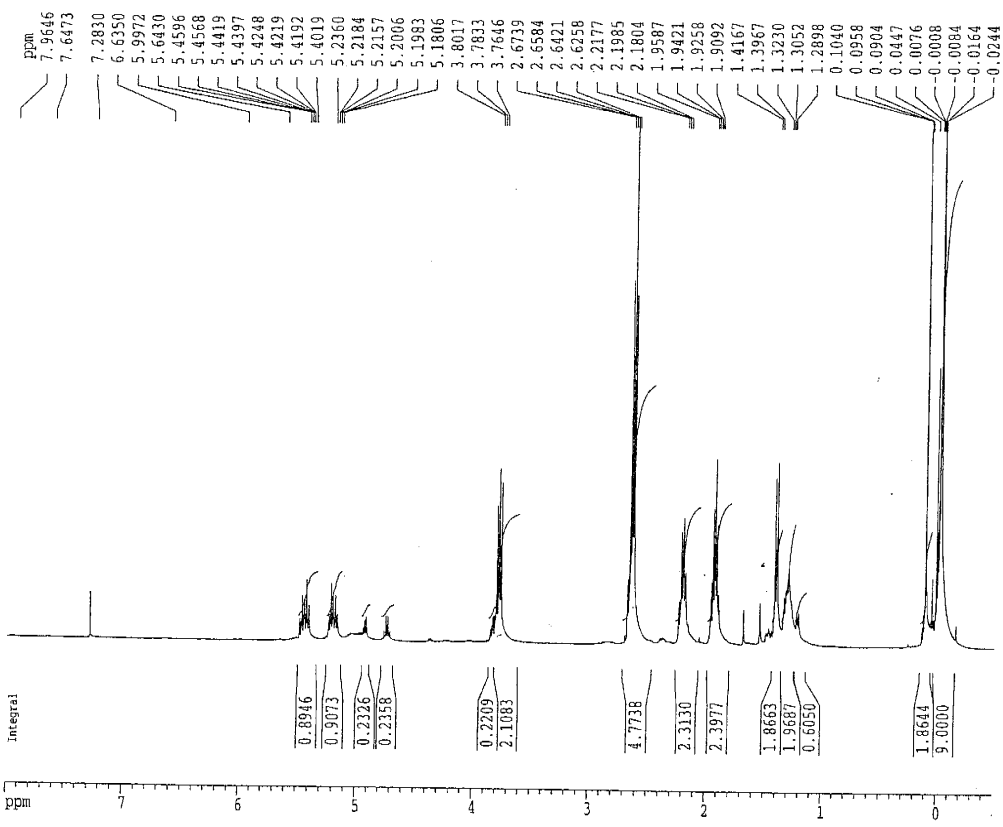


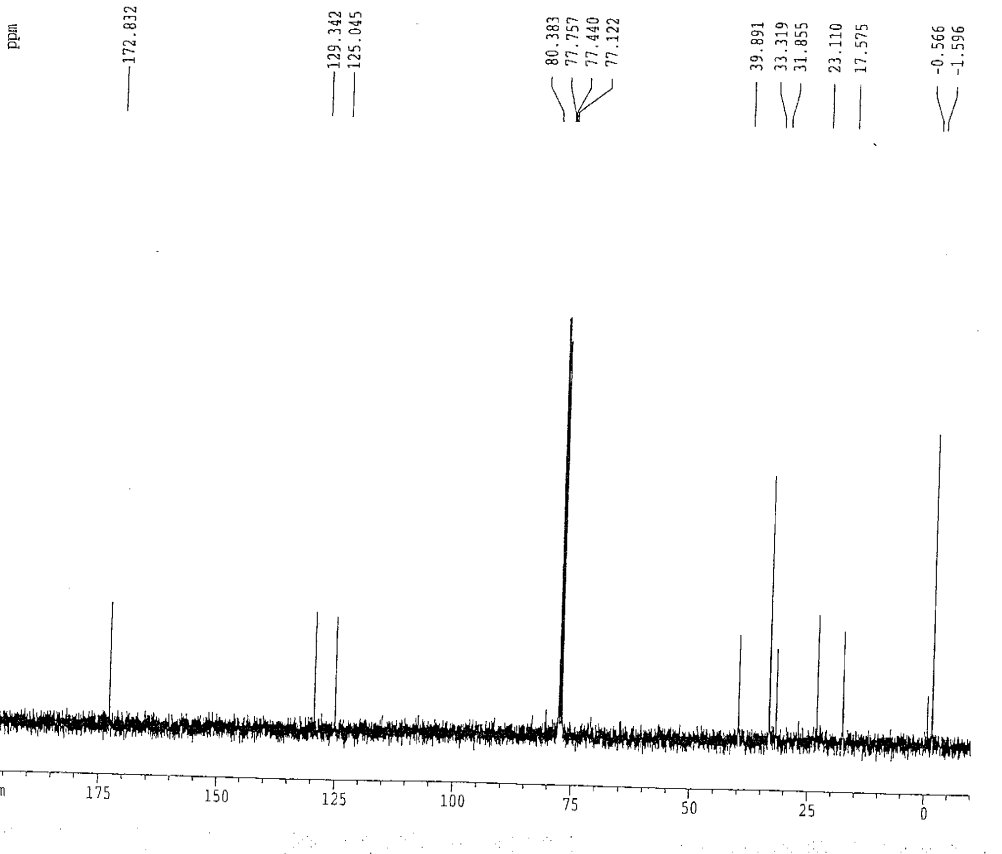
Current Data Parameters
NAME wj3-200
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030122
Time 12.33
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 101.6
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 8.000 ppm
F1 3201.04 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.42500 ppm/cm
HZCM 170.05525 Hz/cm



Current Data Parameters
NAME wj3-200
EXPNO 2
PROCNO 1

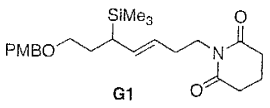
F2 - Acquisition Parameters
Date_ 20030122
Time 12.39
INSTRUM drx400
PROBHD 5 mm Multinuci
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 263
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4155276 sec
RG 574.7
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.6232933 MHz

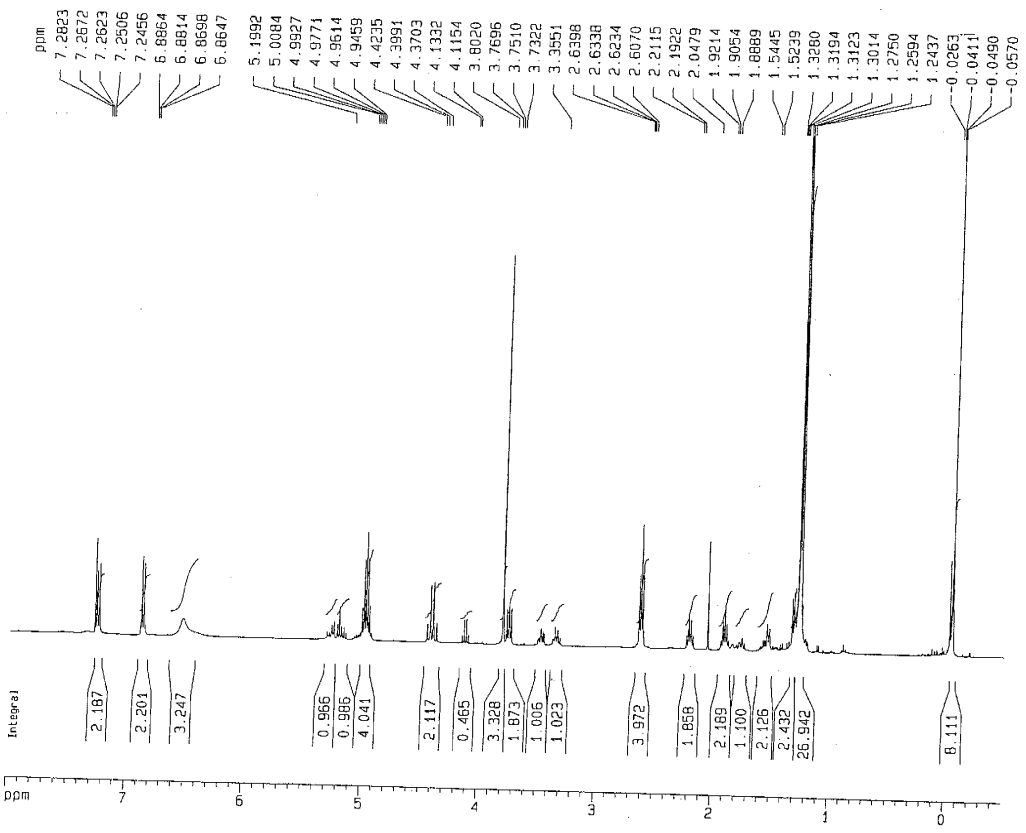
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -10.036 ppm
F2 -1009.74 Hz
PPMCM 10.50179 ppm/cm
HZCM 1056.61401 Hz/cm



G1



Current Data Parameters

NAME	wj5-~~147~~ 16-21
EXPNO	~~149~~ 1
PROCNO	1

F2 - Acquisition Parameters

Date_	20050323
Time	17.18
INSTRUM	spect
PROBHD	5 mm QNP 1H/15
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	4789.272 Hz
FIDRES	0.146157 Hz
AQ	3.4210291 sec
RG	90.5
DW	104.400 usec
DE	6.00 usec
TE	297.1 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

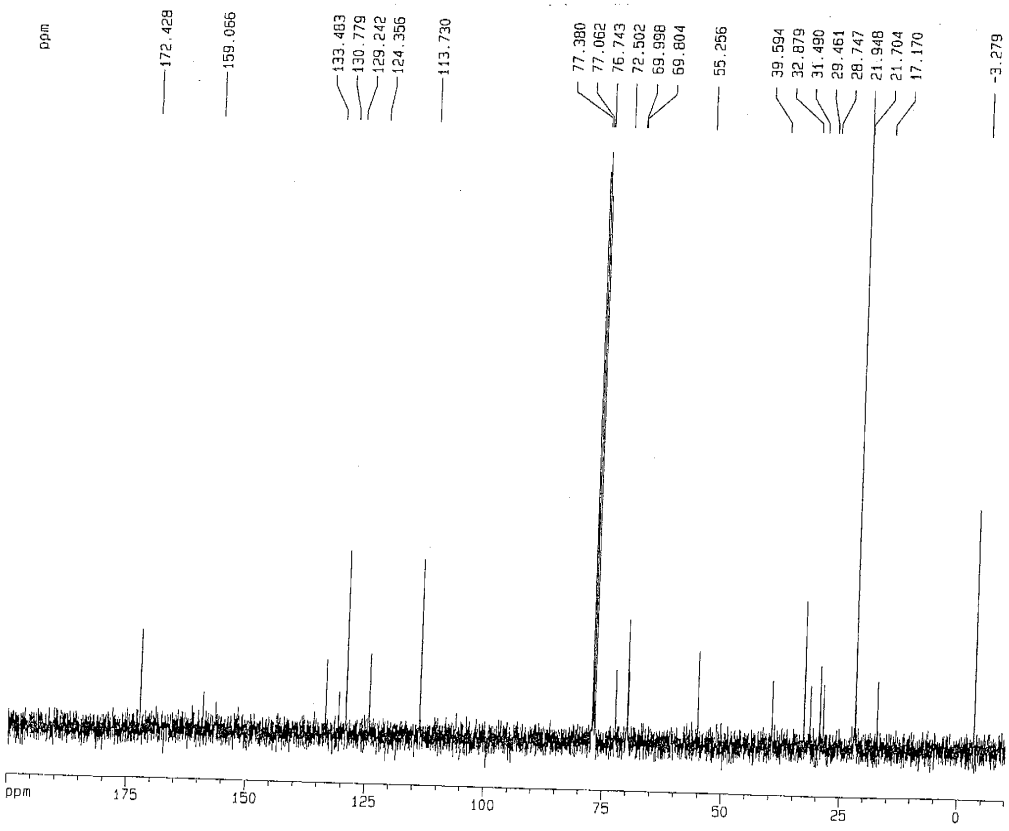
NUC1	1H
P1	7.05 usec
PL1	-3.00 dB
SFO1	400.1320007 MHz

F2 - Processing parameters

SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
CY	0.00 cm
F1P	8.000 ppm
F1	3201.04 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.42500 ppm/cm
HZCM	170.05525 Hz/cm



NAME	wj5-~~147~~ 16-21
EXPNO	~~149~~ 2
PROCNO	1

F2 - Acquisition Parameters

Date_	20050323
Time	17.24
INSTRUM	spect
PROBHD	5 mm QNP 1H/15
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	382
DS	4
SWH	23980.814 Hz
FIDRES	0.366018 Hz
AQ	1.3664756 sec
RG	2048
DW	20.850 usec
DE	6.00 usec
TE	297.2 K
D1	0.15000001 sec
d11	0.03000000 sec
DELTA	0.05000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	7.20 usec
PL1	-2.00 dB
SFO1	100.6228298 MHz

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

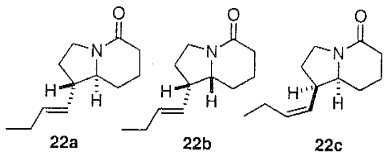
CPDPRG2	waltz16
NUC2	1H
PCPD2	98.40 usec
PL2	-3.00 dB
PL12	19.96 dB
PL13	120.00 dB
SFO2	400.1316005 MHz

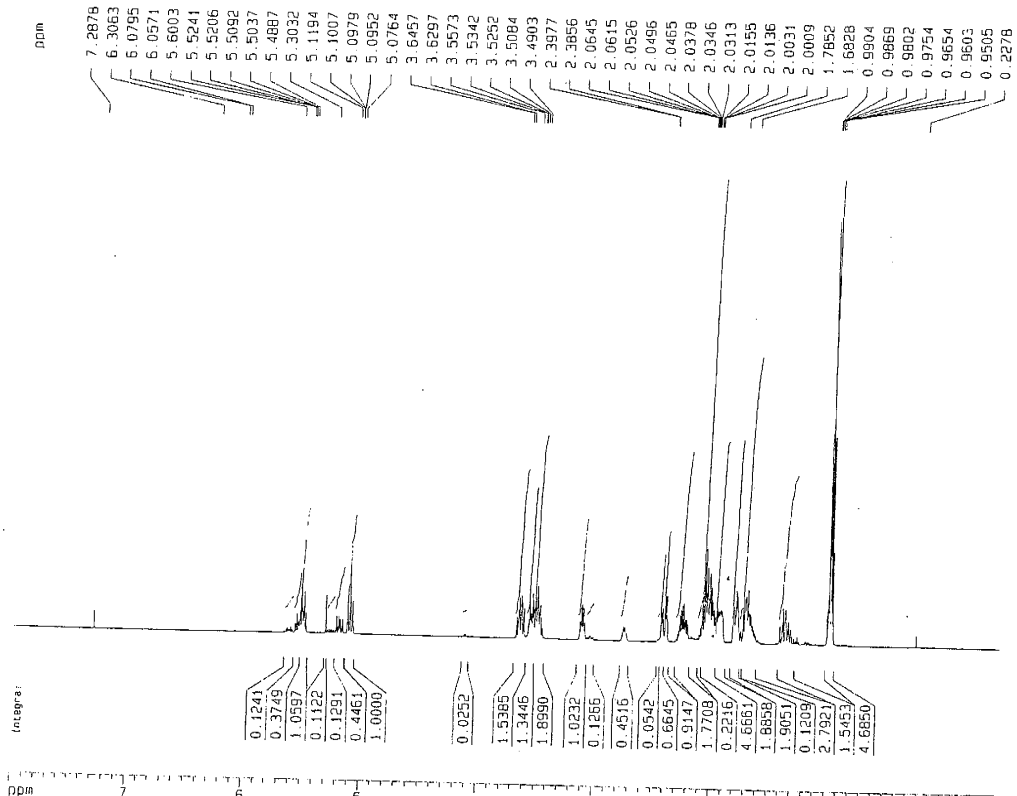
F2 - Processing parameters

SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	0.00 cm
F1P	200.000 ppm
F1	20122.55 Hz
F2P	-10.000 ppm
F2	-1006.13 Hz
PPMCM	10.50000 ppm/cm
HZCM	1056.43408 Hz/cm






```
NAME                wj4-35
EXPNO                    1
PROCNO                   1

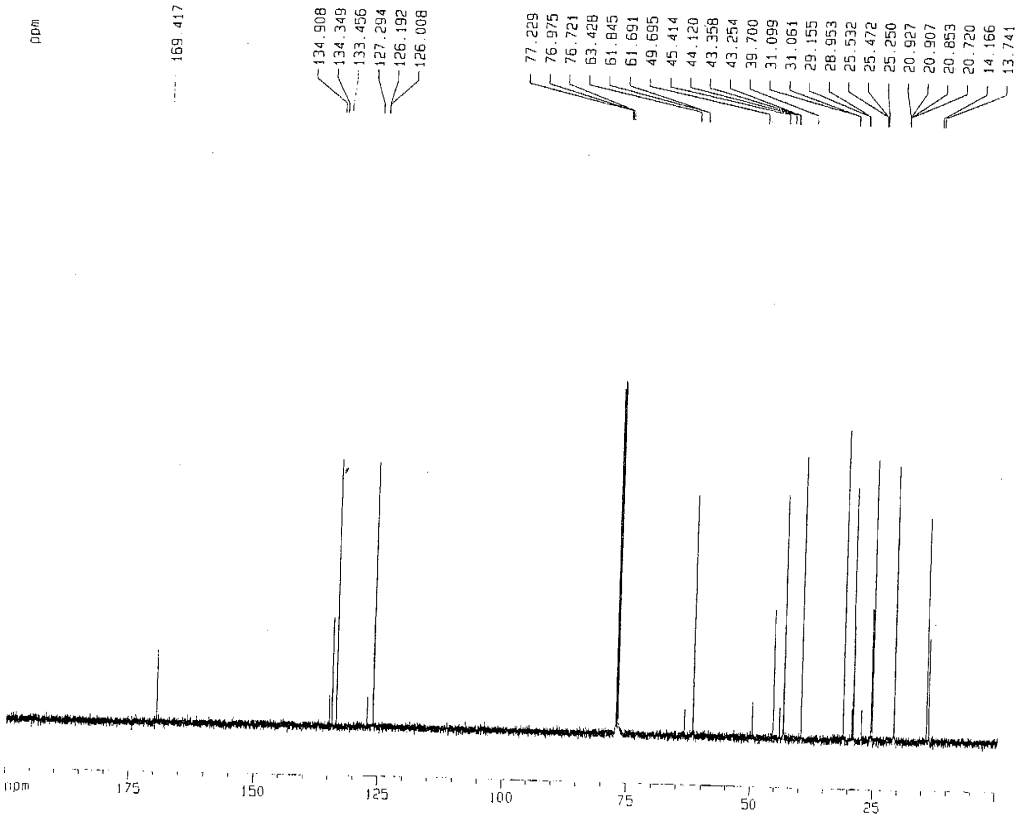
F2 - Acquisition Parameters
Date_             20030628
Time                 16.56
INSTRUM               spect
PROBHD  5 mm BBO BB-1H
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      16
DS                       2
SWH              6009.615 Hz
FIDRES           0.183399 Hz
AQ               2.7264309 sec
RG                    80.6
DW                83.200 usec
DE                 6.00 usec
TE                 300.0 K
D1              0.03000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 7.70 usec
PL1                -4.00 dB
SFO1        500.1320005 MHz

F2 - Processing parameters
SI                   32768
SF          500.1300000 MHz
WDW                     EM
SSB                      0
LB                0.30 Hz
GB                       0
PC                    1.00

F1 - Processing parameters
SI                    1024
MC2                     QF
SF          500.1300000 MHz
WDW                  QSINE
SSB                      0
LB                0.30 Hz
GB                       0

1D NMR plot parameters
CX                  20.00 cm
CY                   0.00 cm
F1P                  8.000 ppm
F1                 4001.04 Hz
F2P                 -0.500 ppm
F2                 -250.07 Hz
PPMCM             0.42500 ppm/cm
HZCM              212.55525 Hz/cm
```




```
Current Data Parameters
NAME                wj4-35
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20030628
Time                 17.02
INSTRUM               spect
PROBHD  5 mm BBO BB-1H
PULPROG           zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      341
DS                       4
SWH             31446.541 Hz
FIDRES           0.479836 Hz
AQ               1.0420883 sec
RG                    2048
DW                15.900 usec
DE                 6.00 usec
TE                 300.0 K
D1              0.10000000 sec
D11             0.03000000 sec
D12             0.00002000 sec

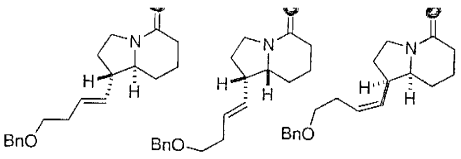
======== CHANNEL f1 ========
NUC1                   13C
P1                 8.50 usec
PL1                 5.00 dB
SFO1        125.7718472 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                   1H
PCPD2              95.00 usec
PL2                -4.00 dB
PL12               19.00 dB
PL13               30.00 dB
SFO2        500.1325000 MHz

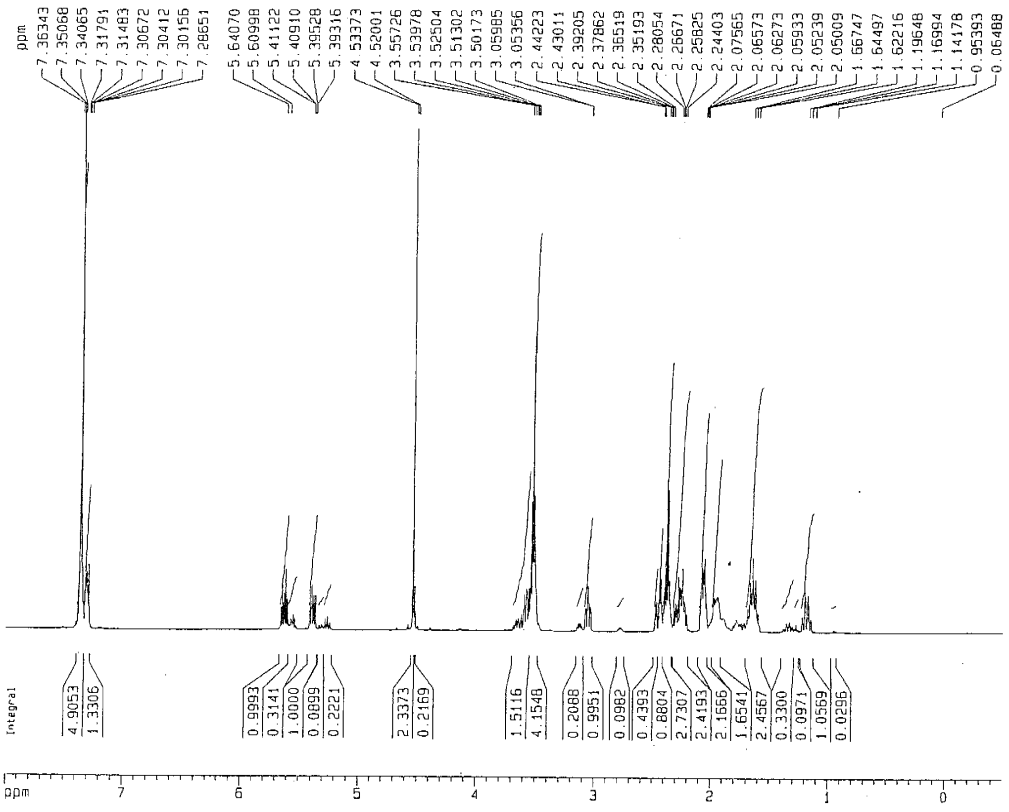
F2 - Processing parameters
SI                   65536
SF          125.7578000 MHz
WDW                     EM
SSB                      0
LB                1.00 Hz
GB                       0
PC                    1.40

F1 - Processing parameters
SI                    1024
MC2                     QF
SF          500.1300000 MHz
WDW                     EM
SSB                      0
LB                0.30 Hz
GB                       0

1D NMR plot parameters
CX                  20.00 cm
CY                   0.00 cm
F1P                200.000 ppm
F1               25131.38 Hz
F2P                 -0.500 ppm
F2                 -62.88 Hz
PPMCM             10.02500 ppm/cm
HZCM              1260.72192 Hz/cm
```



24a **24b** **24c**




```
Current Data Parameters
NAME                  wj3-126
EXPNO                       1
PROCNO                      1

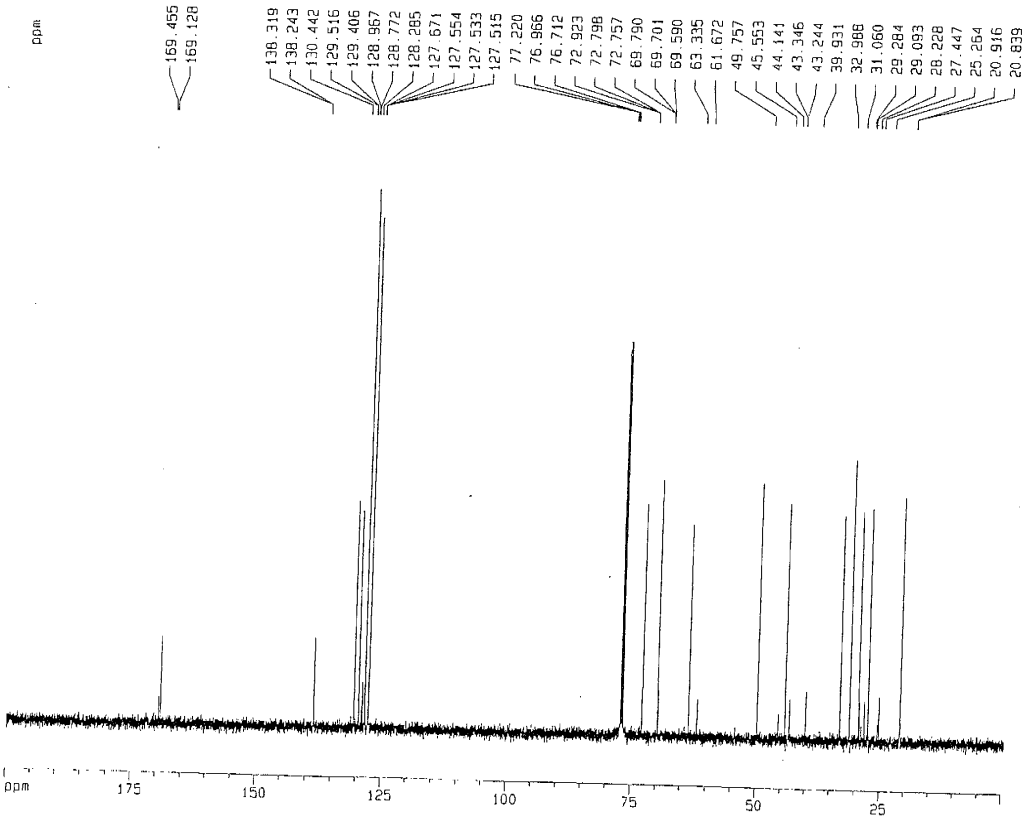
F2 - Acquisition Parameters
Date_              20020920
Time                   11.00
INSTRUM                spect
PROBHD  5 mm BBO BB-1H
PULPROG                zg30
TD                    32768
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                6009.615 Hz
FIDRES             0.183399 Hz
AQ                 2.7264309 sec
RG                       128
DW                  83.200 usec
DE                   6.00 usec
TE                   300.0 K
D1                0.03000000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     7.70 usec
PL1                   -4.00 dB
SFO1           500.1320005 MHz

F2 - Processing parameters
SI                    32768
SF             500.1300000 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00

F1 - Processing parameters
SI                      1024
MC2                       QF
SF             500.1300000 MHz
WDW                    QSINE
SSB                        0
LB                      0.30 Hz
GB                         0

1D NMR plot parameters
CX                     20.00 cm
CY                      0.00 cm
F1P                  8.000 ppm
F1                 4001.04 Hz
F2P                 -0.500 ppm
F2                 -250.07 Hz
PPMCM               0.42500 ppm/cm
HZCM              212.65525 Hz/cm
```




```
Current Data Parameters
NAME                  wj3-126
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20020920
Time                   11.08
INSTRUM                spect
PROBHD  5 mm BBO BB-1H
PULPROG             zgpg30
TD                    65536
SOLVENT                CDCl3
NS                       422
DS                         4
SWH               31446.541 Hz
FIDRES             0.479836 Hz
AQ                 1.0420883 sec
RG                    14596.5
DW                  15.900 usec
DE                   6.00 usec
TE                   300.0 K
D1                0.10000000 sec
D11               0.03000000 sec
D12               0.00002000 sec

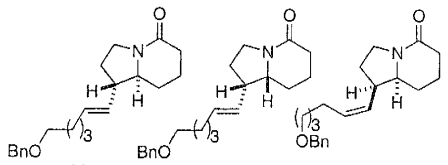
======== CHANNEL f1 ========
NUC1                      13C
P1                     6.50 usec
PL1                    5.00 dB
SFO1           125.7719472 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 95.00 usec
PL2                   -4.00 dB
PL12                  19.00 dB
PL13                  30.00 dB
SFO2           500.1325000 MHz

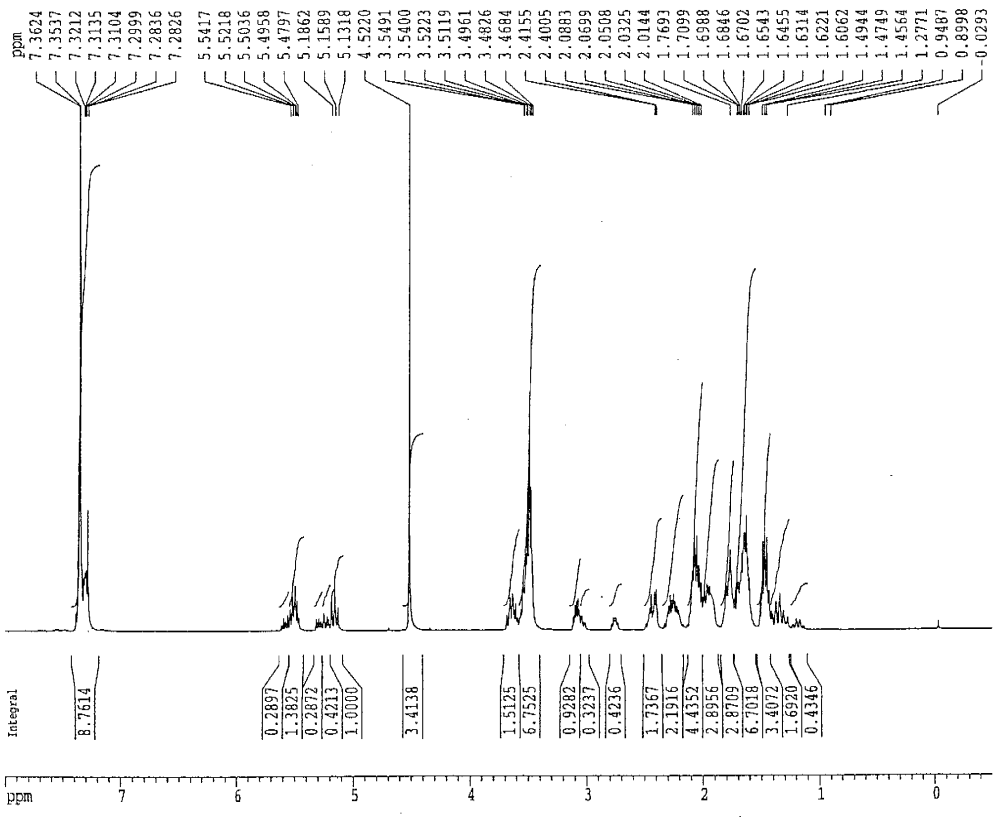
F2 - Processing parameters
SI                    65536
SF             125.7578000 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40

F1 - Processing parameters
SI                      1024
MC2                       QF
SF             500.1300000 MHz
WDW                       no
SSB                        0
LB                      0.30 Hz
GB                         0

1D NMR plot parameters
CX                     20.00 cm
CY                      0.00 cm
F1P                  200.000 ppm
F1                 25151.56 Hz
F2P                 -0.500 ppm
F2                  -62.88 Hz
PPMCM              10.02500 ppm/cm
HZCM              1260.72192 Hz/cm
```



26a **26b** **26c**



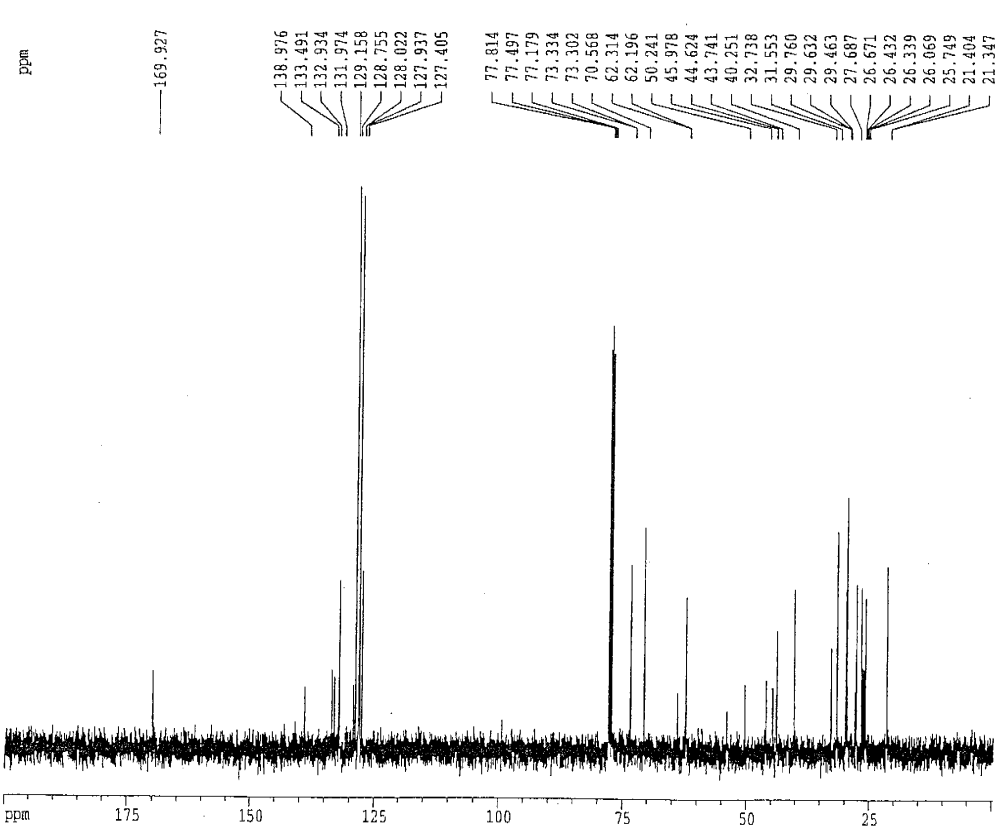
```
Current Data Parameters
NAME                  wj4-53
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20030729
Time                  14.29
INSTRUM              drx400
PROBHD   5 mm Multinucl
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                        16
DS                         2
SWH                4789.272 Hz
FIDRES            0.146157 Hz
AQ                3.4210291 sec
RG                    143.7
DW                  104.400 usec
DE                    4.50 usec
TE                   300.0 K
D1                1.00000000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                      7.70 usec
PL1                    -6.00 dB
SFO1             400.1320007 MHz

F2 - Processing parameters
SI                        16384
SF               400.1300000 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00

1D NMR plot parameters
CX                      20.00 cm
CY                       0.00 cm
F1P                      8.000 ppm
F1                    3201.04 Hz
F2P                     -0.500 ppm
F2                    -200.07 Hz
PPMCM                 0.42500 ppm/cm
HZCM                170.05525 Hz/cm
```



```
Current Data Parameters
NAME                  wj4-53
EXPNO                      2
PROCNO                     1

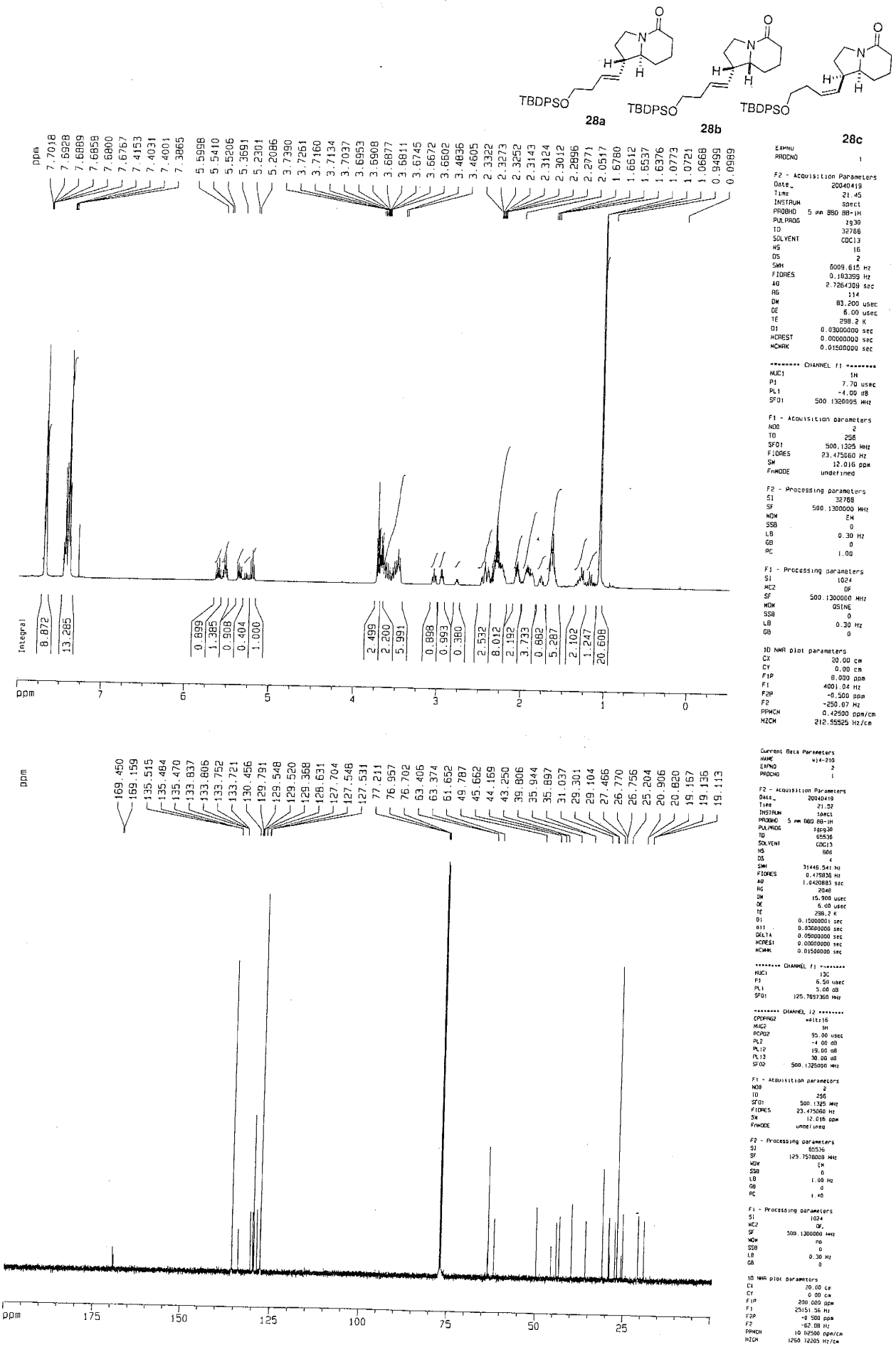
F2 - Acquisition Parameters
Date_              20030729
Time                  16.20
INSTRUM              drx400
PROBHD   5 mm Multinucl
PULPROG            zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       105
DS                         2
SWH               23148.148 Hz
FIDRES            0.353213 Hz
AQ                1.4156276 sec
RG                    32768
DW                  21.600 usec
DE                    4.50 usec
TE                   300.0 K
D1                0.05000000 sec
D11               0.03000000 sec
D12               0.00002000 sec

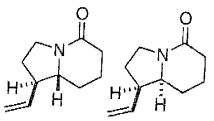
======== CHANNEL f1 ========
NUC1                      13C
P1                     12.30 usec
PL1                     2.00 dB
SFO1             100.6232933 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                 100.00 usec
PL2                     0.00 dB
PL12                   18.00 dB
PL13                   18.00 dB
SFO2             400.1316005 MHz

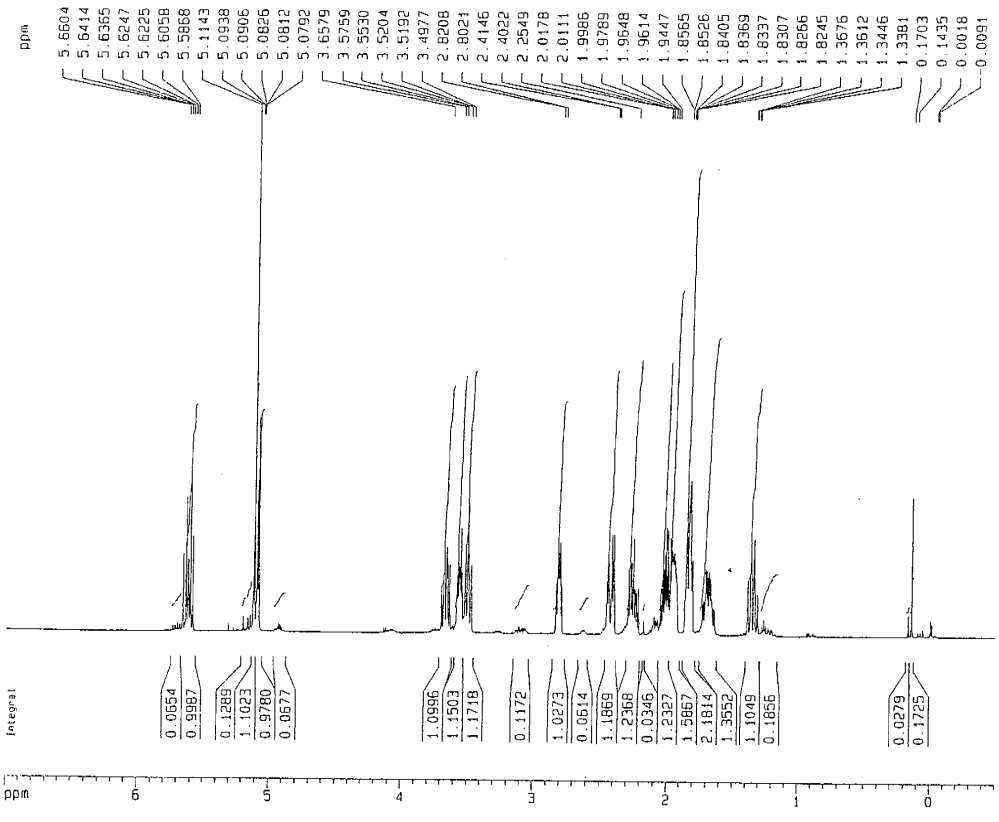
F2 - Processing parameters
SI                        32768
SF               100.6127290 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40

1D NMR plot parameters
CX                      20.00 cm
CY                       0.00 cm
F1P                    200.000 ppm
F1                   20122.55 Hz
F2P                     -0.500 ppm
F2                    -50.31 Hz
PPMCM                10.02500 ppm/cm
HZCM               1008.64264 Hz/cm
```


TBDPSO
28a
TBDPSO
28b
TBDPSO
28c
ppm
integral
Current Data Parameters
Acquisition Parameters
Processing parameters
CHANNEL f1
CHANNEL f2
1D NMR plot parameters



30a **30b**



Current Data Parameters
NAME wj3-242
EXPNO 1
PROCNO 1

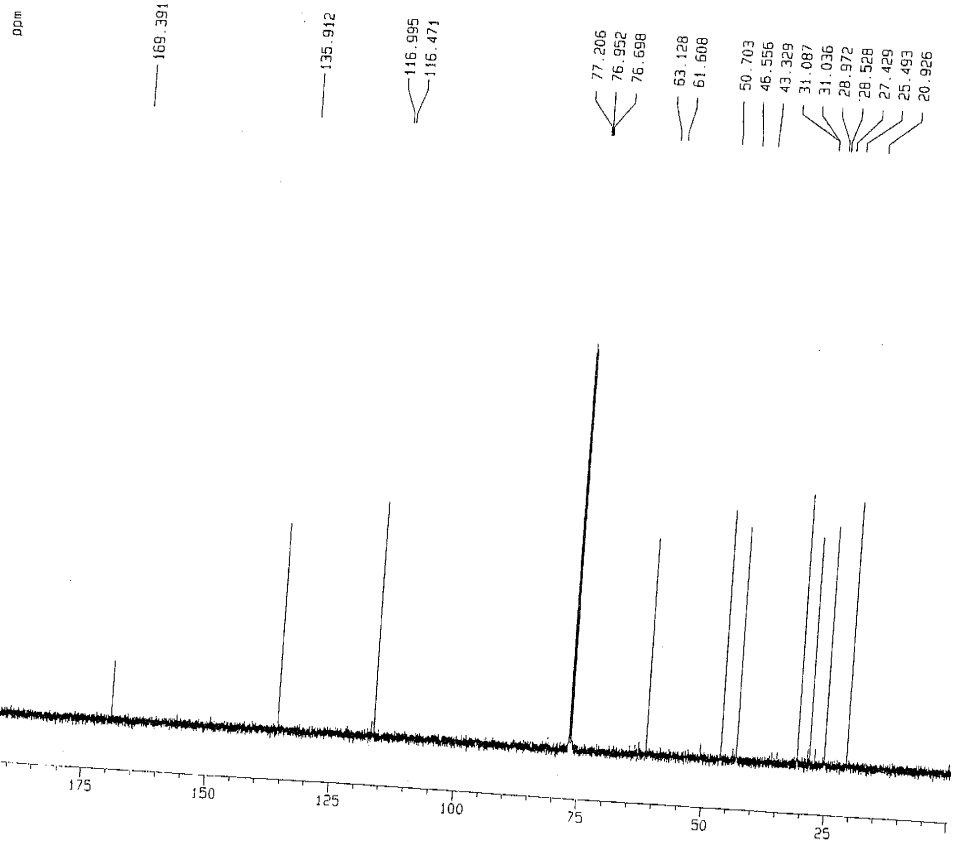
F2 - Acquisition Parameters
Date_ 20030305
Time 18.14
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6009.615 Hz
FIDRES 0.183398 Hz
AQ 2.7264309 sec
RG 161.3
DW 83.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.03000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.70 usec
PL1 -4.00 dB
SFO1 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1300000 MHz
WDW no
SSB 0
LB 0.30 Hz
GB 0

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 7.000 ppm
F1 3500.91 Hz
F2P -0.500 ppm
F2 -250.07 Hz
PPMCM 0.37500 ppm/cm
HZCM 187.54875 Hz/cm



Current Data Parameters
NAME wj3-242
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030305
Time 18.20
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 285
DS 4
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420083 sec
RG 2048
DW 15.900 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

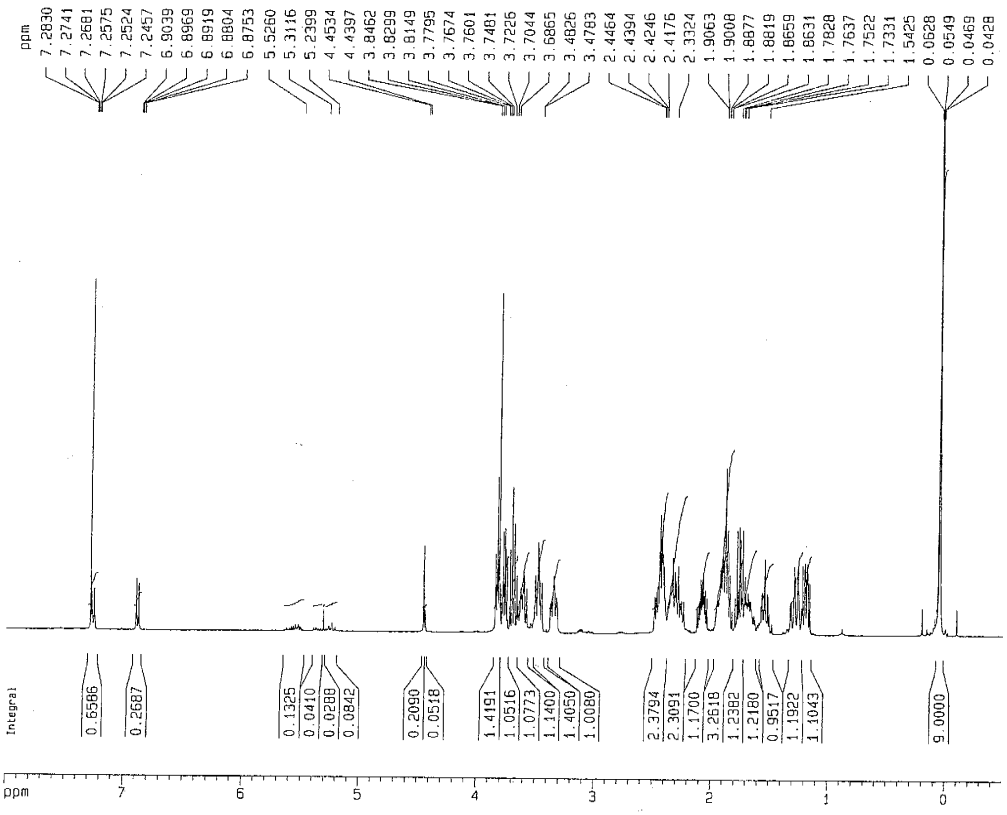
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 5.00 dB
SFO1 125.7719472 MHz

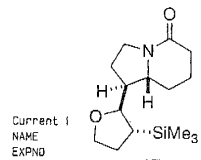
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 -4.00 dB
PL12 19.00 dB
PL13 30.00 dB
SFO2 500.1325000 MHz

F2 - Processing parameters
SI 65536
SF 125.7578008 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1300000 MHz
WDW no
SSB 0
LB 0.30 Hz
GB 0

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 200.000 ppm
F1 25151.56 Hz
F2P -0.500 ppm
F2 -62.86 Hz
PPMCM 10.02500 ppm/cm
HZCM 1260.72192 Hz/cm





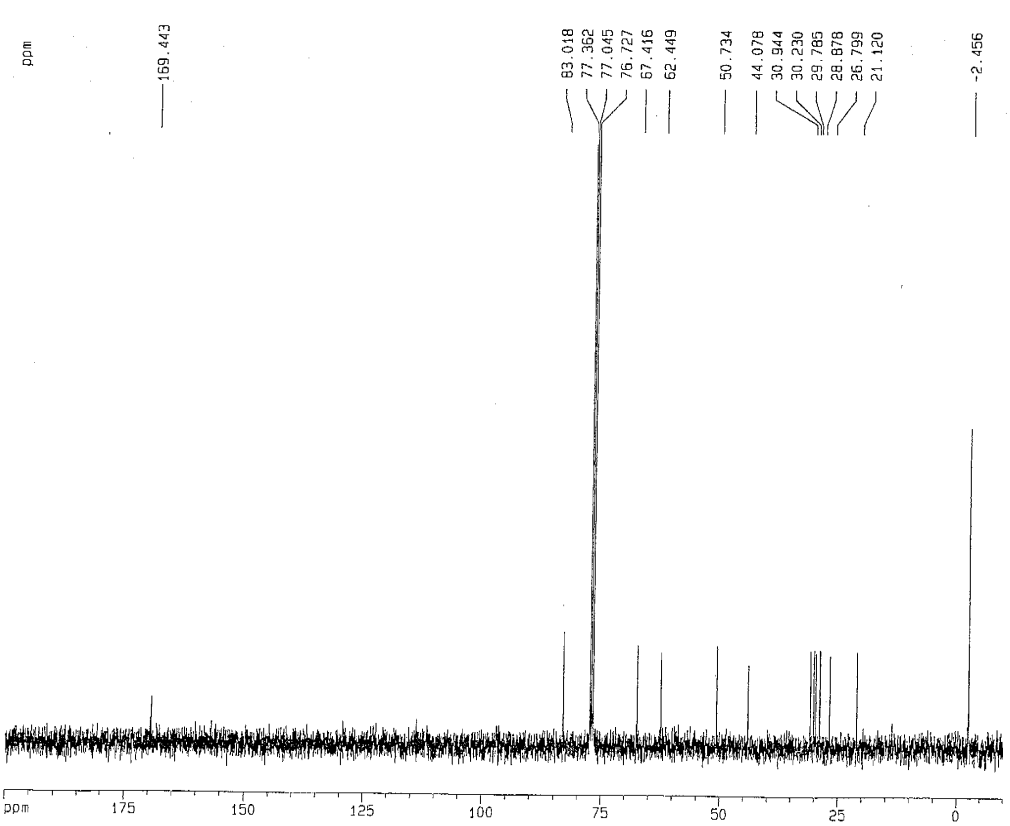
Current Data Parameters
NAME wj8-22.51-59
EXPNO 2
PROCNO 37

F2 - Acquisition Parameters
Date_ 20051015
Time 14.48
INSTRUM spect
PROBHD 5 mm QNP 1H/15
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 203.2
DW 104.400 usec
DE 6.00 usec
TE 295.9 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.05 usec
PL1 -3.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 8.000 ppm
F1 3201.04 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.42500 ppm/cm
HZCM 170.05525 Hz/cm



Current Data Parameters
NAME wj8-22.51-59
EXPNO 2
PROCNO 1

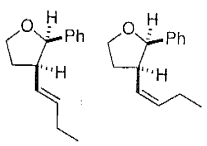
F2 - Acquisition Parameters
Date_ 20051015
Time 14.56
INSTRUM spect
PROBHD 5 mm QNP 1H/15
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 632
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2048
DW 20.850 usec
DE 6.00 usec
TE 296.1 K
D1 0.15000001 sec
d11 0.03000000 sec
DELTA 0.05000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 7.20 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz

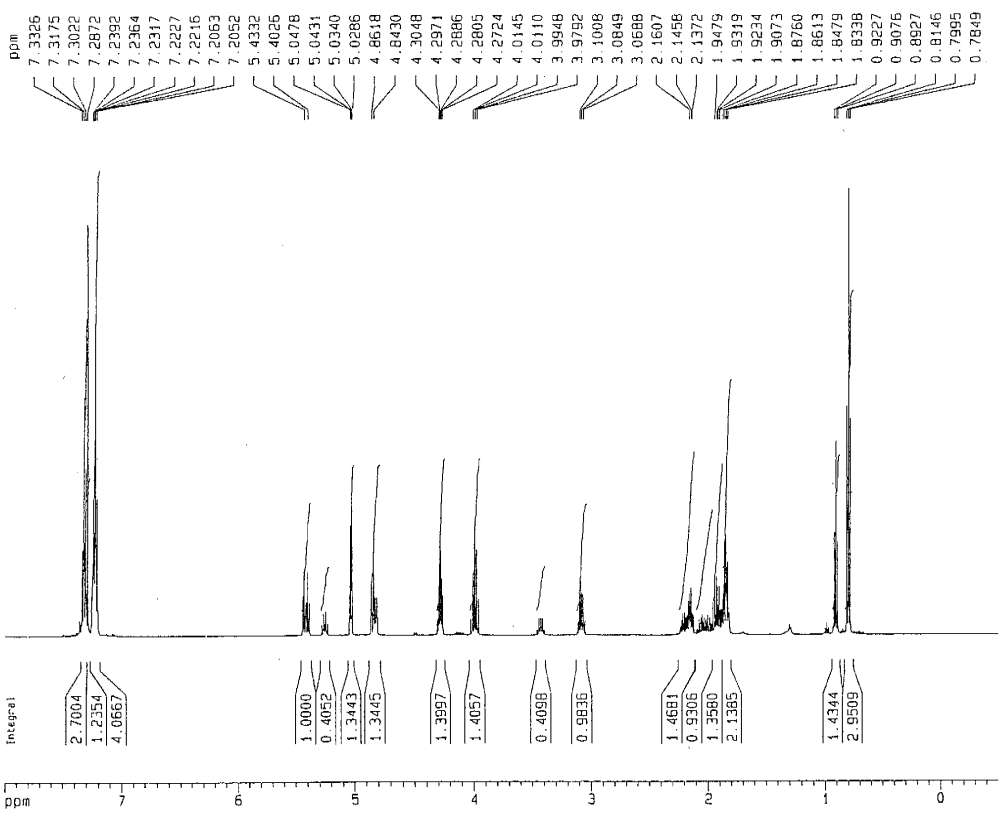
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 98.40 usec
PL2 -3.00 dB
PL12 19.96 dB
PL13 120.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 10.50000 ppm/cm
HZCM 1056.43408 Hz/cm



(*E*)-33a (*Z*)-33b



Current Data Parameters
NAME wj3-274
EXPNO 1
PROCNO 1

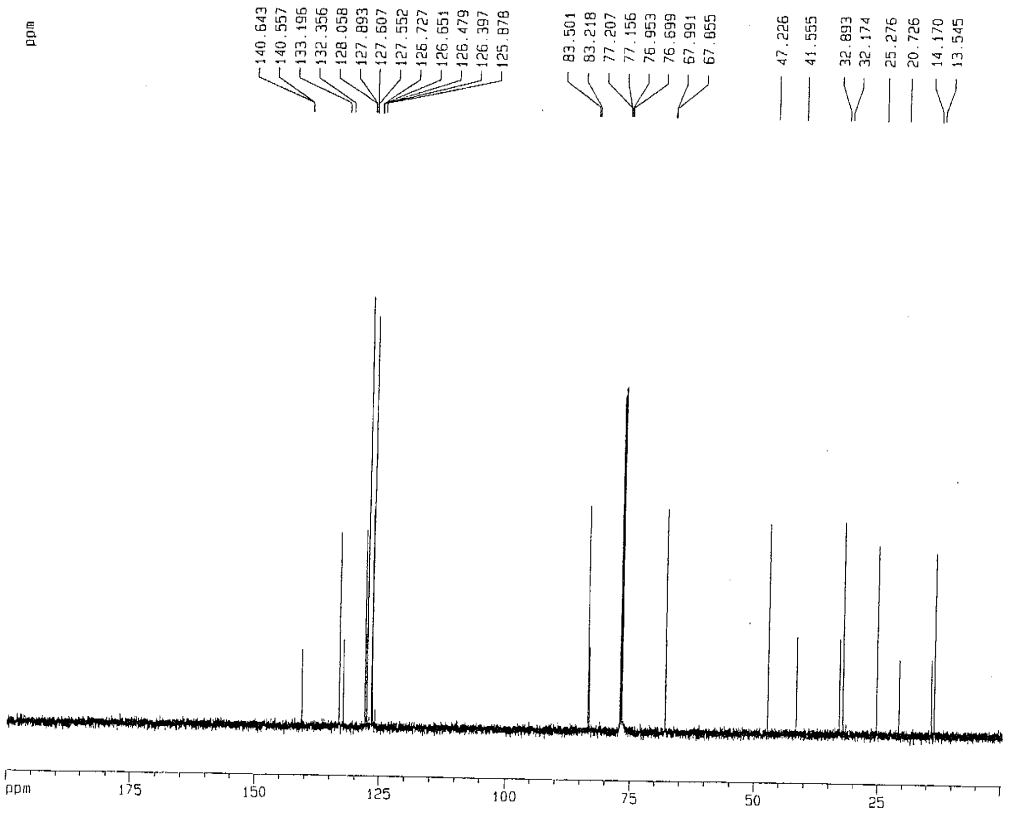
F2 - Acquisition Parameters
Date_ 20030422
Time 23.39
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7264309 sec
RG 90.5
DW 83.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.03000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.70 usec
PL1 -4.00 dB
SFO1 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1300000 MHz
WDW QSINE
SSB 0
LB 0.30 Hz
GB 0

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 8.000 ppm
F1 4001.04 Hz
F2P -0.500 ppm
F2 -250.07 Hz
PPMCM 0.42500 ppm/cm
HZCM 212.55525 Hz/cm



Current Data Parameters
NAME wj3-274
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030422
Time 23.44
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 235
DS 4
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420883 sec
RG 2048
DW 15.900 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

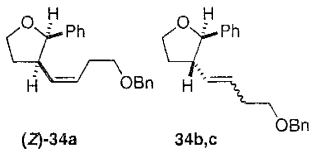
======== CHANNEL f1 ========
NUC1 13C
P1 6.50 usec
PL1 5.00 dB
SFO1 125.7719472 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 -4.00 dB
PL12 19.00 dB
PL13 30.00 dB
SFO2 500.1325000 MHz

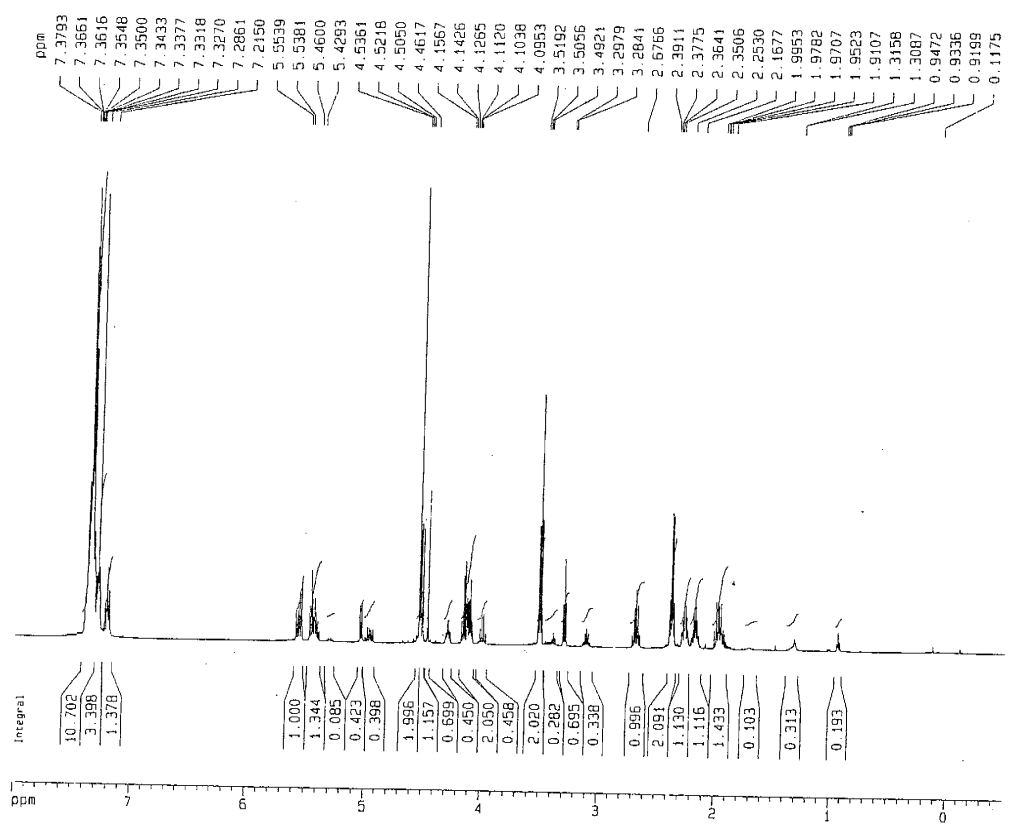
F2 - Processing parameters
SI 65536
SF 125.7578008 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1300000 MHz
WDW no
SSB 0
LB 0.30 Hz
GB 0

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 200.000 ppm
F1 25151.56 Hz
F2P -0.500 ppm
F2 -62.88 Hz
PPMCM 10.02500 ppm/cm
HZCM 1260.72192 Hz/cm



(*Z*)-34a 34b,c




```
Current Data Parameters
NAME                  wj3-270
EXPNO                       1
PROCNO                      1

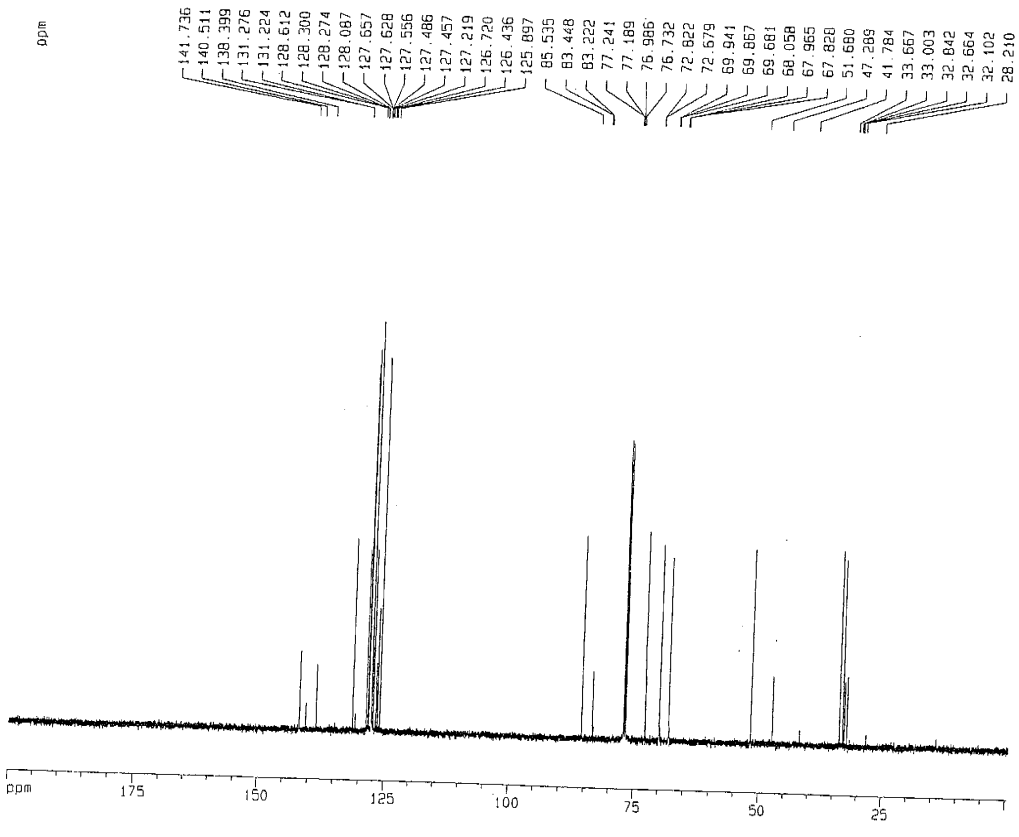
F2 - Acquisition Parameters
Date_              20030422
Time                   23.30
INSTRUM                spect
PROBHD     5 mm BBO BB-1H
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                6009.615 Hz
FIDRES            0.183398 Hz
AQ                2.7264309 sec
RG                     71.8
DW                 83.200 usec
DE                  6.00 usec
TE                   300.0 K
D1                0.03000000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     7.70 usec
PL1                   -4.00 dB
SFO1          500.1320005 MHz

F2 - Processing parameters
SI                        32768
SF                500.1300000 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00

F1 - Processing parameters
SI                         1024
MC2                          QF
SF                500.1300000 MHz
WDW                       QSINE
SSB                           0
LB                         0.30 Hz
GB                            0

1D NMR plot parameters
CX                        20.00 cm
CY                         0.00 cm
F1P                        8.000 ppm
F1                   4001.04 Hz
F2P                       -0.500 ppm
F2                    -250.07 Hz
PPMCM                  0.42500 ppm/cm
HZCM                 212.55525 Hz/cm
```




```
Current Data Parameters
NAME                  wj3-270
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20030422
Time                   23.36
INSTRUM                spect
PROBHD     5 mm BBO BB-1H
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      287
DS                        4
SWH               31446.541 Hz
FIDRES            0.479836 Hz
AQ                1.0420883 sec
RG                     2048
DW                 15.900 usec
DE                  6.00 usec
TE                   300.0 K
D1                0.10000000 sec
d11               0.03000000 sec
d12               0.00002000 sec

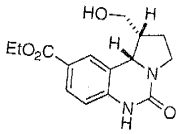
======== CHANNEL f1 ========
NUC1                      13C
P1                     8.50 usec
PL1                    5.00 dB
SFO1          125.7719472 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                 95.00 usec
PL2                   -4.00 dB
PL12                  19.00 dB
PL13                  30.00 dB
SFO2          500.1325000 MHz

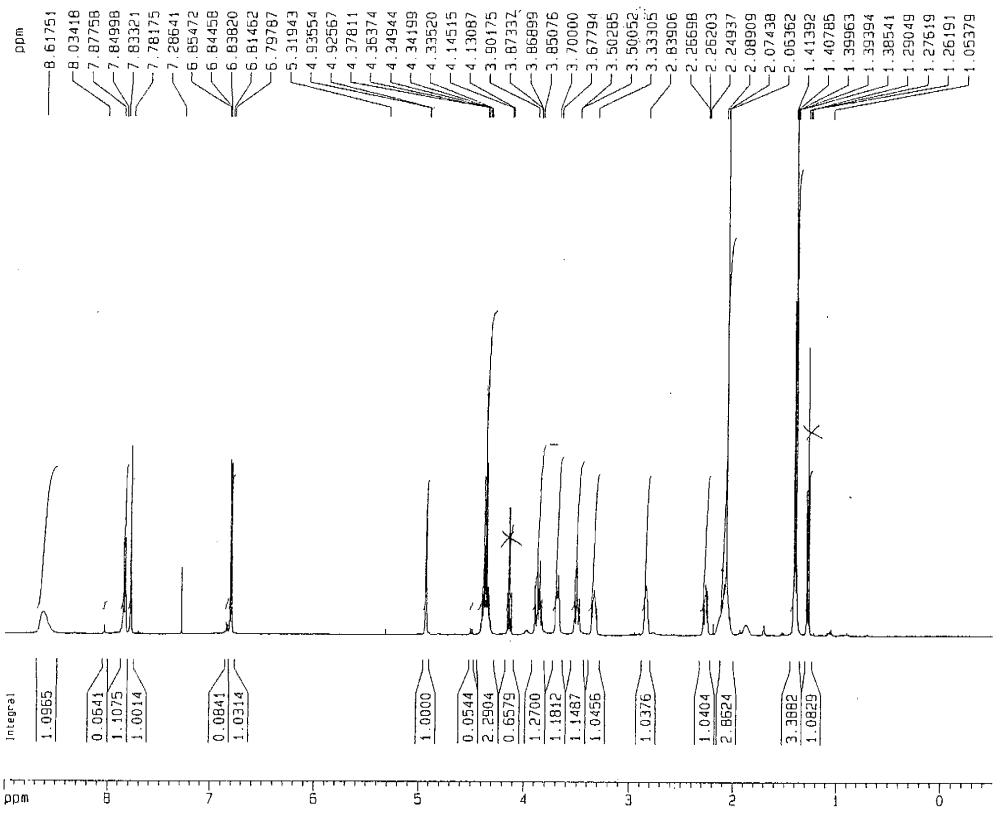
F2 - Processing parameters
SI                        65536
SF                125.7578008 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40

F1 - Processing parameters
SI                         1024
MC2                          QF
SF                500.1300000 MHz
WDW                          no
SSB                           0
LB                         0.30 Hz
GB                            0

1D NMR plot parameters
CX                        20.00 cm
CY                         0.00 cm
F1P                      200.000 ppm
F1                   25151.56 Hz
F2P                       -0.500 ppm
F2                    -62.88 Hz
PPMCM                  10.02500 ppm/cm
HZCM                 1260.72192 Hz/cm
```



19



```
Current Data Parameters
NAME                  wj3-196
EXPNO                       1
PROCNO                      1

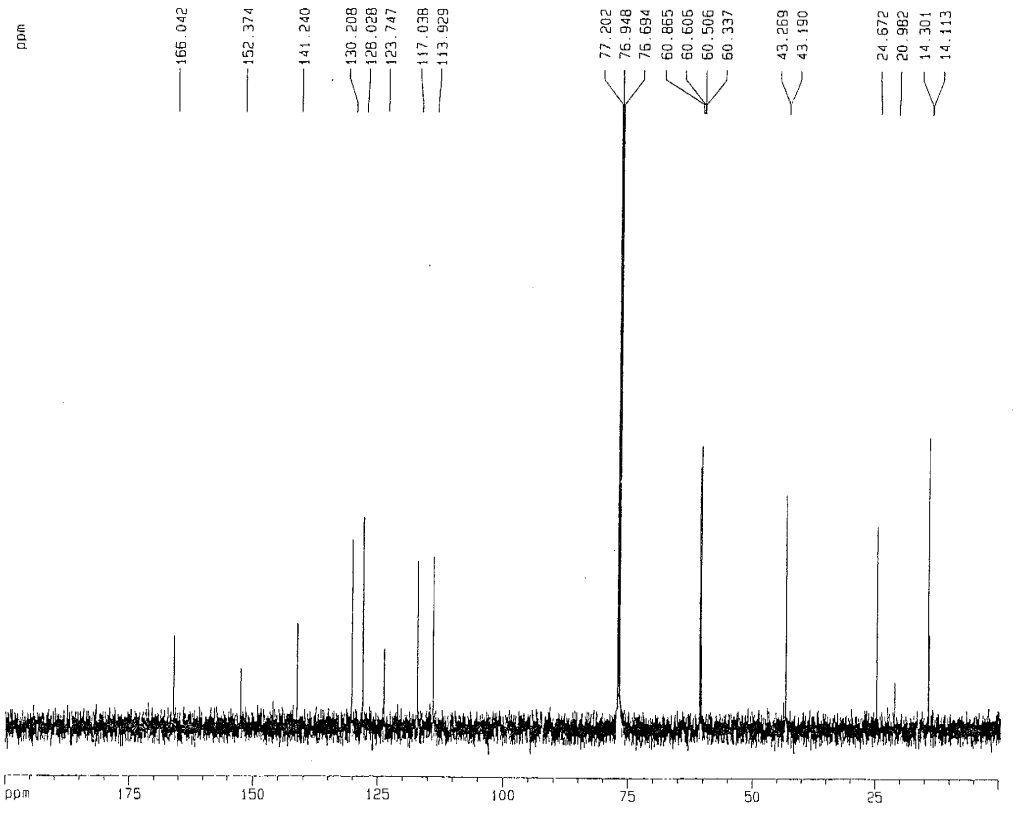
F2 - Acquisition Parameters
Date_              20030115
Time                   13.13
INSTRUM                spect
PROBHD   5 mm BBO BB-1H
PULPROG                zg30
TD                     32768
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                6009.615 Hz
FIDRES             0.183399 Hz
AQ                 2.7264309 sec
RG                     228.1
DW                    83.200 usec
DE                     6.00 usec
TE                    300.0 K
D1                 0.03000000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                      7.70 usec
PL1                    -4.00 dB
SFO1              500.1320005 MHz

F2 - Processing parameters
SI                     32768
SF                500.1300000 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00

F1 - Processing parameters
SI                      1024
MC2                       QF
SF                500.1300000 MHz
WDW                    QSINE
SSB                        0
LB                      0.30 Hz
GB                         0

1D NMR plot parameters
CX                     20.00 cm
CY                      0.00 cm
F1P                  9.000 ppm
F1                 4501.17 Hz
F2P                 -0.500 ppm
F2                 -250.07 Hz
PPMCM               0.47500 ppm/cm
HZCM              237.56175 Hz/cm
```



```
Current Data Parameters
NAME                  wj3-196
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20030115
Time                   13.19
INSTRUM                spect
PROBHD   5 mm BBO BB-1H
PULPROG             zgpg30
TD                     65536
SOLVENT                CDCl3
NS                        362
DS                         4
SWH               31446.541 Hz
FIDRES             0.479836 Hz
AQ                 1.0420883 sec
RG                      8192
DW                   15.900 usec
DE                     6.00 usec
TE                    300.0 K
D1                 0.10000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

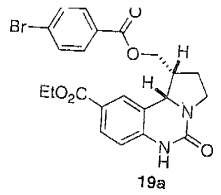
======== CHANNEL f1 ========
NUC1                       13C
P1                      6.50 usec
PL1                    5.00 dB
SFO1             125.7719472 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  95.00 usec
PL2                    -4.00 dB
PL12                   19.00 dB
PL13                   30.00 dB
SFO2              500.1325000 MHz

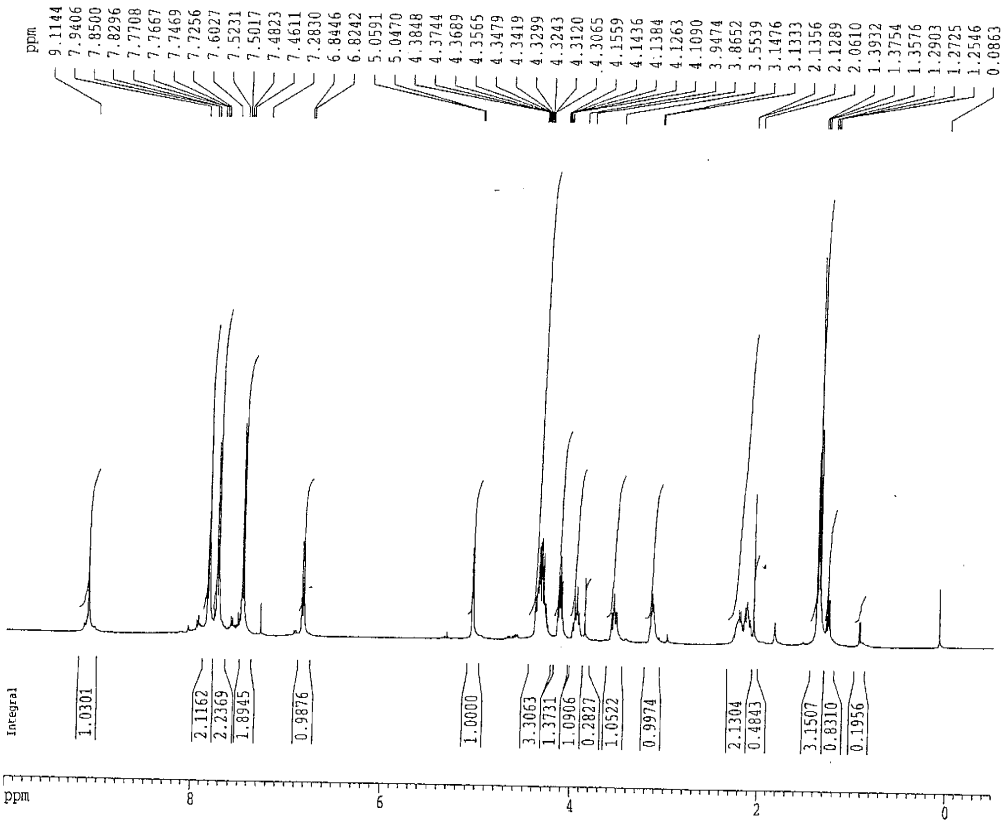
F2 - Processing parameters
SI                     65536
SF               125.7578008 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40

F1 - Processing parameters
SI                      1024
MC2                       QF
SF                500.1300000 MHz
WDW                       no
SSB                        0
LB                      0.30 Hz
GB                         0

1D NMR plot parameters
CX                     20.00 cm
CY                      0.00 cm
F1P                  200.000 ppm
F1                 25151.56 Hz
F2P                 -0.500 ppm
F2                 -62.88 Hz
PPMCM              10.02500 ppm/cm
HZCM             1260.72192 Hz/cm
```



19a



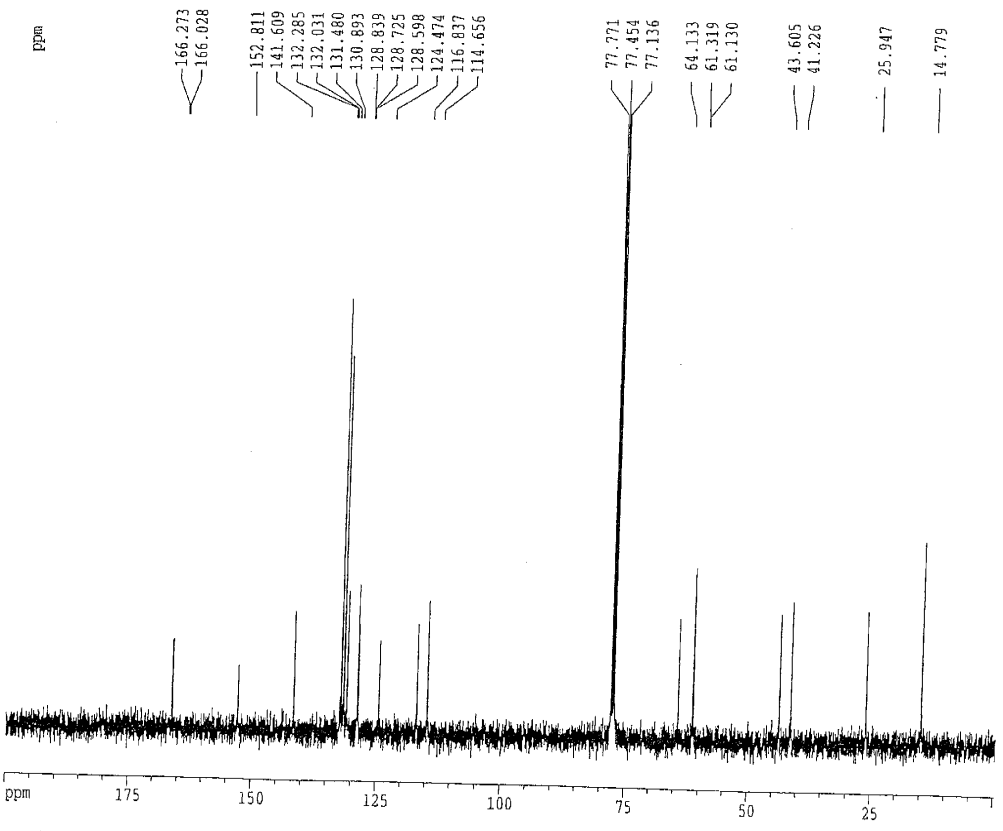

```
NAME                wj3-225
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20030215
Time                  18.23
INSTRUM              drx400
PROBHD   5 mm Multinucl
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        2
SWH              4789.272 Hz
FIDRES           0.146157 Hz
AQ               3.4210291 sec
RG                      114
DW                104.400 usec
DE                  4.50 usec
TE                  300.0 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                   7.70 usec
PL1                  -6.00 dB
SFO1          400.1320007 MHz

F2 - Processing parameters
SI                        16384
SF              400.1300000 MHz
WDW                          EM
SSB                           0
LB                       0.30 Hz
GB                            0
PC                         1.00

1D NMR plot parameters
CX                       20.00 cm
CY                        0.00 cm
F1P                      10.000 ppm
F1                     4001.30 Hz
F2P                     -0.500 ppm
F2                     -200.07 Hz
PPMCM                 0.52500 ppm/cm
HZCM                210.06825 Hz/cm
```




```
Current Data Parameters
NAME                wj3-225
EXPNO                     2
PROCNO                    1

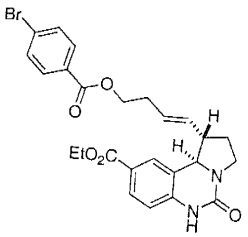
F2 - Acquisition Parameters
Date_              20030215
Time                  18.27
INSTRUM              drx400
PROBHD   5 mm Multinucl
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      435
DS                        2
SWH             23148.148 Hz
FIDRES           0.353213 Hz
AQ               1.4156276 sec
RG                    32768
DW                21.600 usec
DE                  4.50 usec
TE                  300.0 K
D1              0.05000000 sec
d11             0.03000000 sec
d12             0.00002000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                  12.30 usec
PL1                  2.00 dB
SFO1          100.6232933 MHz

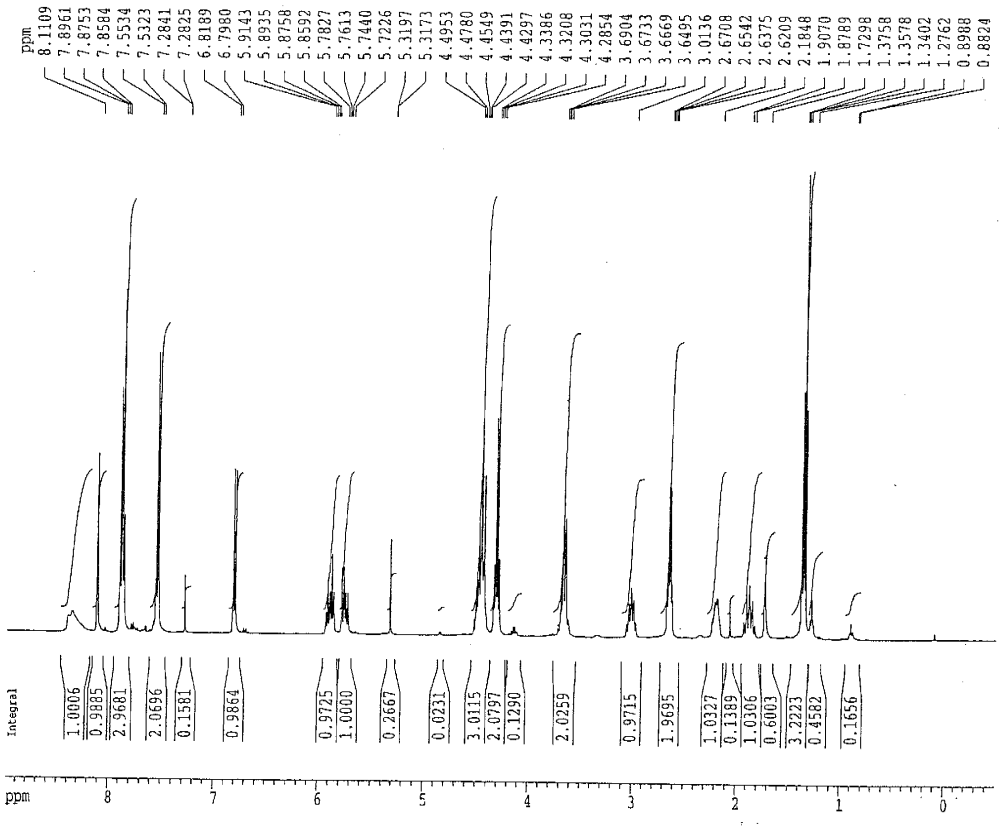
======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2               100.00 usec
PL2                   0.00 dB
PL12                 18.00 dB
PL13                 18.00 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                        32768
SF              100.6127290 MHz
WDW                          EM
SSB                           0
LB                    1.00 Hz
GB                            0
PC                         1.40

1D NMR plot parameters
CX                       20.00 cm
CY                        0.00 cm
F1P                     200.000 ppm
F1                    20122.55 Hz
F2P                     -0.500 ppm
F2                     -50.30 Hz
PPMCM                10.02500 ppm/cm
HZCM              1008.64258 Hz/cm
```



20



F2 - Acquisition Parameters

Date_	20021127
Time	17.01
INSTRUM	drx400
PROBHD	5 mm Multinucl
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	4789.272 Hz
FIDRES	0.146157 Hz
AQ	3.4210291 sec
RG	128
DW	104.400 usec
DE	4.50 usec
TE	300.0 K
D1	1.00000000 sec

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

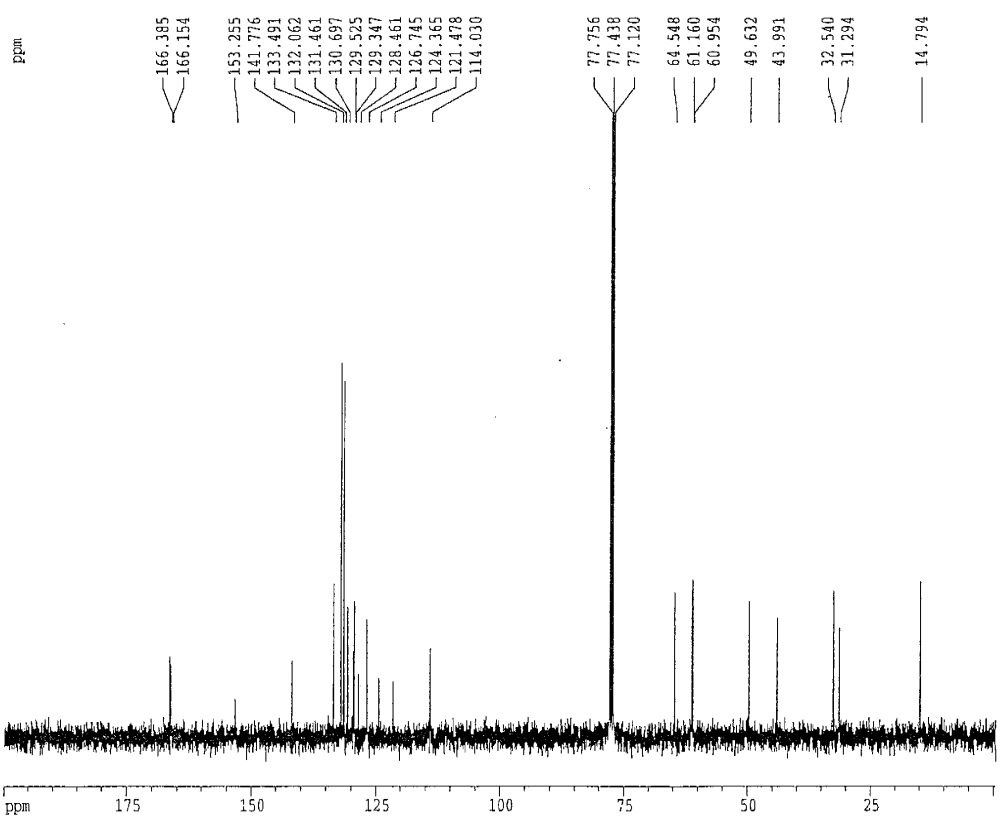
NUC1	1H
P1	7.70 usec
PL1	-6.00 dB
SFO1	400.1320007 MHz

F2 - Processing parameters

SI	16384
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
CY	0.00 cm
F1P	9.000 ppm
F1	3601.17 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.47500 ppm/cm
HZCM	190.06175 Hz/cm



Current Data Parameters

NAME	wj3-165
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20021127
Time	17.07
INSTRUM	drx400
PROBHD	5 mm Multinucl
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	425
DS	2
SWH	23148.148 Hz
FIDRES	0.353213 Hz
AQ	1.4156276 sec
RG	3649.1
DW	21.600 usec
DE	4.50 usec
TE	300.0 K
D1	0.05000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

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

NUC1	13C
P1	12.30 usec
PL1	2.00 dB
SFO1	100.6232933 MHz

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

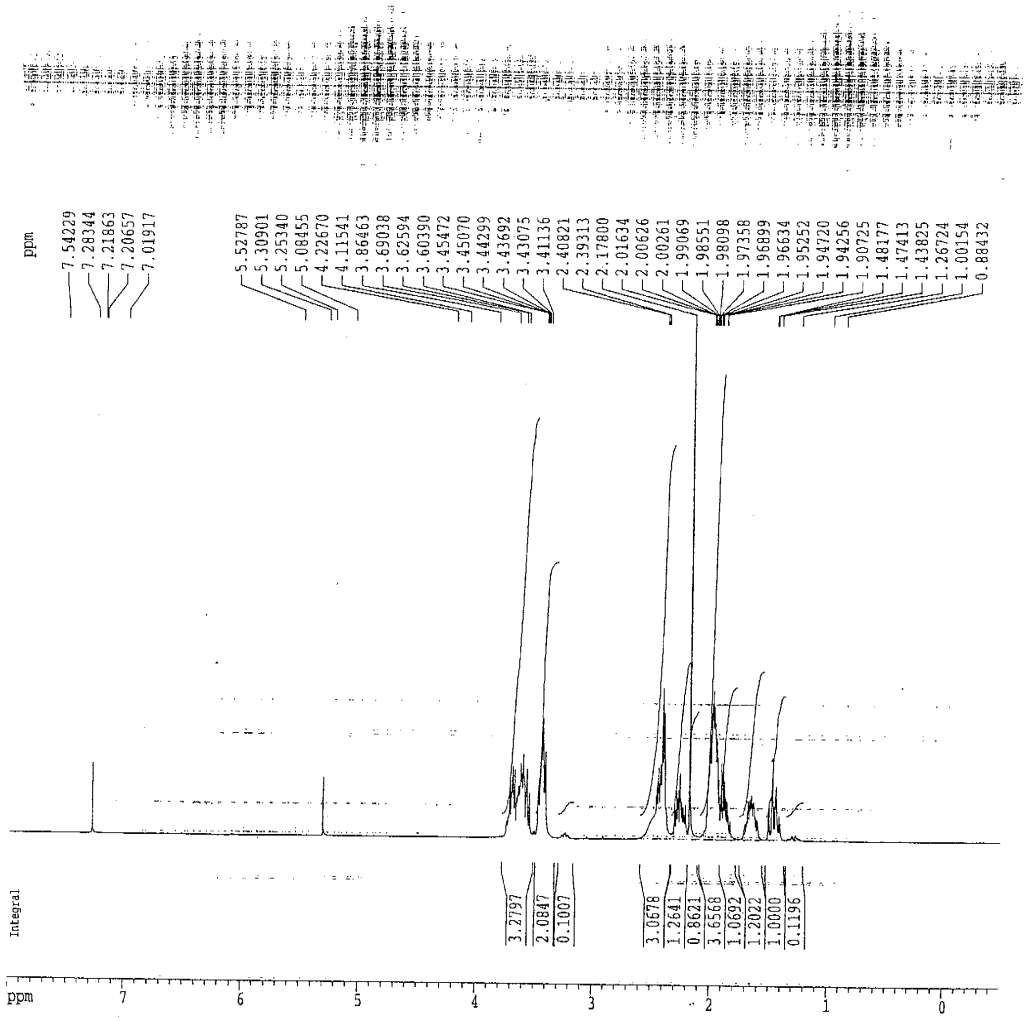
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	0.00 dB
PL12	18.00 dB
PL13	18.00 dB
SFO2	400.1316005 MHz

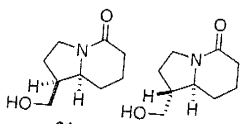
F2 - Processing parameters

SI	32768
SF	100.6127290 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	0.00 cm
F1P	200.000 ppm
F1	20122.55 Hz
F2P	-0.500 ppm
F2	-50.31 Hz
PPMCM	10.02500 ppm/cm
HZCM	1008.64270 Hz/cm





31a 31b

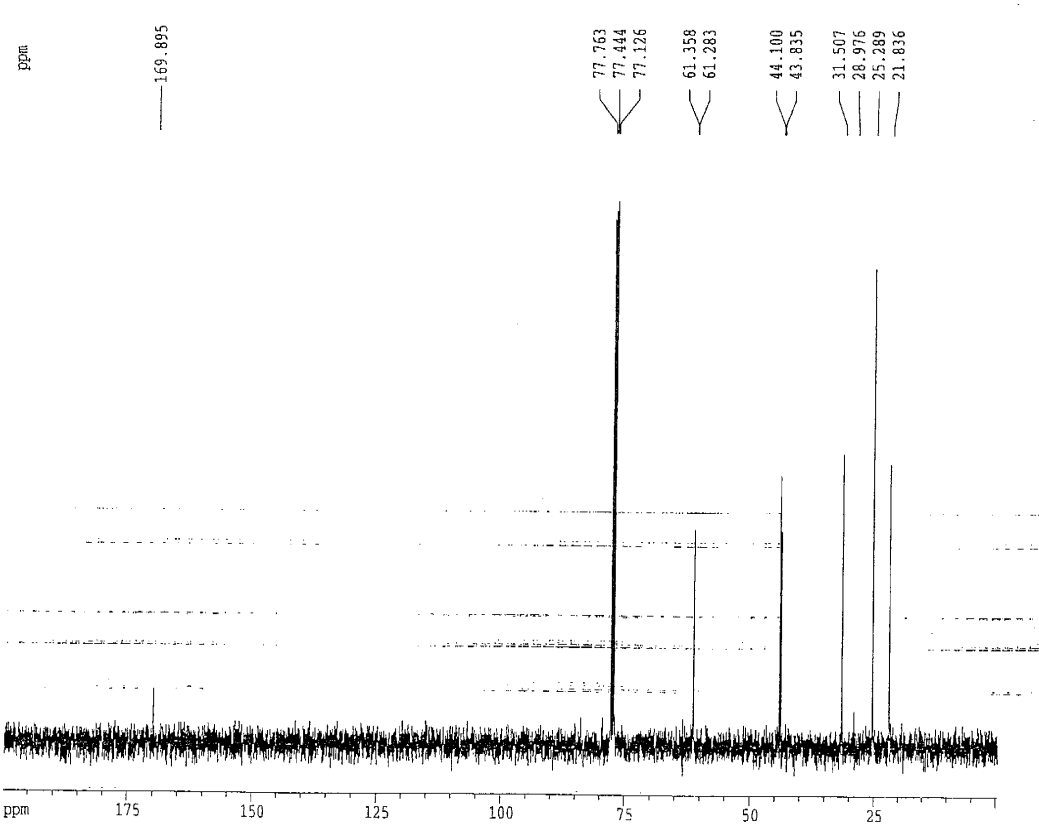
Current Data Parameters
NAME wj4-38
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030702
Time 16.08
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 143.7
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 2
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 8.000 ppm
F1 3201.04 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.42500 ppm/cm
HZCM 170.05525 Hz/cm



Current Data Parameters
NAME wj4-38
EXPNO 2
PROCNO 1

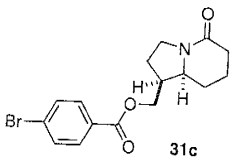
F2 - Acquisition Parameters
Date_ 20030702
Time 16.11
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 175
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 32768
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.6232933 MHz

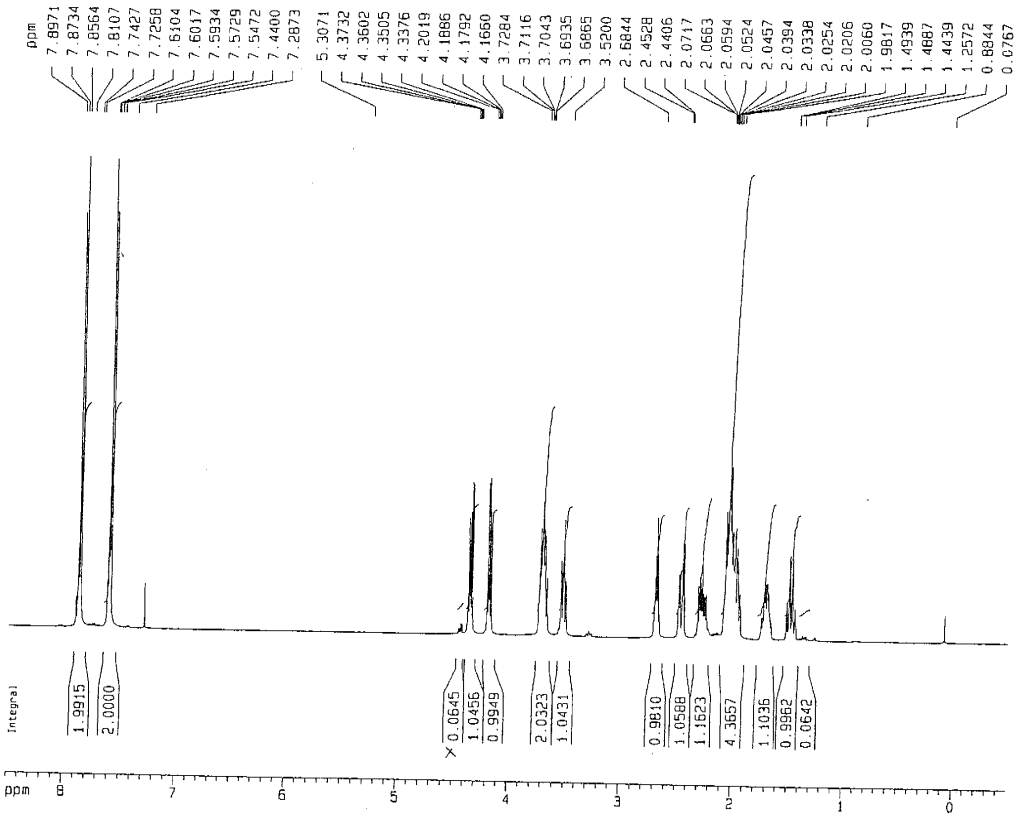
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.5127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -0.500 ppm
F2 -50.31 Hz
PPMCM 10.02500 ppm/cm
HZCM 1008.64264 Hz/cm



31c




```
Current Data Parameters
NAME                  wj4-40
EXPNO                      1
PROCNO                     1

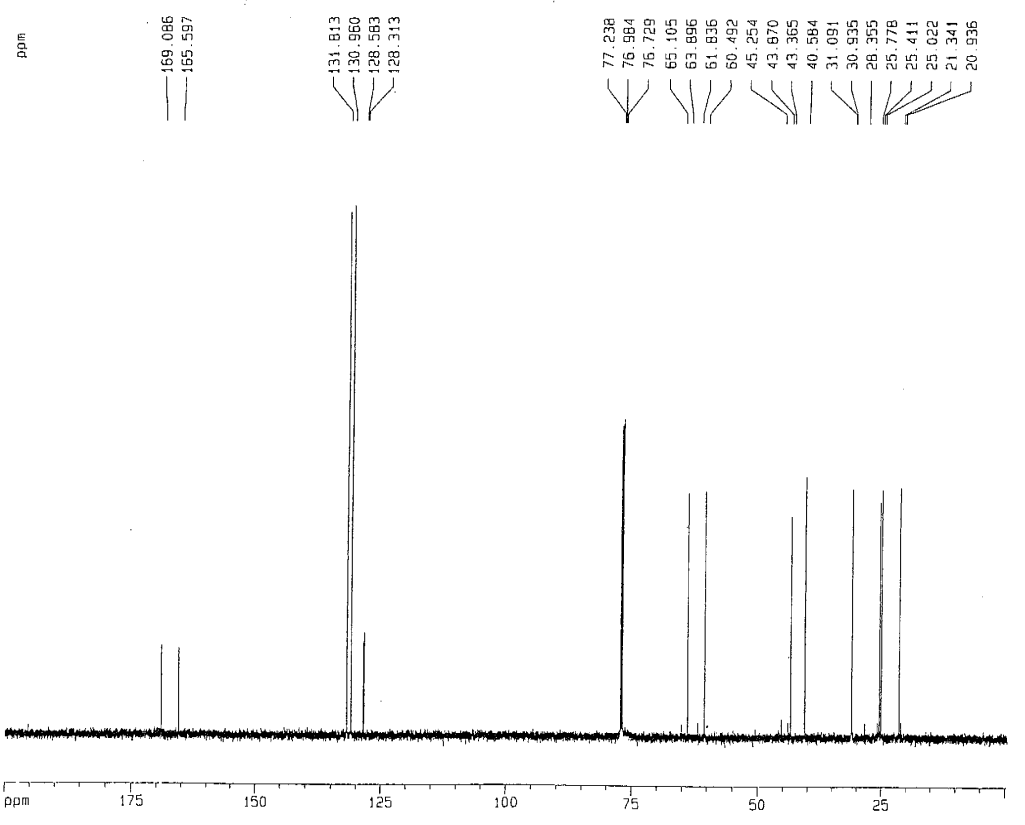
F2 - Acquisition Parameters
Date_              20030807
Time                  19.57
INSTRUM               spect
PROBHD  5 mm BBO BB-1H
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                6009.615 Hz
FIDRES            0.183399 Hz
AQ                2.7264309 sec
RG                      114
DW                 83.200 usec
DE                  6.00 usec
TE                 300.0 K
D1                0.03000000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                    7.70 usec
PL1                  -4.00 dB
SFO1           500.1320005 MHz

F2 - Processing parameters
SI                       32768
SF                500.1300000 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00

F1 - Processing parameters
SI                        1024
MC2                         QF
SF                500.1300000 MHz
WDW                      QSINE
SSB                          0
LB                        0.30 Hz
GB                           0

1D NMR plot parameters
CX                       20.00 cm
CY                        0.00 cm
F1P                       8.500 ppm
F1                     4251.10 Hz
F2P                      -0.500 ppm
F2                     -250.07 Hz
PPMCM                 0.45000 ppm/cm
HZCM                225.05850 Hz/cm
```




```
Current Data Parameters
NAME                  wj4-40
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_              20030807
Time                  20.01
INSTRUM               spect
PROBHD  5 mm BBO BB-1H
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      248
DS                        4
SWH               31446.541 Hz
FIDRES            0.479836 Hz
AQ                1.0420863 sec
RG                     2048
DW                 15.900 usec
DE                  6.00 usec
TE                 300.0 K
D1                0.10000000 sec
D11               0.03000000 sec
D12               0.00002000 sec

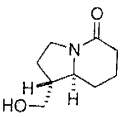
======== CHANNEL f1 ========
NUC1                      13C
P1                    6.50 usec
PL1                   9.00 dB
SFO1           125.7719472 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                95.00 usec
PL2                  -4.00 dB
PL12                 19.00 dB
PL13                 36.00 dB
SFO2           500.1325000 MHz

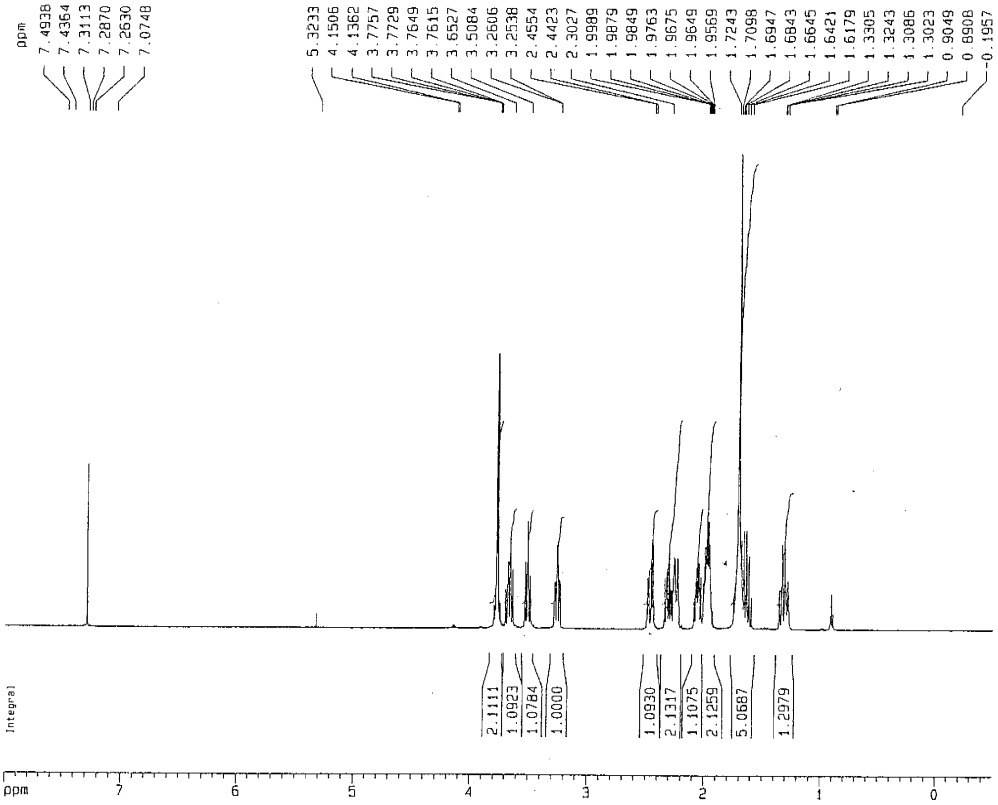
F2 - Processing parameters
SI                       65536
SF                125.7578008 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40

F1 - Processing parameters
SI                        1024
MC2                         QF
SF                500.1300000 MHz
WDW                         no
SSB                          0
LB                        0.30 Hz
GB                           0

1D NMR plot parameters
CX                       20.00 cm
CY                        0.00 cm
F1P                     200.000 ppm
F1                    25151.56 Hz
F2P                      -0.500 ppm
F2                     -62.88 Hz
PPMCM                10.02500 ppm/cm
HZCM               1260.72192 Hz/cm
```



32




```
Current Data Parameters
NAME                  wj3-42.cryst
EXPNO                          1
PROCNO                         1

F2 - Acquisition Parameters
Date_              20020706
Time                   14.18
INSTRUM                spect
PROBHD  5 mm BBO BB-1H
PULPROG                zg30
TD                     32768
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                 6009.615 Hz
FIDRES              0.183399 Hz
AQ                  2.7264309 sec
RG                     574.7
DW                    83.200 usec
DE                     6.00 usec
TE                     300.0 K
D1                  0.03000000 sec

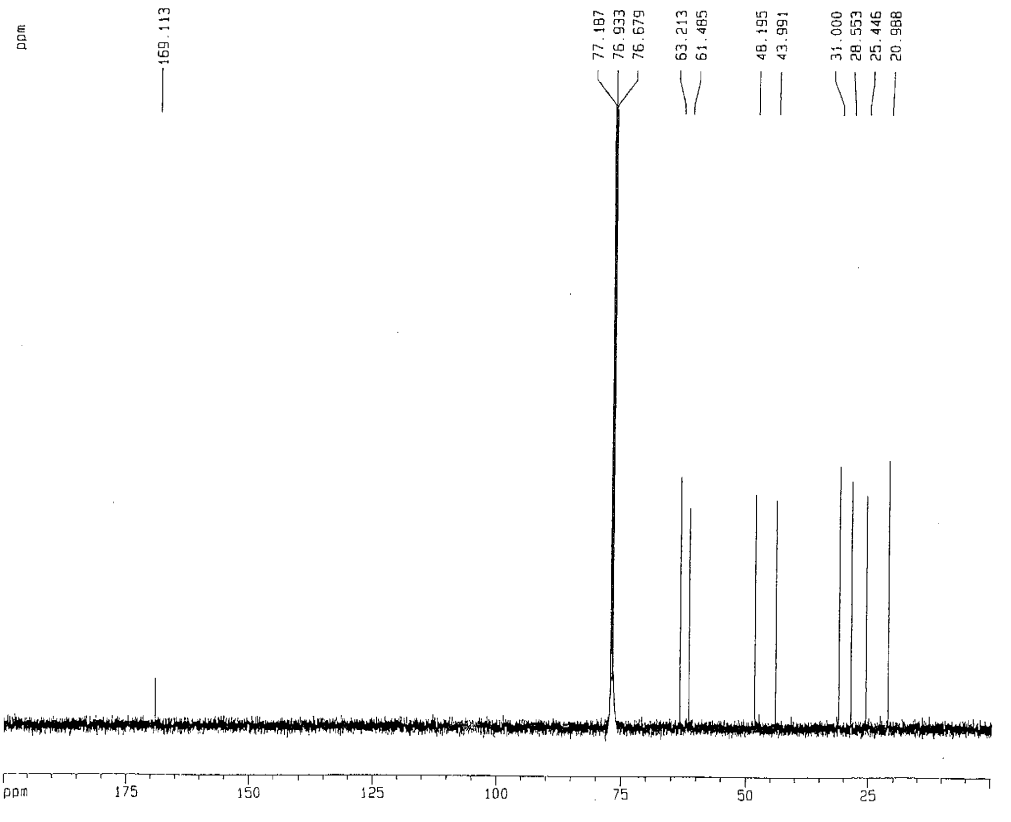
======== CHANNEL f1 ========
NUC1                       1H
P1                      7.70 usec
PL1                    -4.00 dB
SFO1             500.1320005 MHz

F1 - Acquisition parameters
NDO                        2
TD                       256
SFO1             500.1325 MHz
FIDRES           23.475060 Hz
SW                  12.016 ppm

F2 - Processing parameters
SI                     32768
SF               500.1300000 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00

F1 - Processing parameters
SI                      1024
MC2                       QF
SF               500.1300000 MHz
WDW                       no
SSB                        0
LB                      0.30 Hz
GB                         0

1D NMR plot parameters
CX                     20.00 cm
CY                      0.00 cm
F1P                 8.000 ppm
F1                  4001.04 Hz
F2P                -0.500 ppm
F2                 -250.07 Hz
PPMCM               0.42500 ppm/cm
HZCM              212.65525 Hz/cm
```




```
Current Data Parameters
NAME                  wj3-42.cryst
EXPNO                          2
PROCNO                         1

F2 - Acquisition Parameters
Date_              20020706
Time                   15.29
INSTRUM                spect
PROBHD  5 mm BBO BB-1H
PULPROG                zgpg30
TD                     65536
SOLVENT                CDCl3
NS                      3713
DS                         4
SWH                31446.541 Hz
FIDRES              0.479836 Hz
AQ                  1.0420883 sec
RG                     10321.3
DW                    15.900 usec
DE                     6.00 usec
TE                     300.0 K
D1                  0.10000000 sec
d11                 0.03000000 sec
d12                 0.00002000 sec

======== CHANNEL f1 ========
NUC1                       13C
P1                      6.50 usec
PL1                     5.00 dB
SFO1             125.7719472 MHz

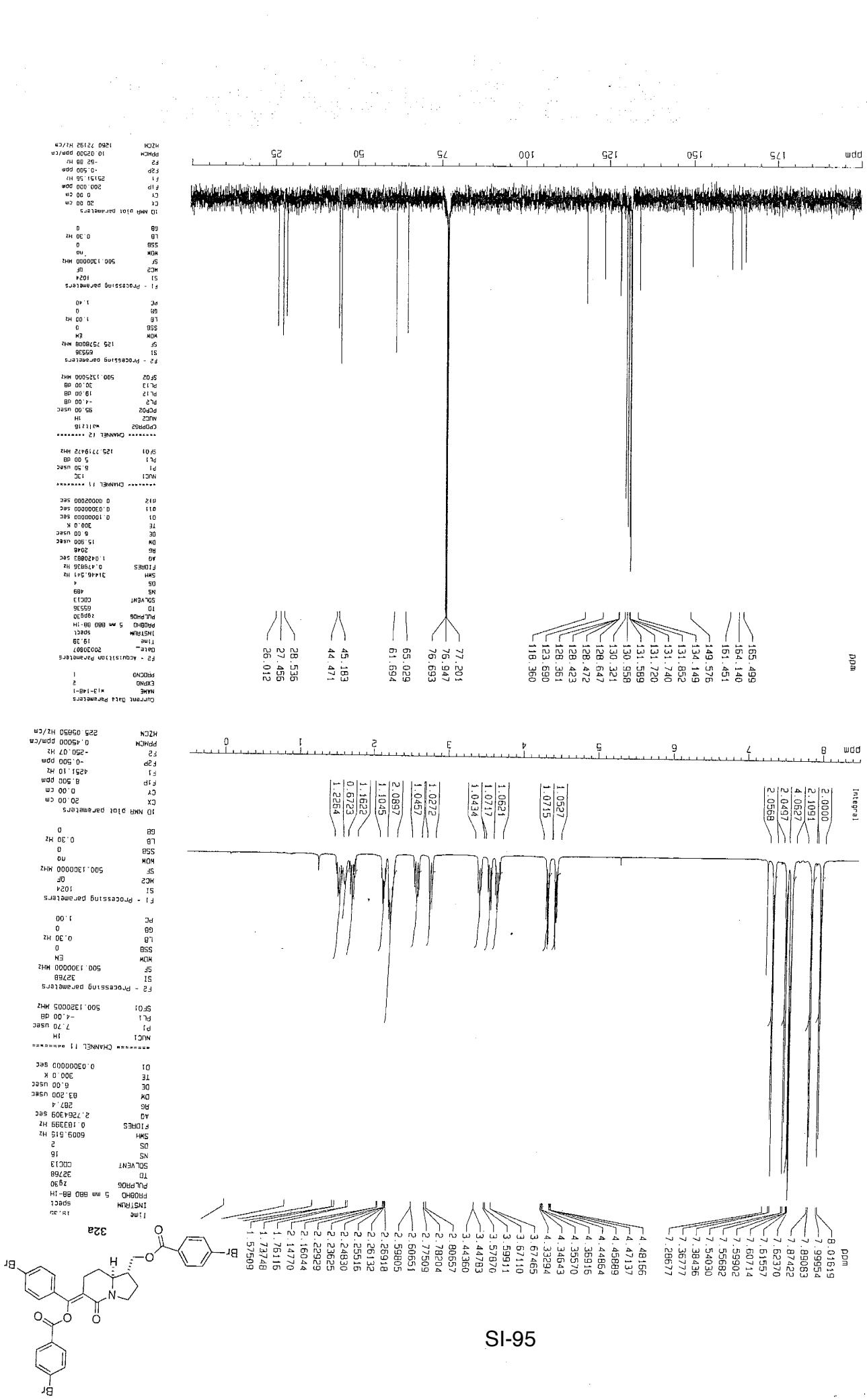
======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  95.00 usec
PL2                    -4.00 dB
PL12                   19.00 dB
PL13                   30.00 dB
SFO2             500.1325000 MHz

F1 - Acquisition parameters
NDO                        2
TD                       256
SFO1             500.1325 MHz
FIDRES           23.475060 Hz
SW                  12.016 ppm

F2 - Processing parameters
SI                     65536
SF               125.7579008 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40

F1 - Processing parameters
SI                      1024
MC2                       QF
SF               500.1300000 MHz
WDW                       no
SSB                        0
LB                      0.30 Hz
GB                         0

1D NMR plot parameters
CX                     20.00 cm
CY                      0.00 cm
F1P                 200.000 ppm
F1                  25151.56 Hz
F2P                -0.500 ppm
F2                 -62.88 Hz
PPMCM              10.02500 ppm/cm
HZCM              1260.72192 Hz/cm
```


32a
Br
Br
Br
O
O
O
O
N
H
ppm
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1.73748
1.76116
2.14770
2.16044
2.22929
2.23625
2.24830
2.25516
2.26132
2.26918
2.59805
2.60651
2.77509
2.78204
2.80657
3.44360
3.44783
3.57070
3.59911
3.67110
3.67465
4.33294
4.34643
4.35570
4.35915
4.44864
4.45889
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ppm