Journal of Organic Chemistry

Supporting Information to Accompany :

A Novel Deoxygenation Reaction of Epoxides by Indium

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General Points:

Infra-red spectra were recorded on FT-IR spectrometers. Proton NMR (^{1}H) spectra were recorded at 400 MHz or at 300MHz. Carbon NMR (^{13}C) spectra were similarly recorded at 100.61 MHz or 75.50 MHz . The two-dimensional (2D) homonuclear (^{1}H-^{1}H) shift correlation experiments were carried out at 400 MHz and two dimensional heteronuclear (^{1}H-^{13}C) shift correlation experiments were performed at 100 MHz. The 1D ^{1}H nOe NMR data and two-dimensional ^{1}H NOESY and DQFCOSY NMR data were acquired at 400.13 MHz using a Dual (BBO) probehead at a temperature of 300 K. The NMR experiments were, in general, carried out in deuterochloroform ($CDCl_3$), unless, otherwise specified. The chemical shifts (δ) are quoted in parts per million (ppm) from TMS as the internal standard. Multiplicities are abbreviated as s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broad. The coupling constants (*J*) are reported in Hertz (Hz).

High and low resolution mass spectra were recorded were recorded using electron impact (EI), chemical ionization (CI), fast atom bombardment (FAB) or electrospray (ES) techniques as stated for each compound.

Gas Chromatography Mass Spectrometry (GC-MS) was performed at Strathclyde University.

Melting points (mp) were carried out and are uncorrected.

Flash chromatography was performed using silica gel 60 (200-400 mesh). Thin layer chromatography (TLC) was performed using aluminium sheets of silica gel 60 F_{254} and was visualized under uv lamp (254 nm). The plates were developed with aqueous potassium permanganate solution, methanolic phosphomolybdic acid (10-20% w/v), acidic *p*-anisaldehyde (10% v/v) or acidic methanolic vanillin solutions.

All reagents were obtained from commercial suppliers and used without further purification unless otherwise stated. As required, organic solvents were dried and / or distilled prior to use. Tetrahydrofuran was dried by distillation from sodium and benzophenone, dichloromethane was dried by distillation over calcium hydride, diethyl ether was dried over sodium wire, and petroleum ether was

distilled at 60-80°C. *n*-Heptane used for flash chromatography was of ≥99.3% purity and was used without further purification. In experiments where sodium hydride (60% suspension in mineral oil) has been utilized as a base, it was washed with petroleum ether or THF at least twice prior to use.

Indium metal power of 99.99% purity (purity excludes ~1% Mg as anticaking agent) or of 99+% purity. Indium(I) chloride (anhydrous) was of 99.99% purity and ammonium chloride from Fisher Scientific of 99+% purity.

trans-Stilbene oxide and *trans*-1,3-diphenyl-2,3-epoxypropan-1-one were each ≥98% purity. *cis*-Stilbene oxide, *exo*-2,3-epoxynorbornane and ethyl 3-phenylglycidate (a mixture of *cis* and *trans*) of 97%, 97% and 92% purity respectively.

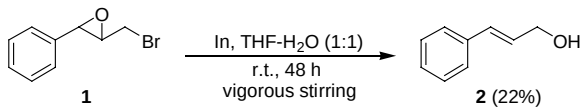
All reactions requiring anhydrous conditions were performed in flame-dried apparatus under a nitrogen or argon atmosphere. Organic extracts were, in general, dried over anhydrous magnesium sulfate ($MgSO_4$) or sodium sulfate (Na_2SO_4).

Preparation of the epoxides

The epoxides 1-bromo-2,3-epoxy-3-phenylpropane[1] **(1)**, ethyl 6-chloro-3,4-epoxy-4-phenyl-1,2,3,4-tetrahydroquinolin-2-one-3-carboxylate[2] **(12)**, 10,11-epoxy-10,11-di-hydrodibenzo[*a*,*d*]cyclohepten-5-one[3] **(14)**, 1a,2,3,7b-tetrahydro-1-oxa-cyclopropa[*a*] naphthalene[4] **(16)**, (*E*)-4-{(1*S*,6*R*)-2,2,6-trimethyl-7-oxa-bicyclo[4.1.0]hept-1-yl}-but-3-en-2-one[5] **(20)**, 2-methyl-2-phenyloxirane[6a] **(22)** (Corey-Chaykovsky Method[6b]), 1,3,3-trimethyl-7-oxa-bicyclo[4.1.0]heptan-2-one[7] **(25)** were prepared according to the respective literature procedures and the spectral data of each of the epoxides were consistent with those reported in the literature.

Deoxygenation of 2-bromomethyl-3-phenyloxirane (1) by indium

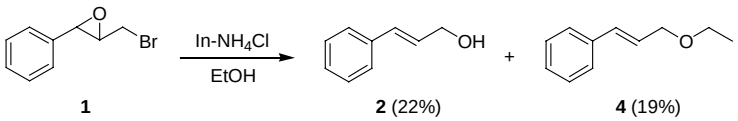
Method I



1 **2** (22%)

1-Bromo-2,3-epoxy-3-phenylpropane **1** (0.213 g, 1.0 mmol, 1.00eq) was dissolved in freshly distilled THF (20 ml) and deionized water (20 ml) was added to the solution. Indium metal powder (0.229 g, 2.0 mmol, 2.00 eq) was then added to the mixture and stirred vigorously for 48 h under a nitrogen atmosphere at r.t. before evaporation to dryness and extraction with ethyl acetate (100 ml). The ethyl acetate layer was washed with deionized water (3 × 75 ml), saturated brine solution (100 ml), evaporated to dryness and chromatographed on silica [ethyl acetate/dichloromethane = 1:9] to give cinnamyl alcohol **2** (0.030g, 22%) as a pale brown semi-solid which later crystallized as prisms on long standing, mp 30-32 ^{o}C (lit.[8a] mp 30-32 ^{o}C) [Found (FAB): MH^{+} 135.0816, $C_9H_{10}O$ requires *MH* 135.0810]; IR (CH_2Cl_2) 3367, 3105, 3082, 3060, 3028, 2921, 2866, 1656, 1599, 1578, 1494, 1449, 1412, 1379, 1296, 1266, 1203, 1092, 1070, 1009, 968, 920, 835, 802, 734, 694 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ2.48 (broad s, 1H,), 4.32 (dd, J = 5.7, 1.5 Hz, 2H), 6.38 (dt, J = 15.9, 5.7 Hz, 1H), 6.62 (d, J = 15.9 Hz, 1H), 7.24-7.30 (m, 1H), 7.31-7.37 (m, 2H), 7.38-7.44 (m, 2H); ^{13}C NMR (100.61 MHz, $CDCl_3$) δ64.3 (CH_2), 127.0 (CH), 128.2 (CH), 129.0 (CH), 129.1 (CH), 131.7 (CH), 137.2 (C); GC-MS retention time 12.08 min., *m/z* (EI) 134 ($M^{+\bullet}$, 26%), 133 (14), 116 (20), 115 (42), 105 (25), 103 (22), 92 (100), 91 (81), 89 (10), 79 (25), 78 (27), 77 (33), 65 (10), 63 (9), 51 (21). The spectroscopic data of the cinnamyl alcohol **2** were consistent with those of an authentic commercial sample and those reported in the literature.[8]

Method II



1 **2** (22%) **4** (19%)

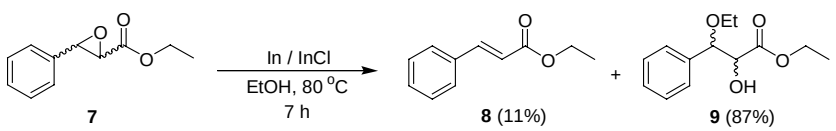
Under nitrogen, 1-bromo-2,3-epoxy-3-phenylpropane **1** (0.320 g, 1.5 mmol, 1.00 eq) was dissolved in ethanol (20 ml) at room temperature, indium metal powder (0.3445 g, 3.0 mmol, 2.00 eq) and

ammonium chloride (0.160 g, 3.0 mmol, 2.00 eq) were added to the solution, which was then heated to reflux at 78 ^{o}C for 6 h under nitrogen. Additional indium powder (0.1148 g, 1.0 mmol, 0.67 eq) was then added and the reflux was continued for another 18 h. When the reaction was complete (TLC), the mixture was filtered through Celite,® evaporated, and the residue partitioned between water (50 ml) and ethyl acetate (50 ml). The aqueous portion was extracted with ethyl acetate (50 ml) and the combined organic phases were washed with water (2 × 50 ml), dried, filtered and evaporated to obtain a brown semi-solid, which was purified by chromatography on silica gel to obtain 1-[(*E*)-3-ethoxyprop-1-enyl]benzene **4** (0.046 g, 19%) as a colorless liquid using dichloromethane/petroleum ether (2:3) as eluent and cinnamyl alcohol **2** (0.044 g, 22%) as a pale brown semi-solid using ethyl acetate/dichloromethane (1:9) as eluent.

1-[(*E*)-3-Ethoxyprop-1-enyl]benzene (4): [Found (ESI): MNH_4^+ 180.1384, $C_{11}H_{14}O$ requires *MNH*$_4$ 180.1383]; IR (neat) 3082, 3060, 3027, 2975, 2929, 2865, 2795, 1599, 1496, 1485, 1449, 1374, 1354, 1132, 1121, 1101, 1080, 1011, 966, 743, 692 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.30 (t, *J* = 7.0 Hz, 3H), 3.59 (q, *J* = 7.0 Hz, 2H), 4.18 (dd, *J* = 6.0, 1.5 Hz, 2H), 6.35 (dt, *J* = 15.9, 6.0 Hz, 1H), 6.65 (d, *J* = 15.9 Hz, 1H), 7.25-7.29 (m, 1H), 7.33-7.37 (m, 2H), 7.41-7.45 (m, 2H); ^{13}C NMR (100.61 MHz, $CDCl_3$) δ 15.4 (CH_3), 65.9 (CH_2), 71.4 (CH_2), 126.6 (CH), 126.7 (CH), 127.8 (CH), 128.7 (CH), 132.3 (CH), 137.0 (C); *m/z* (EI) 162 ($M^{+\bullet}$, 19%), 133 (23), 117 (76), 115 (183), 105 (100), 103 (25), 91 (86), 77 (47), 65 (17), 51 (33), 43 (34). The spectroscopic data of the ether **4** were consistent with literature values.[9]

Cinnamyl alcohol (2): The spectral data for the cinnamyl alcohol **2** were consistent with the data of the same compound obtained from the previous method and those reported in the literature.[8]

Deoxygenation of ethyl-(±)-3-phenylglycidate (7) (following general procedure B)



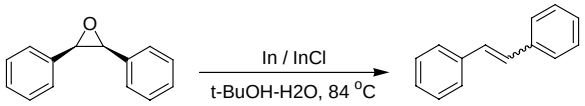
7 **8** (11%) **9** (87%)

Ethyl-(±)-3-phenylglycidate **7** (0.1045 g of 92% mixture of *cis* and *trans*, 0.5 mmol, 1.00 eq), afforded the *trans*-ethyl cinnamate **8** (0.010 g, 11%) and ethyl 3-ethoxy-2-hydroxy-3-phenylpropanoate[10] **9** (0.104 g, 87%) as colorless liquids:

Ethyl 3-ethoxy-2-hydroxy-3-phenylpropanoate[10] (9): [Found (ESI): MNH_4^+ 256.1546, $C_{13}H_{18}O_4$ requires *MNH₄* 256.1543]; IR (neat) 3474, 3088, 3064, 3032, 2978, 2933, 2876, 1739, 1495, 1455, 1370, 1302, 1263, 1203, 1103, 1075, 1024, 940, 888, 867, 753, 702 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.20 (t, *J* = 7.1 Hz, 3H), 1.22 (t, *J* = 7.0 Hz, 3H), 2.90 (broad s, 1H), 3.44 (dq, *J* = 9.3, 7.0 Hz, 1H), 3.52 (dq, *J* = 9.3, 7.0 Hz, 1H), 4.15 (q, *J* = 7.1 Hz, 2H), 4.46 (broad s, 1H), 4.63 and 4.67 (2 × d, *J* = 4.2 and 3.3 Hz, 1H), 7.29-7.37 (5H, m, ArH); ^{13}C NMR (100.61 MHz, $CDCl_3$) δ 14.2 (CH_3, major), 14.3 (CH_3, minor), 15.2 (CH_3, minor), 15.4 (CH_3, major), 61.6 (CH_2, major), 61.7 (CH_2, minor), 65.2 (CH_2, minor), 65.3 (CH_2, major), 74.7 (CH, major), 75.4 (CH, minor), 82.1 (CH, minor), 82.9 (CH, major), 127.4 (CH, minor), 127.6 (CH, major), 128.2 (CH, minor), 128.3 (CH, major), 128.4 (CH, major), 128.5 (CH, minor), 137.3 (C, major), 138.1 (C, minor), 172.1 (C, major), 172.2 (C, minor); *m/z* (CI) 256 [$(M+NH_4)^+$, 100%], 239 (5), 221 (3), 212 (7), 210 (24), 196 (3), 194 (8), 177 (4), 162 (4), 137 (4), 135 (8).

Ethyl cinnamate (8): Data for ethyl cinnamate (**8**) were as reported in the Experimental Section of this paper.

Deoxygenation of *cis*-stilbene oxide (10) (following General Procedure C)



10 **6** (84%, *E* : *Z* = 3 :1)

cis-Stilbene oxide **10** (0.098 g, 0.5 mmol, 1.00 eq), yielded stilbene **6** (0.076 g, 84%) as an inseparable mixture of *cis* and *trans*- isomers in the ratio of 1:3 respectively.

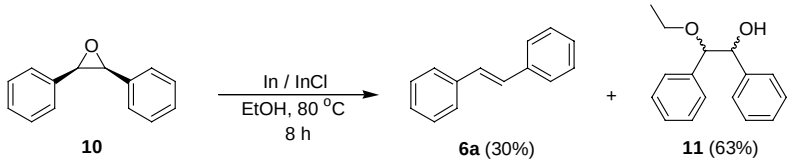
The ratio of the *cis* and *trans* isomers was determined by ^1H-NMR spectral analysis. The presence of *cis* and *trans*-stilbene was further verified by GC-MS analysis comparing with those of an authentic

sample from Aldrich. The spectroscopic data and GC-MS data of the *cis* and *trans*-stilbene were consistent with those of an authentic commercial sample and with literature values.[11]

***trans*-Stilbene:** ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.16 (s, 2H), 7.28-7.34 (m, 2H), 7.37-7.46 (m, 4H), 7.53-7.62 (m, 4H); ^{13}C NMR (100.61 MHz, $CDCl_3$) δ 126.7 (CH), 127.8 (CH), 128.9 (CH), 129.0 (CH), 137.6 (C); GC-MS retention time 14.72 min., *m/z* (EI) 180 [$M^{+\bullet}$, 100%], 179 (86), 178 (57), 177 (8), 176 (8), 166 (13), 165 (37), 153 (4), 152 (9), 139 (1), 115 (1), 102 (2), 89 (4), 77 (2), 76 (5), 63 (2), 51 (2).

***cis*-Stilbene:** ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.65 (s, 2H), 7.20-7.30 (m, 10H); ^{13}C NMR (100.61 MHz, $CDCl_3$) δ 127.3 (CH), 128.4 (CH), 129.1 (CH), 130.4 (CH), 137.4 (C); GC-MS retention time 13.60 min., *m/z* (EI) 180 [$M^{+\bullet}$, 99%], 179 (100), 178 (77), 177 (9), 176 (14), 165 (56), 152 (18), 151 (5), 139 (2), 115 (2), 102 (4), 89 (7), 77 (2), 76 (9), 63 (4), 51 (5).

Deoxygenation of *cis*-stilbene oxide (10) (following General Procedure B)



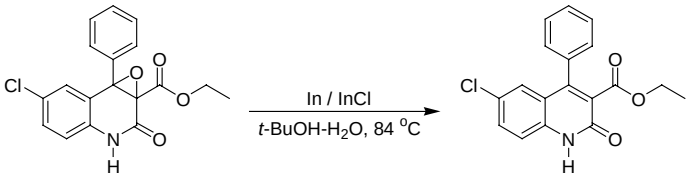
10 **6a** (30%) **11** (63%)

cis-Stilbene oxide **10** was deoxygenated following the general procedure **B** by heating a mixture of *cis*-stilbene oxide **10** (0.098 g, 0.5 mmol, 1.00 eq), indium metal powder (0.201 g, 1.75 mmol, 3.50 eq), indium(I) chloride (0.113 g, 0.75 mmol, 1.50 eq) in absolute ethanol (15 ml) at 80 ^{o}C for 8 h under an argon atmosphere to yield a colorless oil. The crude material was purified by flash chromatography on silica gel to afford *trans*-stilbene **6a** (0.027g, 30%) as a colorless solid using ethyl acetate/petroleum ether (1:49) as the eluant and 2-ethoxy-1,2-diphenylethanol **11** (0.076g, 63%) as a colorless semi-solid using ethyl acetate/petroleum ether (1:9) as the eluant.

***trans*-Stilbene (6a):** The IR, ^{1}H-NMR, ^{13}C-NMR, LRMS and HRMS data of *trans*-stilbene **6a** were consistent with those reported above for the same compound and with literature values.[8a,11]

2-Ethoxy-1,2-diphenylethanol (11): [Found (ESI): MNH_4^+ 260.1645, $C_{16}H_{18}O_2$ requires *MNH*$_4$ 260.1645]; IR (neat) 3550, 3469, 3087, 3063, 3031, 2975, 2928, 2876, 1604, 1494, 1454, 1389, 1341, 1319, 1298, 1259, 1223, 1197, 1157, 1091, 1074, 1060, 1014, 916, 900, 851, 820, 766 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.33 (t, *J* = 7.0 Hz, 3H), 3.47-3.61 (m, 2H), 3.74 (broad s, 1H), 4.32 (d, *J* = 8.3 Hz, 1H), 4.75 (d, *J* = 8.3 Hz, 1H), 7.09-7.17 (m, 4H), 7.24-7.32 (m, 6H); ^{13}C NMR (100.61 MHz, $CDCl_3$) δ 15.5 (CH_3), 64.7 (CH_2), 78.8 (CH), 87.6 (CH), 127.5 (CH), 127.8 (CH), 127.9 (CH), 128.0 (CH), 128.1 (CH), 128.2 (CH), 138.3 (C), 139.5 (C); *m/z* (CI) 260 [$(M+NH_4)^+$, 99%], 242 (27), 225 (100), 214 (24), 196 (50), 135 (21). The ^{1}H NMR data of 2-ethoxy-1,2-diphenylethanol **11** was consistent with those reported in the literature.[12]

Deoxygenation of Ethyl 6-chloro-3,4-epoxy-4-phenyl-1,2,3,4-tetrahydroquinolin-2-one-3-carboxylate (12) (following General Procedure C)

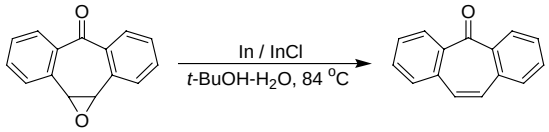


12 **13** (94%)

Ethyl 6-chloro-3,4-epoxy-4-phenyl-1,2,3,4-tetrahydroquinolin-2-one-3-carboxylate **12** (0.103 g, 0.3 mmol, 1.00 eq) was deoxygenated following the general procedure **C** to afford ethyl-6-chloro-1,2-dihydro-2-oxo-4-phenylquinoline-3-carboxylate **13** (0.092 g, 94%) as a white powder, mp 223-224 ^{o}C (lit.[13] mp 223-224 ^{o}C) [Found (ESI): MH^+ 328.0734, $C_{18}H_{14}ClNO_3$ requires *MH* 328.0735]; IR (KBr) 3149, 2986, 2912, 2873, 1739, 1645, 1556, 1485, 1444, 1411, 1374, 1344, 1301, 1277, 1242, 1176,

1142, 1089, 1007, 955, 887, 856, 826, 826, 776, 756, 706 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.98 (t, J = 7.1 Hz, 3H), 4.14 (q, J = 7.1 Hz, 2H), 7.26-7.28 (m, 1H), 7.36-7.42 (m, 2H), 7.48-7.56 (m, 5H), 13.6 (broad s, 1H); ^{13}C NMR (100.61 MHz, $CDCl_3$) δ 14.2 (CH_3), 62.0 (CH_2), 118.9 (CH), 121.1 (C), 127.2 (CH), 127.6 (C), 129.0 (C), 129.1 (CH), 129.2 (CH), 129.8 (CH), 132.4 (CH), 134.4 (C), 137.4 (C), 150.2 (C), 161.6 (C), 165.7 (C); *m/z* (EI) 327 [$M^{+\bullet}$, 25%], 284 (14), 283 (13), 282 (40), 256 (25), 255 (29), 254 (71), 253 (31), 236 (11), 220 (22), 219 (20), 202 (12), 201 (20), 200 (9), 199 (32), 191 (40), 190 (100), 189 (16), 188 (17), 176 (8), 165 (15), 164 (44), 163 (69), 162 (15), 150 (10), 137 (10), 123 (10), 113 (10), 99 (8), 88 (10), 87 (15), 77 (16), 75 (18), 63 (20), 51 (22), 44 (54).

Deoxygenation of 10,11-epoxy-10,11-dihydro-dibenzo[*a*,*d*]cyclohepten-5-one (14) (following General Procedure C)

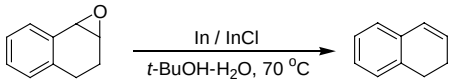


14 **15** (81%)

10,11-Epoxy-10,11-dihydrodibenzo[*a*,*d*]cyclohepten-5-one **14** (0.111 g, 0.5 mmol, 1.00 eq) was deoxygenated following general procedure **C** to afford 10,11-epoxy-10,11-dihydro-dibenzo[*a*,*d*]cyclohepten-5-one **15** (dibenzosuberenone) (0.084 g, 81%) as a pale-yellow solid, mp 88-89 °C (lit.[14a] mp 89 °C) [Found (ESI): MH^+ 207.0805, $C_{15}H_{10}O$ requires *MH* 207.0804]; IR (KBr) 3057, 3024, 2979, 1647, 1590, 1481, 1458, 1427, 1416, 1318, 1300, 1244, 1228, 1208, 1168, 1154, 1113, 1039, 990, 956, 932, 886, 857, 807, 794, 770, 722, 691 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.09 (s, 2H), 7.54-7.62 (m, 4H), 7.66 (ddd, J = 7.5, 7.4, 1.1 Hz, 2H), 8.27 (d, J = 8.0 Hz, 2H); ^{13}C NMR (100.61 MHz, $CDCl_3$) δ 129.3 (CH), 130.7 (CH), 131.3 (CH), 132.2 (CH), 132.4 (CH), 135.4 (C), 139.2 (C), 193.5 (C); *m/z* (EI) 206 [$M^{+\bullet}$, 33%], 178 (94), 176 (24), 152 (20), 139 (6), 126 (7), 113 (7), 102 (8), 98 (10), 89 (16), 87 (21), 76 (24), 74 (38), 63 (57), 50 (79), 39 (100). The spectroscopic data

of the dibenzosuberenone **15** were consistent with those of an authentic commercial sample and with those reported in the literature.[14b]

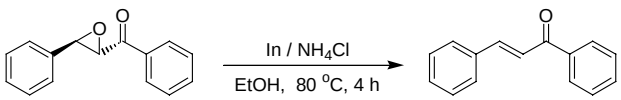
Deoxygenation of 1a,2,3,7b-tetrahydro-1-oxa-cyclopropa[*a*]naphthalene (16) (following General Procedure C)



16 **17** (69%)

1a,2,3,7b-Tetrahydro-1-oxacyclopropa[*a*]naphthalene **16** (0.102 g, 0.7 mmol, 1.00 eq) was deoxygenated following the general procedure **C** to afford 1,2-dihydronaphthalene **17** (0.063 g, 69%) as a colorless liquid with pleasant odour [Found (EI): $M^{+\bullet}$ 130.0783, $C_{10}H_{10}$ requires *M* 130.0782]; IR (neat) 3032, 2931, 2888, 2880, 1484, 1452, 1438, 1428, 1326, 1278, 1223, 1220, 1159, 1116, 1032, 1026, 1010, 939, 885, 868, 781, 744 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 2.31 (m, 2H), 2.79 (t, J = 8.2 Hz, 2H), 6.02 (dt, J = 9.5, 4.3 Hz, 1H), 6.45 (dt, J = 9.5, 1.9 Hz, 1H), 6.98-7.04 (m, 1H), 7.05-7.18 (m, 3H); ^{13}C NMR (75.5 MHz, $CDCl_3$) δ 23.6 (CH_2), 27.9 (CH_2), 126.3 (CH),126.9 (CH), 127.3 (CH), 128.0 (CH), 128.2 (CH), 129.1 (CH), 134.6 (C), 135.9 (C); *m/z* (EI) 130 [$M^{+\bullet}$, 100%], 129 (58), 128 (18), 127 (10), 115 (25), 102 (3), 77 (4). The spectroscopic data of the 1,2-dihydronapthalene **17** were consistent with those of an authentic commercial sample and with those reported in the literature.[15]

Deoxygenation of *trans*-2,3-epoxy-1,3-diphenyl-1-propanone (18) (following General Procedure A)

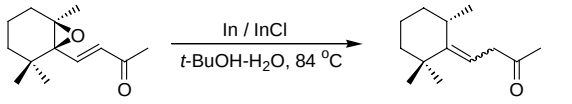


18 **19** (51%)

trans-2,3-Epoxy-1,3-diphenyl-1-propanone **18** (0.561 g, 2.5 mmol, 1.00 eq) was deoxygenated following general procedure **A** to yield (*E*)-1,3-diphenylpropenone **19** (0.264 g, 51%) as a pale-yellow solid, mp 55-56 °C (lit.[16] mp 55-56 °C) [Found (ESI): MH^+ 209.0963, $C_{15}H_{12}O$ requires *MH* 209.0961];

IR (KBr) 3083, 3051, 3023, 1664, 1607, 1575, 1495, 1448, 1337, 1308, 1287, 1215, 1182, 1156, 1033, 1015, 989, 927, 852, 786, 752 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.41-7.46 (m, 3H), 7.49-7.54 (m, 2H), 7.56 (d, *J* = 15.8 Hz, 1H), 7.58-7.63 (m, 1H), 7.65-7.70 (m, 2H), 7.83 (d, *J* = 15.8 Hz, 1H), 8.00-8.07 (m, 2H); ^{13}C NMR (100.61 MHz, $CDCl_3$) δ 122.4 (CH), 128.6 (CH), 128.7 (CH), 128.9 (CH), 129.2 (CH), 130.8 (CH), 133.0 (CH), 135.2 (C), 138.5 (C), 145.1 (CH), 190.8 (C); *m/z* (EI) 208 [$M^{+\bullet}$, 33%], 207 (44), 179 (14), 165 (8), 152 (3), 131 (22), 115 (2), 105 (52), 103 (33), 89 (5), 77 (100), 63 (9), 51 (83), 39 (13). The spectroscopic data of the (*E*)-1,3-diphenylpropenone were consistent with those of an authentic commercial sample and with those reported in the literature.[17]

Deoxygenation of (*E*)-4-{(1*S*,6*R*)-2,2,6-trimethyl-7-oxabicyclo[4.1.0]hept-1-yl}but-3-en-2-one (20) (following General Procedure C)



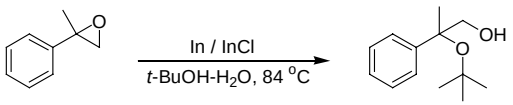
20 **21** (81%, *E* : *Z* = 1 : 0.7)

(*E*)-4-{(1*S*,6*R*)-2,2,6-Trimethyl-7-oxabicyclo[4.1.0]hept-1-yl}but-3-en-2-one **20** (0.104 g, 0.5 mmol, 1.00 eq) was deoxygenated following general procedure **C** and afforded a 0.7 : 1 mixture of (*Z*)- and (*E*)-4-[(*S*)-2,2,6-trimethylcyclohexylidene]butan-2-one **21** (0.079 g, 81%) as a colorless oil with pleasant odour; IR (neat) 2956, 2928, 2869, 1721, 1463, 1357, 1160, 983, 868, 668 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.04 (*Z*) and 1.12 (*E*) (2 × d, *J* = 6.6 and 7.5 Hz, 3H), 1.11 (*E*), 1.13 (*Z*) and 1.27 (*Z*) (3 × s, 6H), 1.28-1.86 (*E*) and (*Z*) (m, 6H), 2.16 (*E*) and 2.17 (*Z*) (2 × s, 3H), 2.28-2.41 (*Z*) and 2.76-2.87 (*E*) (2 × m, 1H), 3.11 (*E*), 3.21 (*E*) and 3.42 (*Z*) (3 × dd, *J* = 17.3, 7.1; 17.3, 7.1 and 7.2, 1.2 Hz, 2H), 5.30 (*Z*) and 5.47 (*E*) (td and t, *J* = 7.2, 1.6 and 7.1 Hz, 1H); ^{13}C NMR (100.61 MHz, $CDCl_3$) (*E* + *Z*) 18.1 (CH_2), 20.9 (CH_3), 21.4 (CH_2), 21.6 (CH_3), 28.7 (CH_3), 28.9 (CH), 30.0 (CH_3), 30.2 (CH), 30.7 (CH_3), 31.3 (CH_3), 32.0 (CH_3), 33.1 (CH_2), 35.5 (CH_2), 35.6 (CH_3), 36.8 (C), 38.3 (C), 41.7 (CH_2), 42.5 (CH_2), 43.2 (CH_2), 44.7 (CH_2), 112.2 (CH), 113.4 (CH), 152.4 (C), 153.7 (C), 208.1 (C),

208.2 (C); (***E)*-isomer**: GC-MS retention time 12.97 min., *m/z* (EI) 195 [$(M+1)^+$, 14%], 194 (2), 179 (20), 177 (73), 176 (52), 161 (100), 151 (4), 149 (3), 147 (4), 137 (7), 136 (20), 133 (20), 123 (19), 122 (8), 121 (76), 120 (13), 119 (34), 109 (23), 107 (22), 106 (36), 105 (30), 95 (40), 94 (11), 93 (37), 91 (26), 81 (40), 79 (33), 77 (16), 67 (32), 65 (8), 55 (6), 43 (21), 41 (26); **(*Z)*-isomer**: GC-MS retention time 13.16 min., *m/z* (EI) 195 [$(M+1)^+$, 11%], 194 (2), 179 (18), 178 (10), 177 (68), 176 (43), 161 (100), 151 (3), 149 (3), 147 (4), 137 (7), 136 (19), 133 (21), 123 (20), 122 (8), 121 (67), 120 (12), 119 (32), 109 (22), 108 (8), 107 (22), 106 (36), 105 (25), 95 (41), 93 (33), 91 (24), 81 (40), 79 (34), 77 (16), 69 (9), 67 (36), 65 (8), 55 (7), 53 (4), 51 (3), 43 (22), 41 (28). The IR and ^{1}H-NMR spectroscopic data of the alkene **21** were in accordance with the literature.[18]

The *E*:*Z* ratio of the alkene **21** was estimated from the ^{1}H-NMR spectrum and further verified by GC-MS analysis. *E* and *Z* isomers and the stereochemistry of the alkene **21** were determined by 1D ^{1}H nOe, 2D ^{1}H NOESY and DQFCOSY NMR experiments.

Attempted deoxygenation reaction of 2-methyl-2-phenyloxirane (22) (following General Procedure C)

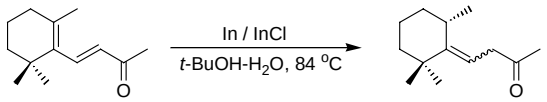


22 **31** (24%)

Attempted deoxygenation reaction of 2-methyl-2-phenyloxirane **22** was performed following general procedure **C** by heating a mixture of 2-methyl-2-phenyloxirane **22** (0.134 g, 1.0 mmol, 1.00 eq), indium metal powder (0.345 g, 3.0 mmol, 3.00 eq), indium(I) chloride (0.225 g, 1.5 mmol, 1.50 eq) in *t*-butanol (4.8 ml) and water (0.2 ml) at 84 ^{o}C for 4 h under an argon atmosphere to yield a colorless oil. Purification of the oil by chromatography on silica [ethyl acetate/petroleum ether = 1:4] afforded 2-*tert*-butoxy-2-phenylpropan-1-ol **31** (0.051 g, 24%) as a colorless oil; [Found (ESI): MNH_4^+ 226.1801, $C_{13}H_{20}O_2$ requires *MNH4* 226.1802]; IR (neat) 3449, 3060, 2976, 2930, 2873, 1492, 1447, 1391, 1365, 1229, 1191, 1168, 1124, 1071, 1046, 1026, 986, 921, 837, 760, 701 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$)

δ 1.17 (s, 9H), 1.84 (s, 3H), 2.34 (broad s, 1H), 3.21 (d, J = 10.6 Hz, 1H), 3.56 (d, J = 10.6 Hz, 1H), 7.25-7.30 (m, 1H), 7.31-7.37 (m, 2H), 7.46-7.53 (m, 2H); ^{13}C NMR (100.61 MHz, $CDCl_3$) δ 22.2 (CH_3), 31.8 (CH_3), 75.0 (CH_2), 76.0 (C), 79.3 (C), 127.0 (CH), 127.6 (CH), 128.4 (CH), 146.6 (C); *m/z* (CI) 226 [$(M+NH_4)^+$, 50%], 191 (57), 152 (100), 134 (4).

Reduction of β-ionone (26) by indium and indium(I) chloride



26 **21** (84%, *E* : *Z* = 1 : 0.8)

Indium metal powder (0.230 g, 2.0 mmol, 4.00 eq), indium(I) chloride (0.150 g, 1.0 mmol, 2.00 eq) were added to a stirred solution of β-ionone **26** (0.096 g, 0.5 mmol, 1.00 eq) in *t*-butanol (4.8 ml) and water (0.2 ml) at 30 ^{o}C. The mixture was heated to reflux at 84 ^{o}C under argon. A white turbidity appeared in the reaction mixture intially which later turned to deep white-grey precipitate. After 30 h, the reaction mixture was cooled and washed through Celite® with ethyl acetate (100 ml) using sintered funnel. The filterate was evaporated to dryness under reduced pressure to a brown oily residue. This was redissolved in ethyl acetate (100 ml) and washed with brine solution (3 × 100 ml) using a separating funnel. The organic layer was separated, dried over anhydrous sodium sulfate, filtered, concentrated *in vacuo* to obtain a brown oil residue. Purification of the crude material by flash chromatography on silica [ethyl acetate/petroleum ether = 7:93] afforded an inseparable 0.8 : 1 mixture of (*Z*)- and (*E*)-4-[(*S*)-2,2,6-trimethylcyclo hexylidene]butan-2-one **21** (0.082 g, 84%) as a colorless oil with a pleasant odour. The spectral data of the alkene **21** were consistent with those of the same compound reported earlier and with the literature.[18]

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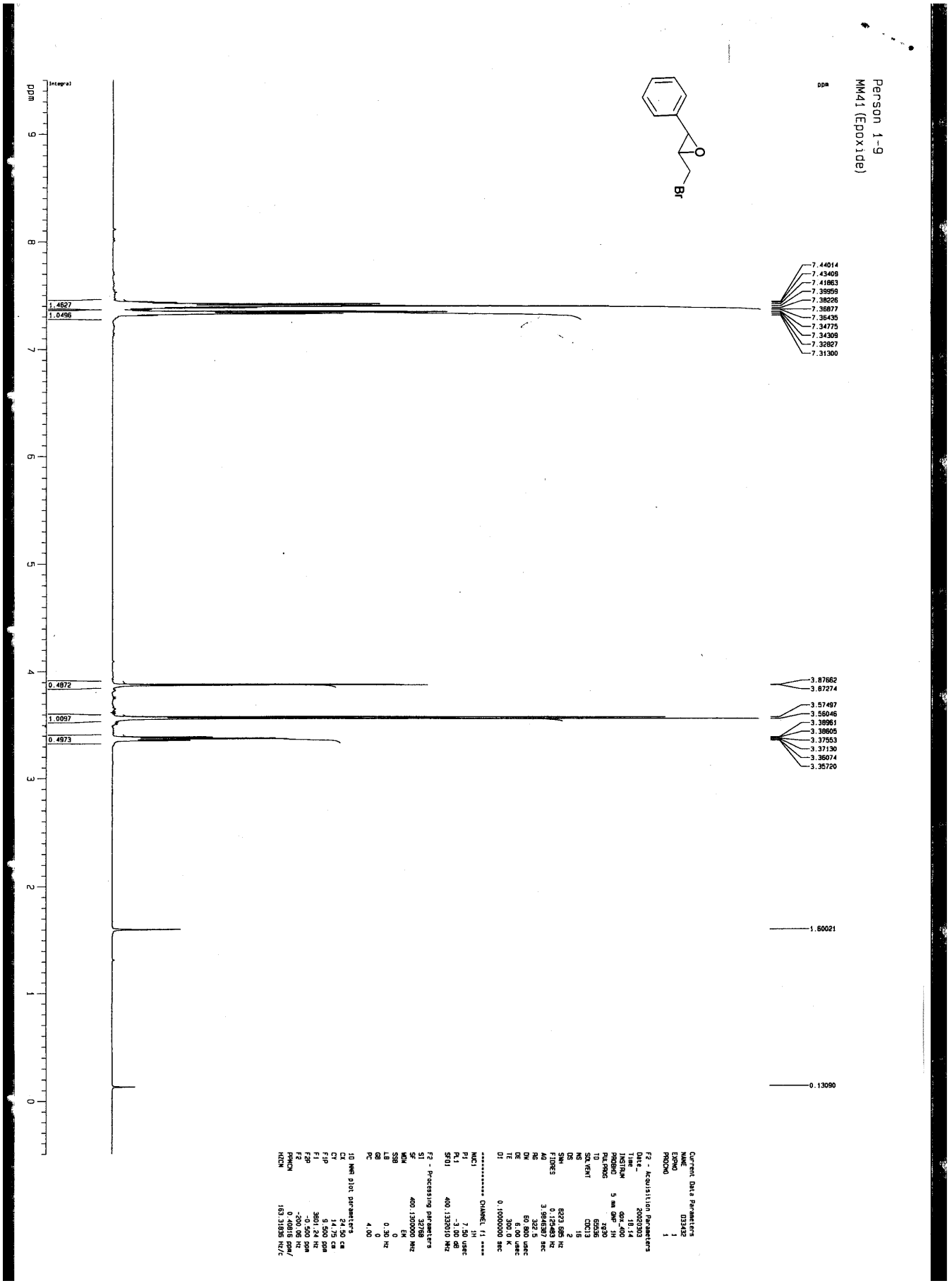
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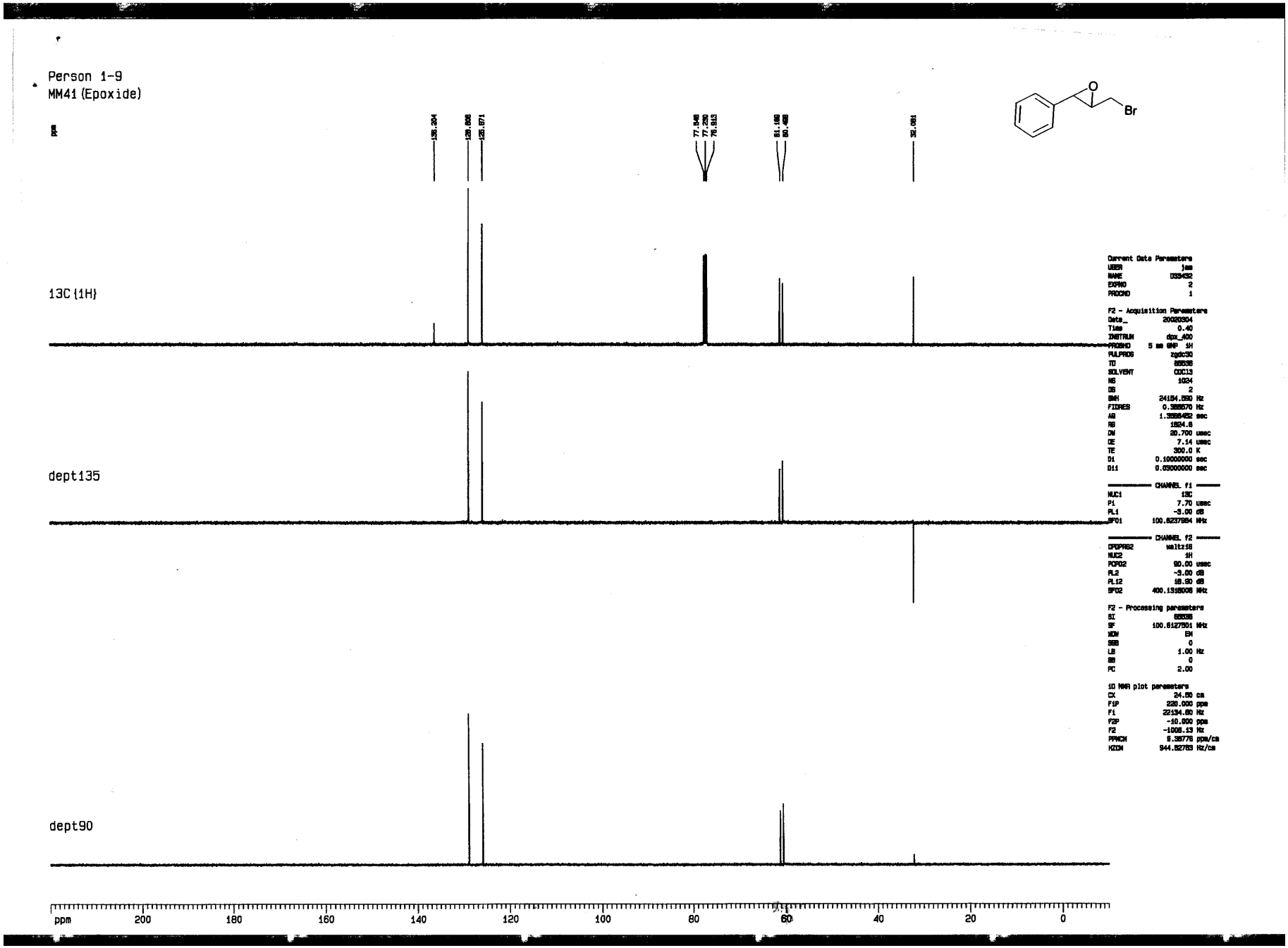
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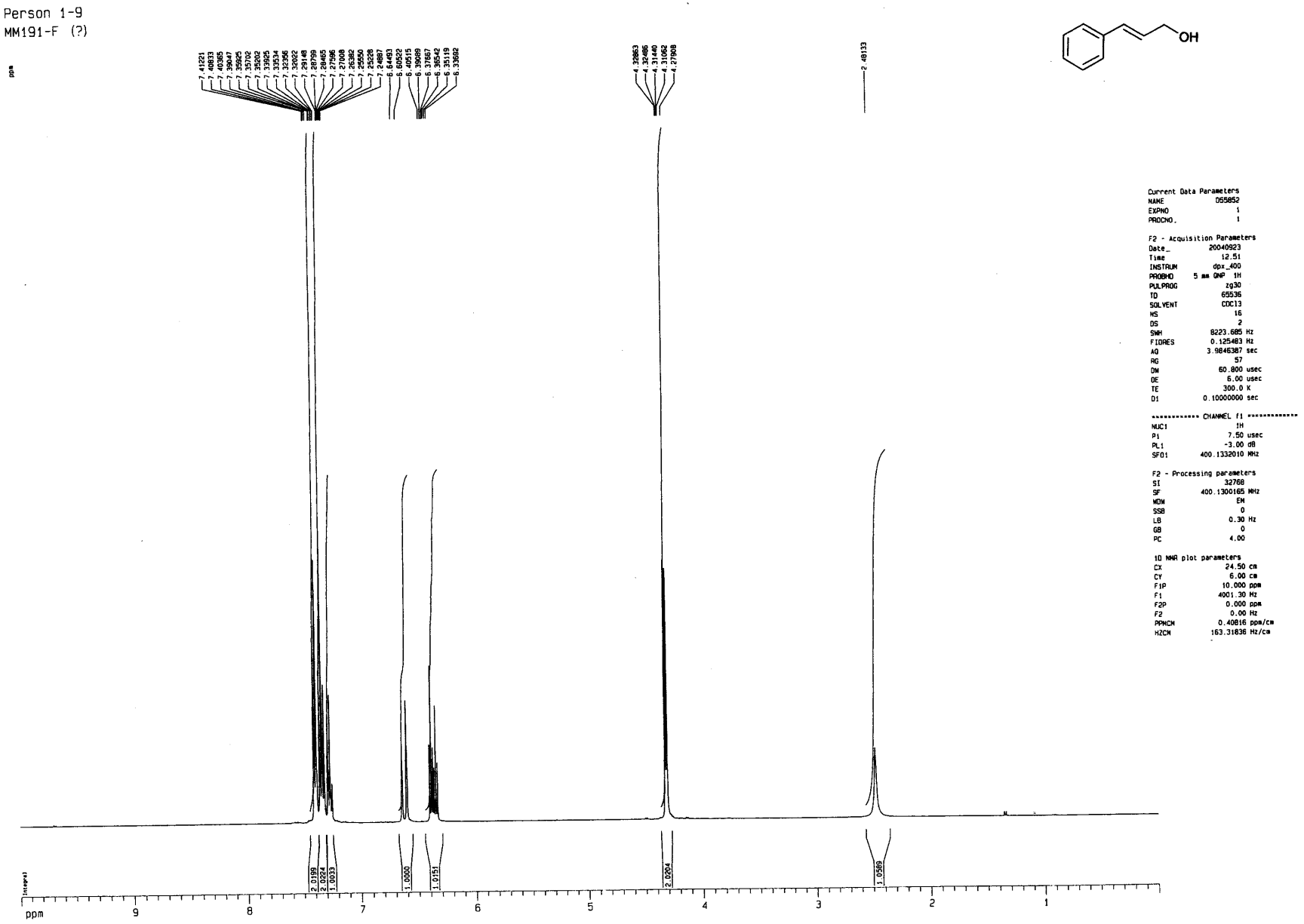

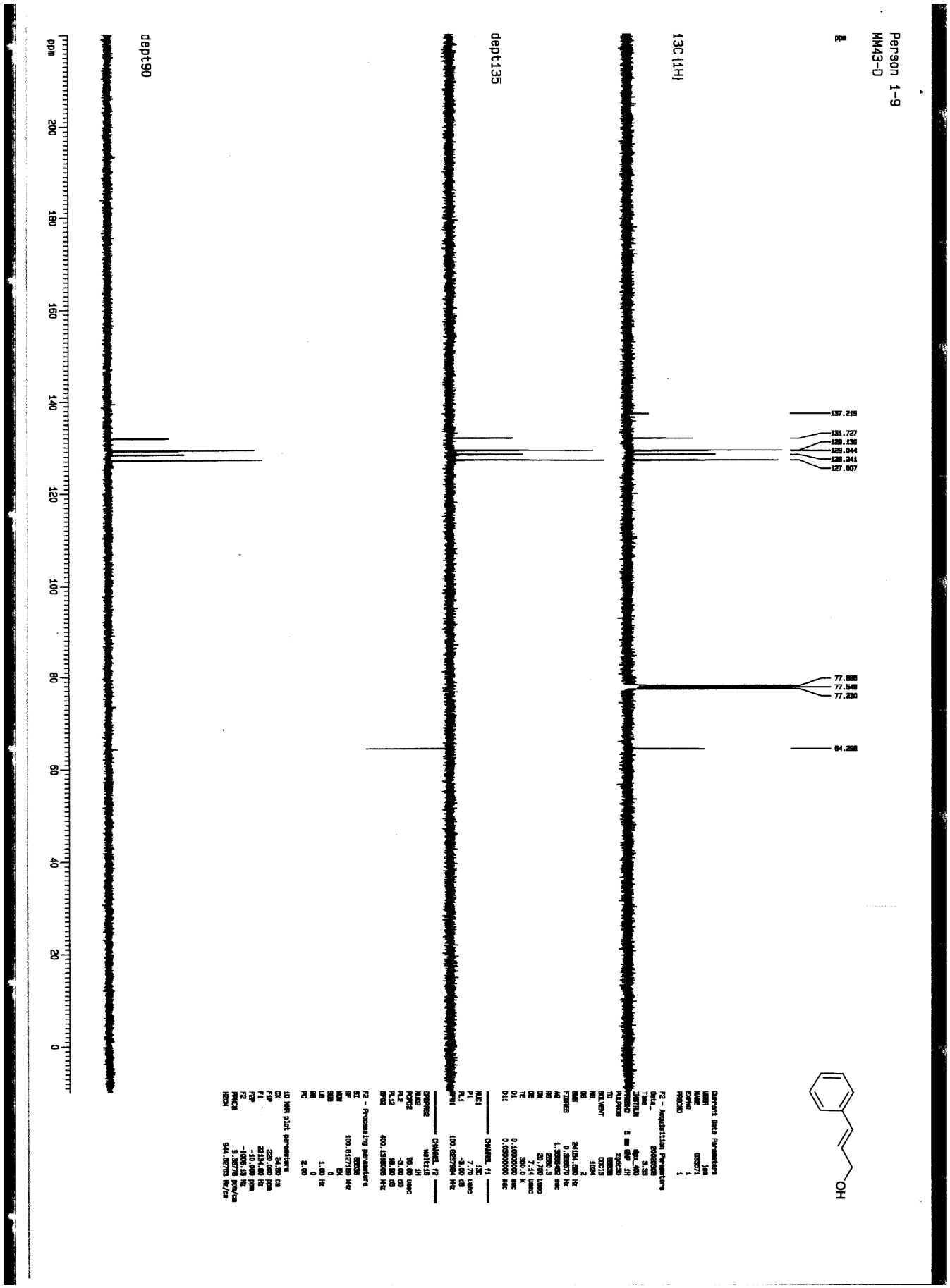
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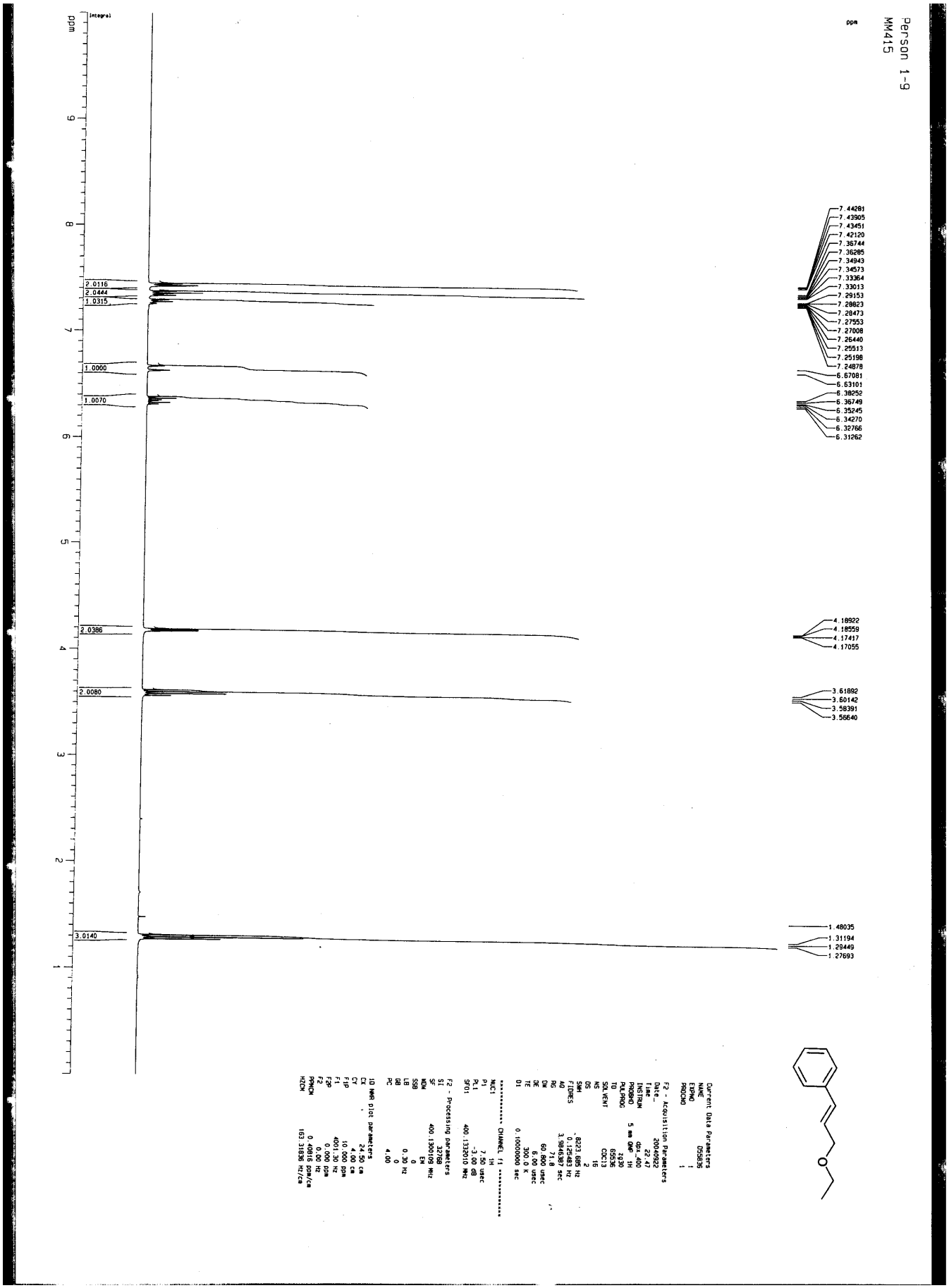
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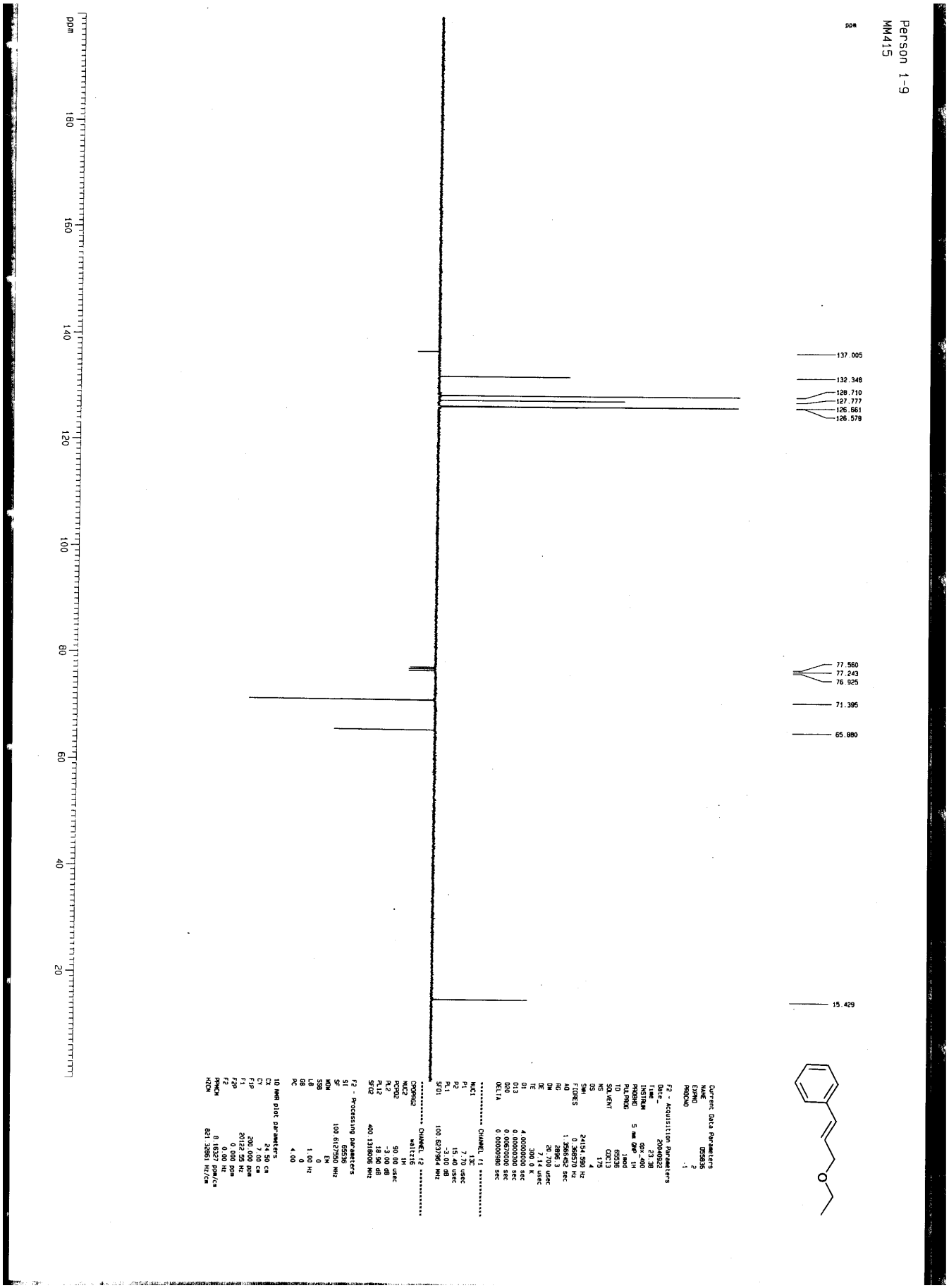
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21 Apr 2004

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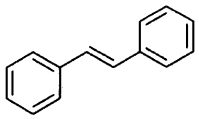

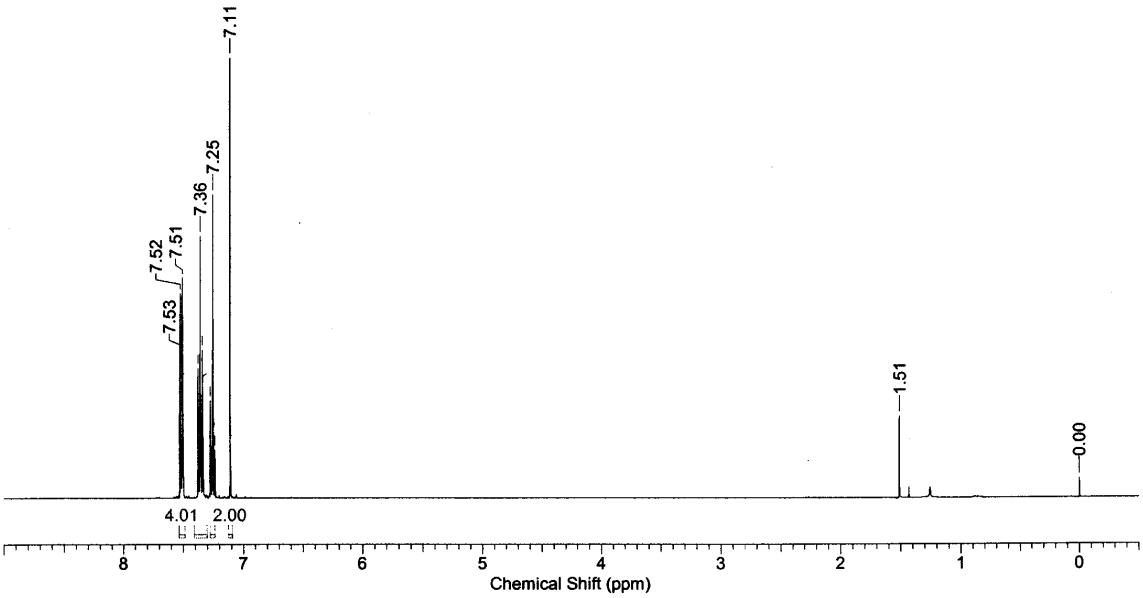

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16 Apr 2004

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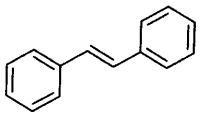
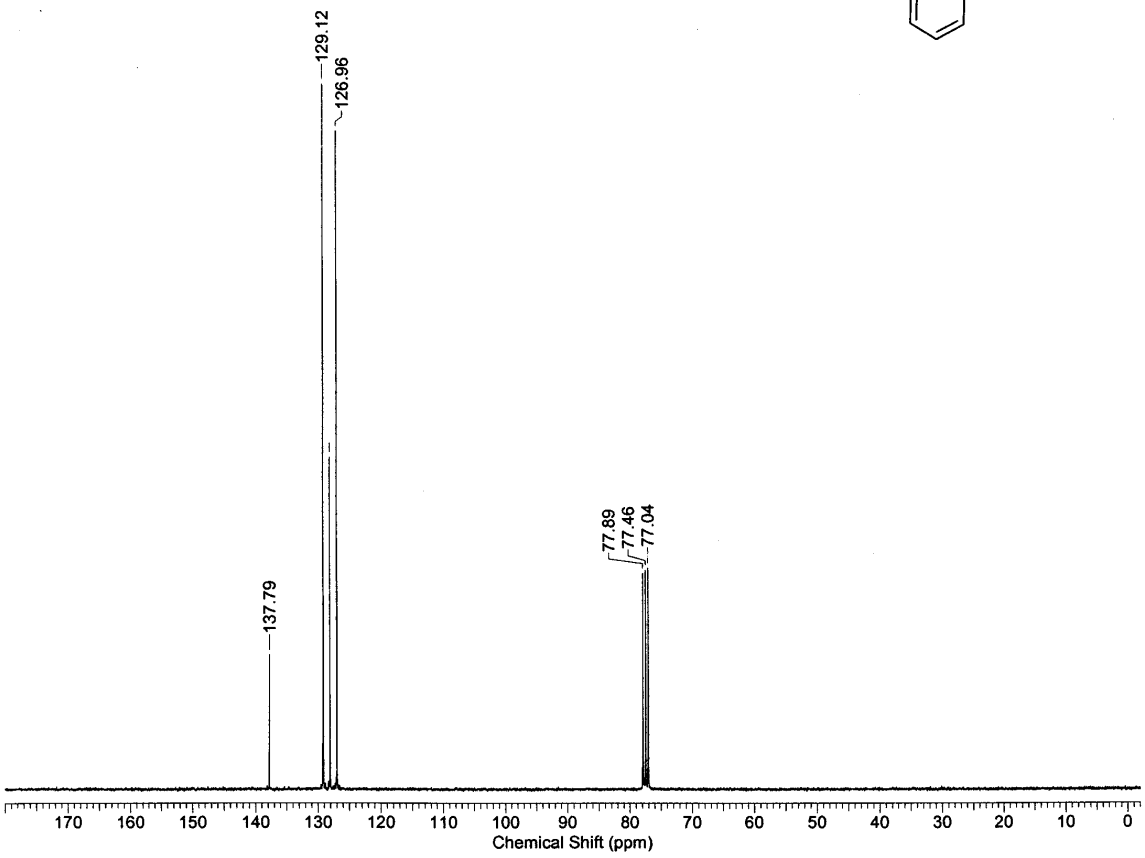
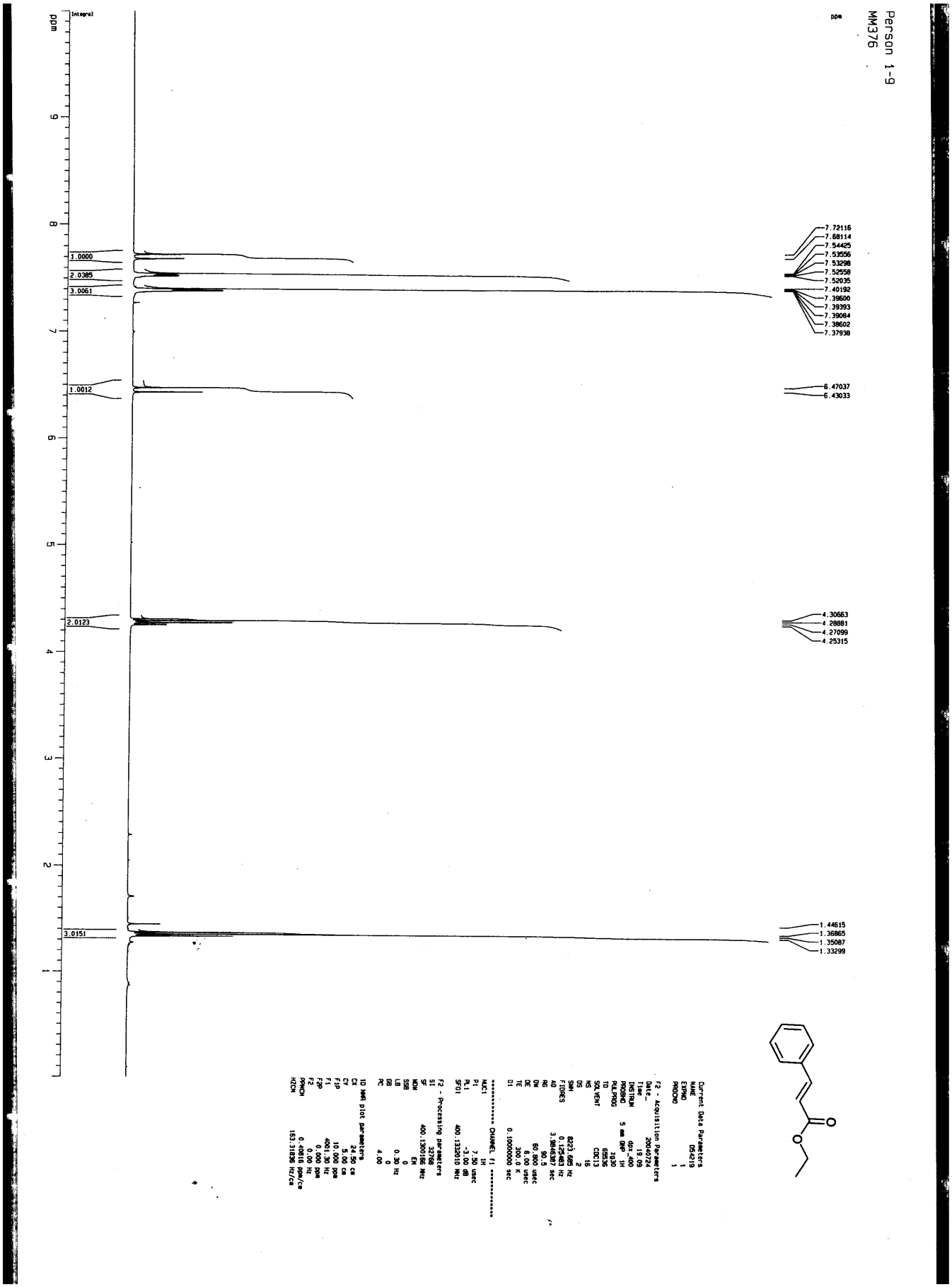
Points Count	32768	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	18939.39		

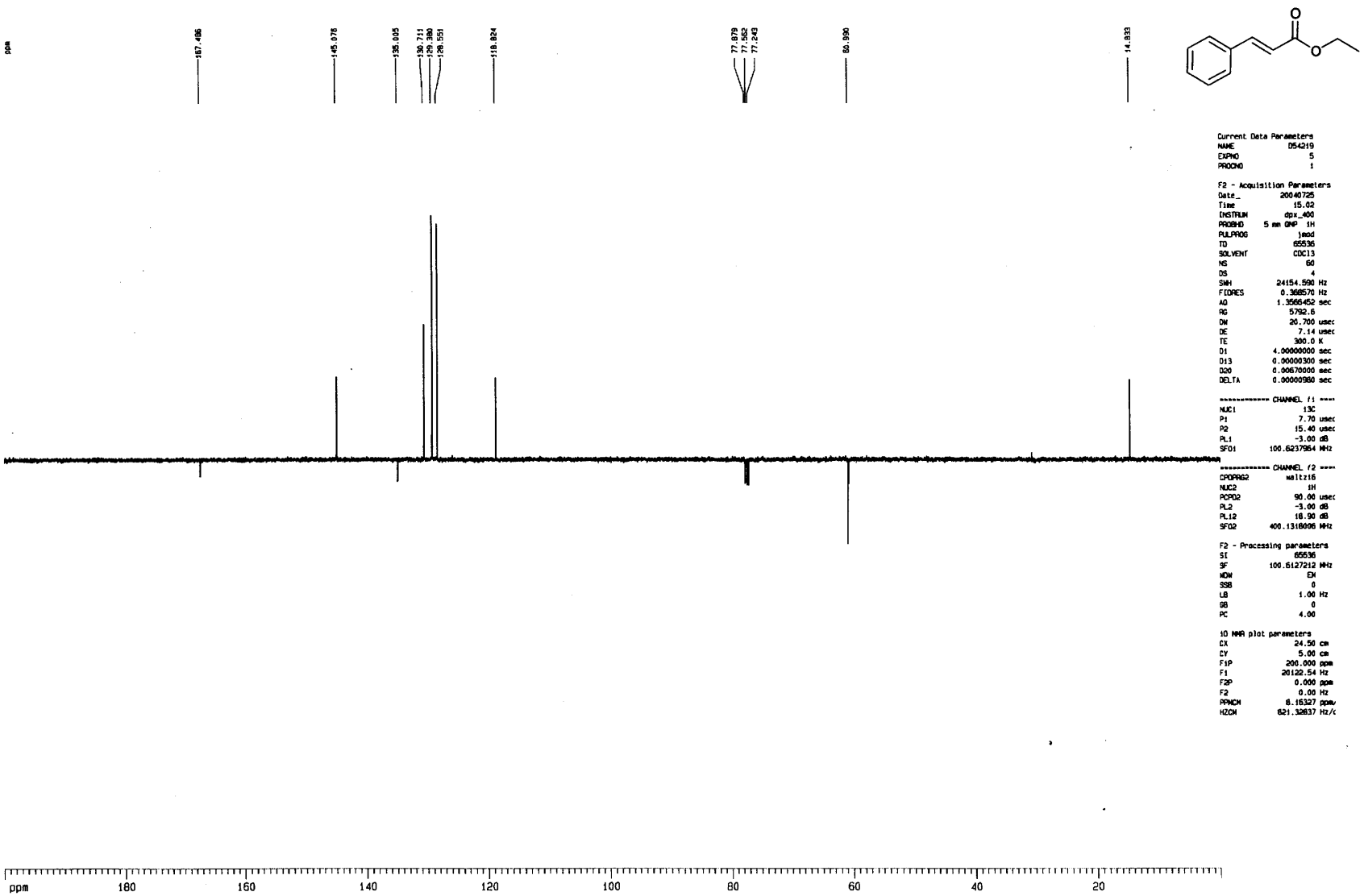


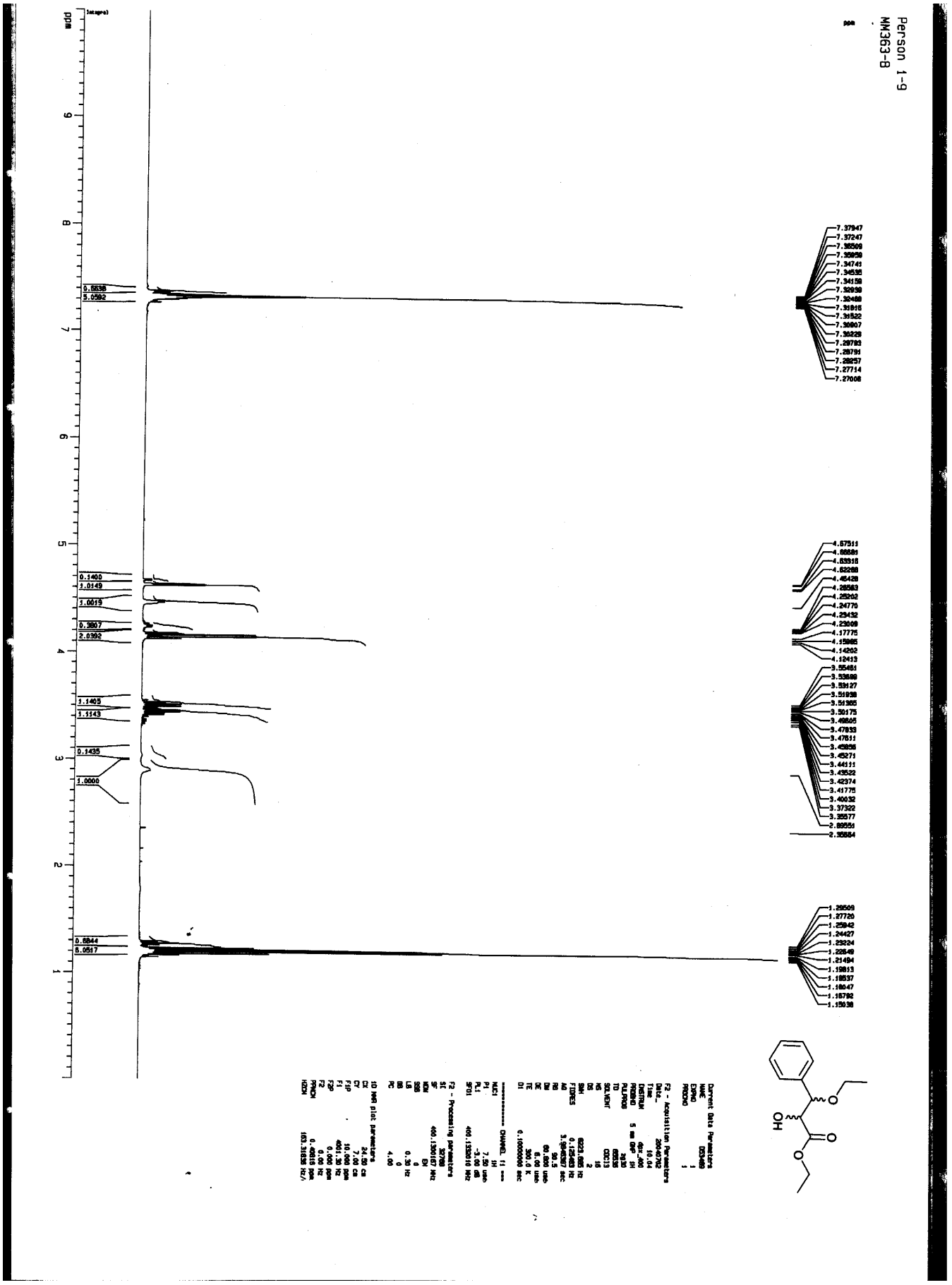


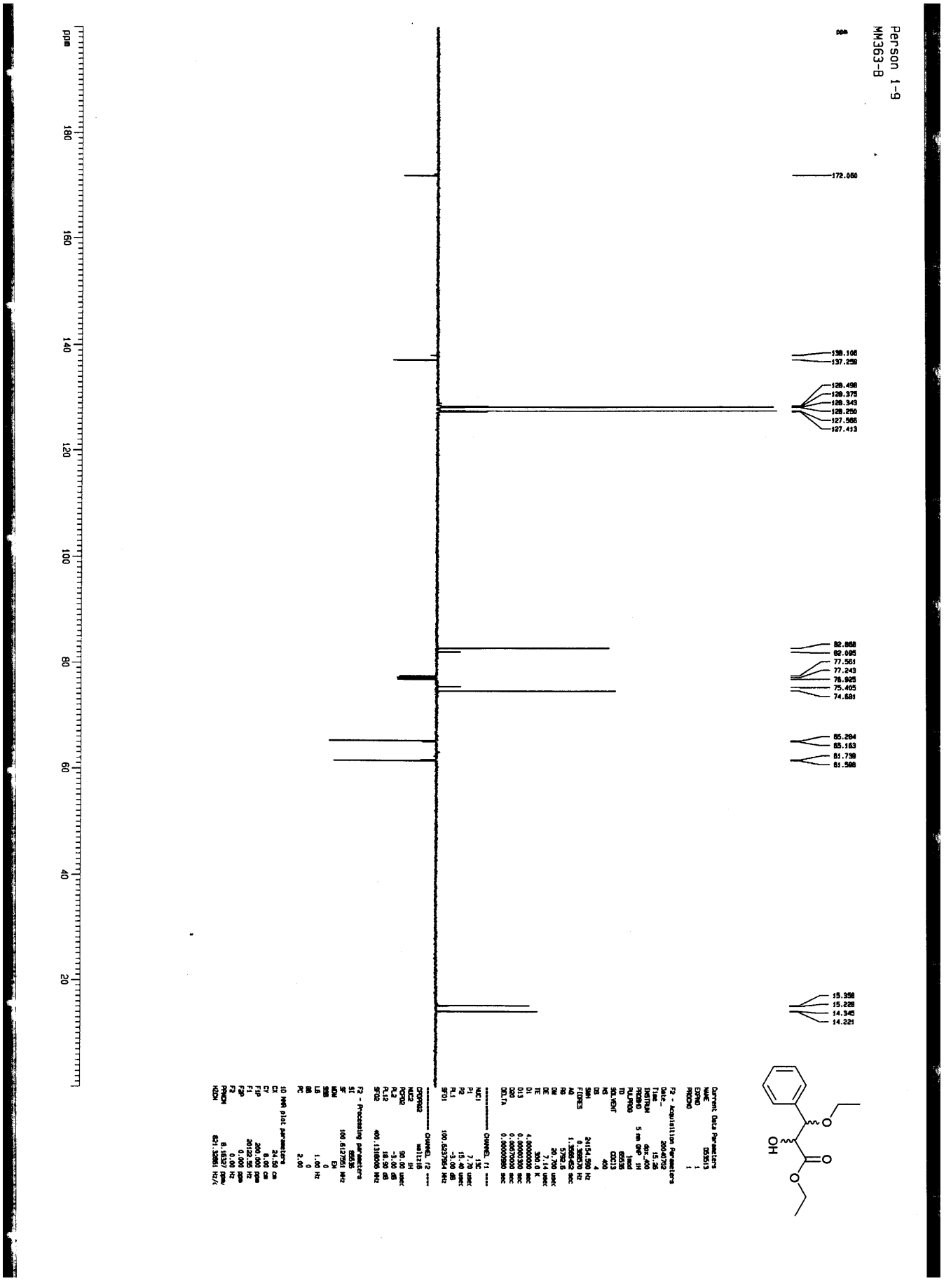
No.	(ppm)	(Hz)	Height
1	77.04	5814.8	0.3127
2	77.46	5846.6	0.3096
3	77.89	5879.0	0.3051
4	126.96	9582.2	0.9338
5	128.06	9665.5	0.4710
6	129.12	9745.2	1.0000
7	129.16	9748.1	0.5507
8	137.79	10399.5	0.1901


Person 1-9
MM376
ppm
Integral
1.0000
2.0385
3.0061
1.0012
2.0123
3.0151
7.72116
7.68114
7.54425
7.53556
7.53298
7.52558
7.52035
7.40192
7.39600
7.39393
7.39084
7.38602
7.37938
6.47037
6.43033
4.30663
4.28881
4.27099
4.25315
1.44615
1.36865
1.35087
1.33299
Current Data Parameters
NAME EtPMO
EXPNO 254219
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040724
Time 19.09
INSTRUM dpx_400
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.984387 sec
RG 90.51
DW 60.800 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 7.35 usec
PL1 -3.00 dB
SFO1 400.1320010 MHz
F2 - Processing parameters
SI 32768
SF 400.1300186 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 24.50 cm
CY 5.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.40816 ppm/cm
HZCM 163.31836 Hz/cm

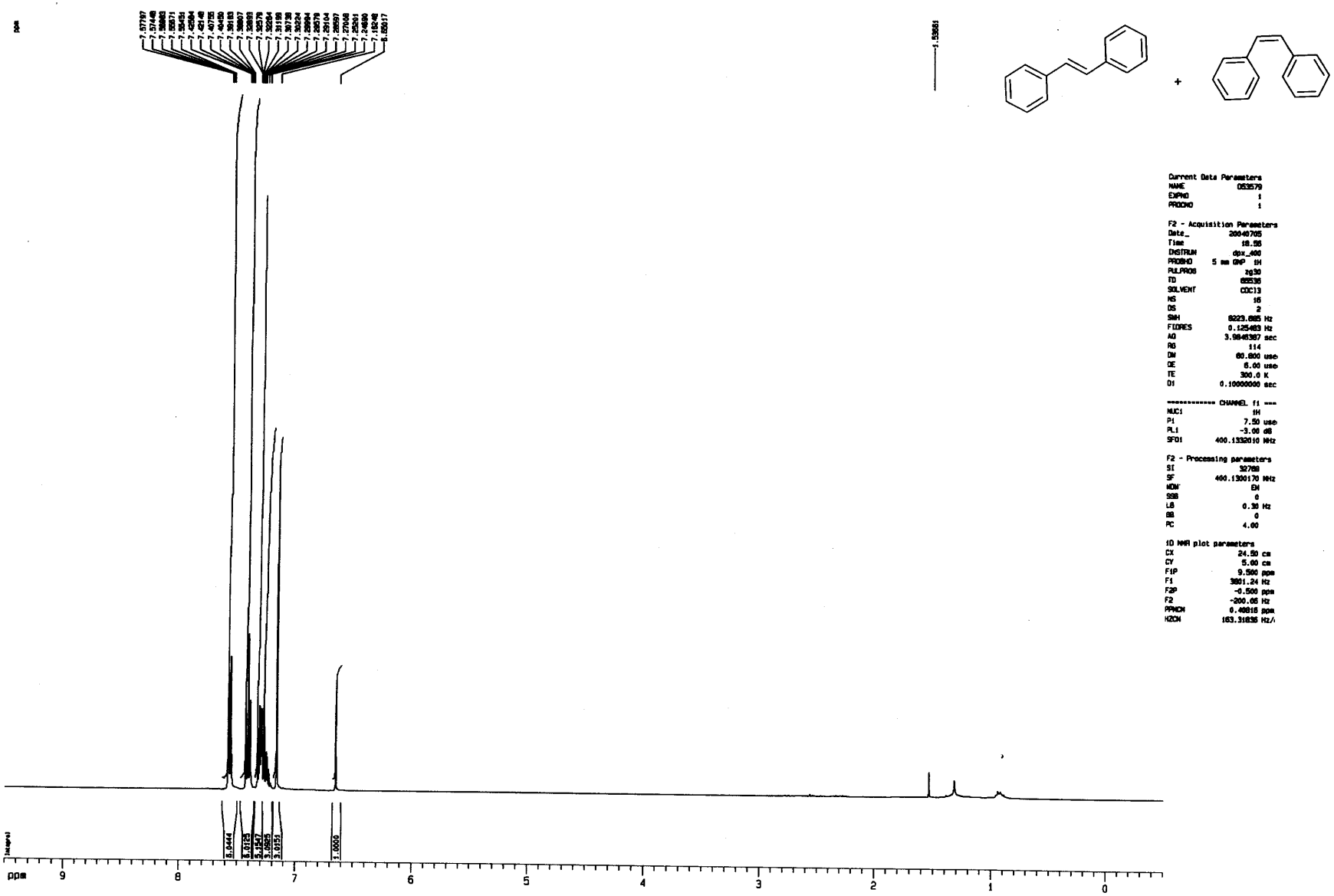
Person 1-9
MM376




Person 1-9
MM363-B
ppm
Integral
0.6838
5.0592
0.1400
1.0149
1.0019
0.3807
2.0392
1.1405
1.1543
0.1435
1.0000
0.6844
5.0517
7.37947
7.37247
7.35509
7.35059
7.34741
7.34538
7.34159
7.32939
7.32488
7.31915
7.31522
7.30907
7.30229
7.29783
7.28791
7.28257
7.27714
7.27008
4.57511
4.56681
4.53918
4.52288
4.48429
4.26563
4.25202
4.24770
4.23432
4.23009
4.17775
4.15965
4.14202
4.12413
3.55481
3.53699
3.53127
3.51938
3.51365
3.50175
3.49565
3.47833
3.47831
3.46256
3.45271
3.44111
3.43522
3.42374
3.41775
3.40032
3.37322
3.35577
2.80551
2.35554
1.28509
1.27720
1.25942
1.24427
1.23224
1.22549
1.21494
1.19813
1.18537
1.18047
1.16792
1.15038
HO
O
O
O
Current Data Parameters
NAME CDCl3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040702
Time 10.24
INSTRUM spect
PROBHD 5 mm CHP BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125473 Hz
AQ 3.9846320 sec
RG 90.5
DW 60.800 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 -3.00 dB
SFO1 400.1330010 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 20.00 cm
CY 24.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.80315 ppm/cm
HZCM 321.31836 Hz/cm


Person 1-9
MM363-B
ppm
172.060
138.106
137.259
128.498
128.375
128.343
128.250
127.506
127.413
82.868
82.095
77.561
77.243
76.925
75.405
74.681
65.204
65.163
61.739
61.508
15.358
15.228
14.345
14.221
OH
O
O
O

Person 1-9
MM372-A (Trans and cis?)



Current Data Parameters
NAME 053579
EXPNO 1
PROCNO 1

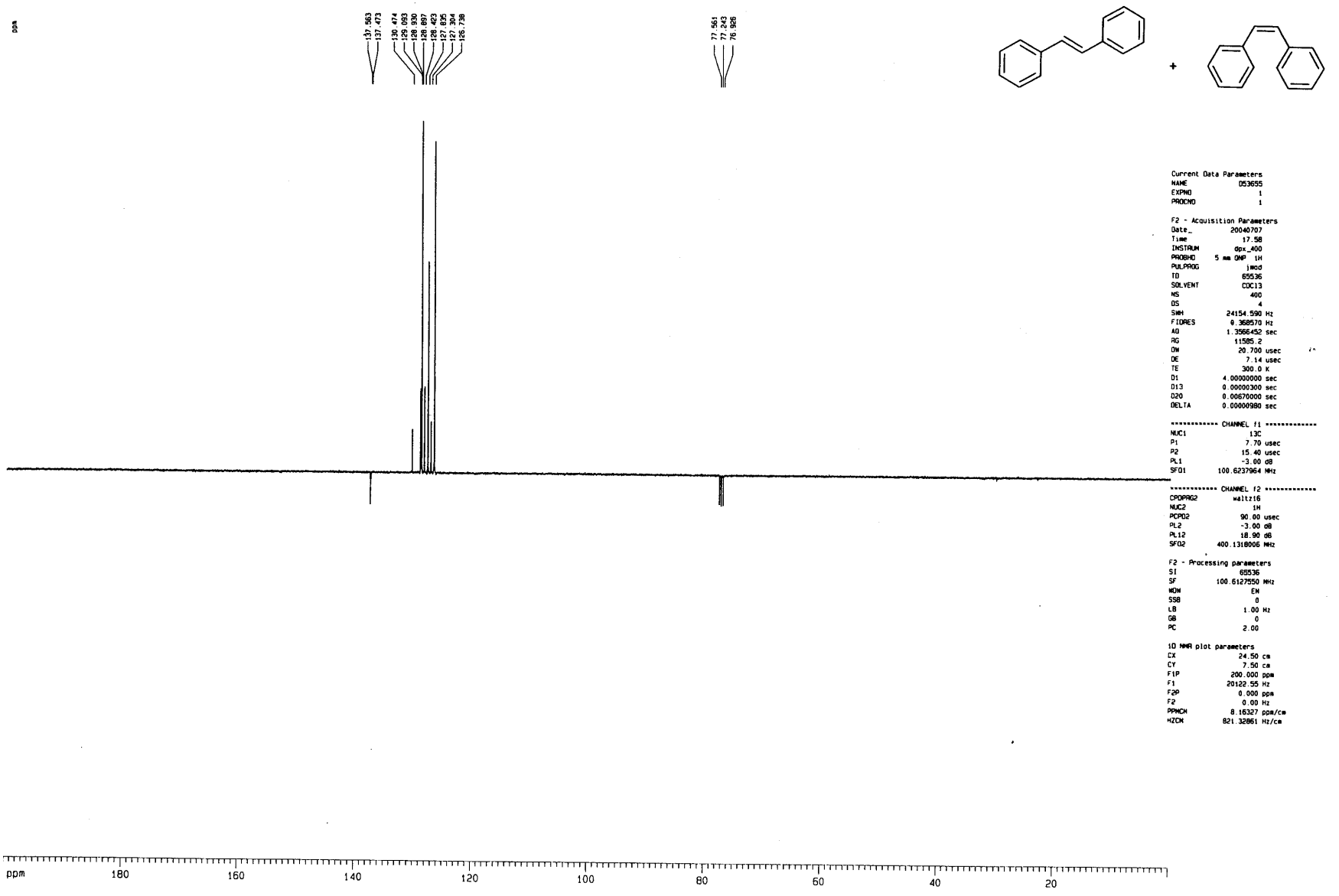
F2 - Acquisition Parameters
Date_ 20040705
Time 18.56
INSTRUM dpx_400
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845307 sec
RG 114
DW 60.800 use
DE 6.00 use
TE 300.0 K
D1 0.10000000 sec

========== CHANNEL f1 ===
NUC1 1H
P1 7.50 use
PL1 -3.00 dB
SFO1 400.1332010 MHz

F2 - Processing parameters
SI 32768
SF 400.1300170 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 24.50 cm
CY 5.00 cm
F1P 9.500 ppm
F1 3801.24 Hz
F2P -0.500 ppm
F2 -200.05 Hz
PPMCM 0.40816 ppm
HZCM 163.31835 Hz/

Person 1-9
MM373-A (Trans and cis)



Current Data Parameters
NAME 053655
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040707
Time 17.58
INSTRUM dpx_400
PROBHD 5 mm QNP 1H
PULPROG lmod
TD 65536
SOLVENT CDCl3
NS 400
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 11585.2
DW 20.700 usec
DE 7.14 usec
TE 300.0 K
D1 4.00000000 sec
D13 0.00000300 sec
D20 0.00670000 sec
DELTA 0.00000980 sec

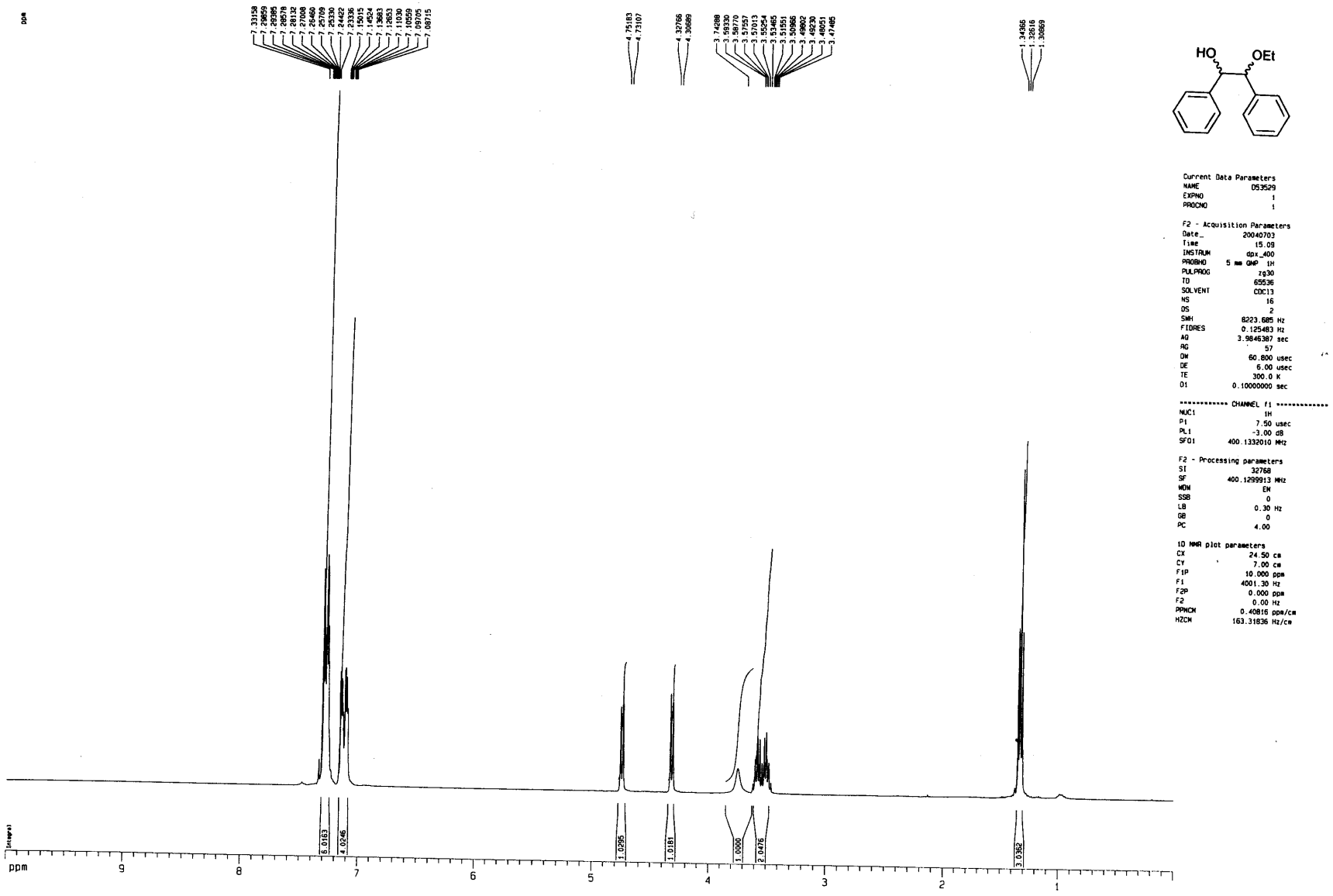
========== CHANNEL f1 ==========
NUC1 13C
P1 7.70 usec
P2 15.40 usec
PL1 -3.00 dB
SFO1 100.6237964 MHz

========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 18.90 dB
SFO2 400.1318006 MHz

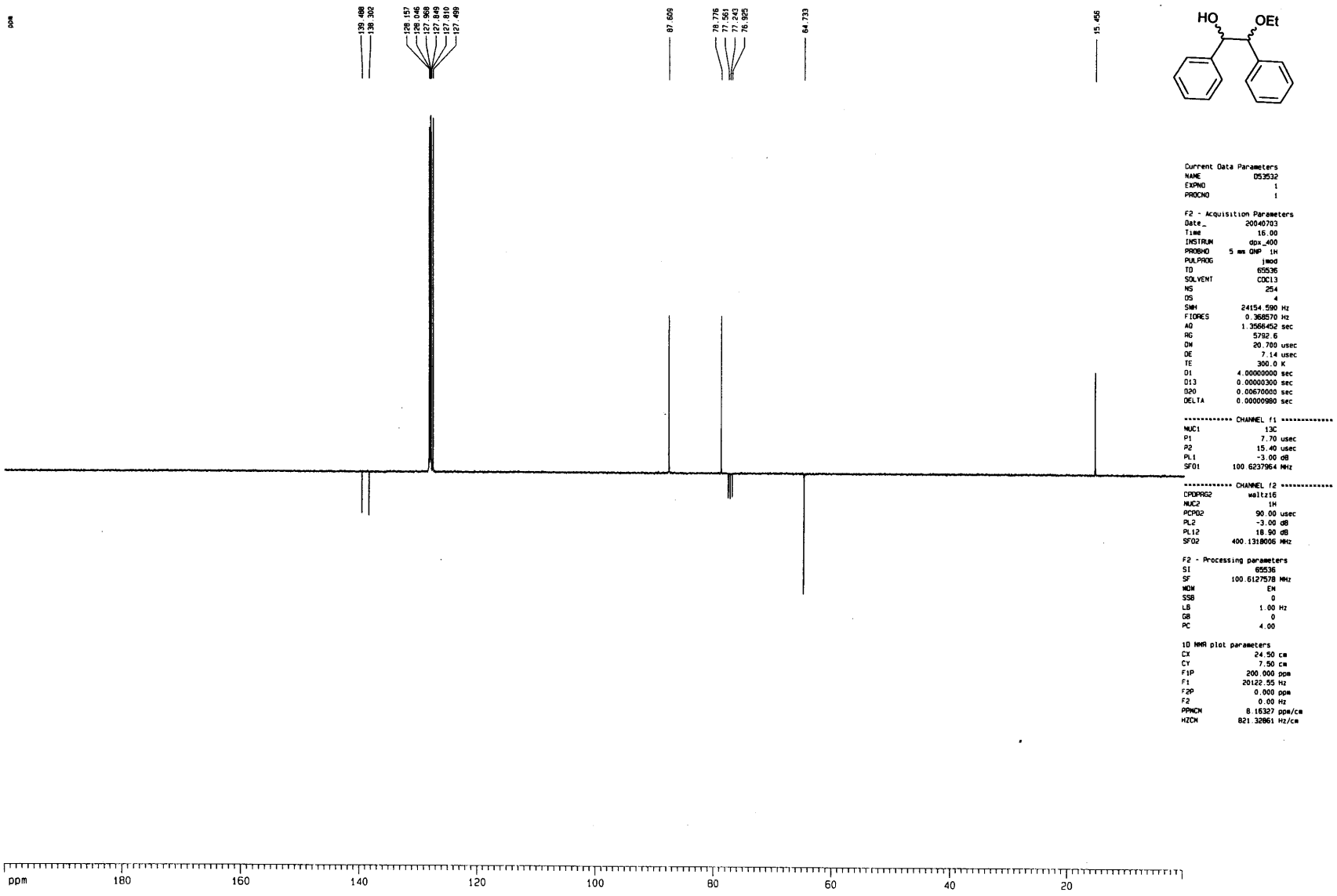
F2 - Processing parameters
SI 65536
SF 100.6127550 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

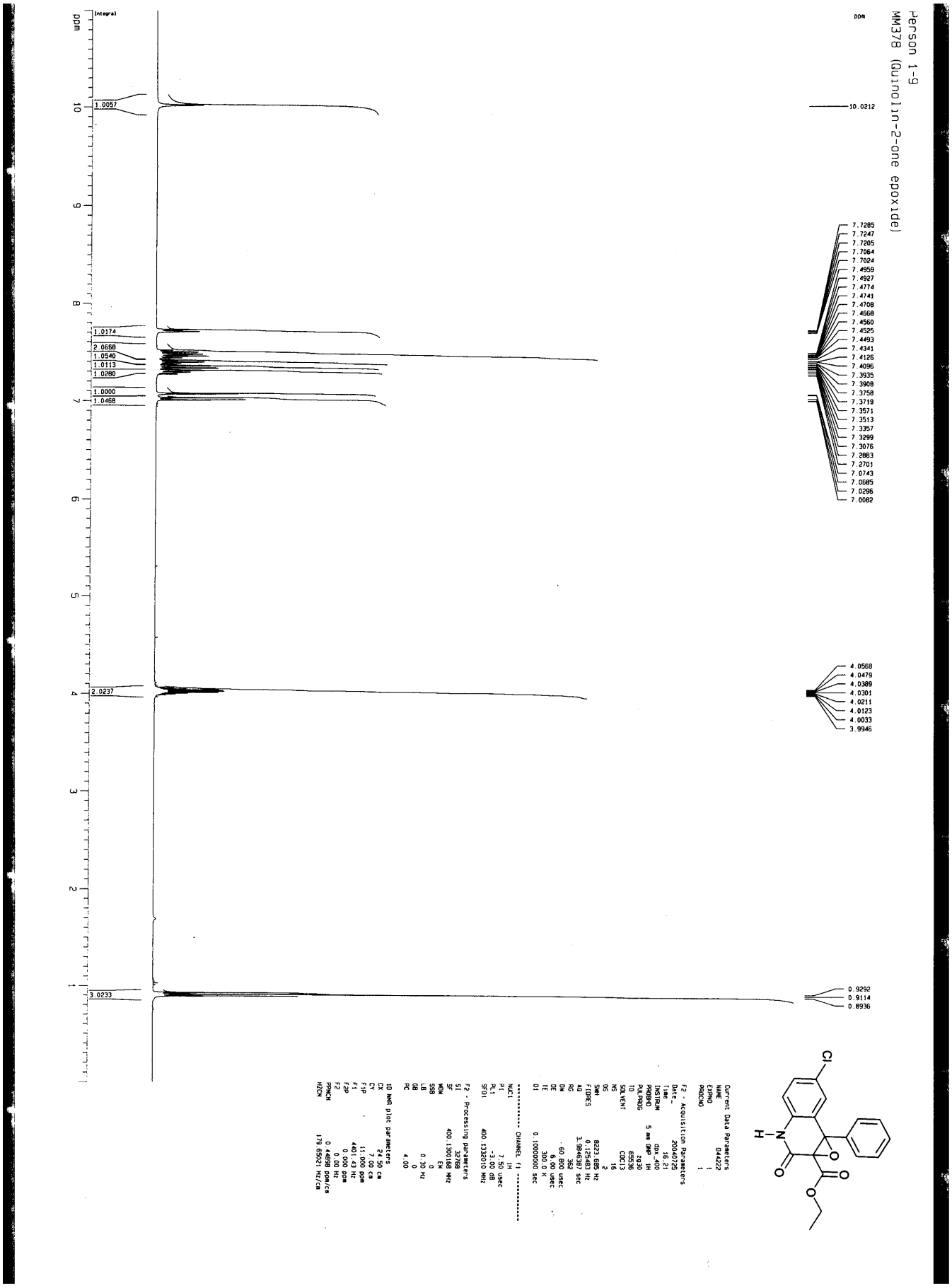
1D NMR plot parameters
CX 24.50 cm
CY 7.50 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 8.16327 ppm/cm
HZCM 821.32861 Hz/cm

Person 1-9
MM362-B



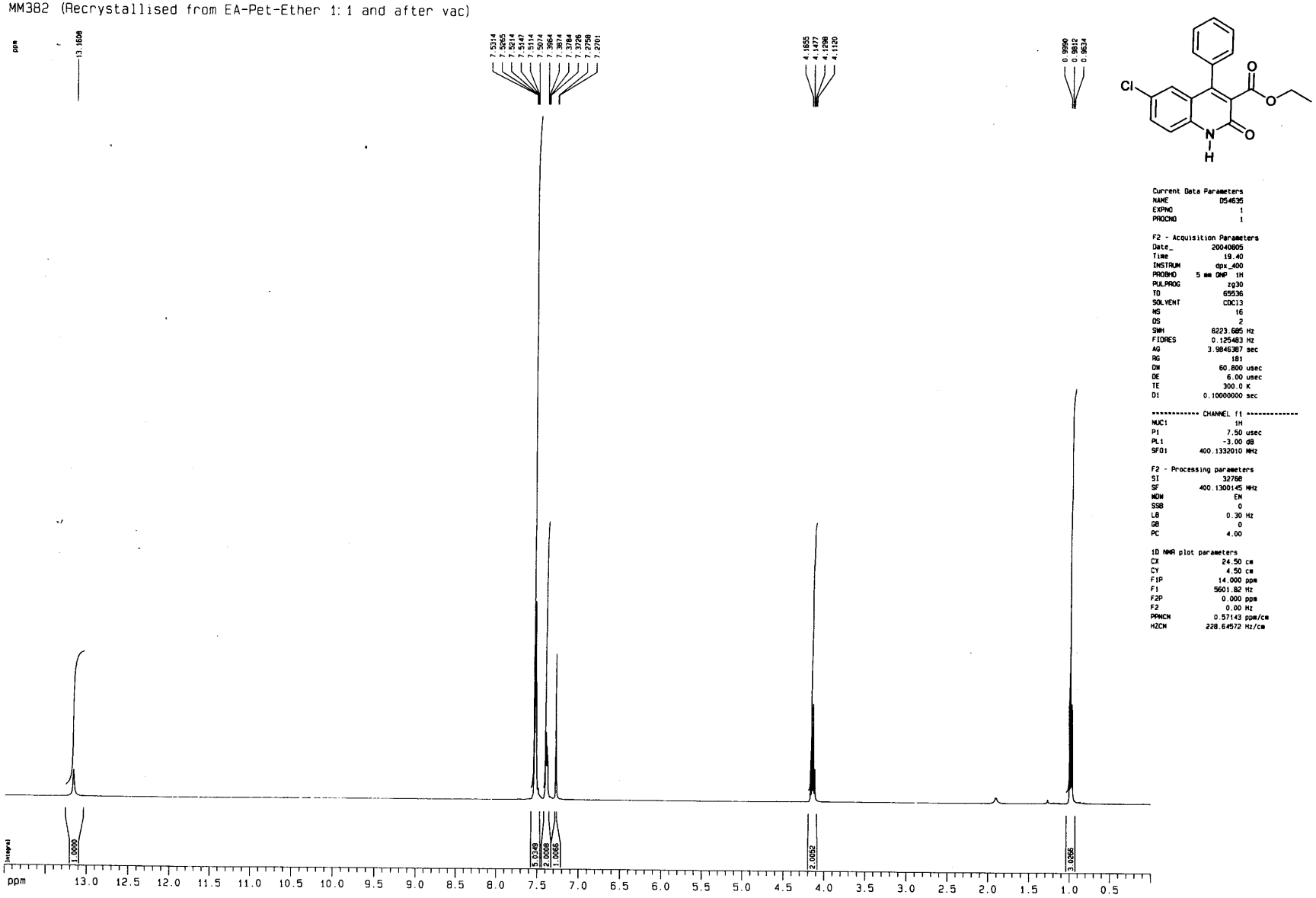
Person 1-9
MM362-B

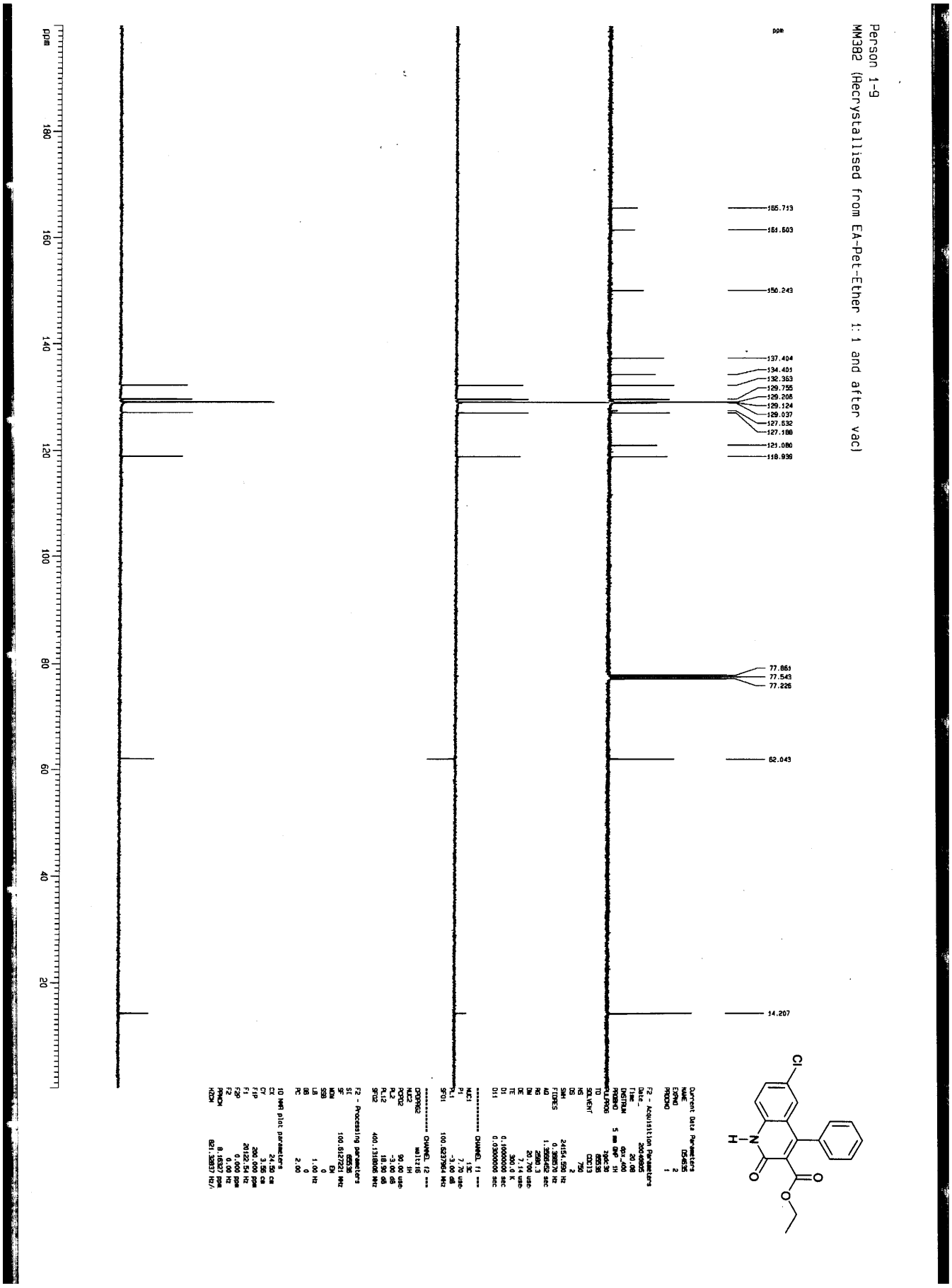



Person 1-9
MM378 (Quinolin-2-one epoxide)
ppm
Integral
1.0057
1.0174
2.0668
1.0540
1.0113
1.0280
1.0000
1.0468
2.0237
3.0233
10.0212
7.7285
7.7247
7.7205
7.7064
7.7024
7.4959
7.4927
7.4774
7.4741
7.4708
7.4668
7.4560
7.4525
7.4493
7.4341
7.4126
7.4096
7.3935
7.3908
7.3758
7.3719
7.3571
7.3513
7.3357
7.3299
7.3076
7.2883
7.2701
7.0743
7.0685
7.0296
7.0082
4.0568
4.0479
4.0389
4.0301
4.0211
4.0123
4.0033
3.9946
0.9292
0.9114
0.8936
Cl
H
N
O
O
O
Current Data Parameters
NAME D44222
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040725
Time 16.21
INSTRUM dbx_400
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845387 sec
RG 362
DW 60.800 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 -3.00 dB
SFO1 400.1332010 MHz
F2 - Processing parameters
SI 32768
SF 400.1300168 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 24.50 cm
CY 7.00 cm
F1P 11.000 ppm
F1 4401.43 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.44898 ppm/cm
HZCM 179.65921 Hz/cm

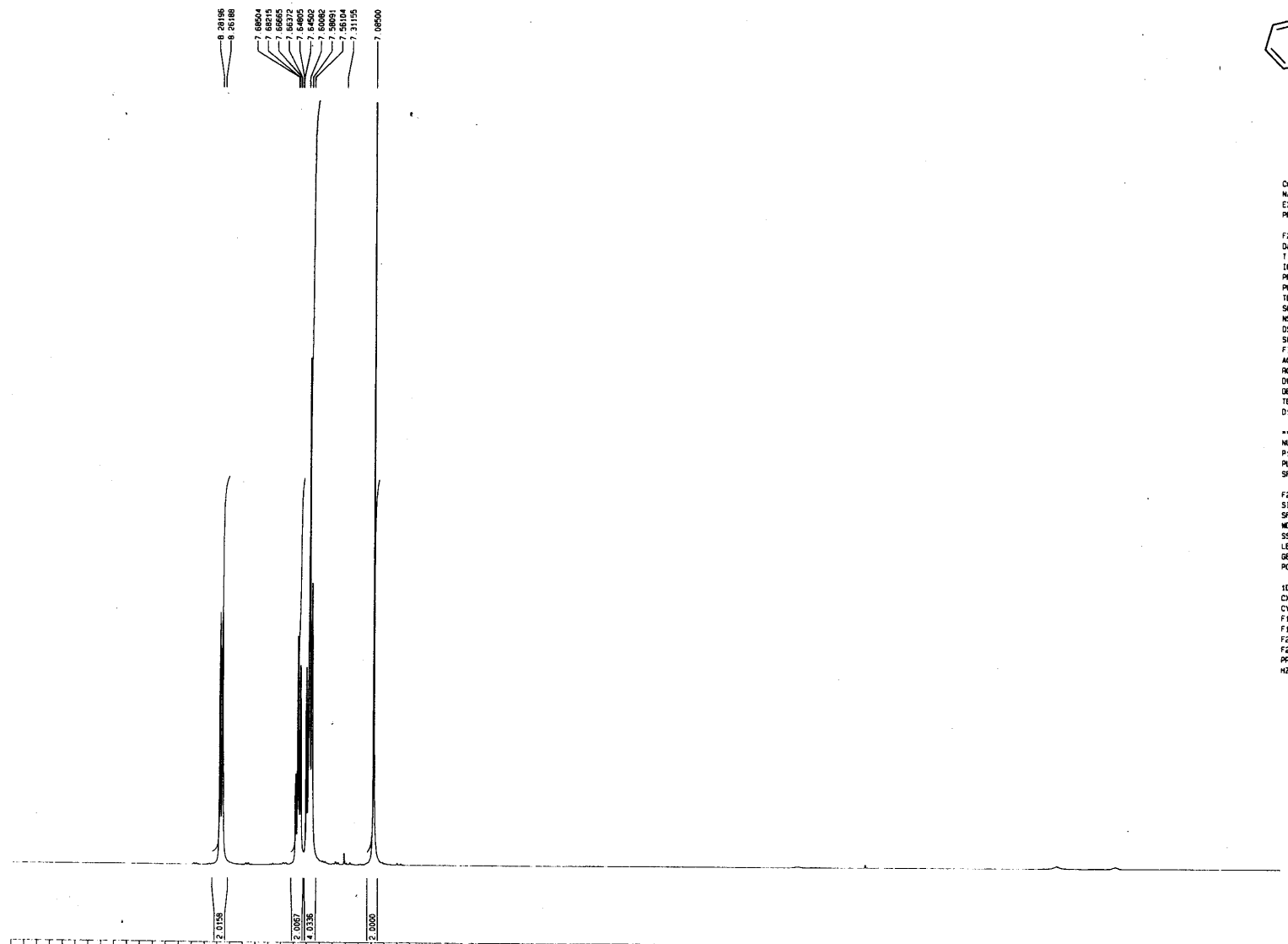

Person 1-9
MM378 (Quinolin-2-one epoxide)
ppm
165.329
163.366
135.254
131.769
131.369
130.812
130.096
129.252
129.220
129.029
128.695
128.137
122.699
118.386
77.837
77.519
77.202
68.556
65.406
62.536
14.146
Cl
H
N
O
O
O
O
Current Data Parameters
NAME EM-378
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040725
Time 16.40
INSTRM dpx_400
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 750
DS 2
SWH 24154.590 Hz
FIDRES 0.368670 Hz
AQ 1.3558432 sec
RG 1572.0
DW 20.700 usec
DE 7.14 usec
TE 300.0 K
D1 0.10000000 sec
D11 0.03000000 sec
========== CHANNEL f1 ===
NUC1 13C
P1 7.70 usec
PL1 -3.00 dB
SFO1 100.6237564 MHz
========== CHANNEL f2 ===
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 18.90 dB
SFO2 400.1318016 MHz
F2 - Processing parameters
SI 65536
SF 100.6122221 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 24.50 cm
CY 21.35 cm
F1P 200.000 ppm
F1 20123.54 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 8.16327 ppm
HZCM 821.33837 Hz/cm
ppm
20
40
60
80
100
120
140
160
180

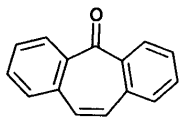
Person 1-9
MM382 (Recrystallised from EA-Pet-Ether 1:1 and after vac)



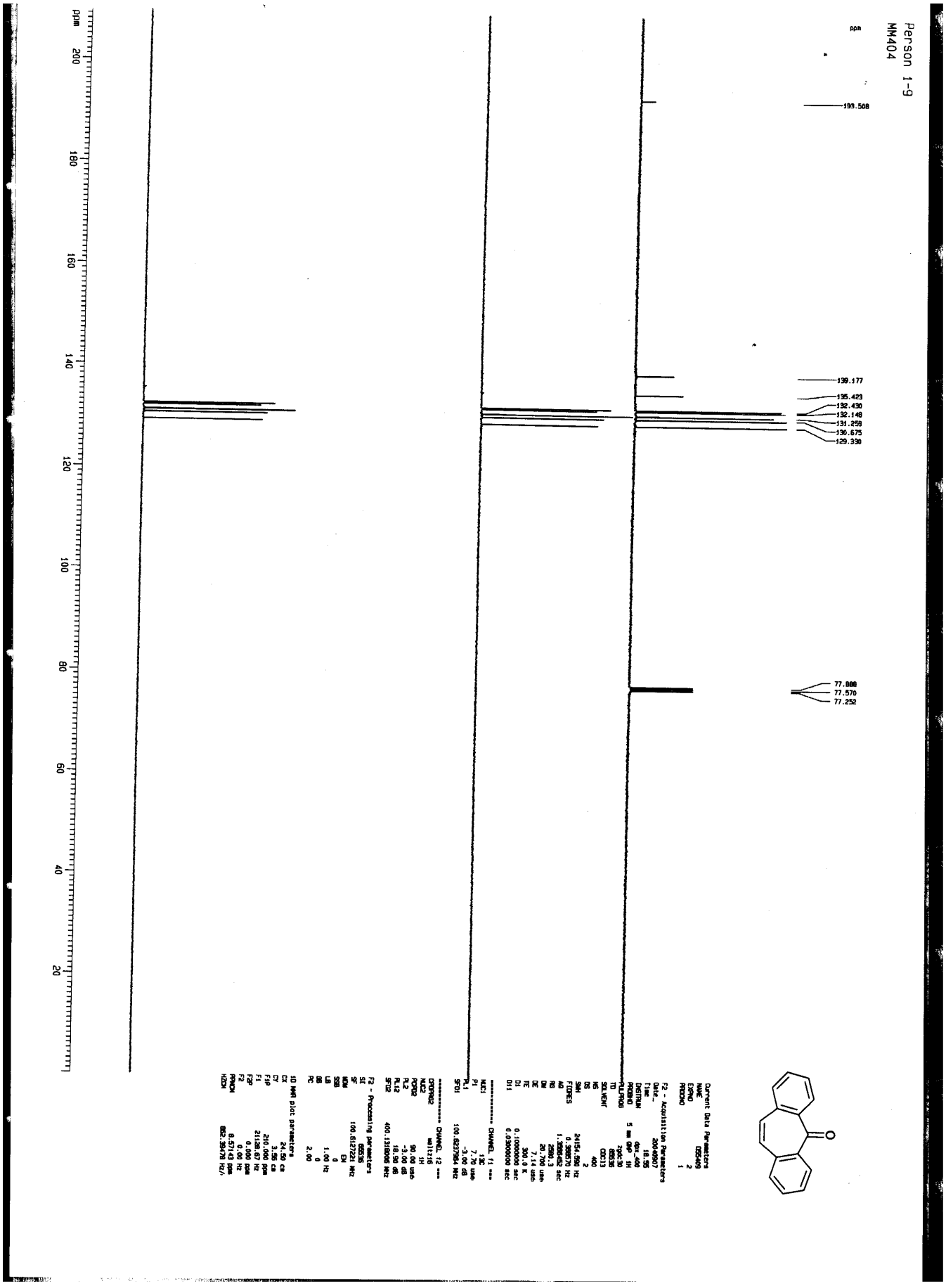

Person 1-9
MN382 (Recrystallised from EA-Pet-Ether 1:1 and after vac)
ppm
165.713
161.503
150.243
137.404
134.401
132.363
129.755
129.205
129.124
129.037
127.532
127.188
121.080
118.939
77.861
77.543
77.226
62.043
14.207
Cl
H
N
O
O
O
Current Data Parameters
NAME
EXPNO
PROCNO
F2 - Acquisition Parameters
Date_
TIME
INSTRUM
PROBHD
PULPROG
TD
SOLVENT
NS
DS
SWH
FIDRES
AQ
RG
DW
DE
TE
D1
D11
======== CHANNEL f1 ========
NUC1
P1
PL1
SFO1
======== CHANNEL f2 ========
DPWR62
NUC2
PCPD2
PL2
PL12
SFO2
F2 - Processing parameters
SI
SF
WDW
SSB
LB
GB
PC
1D NMR plot parameters
CX
CY
F1P
F2P
PPMCM
HZCM
ppm
180
160
140
120
100
80
60
40
20

rson 1-9
404



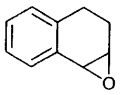


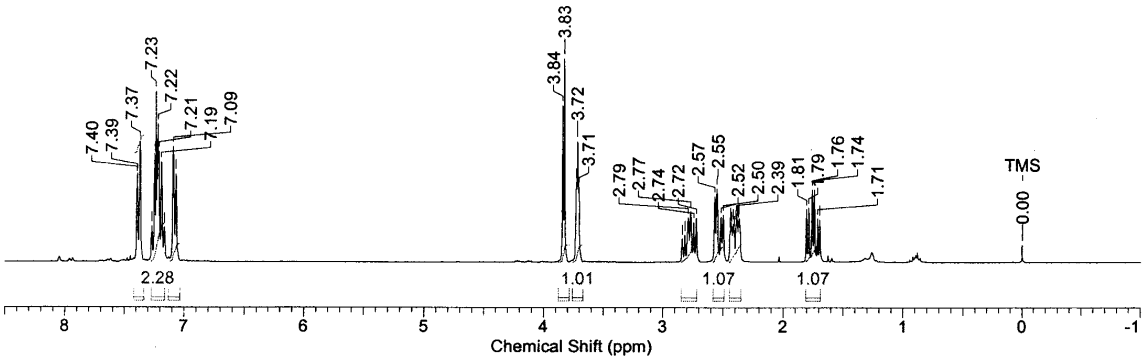

```
Current Data Parameters
NAME                  055409
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20040907
Time                  17.49
INSTRUM             dpx_400
PROBHD     5 mm GNP  1H
PULPROG                zg30
TD                    65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                       128
DW                  60.800 usec
DE                   6.00 usec
TE                   300.0 K
D1                 0.10000000 sec

============ CHANNEL f1 =============
NUC1                      1H
P1                    7.50 usec
PL1                  -3.00 dB
SFO1            400.1332010 MHz

F2 - Processing parameters
SI                        32768
SF                 400.1300000 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                    4.00

1D NMR plot parameters
CX                   24.50 cm
CY                   14.75 cm
F1P                 10.000 ppm
F1                  4001.30 Hz
F2P                  0.000 ppm
F2                    0.00 Hz
PPMCM               0.40816 ppm/cm
HZCM              163.31836 Hz/cm
```


Person 1-9
MM404
ppm
193.508
139.177
135.423
132.430
132.148
131.259
130.675
129.330
77.888
77.570
77.252
Current Data Parameters
NAME
EXPNO
PROCNO
F2 - Acquisition Parameters
Date_
Time
INSTRUM
PROBHD
PULPROG
TD
SOLVENT
NS
DS
SWH
FIDRES
AQ
RG
DW
DE
TE
D1
D11
CHANNEL f1
NUC1
P1
PL1
SFO1
CHANNEL f2
NUC2
PCPD2
PL2
PL12
SFO2
F2 - Processing parameters
SI
SF
WDW
SSB
LB
GB
PC
1D NMR plot parameters
CX
CY
F1P
F1
F2P
F2
PPMCM
HZCM

17 Feb 2004

Acquisition Time (sec)	5.2691		
Comment	Sample ID MM280 (Epoxide) Name MohanM Department PRBD-CM Phone 624 1H_16 CDCl3 u oanmr3 58		
Date	17 Feb 2004 14:03:24	***Date Stamp***	17 Feb 2004 14:03:24
File Name	\\PR-BA-DATA4\MOHANM.:.MY DOCUMENTS\NMR\U56318.1.DX		
Frequency (MHz)	300.23	***Nucleus***	1H
Number of Transients	16	***Origin***	Bruker BioSpin GmbH
Original Points Count	32768	***Owner***	service
Points Count	32768	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	6218.91		





No.	(ppm)	(Hz)	Height
1	0.00	0.0	0.0838
2	1.70	509.0	0.2076
3	1.71	514.7	0.2141
4	1.74	522.7	0.3574
5	1.76	528.2	0.3587
6	1.79	536.7	0.2686
7	1.81	542.4	0.2607
8	2.36	708.5	0.1738
9	2.37	710.6	0.2158
10	2.37	712.9	0.1998
11	2.38	714.8	0.2085
12	2.39	717.2	0.2327
13	2.40	719.3	0.1878
14	2.41	722.9	0.1575
15	2.41	725.0	0.1891
16	2.42	727.3	0.1754
17	2.43	729.4	0.1806

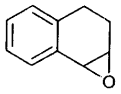
No.	(ppm)	(Hz)	Height
18	2.44	731.5	0.1989
19	2.44	733.7	0.1513
20	2.50	750.4	0.2336
21	2.52	755.9	0.2213
22	2.55	765.8	0.3625
23	2.57	771.5	0.3249
24	2.72	817.6	0.2164
25	2.74	824.1	0.1958
26	2.77	831.5	0.2509
27	2.79	837.9	0.2183
28	2.82	846.5	0.1339
29	2.84	852.9	0.1155
30	3.71	1112.7	0.3711
31	3.72	1116.4	0.5947
32	3.73	1119.4	0.3939
33	3.83	1149.2	1.0000
34	3.84	1153.4	0.7689

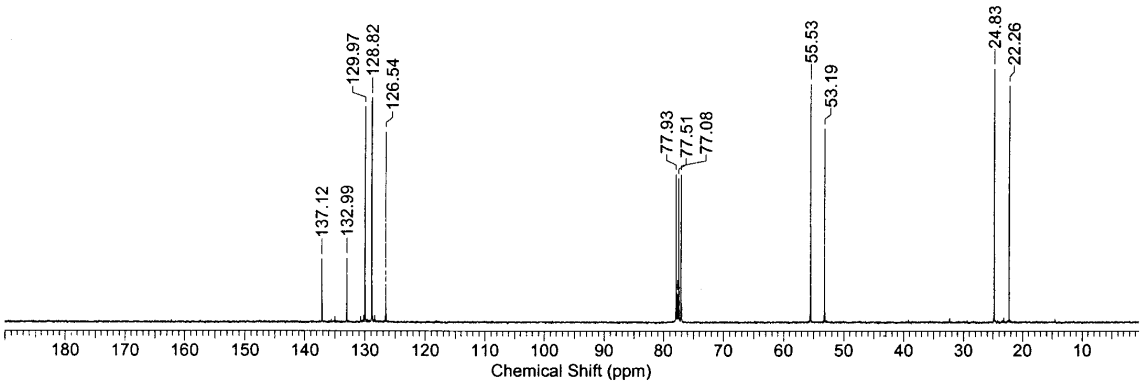
No.	(ppm)	(Hz)	Height
35	7.07	2122.3	0.4319
36	7.09	2129.3	0.5656
37	7.17	2151.5	0.1692
38	7.19	2158.9	0.4919
39	7.21	2165.3	0.5419
40	7.21	2166.1	0.5270
41	7.22	2167.8	0.6761
42	7.23	2169.7	0.5992
43	7.23	2172.2	0.8376
44	7.25	2175.4	0.5203
45	7.25	2176.9	0.4603
46	7.27	2182.8	0.1741
47	7.27	2184.1	0.1373
48	7.37	2212.6	0.5924
49	7.37	2214.1	0.5526
50	7.39	2219.8	0.4362
51	7.40	2221.1	0.4208

No.	Annotation	(ppm)
1	TMS	0.00

No.	(ppm)
1	[1.69 .. 1.81]
2	[2.35 .. 2.45]
3	[2.49 .. 2.58]
4	[2.72 .. 2.85]
5	[3.67 .. 3.76]
6	[3.78 .. 3.88]
7	[7.04 .. 7.13]
8	[7.16 .. 7.28]
9	[7.34 .. 7.42]

Acquisition Time (sec)	1.7302		
Comment	Sample ID MM280 Name MohanM Department PRBD-CM Phone 624 13C_CPD CDCl3 u oanmr3 39		
Date	20 Feb 2004 00:10:49	***Date Stamp***	20 Feb 2004 00:10:49
File Name	C:\DOCUMENTS AND SETTINGS\MOHANM\LOCAL SETTINGS\TEMPORARY INTERNET FILES\OLK62\U56449_1.DX		
Frequency (MHz)	75.50	***Nucleus***	13C
Number of Transients	1024	***Origin***	oanmr3
Original Points Count	32768	***Owner***	oanmr3
Points Count	32768	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	18939.39		

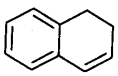


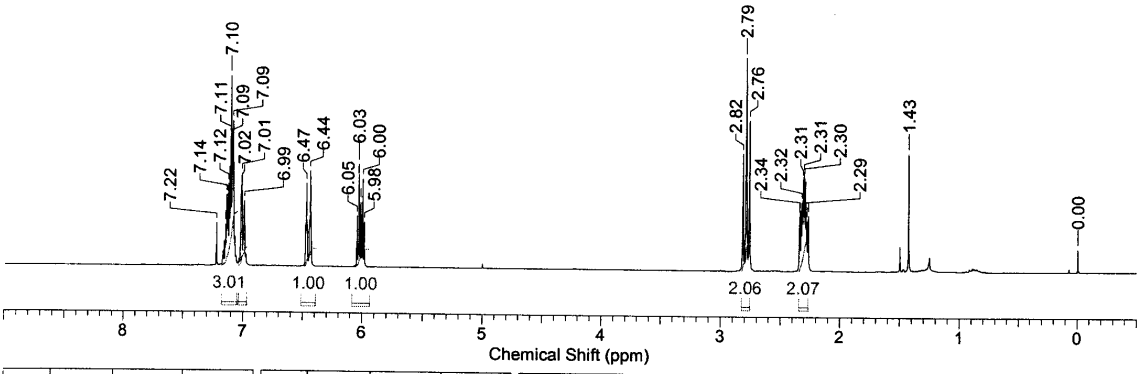


No.	(ppm)	(Hz)	Height
1	22.26	1680.8	0.9340
2	24.83	1875.0	1.0000
3	53.19	4015.9	0.7657
4	55.53	4192.2	0.9405
5	77.08	5819.9	0.5824
6	77.51	5852.3	0.5682
7	77.71	5867.3	0.1080
8	77.93	5884.1	0.5844
9	126.54	9553.8	0.7513
10	128.82	9726.0	0.8846
11	128.86	9728.9	0.8471
12	129.97	9812.7	0.8484
13	132.99	10041.0	0.2491
14	137.12	10352.6	0.2442

2 Mar 2004

Acquisition Time (sec)	5.2691		
Comment	Sample ID MM282-A (Alkene) Name Mahesh Department PRBD-CM Phone 624 1H_16 CDCl3 u oanmr3 57		
Date	02 Mar 2004 17:25:37	***Date Stamp***	02 Mar 2004 17:25:37
File Name	\\PR-BA-DATA4\MOHANM.:MY DOCUMENTS\NMR\U57067.1.DX		
Frequency (MHz)	300.23	***Nucleus***	1H
Number of Transients	16	***Origin***	Bruker BioSpin GmbH
Original Points Count	32768	***Owner***	service
Points Count	32768	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	6218.91		





No.	(ppm)	(Hz)	Height
1	-0.00	-0.1	0.1076
2	1.43	428.8	0.5548
3	2.27	680.9	0.2610
4	2.27	682.6	0.2309
5	2.28	685.1	0.2367
6	2.29	687.0	0.2796
7	2.29	688.7	0.4237
8	2.30	690.6	0.4403
9	2.31	693.0	0.4590
10	2.31	694.9	0.4334
11	2.32	697.2	0.3257
12	2.33	699.1	0.2879
13	2.34	701.6	0.2796
14	2.34	703.5	0.2593

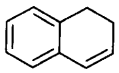
No.	(ppm)	(Hz)	Height
15	2.76	829.1	0.7038
16	2.79	837.5	1.0000
17	2.82	845.6	0.5503
18	5.98	1796.9	0.2108
19	6.00	1801.2	0.4145
20	6.02	1806.2	0.3043
21	6.03	1810.7	0.4846
22	6.05	1815.1	0.2403
23	6.43	1930.7	0.2567
24	6.44	1932.4	0.4504
25	6.44	1934.3	0.2369
26	6.46	1940.2	0.2196
27	6.47	1942.1	0.3900
28	6.47	1943.9	0.2000

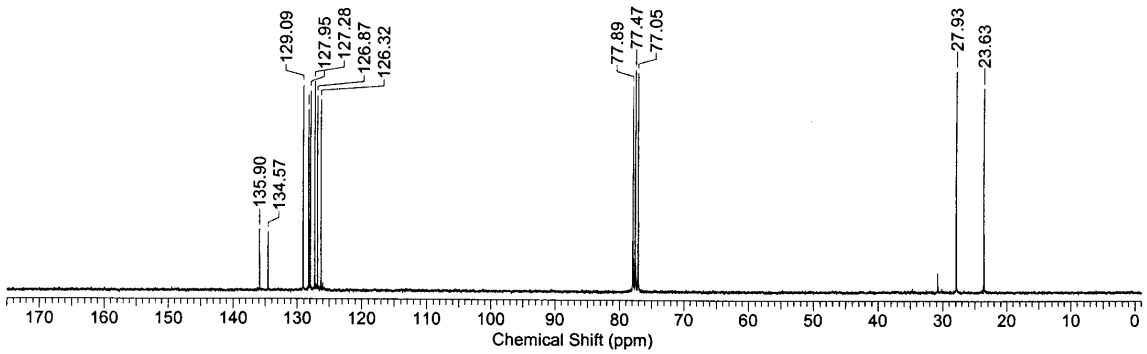
No.	(ppm)	(Hz)	Height
29	6.99	2099.2	0.3052
30	7.01	2105.3	0.4345
31	7.02	2107.4	0.3777
32	7.08	2126.3	0.4869
33	7.09	2127.9	0.6803
34	7.09	2129.8	0.5930
35	7.10	2132.6	0.8892
36	7.11	2134.3	0.6104
37	7.12	2136.4	0.4661
38	7.12	2139.1	0.3863
39	7.13	2141.5	0.2355
40	7.14	2143.0	0.3344
41	7.15	2146.1	0.1956
42	7.22	2168.5	0.2008

No.	(ppm)	Value	Absolute Value
1	[2.27 .. 2.34]	2.073	1.16148e+10
2	[2.75 .. 2.82]	2.063	1.15569e+10
3	[5.94 .. 6.09]	1.005	5.63042e+9
4	[6.39 .. 6.51]	1.000	5.60338e+9
5	[6.97 .. 7.04]	1.023	5.73300e+9
6	[7.05 .. 7.18]	3.011	1.68722e+10

3 Mar 2004

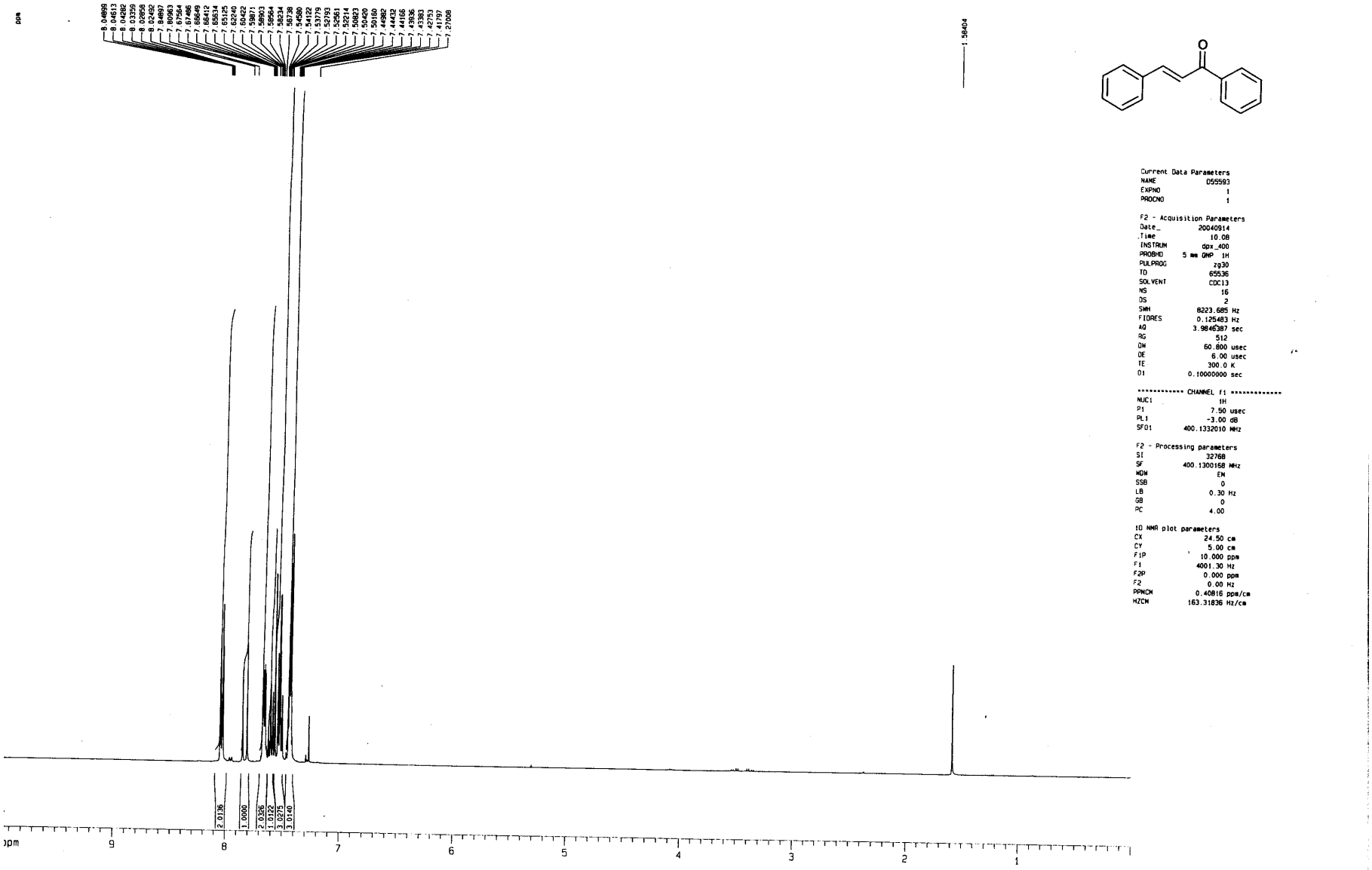
Acquisition Time (sec)	1.7302		
Comment	Sample ID MM282-A Name Mahesh Department PRBD-CM Phone 624 13C_CPD CDCl3 u oanmr3 59		
Date	03 Mar 2004 00:46:04	***Date Stamp***	03 Mar 2004 00:46:04
File Name	\\PR-BA-DATA4\MOHANM.:.MY DOCUMENTS\NMR\U57069_1.DX		
Frequency (MHz)	75.50	***Nucleus***	13C
Number of Transients	1024	***Origin***	oanmr3
Original Points Count	32768	***Owner***	oanmr3
Points Count	32768	***Solvent***	CHLOROFORM-D
Sweep Width (Hz)	18939.39		





No.	(ppm)	(Hz)	Height
1	23.63	1784.3	0.9224
2	27.93	2109.1	0.9971
3	77.05	5817.6	0.9851
4	77.47	5849.4	1.0000
5	77.89	5881.2	0.9299
6	126.32	9537.0	0.8584
7	126.87	9578.6	0.8764
8	127.28	9609.9	0.9402
9	127.95	9660.7	0.8982
10	128.22	9680.9	0.8170
11	129.09	9746.3	0.9235
12	134.57	10160.1	0.2600
13	135.90	10260.7	0.2715

Person 1-9
MM286-A



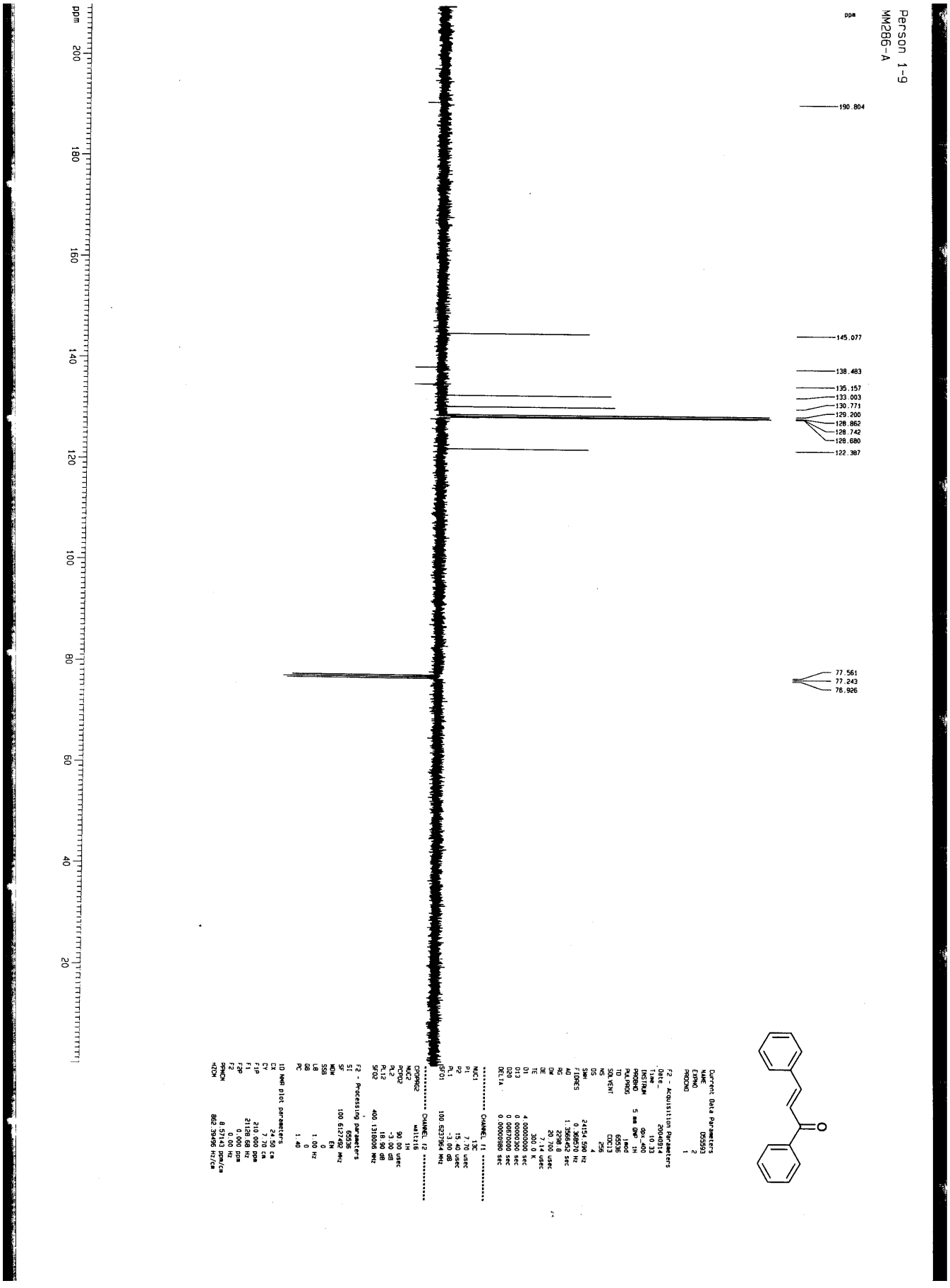
Current Data Parameters
NAME 055593
EXPNO 1
PROCNO 1

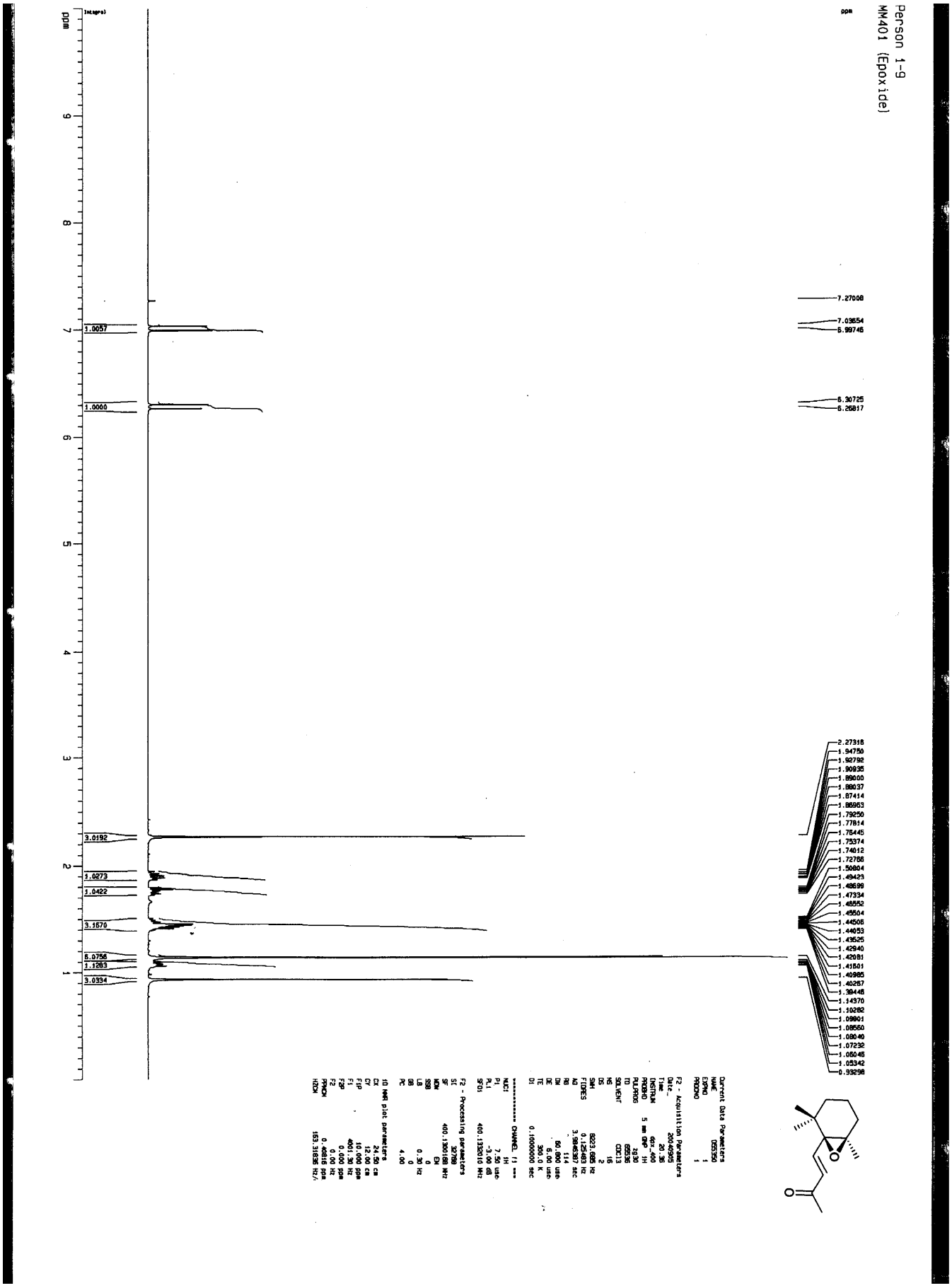
F2 - Acquisition Parameters
Date_ 20040914
Time 10.08
INSTRUM dps_400
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 512
DW 60.800 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

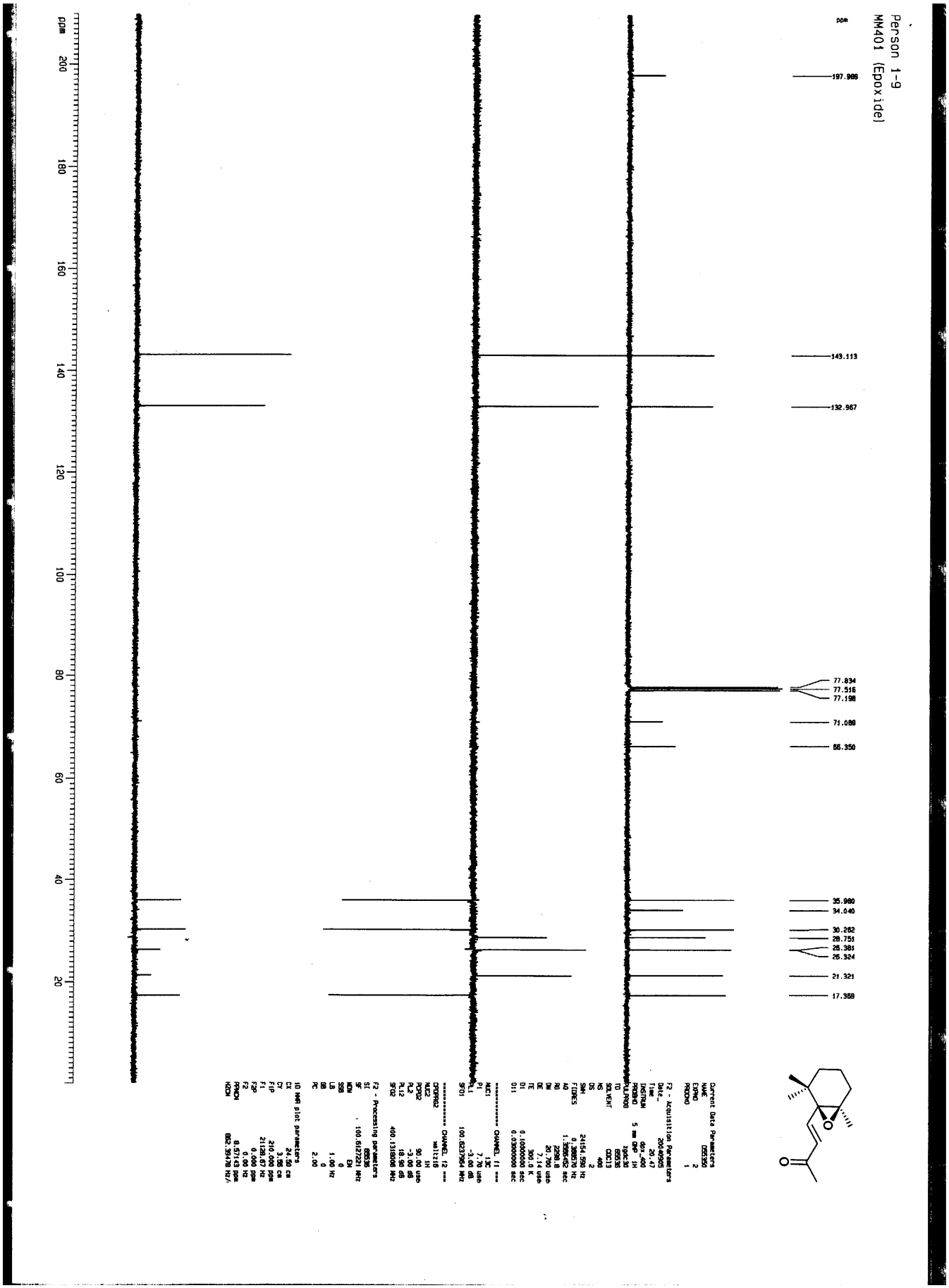
********** CHANNEL f1 **********
NUC1 1H
P1 7.50 usec
PL1 -3.00 dB
SFO1 400.1332010 MHz

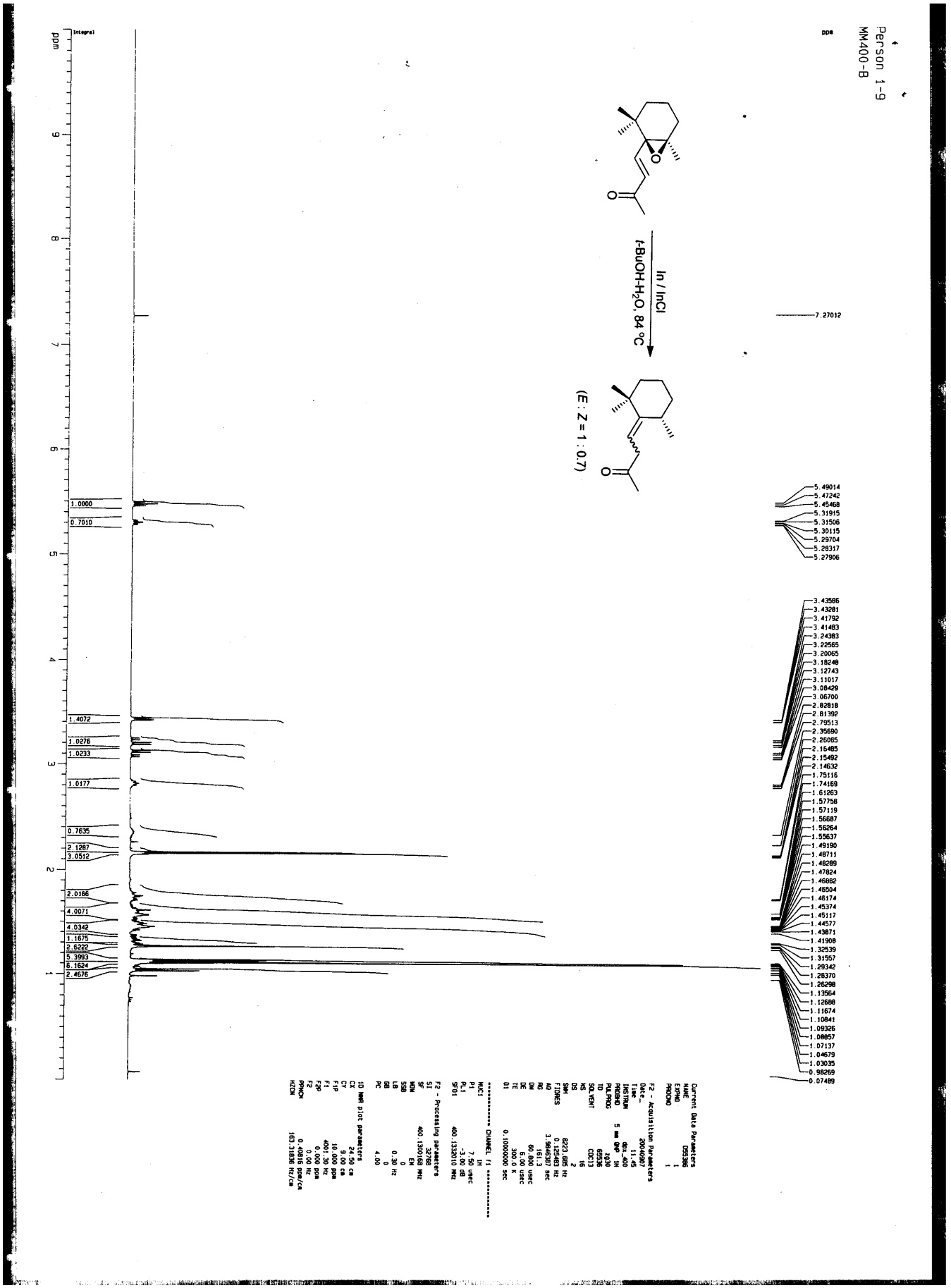
F2 - Processing parameters
SI 32768
SF 400.1300168 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 24.50 cm
CY 5.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.40816 ppm/cm
HZCM 163.31836 Hz/cm

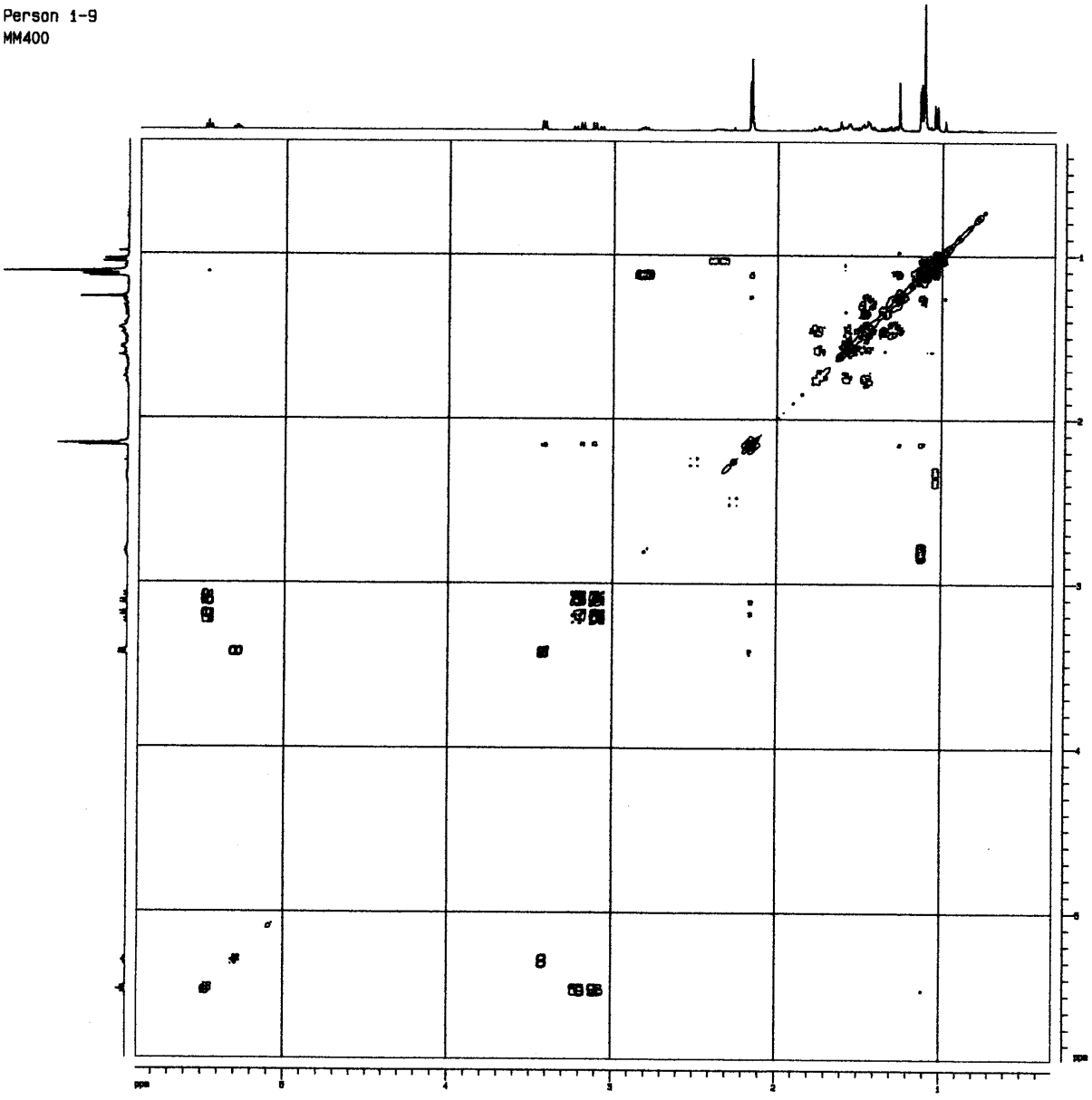

Person 1-9
MM286-A
ppm
190.804
145.077
138.483
135.157
133.003
130.771
129.200
128.862
128.742
128.680
122.387
77.561
77.243
76.926
O
Current Data Parameters
NAME 655593
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140314
Time 10.13
INSTRUM qnp 400
PROBHD 5 mm QNP 1H
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24154.590 Hz
FIDRES 0.368670 Hz
AQ 1.3566452 sec
RG 2298.8
DW 20.700 usec
DE 7.11 usec
TE 300.0 K
D1 4.00000000 sec
D13 0.00000300 sec
D20 0.00670000 sec
DELTA 0.00000980 sec
CHANNEL f1 13C
NUC1 13C
P1 7.70 usec
P2 15.40 usec
PL1 -3.00 dB
SFO1 100.6237954 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 18.90 dB
SFO2 400.1318006 MHz
F2 - Processing parameters
SI 65536
SF 100.6127490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 24.50 cm
CY 7.70 cm
F1P 210.000 ppm
F1 21128.68 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 8.57143 ppm/cm
HZCM 862.39496 Hz/cm
ppm
200
180
160
140
120
100
80
60
40
20

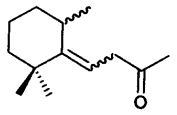

Person 1-9
MM401 (Epoxide)
ppm
7.27008
7.03654
6.99746
6.30725
6.26817
2.27316
1.94750
1.92792
1.90935
1.89000
1.88037
1.87414
1.86963
1.79250
1.77814
1.76445
1.75374
1.74012
1.72786
1.50804
1.49423
1.48699
1.47334
1.46552
1.45504
1.44506
1.44053
1.43525
1.42940
1.42081
1.41501
1.40985
1.40257
1.39446
1.14370
1.10282
1.09801
1.08550
1.08040
1.07232
1.06046
1.05342
0.93298
1.0057
1.0000
3.0192
1.0273
1.0422
3.1870
6.0756
1.1283
3.0334
Current Data Parameters
NAME
EXPNO
PROCNO
F2 - Acquisition Parameters
Date_
Time
INSTRUM
PROBHD
PULPROG
TD
SOLVENT
NS
DS
SWH
FIDRES
AQ
RG
DW
DE
TE
D1
MNC1
P1
PL1
SFO1
F2 - Processing parameters
SI
SF
WDW
SSB
LB
GB
PC
1D NMR plot parameters
CX
CY
F1P
F1
F2P
F2
PPMCM
HZCM
5 mm QNP 1H
CDCl3
8223.685 Hz
0.125488 Hz
3.9845387 sec
60.000 usec
6.00 usec
300.0 K
0.10000000 sec
CHANNEL f1
7.50 usec
-3.00 dB
400.1320010 MHz
32768
400.1300000 MHz
EM
0.30 Hz
4.00
24.50 cm
12.00 cm
10.000 ppm
4001.30 Hz
0.000 ppm
0.00 Hz
0.40816 ppm
163.31838 Hz/


Person 1-9
MN401 (Epoxide)
ppm
197.909
149.113
132.967
77.834
77.516
77.198
71.089
66.350
35.980
34.040
30.282
28.751
26.381
25.924
21.321
17.369
Current Data Parameters
NAME DSE350
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Time 20.47
INSTRUM 5 mm QNP 1H
PROBHD 720C-30
PULPROG DSE35
TD 65536
SOLVENT CDCl3
NS 400
DS 4
SWH 24154.596 Hz
FIDRES 0.368570 Hz
AQ 1.3565452 sec
RG 2288.8
DW 20.700 usec
DE 7.14 usec
TE 300.0 K
D1 0.10000000 sec
D11 0.03000000 sec
========== CHANNEL f1 ==========
NUC1 13C
P1 7.70 usec
PL1 -3.00 dB
SFO1 100.6232454 MHz
========== CHANNEL f2 ==========
OBNUC2 waltz16 1H
PCPD2 90.00 usec
PL2 18.50 dB
PL12 -3.00 dB
SFO2 400.1318008 MHz
F2 - Processing parameters
SI 65536
SF 100.6122221 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 24.50 cm
CY 3.95 cm
F1P 210.000 ppm
F1 21128.67 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 8.57143 ppm/cm
HZCM 862.39478 Hz/cm
ppm
200
180
160
140
120
100
80
60
40
20


Person 1-9
MM400-B
In / InCl
t-BuOH-H2O, 84 °C
(E : Z = 1 : 0.7)
7.27012
5.49014
5.47242
5.45468
5.31915
5.31506
5.30115
5.29704
5.28317
5.27906
3.43586
3.43281
3.41792
3.41483
3.24383
3.22565
3.20065
3.18248
3.12743
3.11017
3.08429
3.06700
2.82818
2.81392
2.79513
2.35690
2.26065
2.15485
2.15492
2.14632
1.75116
1.74169
1.61263
1.57758
1.57119
1.56687
1.56264
1.55637
1.49190
1.48711
1.48289
1.47824
1.46882
1.46504
1.46174
1.45374
1.45117
1.44577
1.43871
1.41908
1.32539
1.31557
1.29342
1.28370
1.26298
1.13564
1.12688
1.11674
1.10841
1.09326
1.08857
1.07137
1.04679
1.03035
0.98269
0.07489
1.0000
0.7010
1.4072
1.0276
1.0233
1.0177
0.7635
2.1287
3.0512
2.0166
4.0071
4.0342
1.1675
2.6222
5.3993
6.1624
2.4676
Integral
ppm
Current Data Parameters
NAME DS5386
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040907
Time 11.45
INSTRUM qhs_400
PROBHD 5 mm QNP 1H
PULPROG 2930
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845387 sec
RG 161.3
DW 60.800 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 -3.00 dB
SFO1 400.1332010 MHz
F2 - Processing parameters
SI 32768
SF 400.1301168 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 24.50 cm
CY 9.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.40815 ppm/cm
HZCM 163.31836 Hz/cm

Person 1-9
MM400





E and *Z*

Current Data Parameters

USER	jan
NAME	080408
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

Date_	20040808
Time	11.41
INSTRUM	dpx_400
PROBHD	5 mm BBF 1H
PULPROG	cosygp
TD	2048
SOLVENT	CDCl3
NS	2
DS	16
SWH	2232.143 Hz
FIDRES	1.088813 Hz
AQ	0.4585025 sec
RG	64
DW	224.000 usec
DE	6.00 usec
TE	300.0 K
D0	0.00000000 sec
D1	0.70947888 sec
D13	0.00000000 sec
D16	0.00010000 sec
IN0	0.0004500 sec

CHANNEL f1

NUC1	1H
P0	7.50 usec
P1	7.50 usec
PL1	-3.00 dB
SFO1	400.1312883 MHz

GRADIENT CHANNEL

GPNAM1	sine.100
GPNAM2	sine.100
GPX1	0.00 %
GPX2	0.00 %
GPY1	0.00 %
GPY2	0.00 %
GPZ1	10.00 %
GPZ2	10.00 %
P16	1000.00 usec

F1 - Acquisition parameters

NDO	1
TD	256
SFO1	400.1313 MHz
FIDRES	8.718308 Hz
SW	5.578 ppm

F2 - Processing parameters

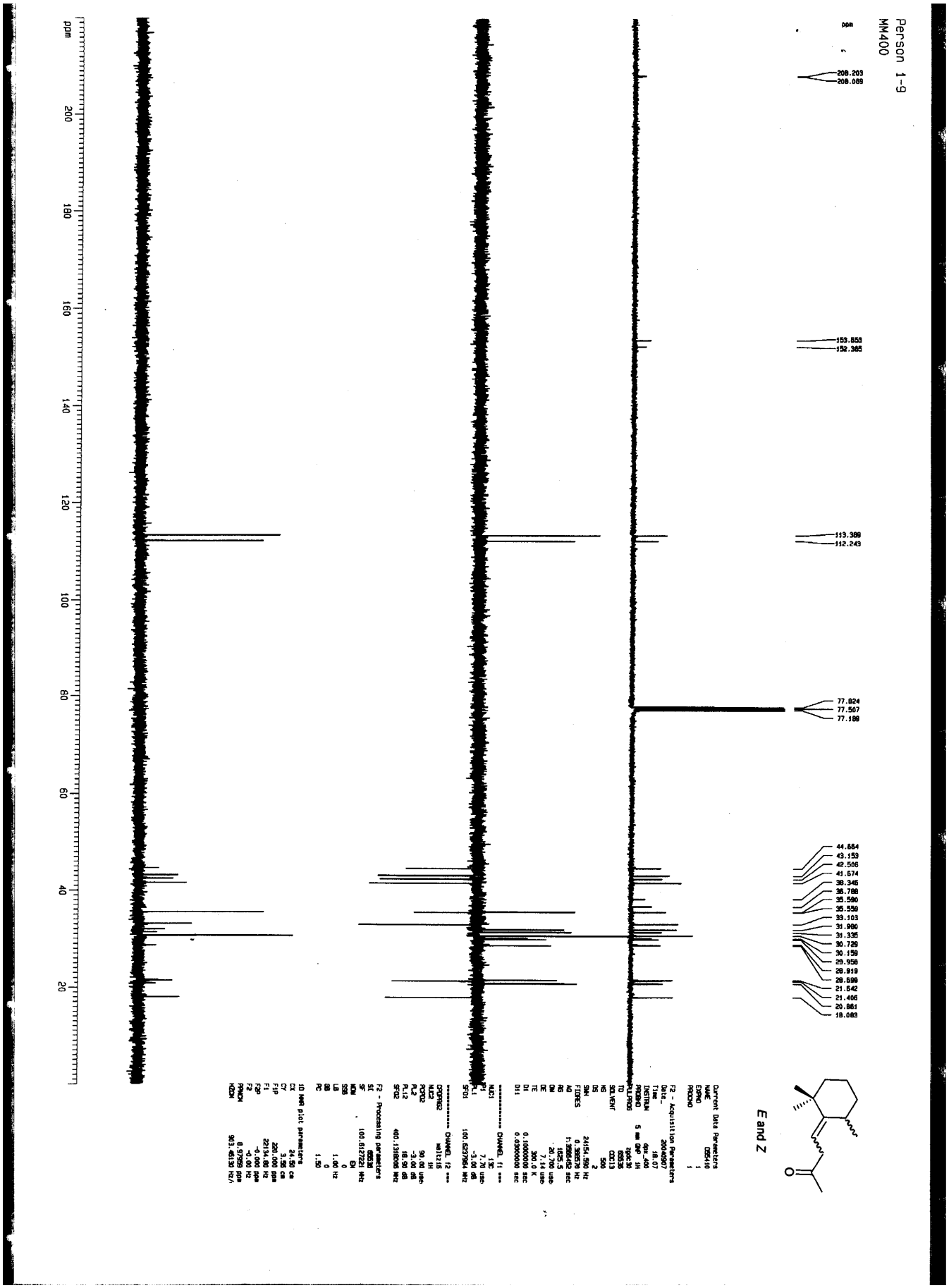
SI	1024
SF	400.1300170 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

F1 - Processing parameters

SI	1024
MC2	QF
SF	400.1300170 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

2D NMR plot parameters

CX2	16.00 cm
CY1	16.00 cm
F2PLO	0.887 ppm
F2LO	2355.44 Hz
F2PHI	0.309 ppm
F2HI	123.30 Hz
F1PLO	0.887 ppm
F1LO	2355.44 Hz
F1PHI	0.309 ppm
F1HI	123.30 Hz
F2PPMCM	0.33808 ppm/cm
F2HZCM	135.28068 Hz/cm
F1PPMCM	0.33808 ppm/cm
F1HZCM	135.28068 Hz/cm


Person 1-9
MM400
E and Z
208.203
208.089
159.853
152.385
113.389
112.243
77.824
77.507
77.189
44.684
43.153
42.506
41.574
38.345
36.788
35.590
35.559
33.103
31.980
31.335
30.729
30.159
29.958
28.919
28.599
21.542
21.406
20.861
18.083
ppm

NMR studies of the (*Z*)- and (*E*)-4-[(*S*)-2,2,6-trimethylcyclohexylidene]butan-2-one (21) to determine the stereochemistry of the molecule

Experimental Details

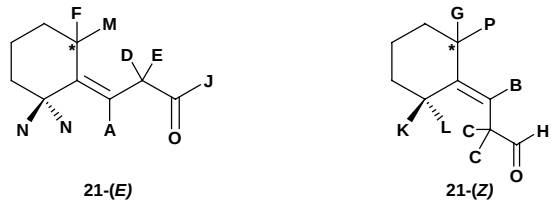
NMR data were acquired at 400.13 MHz on at a temperature of 300 K. The sample was freshly prepared, dissolved in $CDCl_3$ and admitted to a 5 mm NMR tube.

1D ^1H nOe NMR data were acquired using the pulse programme noemul driven by the au program noemult. Frequency lists, containing 6 irradiation points each, were established for 9 independent irradiations including one control. Each irradiation was carried out during 70 ms for each frequency per list and each list was cycled through 100 times to provide an overall irradiation time of 7 s per list. Irradiation power was set at 90 dB below maximum power using the decoupler channel. The overall experiment time was 5 hrs.

2D ^1H NOESY and DQFCOSY NMR data were acquired to assist with the interpretation of the 1D ^1H nOe data. NMR data were acquired over frequency widths of 6 ppm (2.4 kHz) in both ω_2 and ω_1 into 2 K complex data points for each of 256 t_1 increments acquired in TPPI mode (acquisition time = 426 ms). Fourier transformation was carried out using $\pi/2$-shifted squared sine-bell window functions in both dimensions. Data were imported into SPARKY version 3.111 for ease of handling and interpretation.

All NMR data were processed offline on a Dell Precision 340 Workstation operating under Windows 2000 Professional using Xwin-NMR version 3.0.

FIGURE 1. 1D ^{1}H nOe difference spectra compared with the 1D ^{1}H NMR spectrum of alkene **21**



21-(*E*)
N = **M** = **J** = CH_3
A = **D** = **E** = **F** = H

21-(*Z*)
H = **K** = **L** = **P** = CH_3
B = **C** = **G** = H

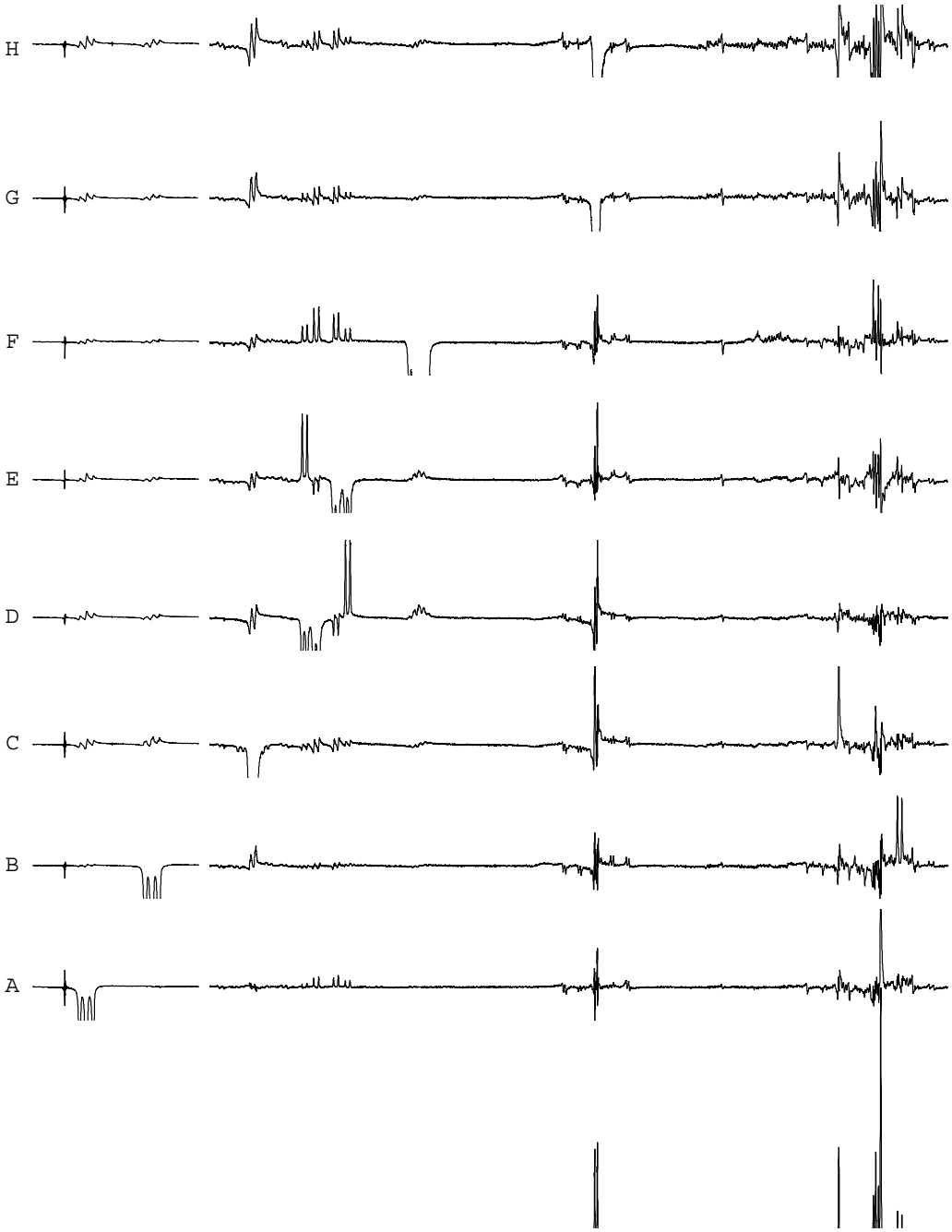
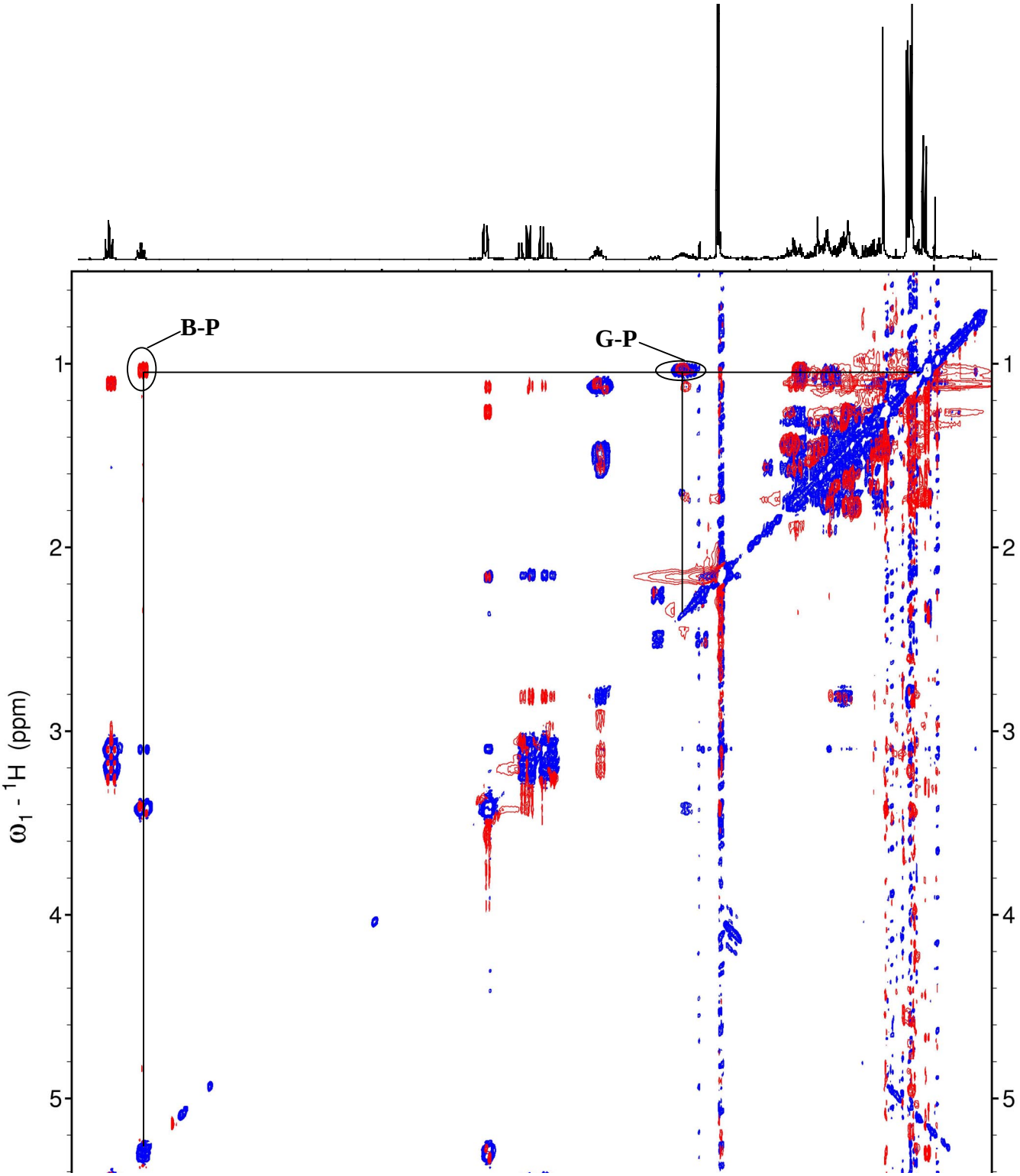
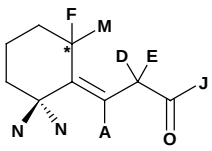


FIGURE 2. Overlay of 2D ^{1}H NOESY (Red) and COSY NMR (Blue) spectra of alkene **21** acquired at 400.13 MHz. Labelling scheme as for Figure 1.



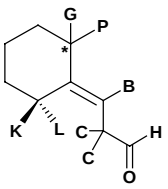
Result

The solution to the NMR data interpretation problem with regard to the stereochemistry of the alkene **21** is summarized below:



21-(*E*)

N = **M** = **J** = CH_3
A = **D** = **E** = **F** = H



21-(*Z*)

H = **K** = **L** = **P** = CH_3
B = **C** = **G** = H

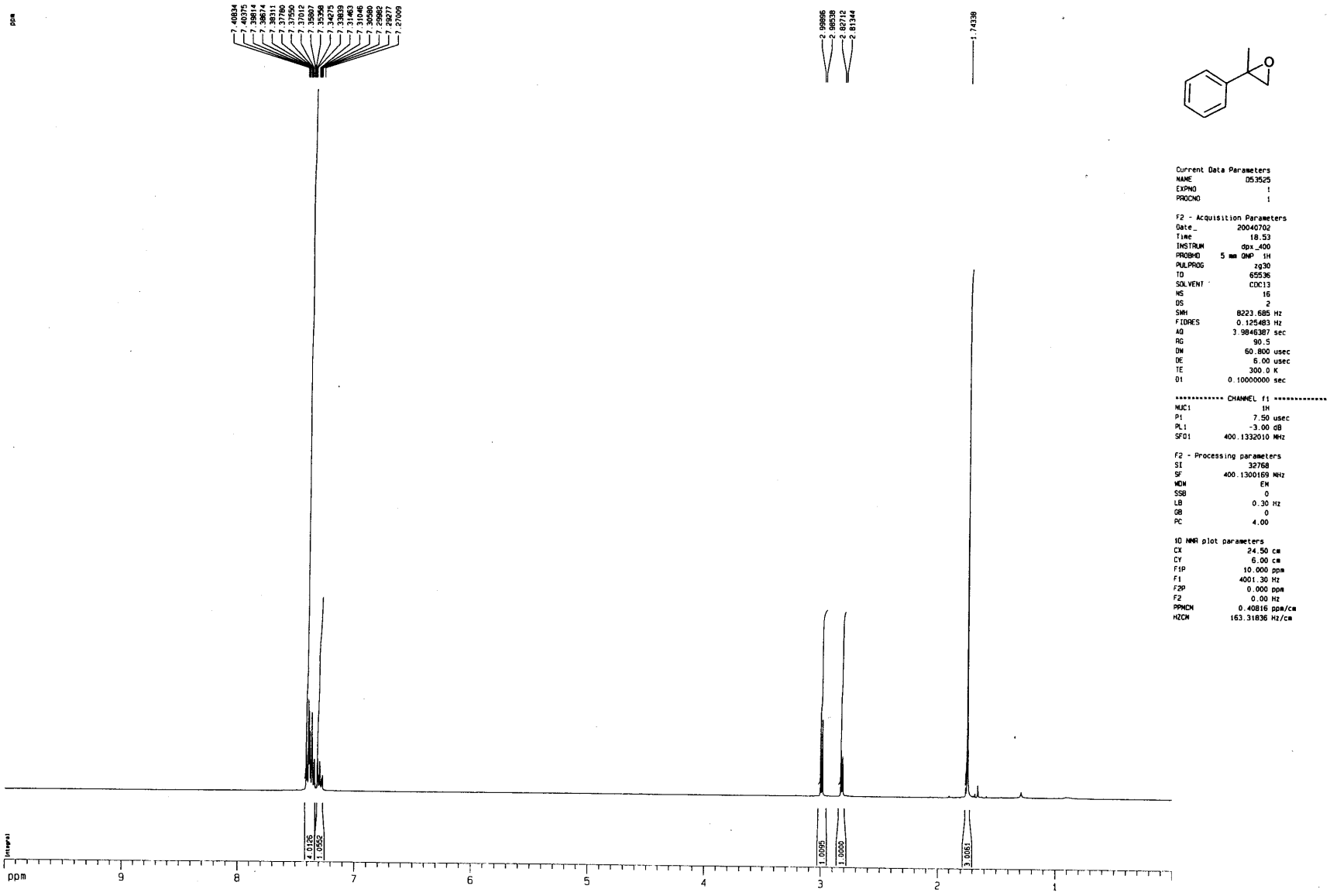
The rationale behind the assignments is given below.

Signal **C** integrates for two protons and corresponds to two magnetically equivalent geminal protons in one isomer. Signals **D** and **E** correspond to a geminal pair of non-magnetically equivalent protons.

Irradiating at **A** results in an nOe to **D** and **E** and to a sharp singlet methyl signal **N**, which integrates for 6 protons relative to signal **A**. Signal **A** is assigned to the proton attached to the double bond of the *E* isomer, signal **N** corresponding to two magnetically equivalent methyl groups associated with the six-membered ring. Irradiating **F** results in an nOe to the methyl doublet **M** and strong nOes to both **D** and **E**. The relative intensities of the nOes **F** to **D**/**E** and **F** to **M** in the 2D NOESY data indicate that methyl **M** is further away from the **D**/**E** geminal proton pair than proton **F**. From model building, these data are consistent with the stereochemistry at * as shown.

Irradiating at proton **B** results in a strong nOe to doublet **P**, thereby locating proton **B** and methyl **P** in the *Z* isomer. Irradiaton of **C** results in an nOe to **K** (strong) and to **L** (weak). From the 2D COSY data, proton **C** shows a $^{4}J_{HH}$ coupling to proton **H**. By the same token, protons **D** and **E** show similar couplings to methyl protons **J**.

Person 1-9
MM369 (from acetophenone)



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PROCNO 1

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TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 90.5
DW 60.800 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

********** CHANNEL f1 **********
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P1 7.50 usec
PL1 -3.00 dB
SFO1 400.1332010 MHz

F2 - Processing parameters
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SF 400.1300169 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
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F2 0.00 Hz
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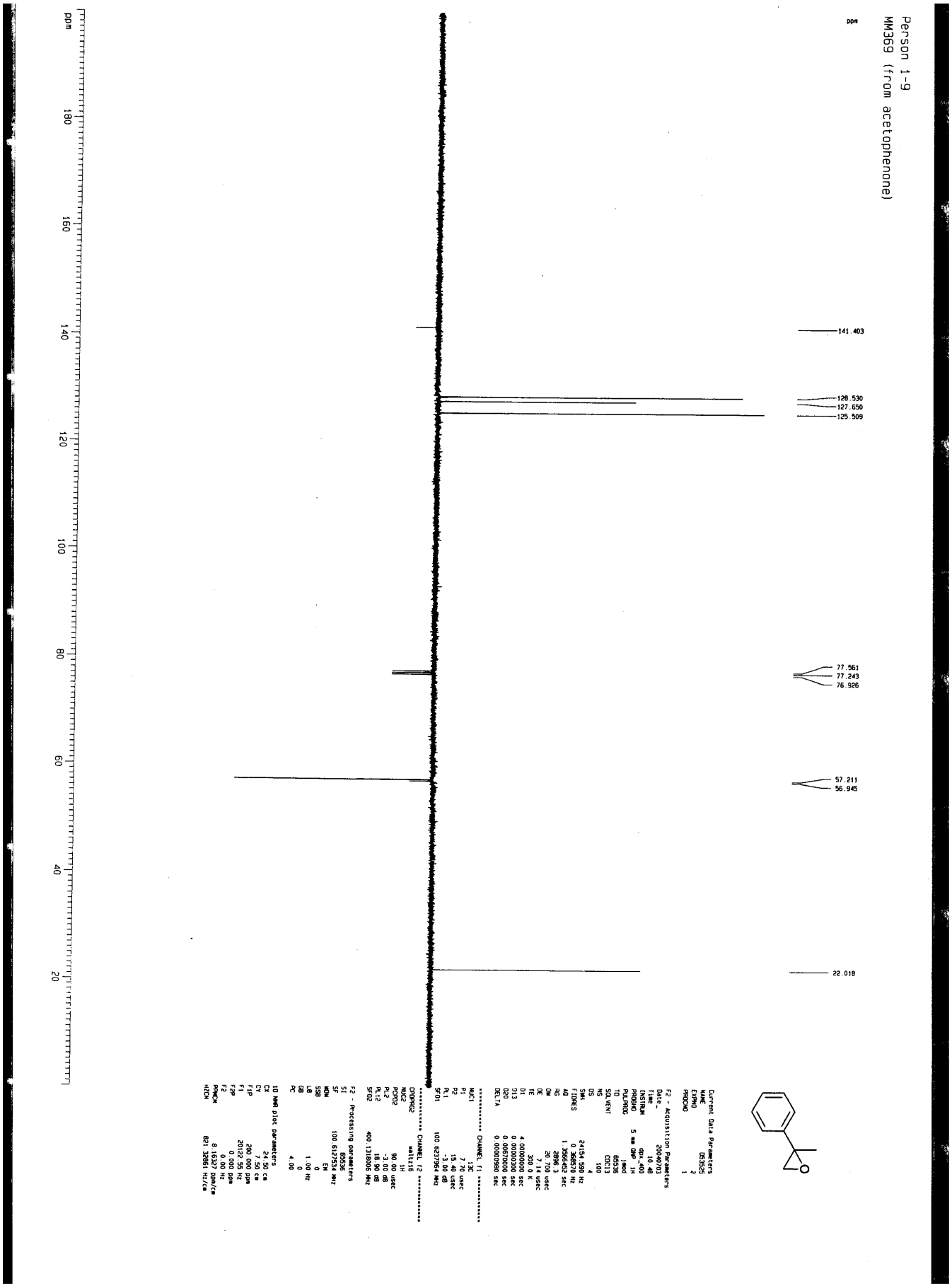
HZCM 163.31836 Hz/cm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22.018
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PROCNO 1
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TD 65536
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FIDRES 0.368549 Hz
AQ 1.3564629 sec
RG 2896.3
DW 20.700 usec
DE 7.14 usec
TE 300.0 K
D1 4.00000000 sec
D11 0.03000000 sec
D20 0.06570000 sec
DELTA 0.00009980 sec
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P1 7.70 usec
PL1 -1.50 dB
SF01 100.6232964 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL12 18.06 dB
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F2 - Processing parameters
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SF 100.6125764 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
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F1 20112.55 Hz
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HZCM 821.32861 Hz/cm
ppm
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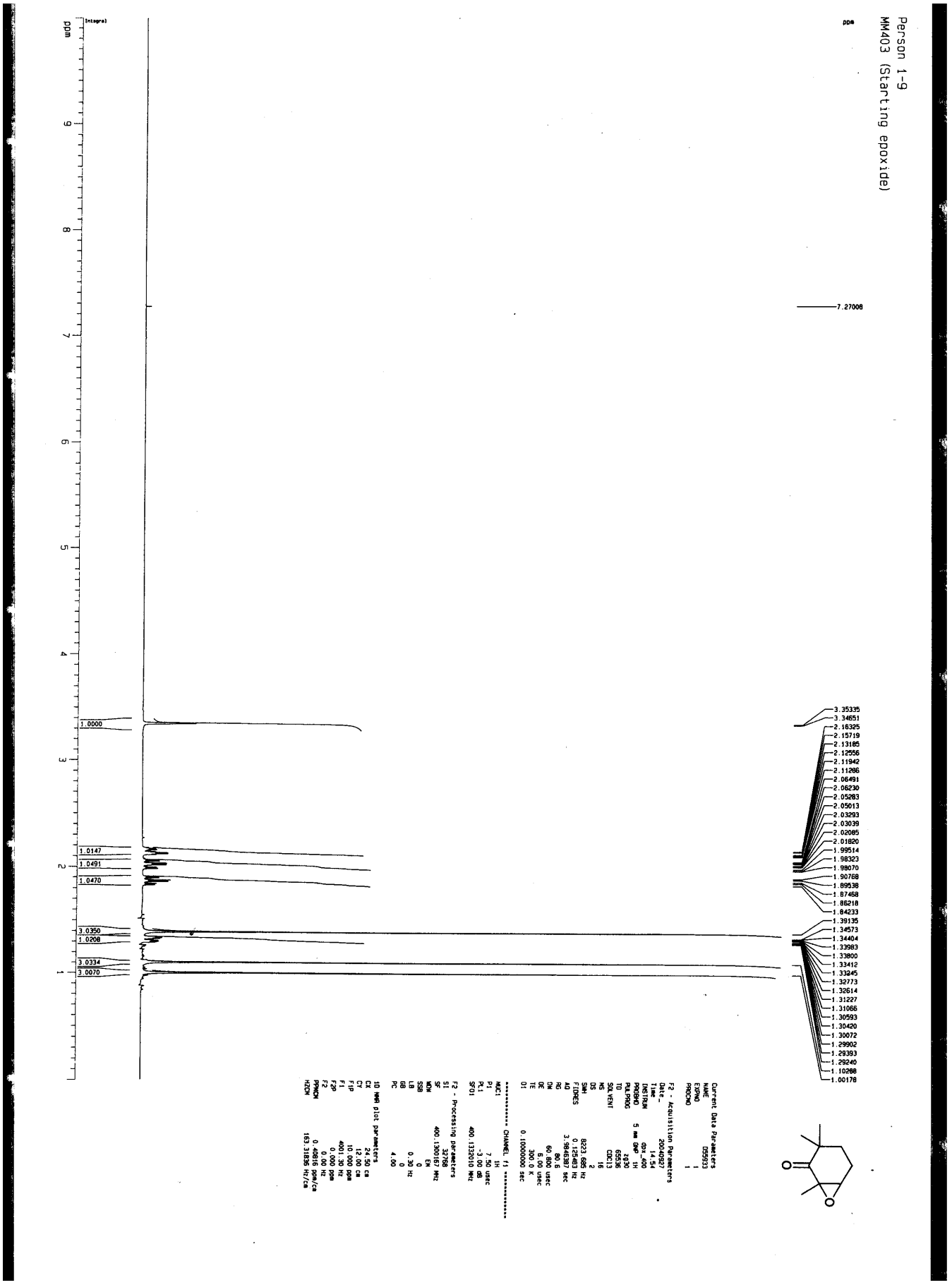
160
140
120
100
80
60
40
20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3.0070
7.27008
3.35335
3.34651
2.16325
2.15719
2.13185
2.12556
2.11942
2.11286
2.06491
2.06230
2.05283
2.05013
2.03293
2.03039
2.02085
2.01820
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1.98323
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1.32614
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PROCNO 1
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125463 Hz
AQ 3.9845387 sec
RG 80.6
DW 60.800 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 -3.00 dB
SFO1 400.1320010 MHz
F2 - Processing parameters
SI 32768
SF 400.1300167 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 24.50 cm
CY 12.00 cm
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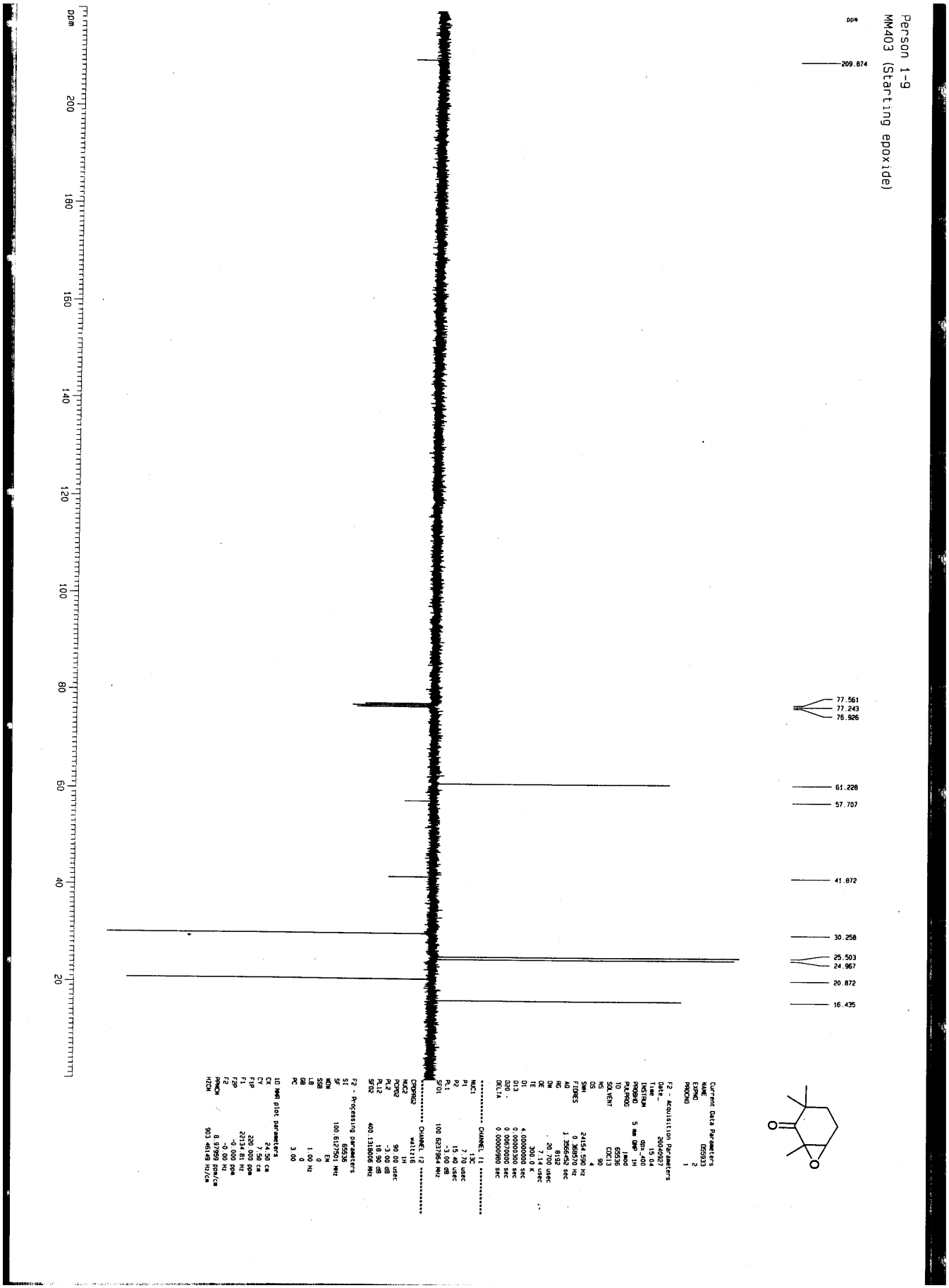
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.40000 ppm/cm
HZCM 160.31836 Hz/cm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20.872
16.435
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EXPNO 2
PROCNO 1
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Date_ 20040127
Time 14.15.24
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PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 90
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SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 8192
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DE 30.14 usec
TE 300.0 K
D1 4.00000009 sec
D13 0.00000300 sec
D20 0.00670000 sec
DELTA 0.00000980 sec
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NUC1 1H
P1 7.70 usec
P2 15.40 usec
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SFO1 100.6237964 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 18.90 dB
SFO2 400.1318006 MHz
F2 - Processing parameters
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SF 100.6127501 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 3.00
1D NMR plot parameters
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CY 7.50 cm
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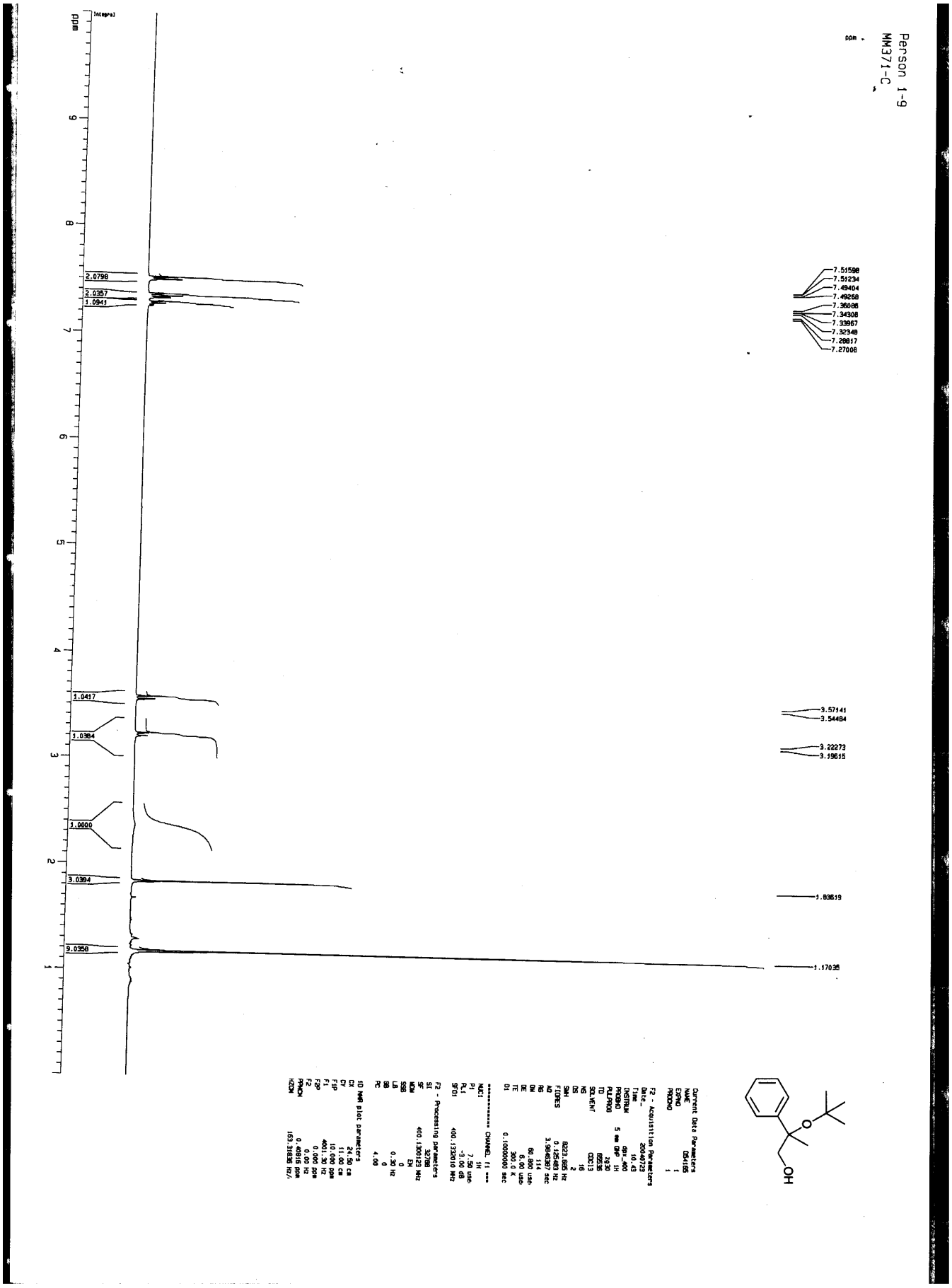
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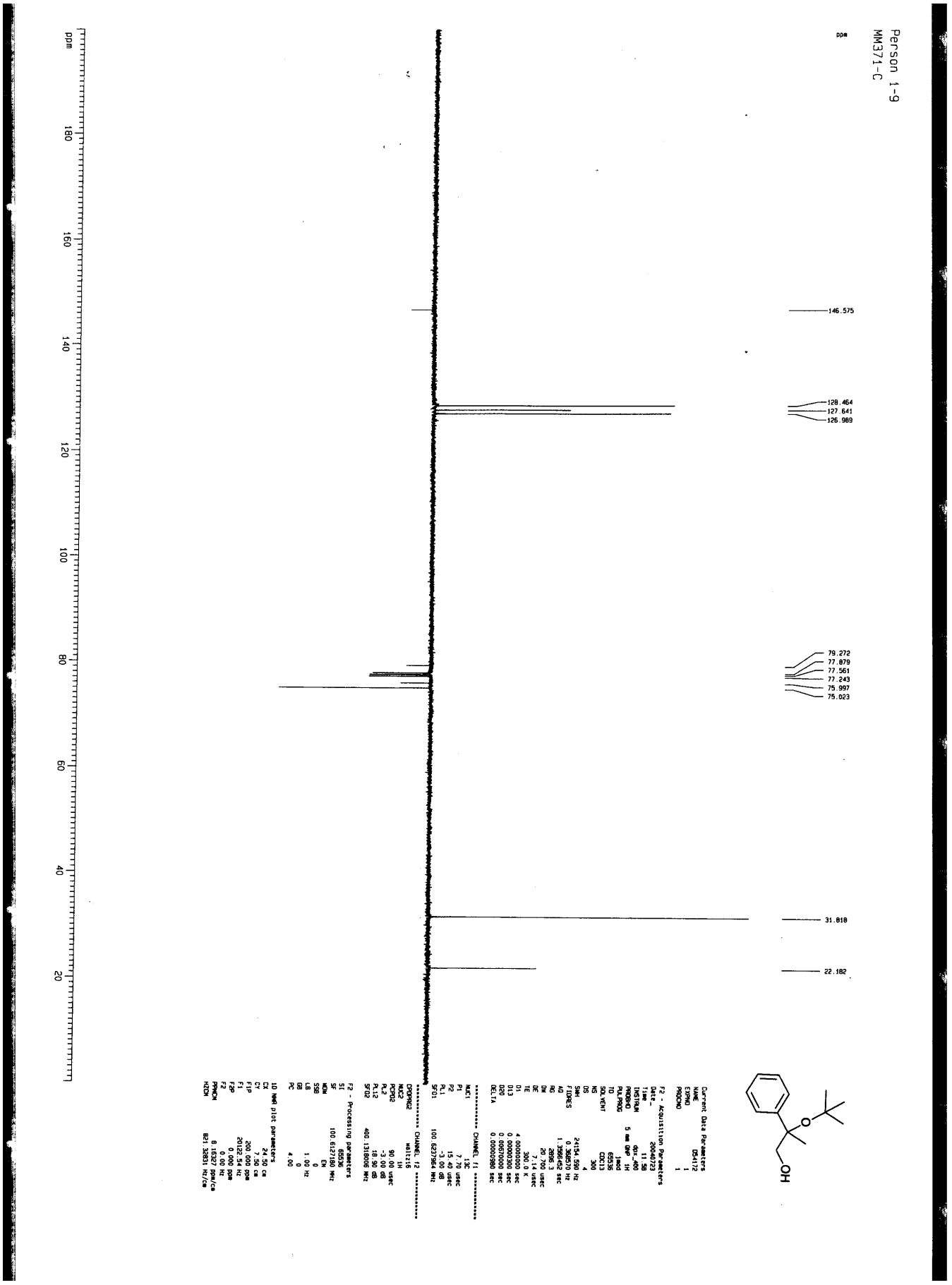
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22.182
OH
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PROCNO 1
F2 - Acquisition Parameters
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Time 11.58
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RG 203.3
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TE 300.0 K
D1 4.00000000 sec
D13 0.00000300 sec
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P2 15.40 usec
PL1 -3.00 dB
SFO1 100.6237954 MHz
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NUC2 1H
PCPD2 90.00 usec
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PL12 18.06 dB
SFO2 400.1318098 MHz
F2 - Processing parameters
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SF 100.6127690 MHz
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SSB 0
LB 1.00 Hz
GB 0
PC 4.00
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F2P 0.000 ppm
F2 0.00 Hz
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HZCM 821.32831 Hz/cm