# Cinchona Alkaloid-Lewis Acid Catalyst Systems for Enantioselective Ketene-Aldehyde Cycloadditions

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## Supporting Information

**General Information:** Optical rotations were measured on a Perkin-Elmer 241 digital polarimeter with a sodium lamp at ambient temperature and are reported as follows: $[\alpha]_\lambda$ (c g/100mL). Infrared spectra were recorded on a Nicolet Avatar 360 FT-IR spectrometer. NMR spectra were recorded on a Bruker Avance-300 ($^1$H: 300 MHz; $^{13}$C: 75 MHz) spectrometer with chemical shifts reported relative to residual $CHCl_3$ (7.27 ppm) for $^1$H and $CDCl_3$ (77.0 ppm) for $^{13}$C NMR spectra. Analytical high performance liquid chromatography (HPLC) was performed on a Hewlett Packard 1100 liquid chromatograph equipped with a variable wavelength UV detector (deuterium lamp, 190-600 nm) using a Daicel Chiracel™ OD-H column (250 x 4.6 mm) (Daicel Inc.) or Daicel Chiralpak™ AD column (250 x 4.6 mm) (Daicel Inc.) and HPLC-grade isopropanol and hexanes as the eluting solvents. Analytical gas-liquid chromatography (GLC) was performed on a Varian 3900 gas chromatography equipped with a flame ionization detector and split mode capillary injection system using a Chiraldex™ G-TA column (20 m x 0.25 mm) (Advanced Separation Technologies Inc.). Helium was used as the carrier gas at the indicated pressures. Unless otherwise stated, all reactions were carried out in dry glassware under a nitrogen atmosphere using standard inert atmosphere techniques for the manipulation of solvents and reagents. Anhydrous solvents ($CH_2Cl_2$ and diethyl ether) were obtained by passage through successive alumina- and Q5 reactant-packed columns on a solvent purification system. *N*, *N*-Diisopropylethylamine was distilled under nitrogen from $CaH_2$. All commercially available aldehydes were redistilled under $N_2$. Commercially available acetyl chloride and propionyl chloride were redistilled under $N_2$. Anhydrous $LiClO_4$ (ReagentPlus) was purchased from Aldrich Chemical Co. and weighed out in a $N_2$-filled glovebox. *O*-Trimethylsilylquinidine (TMS*Q*) and *O*-trimethylsilylquinine (TMS*q*) were prepared according to the literature procedure (TMSCl was added at 0°C for large scale preparations).[1] Flash chromatography was performed as previously described on EM silica gel 60 (230-240 mesh).[2] Characterization data for β-lactones **7a,b**,[3] **7c,d**[4] **7e,f,j**,[5] and **7g**[6] have been reported previously.

**General procedure for asymmetric ketene-aldehyde cycloadditions:** To a solution of TMS*Q* (or TMS*q*) (0.10 mmol) and $LiClO_4$ (0.3-3.0 mmol) in 1.0 ml of diethyl ether was added 2.0 ml of $CH_2Cl_2$ and the reaction mixture was cooled to the indicated reaction temperature (–78 or –40 °C). To the resulting mixture was added 0.44 ml of *N*, *N*-diisopropylethylamine (2.5 mmol) followed by the aldehyde (1.0 mmol). A solution of 2.0 mmol of acid chloride in 0.5 ml of $CH_2Cl_2$ was then added over 1-4 h by syringe pump. The reaction mixture was stirred for 7-16 h then was quenched at the reaction temperature by adding 10 ml of $Et_2O$ and the resulting mixture was filtered through silica gel eluting with $Et_2O$ (3 x 20 mL). The filtrate was concentrated *in vacuo* and the crude product mixture was purified by flash chromatography.

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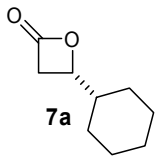
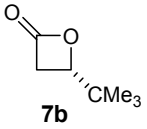
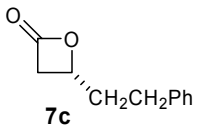


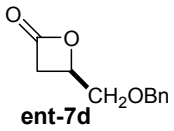

***(S)*-4-Cyclohexyloxetan-2-one (7a)**: The General Procedure was followed employing 40 mg of TMS*Q* (0.10 mmol, 10 mol%), 212 mg $LiClO_4$ (2.0 mmol, 200 mol%) and 120 µL of cyclohexanecarboxaldehyde (1.0 mmol) at a reaction temperature of –40 °C. Purification by flash chromatography (15% diethyl ether in pentane) gave 131 mg (85%) of the title compound as a colorless crystalline solid. Separation of the enantiomers by chiral GLC [Chiraldex™ G-TA column 20 m x 0.25 mm, flow rate 0.5 mL/min, method: 100 °C for 10 min, ramp @ 15.0 °C/min to 130 °C for 8.0 min, ramp @ 15.0 °C/min to 160 °C for 10.0 min, $T_r$: 24.5 min (4*S*), $T_r$: 26.2 min (4*R*)] provided the enantiomer ratio (4*S*):(4*R*) = 97:3 (94% ee). $[\alpha]_D$ +19.8 (*c* 1.48, $CHCl_3$).



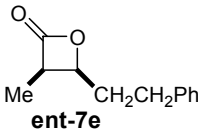

***(S)*-4-*tert*-Butyloxetan-2-one (7b)**: The General Procedure was followed employing 40 mg of TMS*Q* (0.10 mmol, 10 mol%), 318 mg $LiClO_4$ (3.0 mmol, 300 mol%) and 109 µL of pivalaldehyde (1.0 mmol) at a reaction temperature of –78 °C. Purification by flash chromatography (15% diethyl ether in pentane) gave 91.3 mg (71%) of the title compound as a colorless oil. Separation of the enantiomers by chiral GLC [Chiraldex™ G-TA column 20 m x 0.25 mm, flow rate 0.6 mL/min, method: 80 °C for 5.0 min, ramp @ 5.0 °C/min to 100 °C for 10.0 min, ramp @ 5.0 °C/min to 130 °C for 5.0 min, ramp @ 15.0 °C/min to 150 °C for 5.0 min, $T_r$: 17.8 min (4*R*), $T_r$: 19.0 min (4*S*)] provided the enantiomer ratio (4*S*):(4*R*) = 98:2 (96% ee). $[\alpha]_D$ +20.7 (*c* 2.00, $CHCl_3$).



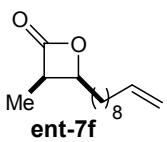

***(R)*-4-Phenethyloxetan-2-one (7c)**: The General Procedure was followed employing 40 mg of TMS*Q* (0.10 mmol, 10 mol%), 53 mg $LiClO_4$ (0.5 mmol, 50 mol%) and 132 µL of hydrocinnamaldehyde (1.0 mmol) at a reaction temperature of –78 °C. Purification by flash chromatography (25% diethyl ether in pentane) gave 141 mg (80%) of the title compound as a colorless oil. Separation of the enantiomers by chiral HPLC [Daicel Chiracel™ OD-H column, flow rate 1.0 mL/min, 10% $^i$PrOH, 90% hexane, $T_r$ 15.5 min (*S*) and 17.3 min (*R*)] provided the enantiomer ratio: (*R*):(*S*) = 96:4 (92% ee). $[\alpha]_D$ +48.9 (*c* 1.20, $CHCl_3$)




***(R)*-4-(Benzyloxy)methyl)oxetan-2-one (ent-7d)**: The General Procedure was followed employing 40 mg of TMS*q* (0.10 mmol, 10 mol%), 32 mg $LiClO_4$ (0.3 mmol, 30 mol%) and 140 µL of benzyloxyacetaldehyde (1.0 mmol) at a reaction temperature of –78 °C. Purification by flash chromatography (25% ethyl acetate in hexane) gave 134 mg (70%) of the title compound as a colorless oil. Separation of the enantiomers by chiral HPLC [Daicel Chiracel™ OD-H column, flow rate 0.9 mL/min, 15% $^i$PrOH, 85% hexane, $T_r$ 13.9 min (*R*) and 24.8 min (*S*)] provided the enantiomer ratio: (*R*):(*S*) = 92:8 (84% ee). $[\alpha]_D$ –13.9 (*c* 1.80, $CHCl_3$).

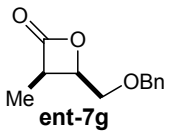



***(3R, 4S)*-3-Methyl-4-phenethyloxetan-2-one (ent-7e)**: The General Procedure was followed employing 40 mg of TMS*q* (0.10 mmol, 10 mol%), 53 mg $LiClO_4$ (0.5 mmol, 50 mol%) and 132 µL of hydrocinnamaldehyde (1.0 mmol) at a reaction temperature of –78 °C. Purification by flash chromatography (10% ethyl acetate in hexane) gave 161 mg (84%) of the title compound as a colorless oil. Separation of enantiomers by Chiral HPLC [Daicel Chiracel™ OD-H column, flow rate 1.0 mL/min, 5% $^i$PrOH, 95% hexane, $T_r$: 12.9 min (3*S*, 4*R*), 14.0 min (3*R*, 4*S*)] provided only one enantiomer (3*R*, 4*S*) (>99% ee). $[\alpha]_D$ –47.2 (*c* 2.04, $CHCl_3$).



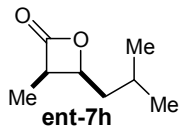

***(3R, 4S)*-4-(Dec-9-enyl)-3-methyloxetan-2-one (ent-7f)**: The General Procedure was followed employing 40 mg of TMS*q* (0.10 mmol, 10 mol%), 106 mg $LiClO_4$ (1.0 mmol, 100 mol%) and 208 µL of undecylenic aldehyde (1.0 mmol) at a reaction temperature of –78 °C. Purification by flash chromatography (5% ethyl acetate in hexane) gave 165 mg (74%) of the title compound as a colorless oil. Separation of enantiomers by chiral GLC [Chiraldex™ G-

TA column 20 m x 0.25 mm, flow rate 1.0 mL/min, method: 100 °C ramp @ 10.0 °C/min to 150 °C for 30.0 min, ramp @ 10.0 °C/min to 160 °C for 10.0 min, $T_r$: 29.2 min (3*S*, 4*R*) min and 30.0 min (3*R*, 4*S*)] provided the enantiomer ratio (3*R,* 4*S*):(3*S,* 4*R*) = 99.4:0.6 (99% ee). $[\alpha]_D$ –27.5 (*c* 1.99, $CHCl_3$).



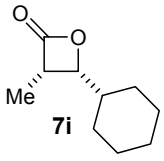
**ent-7g**

***(3R, 4R)*-4-(Benzyloxy)methyl-3-methyloxetan-2-one (ent-7g):** The General Procedure was followed employing 40 mg of TMS*q* (0.10 mmol, 10 mol%), 32 mg $LiClO_4$ (0.3 mmol, 30 mol%) and 140 µL of benzyloxyacetaldehyde (1.0 mmol) at a reaction temperature of –78 °C. Purification by flash chromatography (20% ethyl acetate in hexane) gave 140 mg (68%) of the title compound as a colorless oil. Separation of the enantiomers by chiral HPLC [Daicel Chiracel™ OD-H column, flow rate 1.0 mL/min, 10% $^i$PrOH, 90% hexane, $T_r$ 13.4 min (3*R,* 4*R*) and 31.4 min (3*S,* 4*S*)] provided the enantiomer ratio: (3*R,* 4*R*):(3*S,* 4*S*) = 99.5:0.5 (99% ee). $[\alpha]_D$ –7.6 (*c* 1.58, $CHCl_3$).



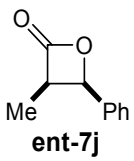
**ent-7h**

***(3R, 4S)*-4-Isobutyl-3-methyloxetan-2-one (ent-7h)**: The General Procedure was followed employing 40 mg of TMS*q* (0.10 mmol, 10 mol%), 212 mg $LiClO_4$ (2.0 mmol, 200 mol%) and 109 µL of isovaleraldehyde (1.0 mmol) at a reaction temperature of –78 °C. Purification by flash chromatography (20% diethyl ether in pentane) gave 103 mg (72%) of the title compound as a colorless oil. Separation of enantiomers by chiral GLC [Chiraldex™ G-TA column 20 m x 0.25 mm, flow rate 0.6 mL/min, method: 80 °C for 5.0 min, ramp @ 5.0 °C/min to 100 °C for 10.0 min, ramp @ 5.0 °C/min to 130 °C for 5.0 min, $T_r$: 23.9 min (3*R,* 4*S*) and 25.0 min (3*S,* 4*R*)] provided the enantiomer ratio (3*R,* 4*S)*: (3*S,* 4*R*) = 99.7:0.3 (99.4% ee). $[\alpha]_D$ −41.8 (*c* 1.88, $CHCl_3$). IR (thin film): 1824, 1465, 1124 $cm^{-1}$; $^1$H NMR (300 MHz, $CDCl_3$) δ 4.67 (ddd, J = 9.7, 6.5, 3.8 Hz, 1H), 3.77 (qd, J = 7.8, 6.5 Hz, 1H), 1.82 (m, 1H), 1.71 (ddd, J = 14.3, 9.7, 6.0 Hz, 1H), 1.51 (ddd, J = 14.3, 7.6, 3.8 Hz, 1H), 1.29 (d, J = 7.8 Hz, 3H), 1.01 (d, J = 6.6 Hz, 3H), 0.99 (d, J = 6.6 Hz, 3H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 172.7, 74.2, 47.4, 38.5, 25.2, 22.8, 22.0, 8.1; Anal. calcd for $C_8H_{14}O_2$: C, 67.57; H, 9.92; found: C, 67.44; H, 10.20.



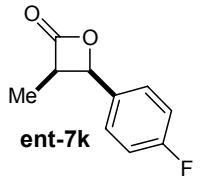
**7i**

***(3S, 4R)*-4-Cyclohexyl-3-methyloxetan-2-one (7i)**: The General Procedure was followed employing 40 mg of TMS*Q* (0.10 mmol, 10 mol%), 212 mg $LiClO_4$ (2.0 mmol, 200 mol%) and 120 µL of cyclohexanecarboxaldehyde (1.0 mmol) at a reaction temperature of –40 °C. Purification by flash chromatography (15% diethyl ether in pentane) gave 124 mg (74%) of the title compound as a colorless crystalline solid. Separation of enantiomers by chiral GLC [Chiraldex™ G-TA column 20 m x 0.25 mm, flow rate 0.5 mL/min, method: 100 °C for 10 min, ramp @ 15.0 °C/min to 130 °C for 8.0 min, ramp @ 15.0 °C/min to 160 °C for 15.0 min, $T_r$: 26.8 min (3*S*, 4*R*), $T_r$: 27.7 min (3*R*, 4*S*)] provided the enantiomer ratio (3*S*, 4*R*): (3*R*, 4*S*) = 98.5: 1.5 (97% ee). $[\alpha]_D$ –13.4 (*c* 1.45, $CHCl_3$). IR (thin film): 2932, 2854, 1825, 1132$cm^{-1}$; $^1$H NMR (300 MHz, $CDCl_3$) δ 4.18 (dd, J = 10.6, 6.3 Hz, 1H), 3.72 (qd, J = 7.8, 6.3 Hz, 1H), 2.01-1.92 (m, 1H), 1.82-1.52 (m, 5H), 1.34 (d, J = 7.8 Hz, 3H), 1.32-1.19 (m, 3H), 1.07-0.88 (m, 2H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 172.9, 79.0, 46.8, 37.7, 28.9, 28.1, 26.0, 25.0 (2C), 8.4; HRMS *m/z* calcd for $C_{10}H_{16}O_2$: 168.1150; found: 168.1158.

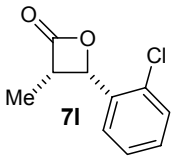


**ent-7j**

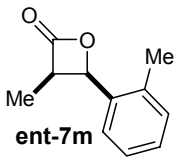
***(3R, 4R)*-3-Methyl-4-phenyloxetan-2-one (ent-7j)**: The General Procedure was followed employing 40 mg of TMS*q* (0.10 mmol, 10 mol%), 212 mg $LiClO_4$ (2.0 mmol, 200 mol%) and 102 µL of benzaldehyde (1.0 mmol) at a reaction temperature of –78 °C. Purification by flash chromatography (10% ethyl acetate in hexane) gave 126 mg (78%) of the title compound as a colorless oil. Separation of the enantiomers by chiral HPLC [Daicel Chiracel™ OD-H column, flow rate 1.0 mL/min, 3% $^i$PrOH, 97% hexane, $T_r$ 10.8 min (3*R,* 4*R*), 11.7 min (3*S,* 4*S*)] provided only one enantiomer (3*R,* 4*R*) (>99% ee). $[\alpha]_D$ +139 (*c* 1.79, $CHCl_3$).




**(3*R*, 4*R*)-4-(4-Fluorophenyl)-3-methyloxetan-2-one (ent-7k):** The General Procedure was followed employing 40 mg of TMS*q* (0.10 mmol, 10 mol%), 212 mg $LiClO_4$ (2.0 mmol, 200 mol%) and 107 μL of 4-fluorobenzaldehyde (1.0 mmol) at a reaction temperature of –78 °C. Purification by flash chromatography (20% ethyl acetate in hexane) gave 153 mg (85%) of the title compound as a colorless oil. Separation of enantiomers by chiral HPLC [Daicel Chiralpak™ AD column, flow rate 1.0 mL/min, 5% $^{i}$PrOH, 95% hexane, $T_r$ 9.0 min (3*S,* 4*S*), 9.7 min (3*R,* 4*R*)] provided only one enantiomer (3*R,* 4*R*) (>99% ee). $[\alpha]_D$ +124 (*c* 1.03, $CHCl_3$); IR (thin film): 1828, 1510, 1228cm$^{-1}$; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.30 (m, 2H), 7.13 (t J = 8.6 Hz, 2H), 5.65 (d, J = 6.4 Hz, 1H), 4.06 (qd, J = 7.8, 6.4 Hz, 1H), 0.95 (d, J = 7.8 Hz, 3H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 171.8, 162.7 (d, J = 246 Hz), 130.4 (d, J = 2.9 Hz), 127.5 (d, J = 8.3 Hz), 115.6 (d, J = 21.7 Hz), 74.6, 50.1, 9.4; HRMS *m/z* calcd for $C_{10}H_9FO_2$: 180.0587; found: 180.0592.




**(3*S*, 4*S*)-4-(2-Chlorophenyl)-3-methyloxetan-2-one (7l):** The General Procedure was followed employing 40 mg of TMS*Q* (0.10 mmol, 10 mol%), 212 mg $LiClO_4$ (2.0 mmol, 200 mol%) and 113 μL of 2-chlorobenzaldehyde (1.0 mmol) at a reaction temperature of –78 °C. Purification by flash chromatography (10% diethyl ether in pentane) gave 158 mg (80%) of the title compound as a colorless oil. Separation of enantiomers by chiral HPLC [Daicel Chiracel™ OD-H column, flow rate 0.6 mL/min, 2% $^{i}$PrOH, 98% hexane, $T_r$ 13.4 min (3*R,* 4*R*), 14.1 min (3*S,* 4*S*)] provided only one enantiomer (3*S,* 4*S*) (>99% ee). $[\alpha]_D$ –201 (*c* 1.68, $CHCl_3$); IR (thin film): 2979, 1827, 1472, 1444, 1289, 1055, 946, 880, 755cm$^{-1}$; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.55-7.52 (m, 1H), 7.43-7.31 (m, 3H), 5.86 (d, J = 6.3 Hz, 1H), 4.16 (qd, J = 7.7, 6.3 Hz, 1H), 0.98 (d, J = 7.7 Hz, 3H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 171.5, 133.0, 131.2, 129.6, 129.3, 127.2, 126.9, 73.3, 50.3, 9.1; HRMS *m/z* calcd for $C_{10}H_9ClO_2$: 196.0291; found: 196.0299.




**(3*R*, 4*R*)-3-Methyl-4-*ortho*-tolyloxetan-2-one (ent-7m):** The General Procedure was followed employing 40 mg of TMS*q* (0.10 mmol, 10 mol%), 212 mg $LiClO_4$ (2.0 mmol, 200 mol%) and 116 μL of 2-methylbenzaldehyde (1.0 mmol) at a reaction temperature of –40 °C. Purification by flash chromatography (10% ethyl acetate in hexane) gave 134 mg (76%) of the title compound as colorless oil. Separation of enantiomers by chiral HPLC [Daicel Chiracel™ OD-H column, flow rate 0.5 mL/min, 4% $^{i}$PrOH, 96% hexane, $T_r$ 18.1 min (3*R,* 4*R*), 19.1 min (3*S,* 4*S*)] provided only one enantiomer (3*R,* 4*R*) (>99% ee). $[\alpha]_D$ +202 (*c* 1.49, $CHCl_3$); IR (thin film): 2978, 1829,1493, 1260cm$^{-1}$; $^1$H NMR (300 MHz, $CDCl_3$) δ 7.48-7.45 (m, 1H), 7.31-7.20 (m, 3H), 5.78 (d, J = 6.3 Hz, 1H), 4.08 (qd, J = 7.7, 6.3 Hz, 1H), 2.22 (s, 3H), 0.91 (d, J = 7.7 Hz, 3H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 171.9, 133.8, 132.9, 130.2, 128.2, 126.3, 124.9, 73.7, 49.5, 18.7, 9.2; HRMS *m/z* calcd for $C_{11}H_{12}O_2$: 176.0837; found: 176.0831.

**Stereochemical proofs for β-lactones**: The absolute configuration of β-lactones **ent-7h** and **7i** was established by conversion to the corresponding methyl esters **8h** and **8i** using the published procedure ($La(O^tBu)_3$, MeOH)[7] and correlating the ester's specific rotations to those of authentic samples of known configuration: **8h** $[\alpha]_D^{23}$ = –21 (2*R*,3*S*) (*c* 0.58, $CHCl_3$) [lit $[\alpha]_D^{23}$ = +16 (2*S*,3*R*) (*c* 0.48, $CHCl_3$)];[8] **8i** $[\alpha]_D^{23}$ = +5.2 (2*S*,3*R*) (*c* 1.64, $CHCl_3$) [lit $[\alpha]_D^{23}$ = –5.5 (2*R*,3*S*) (*c* 0.3, $CHCl_3$)].[9] The configuration of lactone **ent-7j** was established similarly by conversion to the corresponding ethyl ester **8j** according to the literature procedure [$La(O^tBu)_3$, EtOH]: **8j** $[\alpha]_D^{23}$ = +20.6 (2*R*,3*R*) (*c* 1.07, $CHCl_3$) [lit $[\alpha]_D^{23}$ = –22.0 (2*S*,3*S*) (*c* 0.87, $CHCl_3$)].[10] The absolute configuration of β-lactone **ent-7e**

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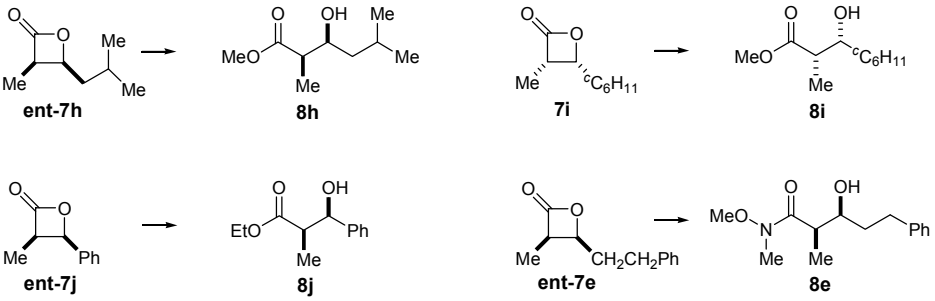
9 Carda, M.; Murga, J.; Falomir, E.; Gonzalez, F.; Marco, J. A. *Tetrahedron: Asymm.* **2000**, *11*, 3211-3220.

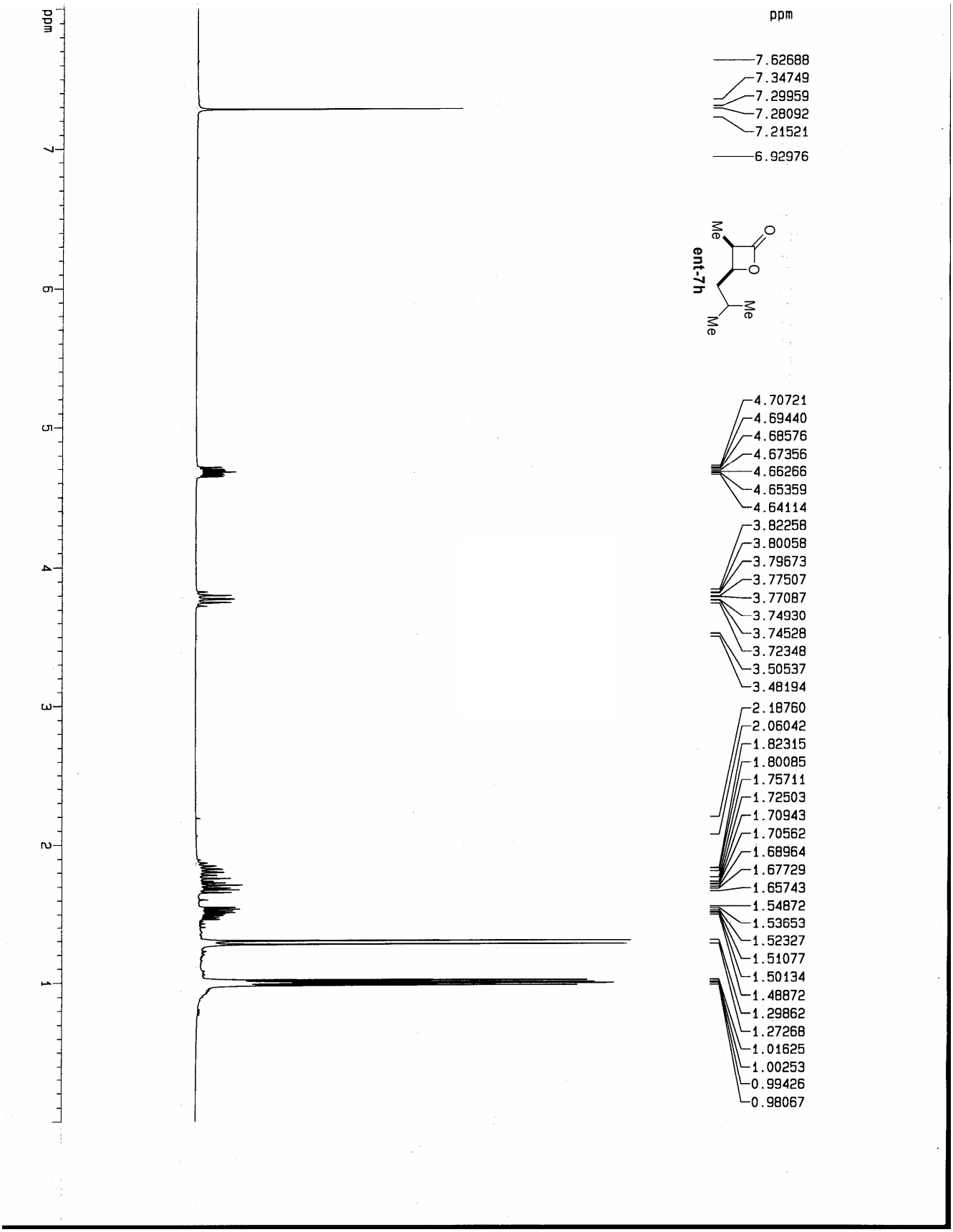
10 Hena, M. A.; Terauchi, S.; Kim, C.-S.; Horiike, M.; Kiyooka, S.-i. *Tetrahedron: Asymm.* **1998**, *9*, 1883-1890.

was established similarly by conversion to the corresponding *N*-methoxy-*N*-methyl amide **8e** using the published procedure [$MeO(Me)NH_2Cl$, $AlMe_3$]:[11] $[\alpha]_D^{24} = -28.8$ (2*R*,3*S*) (*c* 1.56, $CHCl_3$) [lit $[\alpha]_D^{25} = +24.4$ (2*S*,3*R*) (*c* 1.33, $CHCl_3$)].[12] The configuration of the remaining β-lactones **7** and **ent-7** was assigned by analogy to these determinations.




[11] Nelson, S. G.; Cheung, W. S.; Kassick, A. J. Hilfiker, M. A. *J. Am. Chem. Soc.* **2002**, *124*, 13654-13655.

[12] Supporting Information for Marshall, J. A.; Adams, N. D. *J. Org. Chem.* **1999**, *64*, 5201-5204.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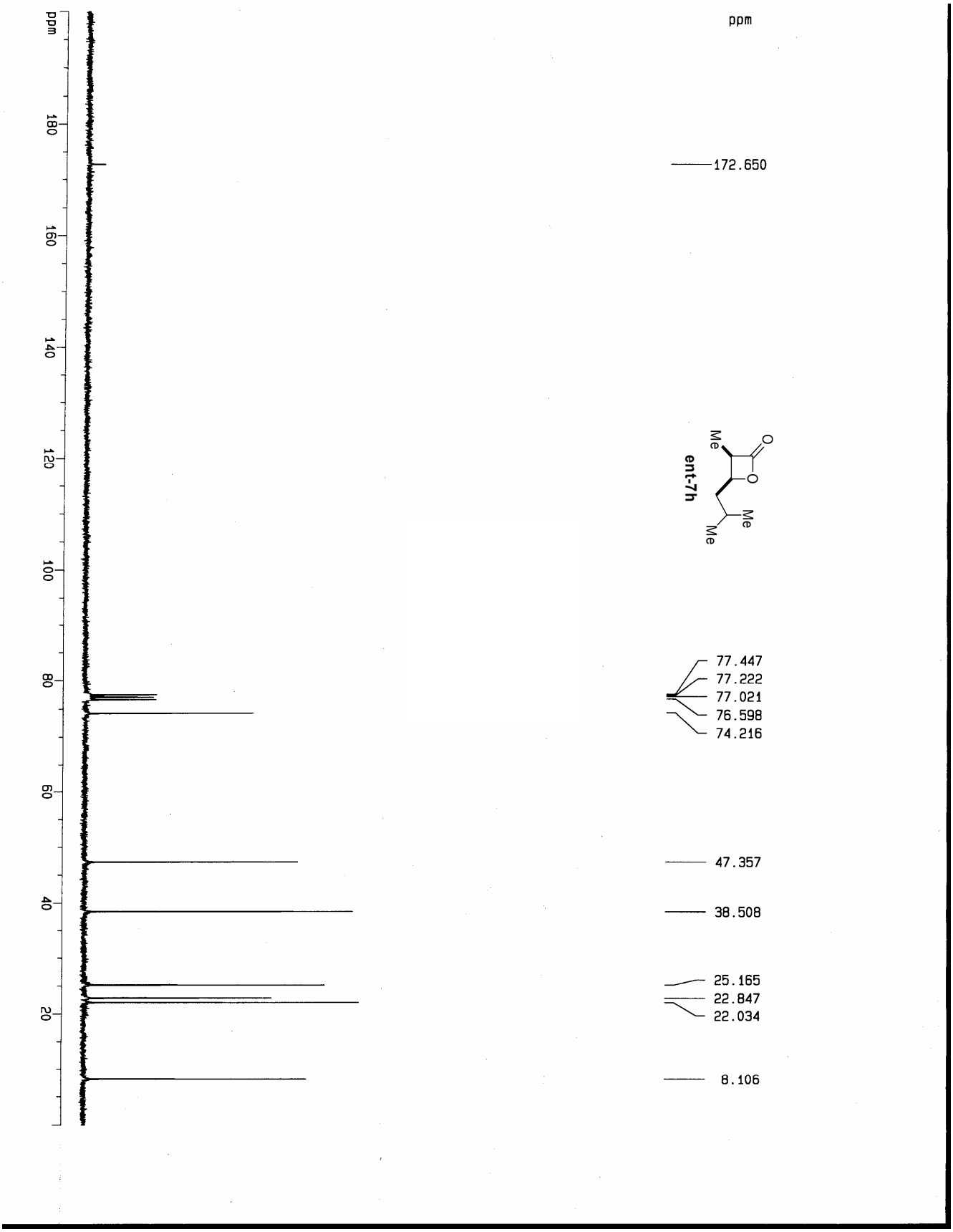
7.29959
7.28092
7.21521
6.92976
Me
O
O
Me
Me
ent-7h
4.70721
4.69440
4.68576
4.67356
4.66266
4.65359
4.64114
3.82258
3.80058
3.79673
3.77507
3.77087
3.74930
3.74528
3.72348
3.50537
3.48194
2.18760
2.06042
1.82315
1.80085
1.75711
1.72503
1.70943
1.70562
1.68964
1.67729
1.65743
1.54872
1.53653
1.52327
1.51077
1.50134
1.48872
1.29862
1.27268
1.01625
1.00253
0.99426
0.98067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180
160
140
120
100
80
60
40
20
ppm
172.650
Me
O
O
Me
Me
ent-7h
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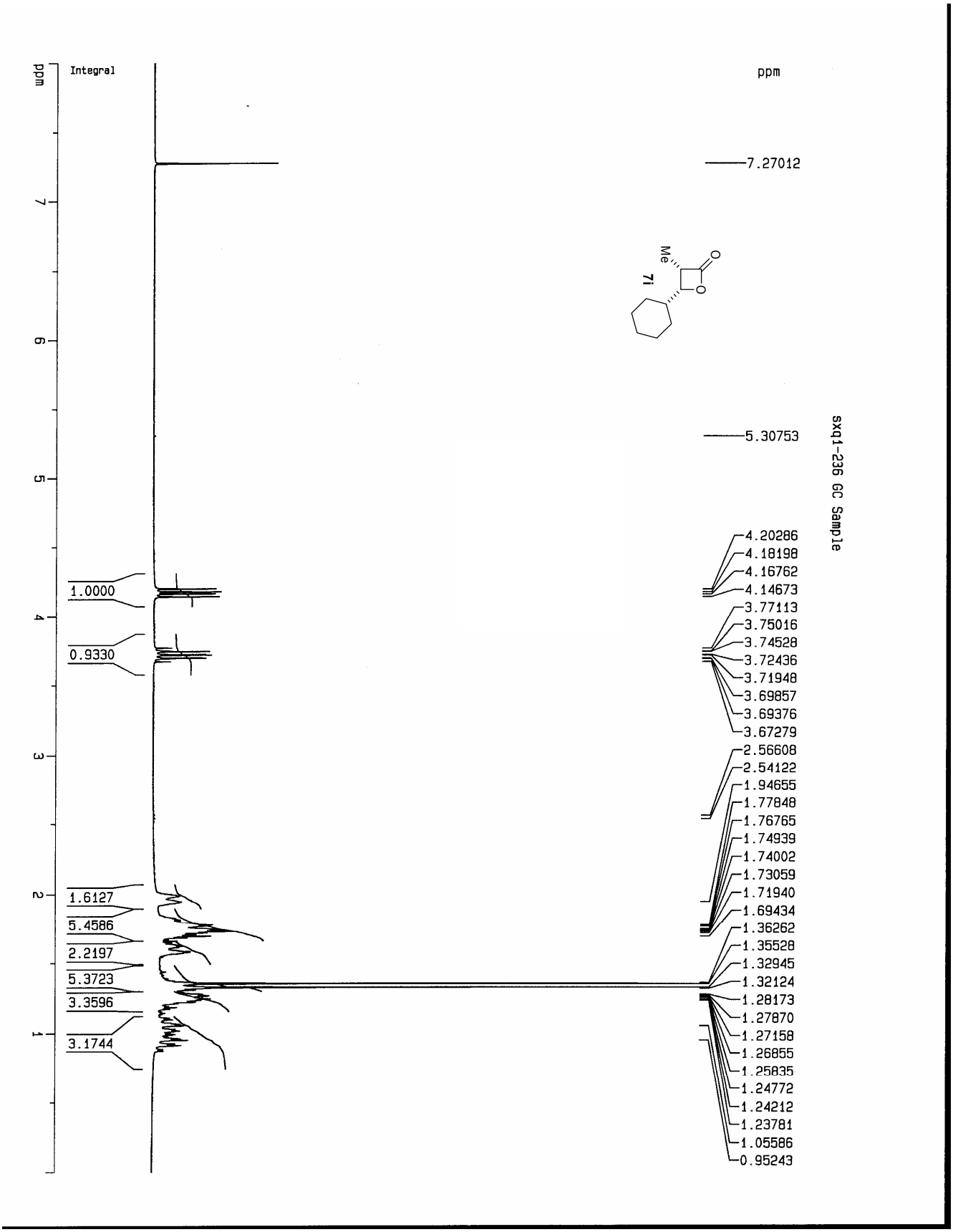
77.222
77.021
76.598
74.216
47.357
38.508
25.165
22.847
22.034
8.106


Integral
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5.30753
Me
O
O
7i
sxq1-236 GC Sample
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4.18198
4.16762
4.14673
3.77113
3.75016
3.74528
3.72436
3.71948
3.69857
3.69376
3.67279
2.56608
2.54122
1.94655
1.77848
1.76765
1.74939
1.74002
1.73059
1.71940
1.69434
1.36262
1.35528
1.32945
1.32124
1.28173
1.27870
1.27158
1.26855
1.25835
1.24772
1.24212
1.23781
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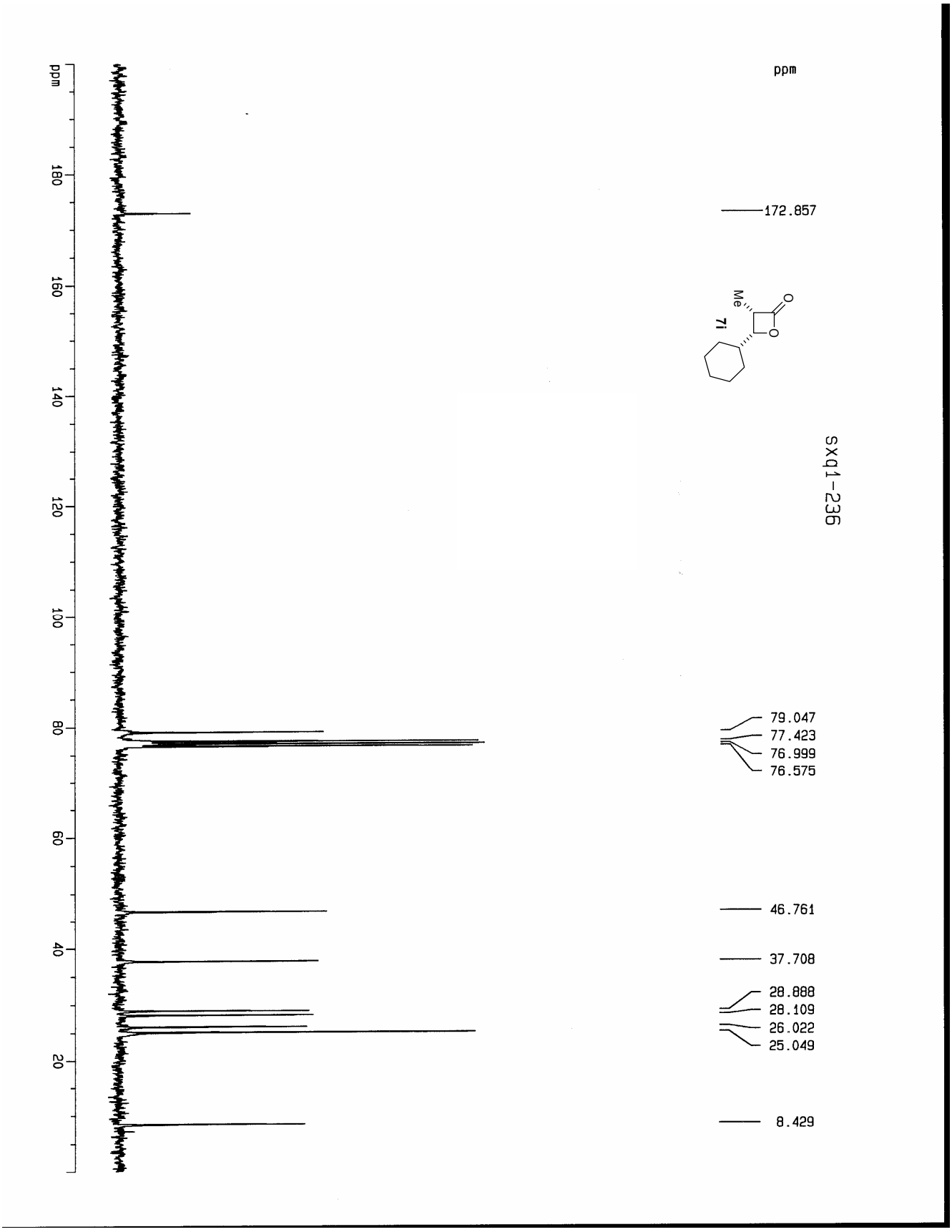
2.2197
5.3723
3.3596
3.1744


ppm
180
160
140
120
100
80
60
40
20
ppm
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Me
O
O
7i
sxq1-236
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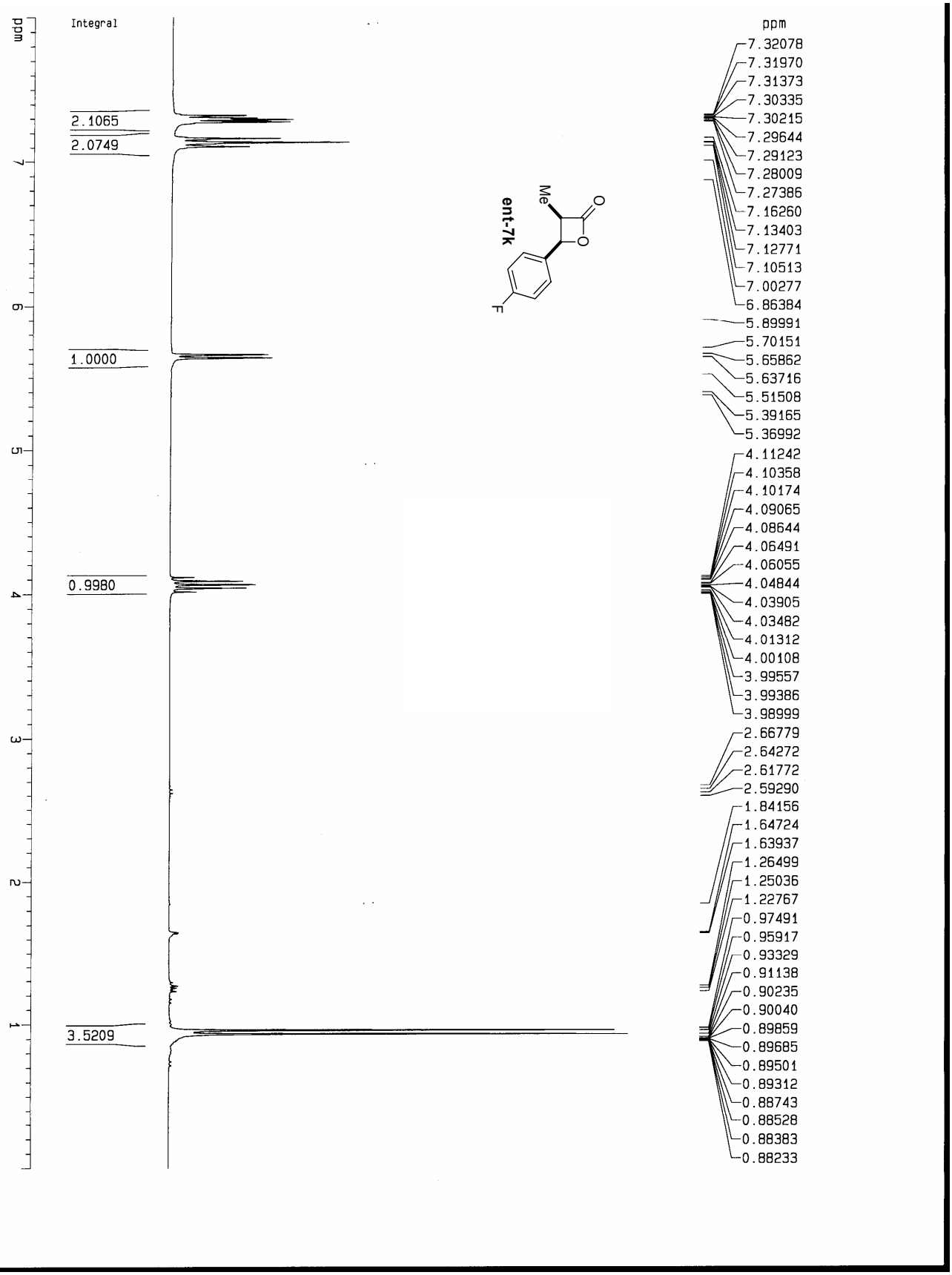
37.708
28.888
28.109
26.022
25.049
8.429


ppm
Integral
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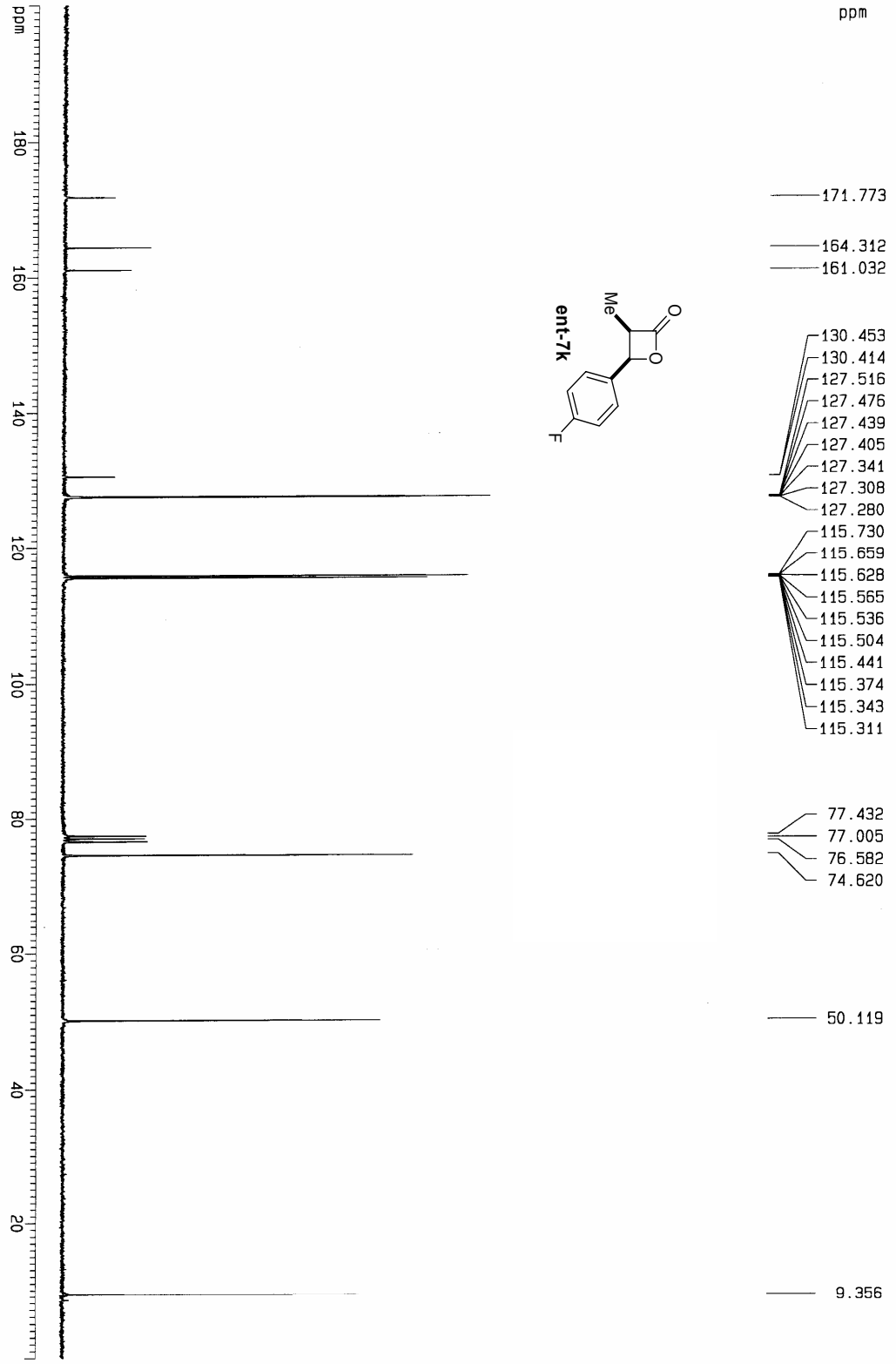
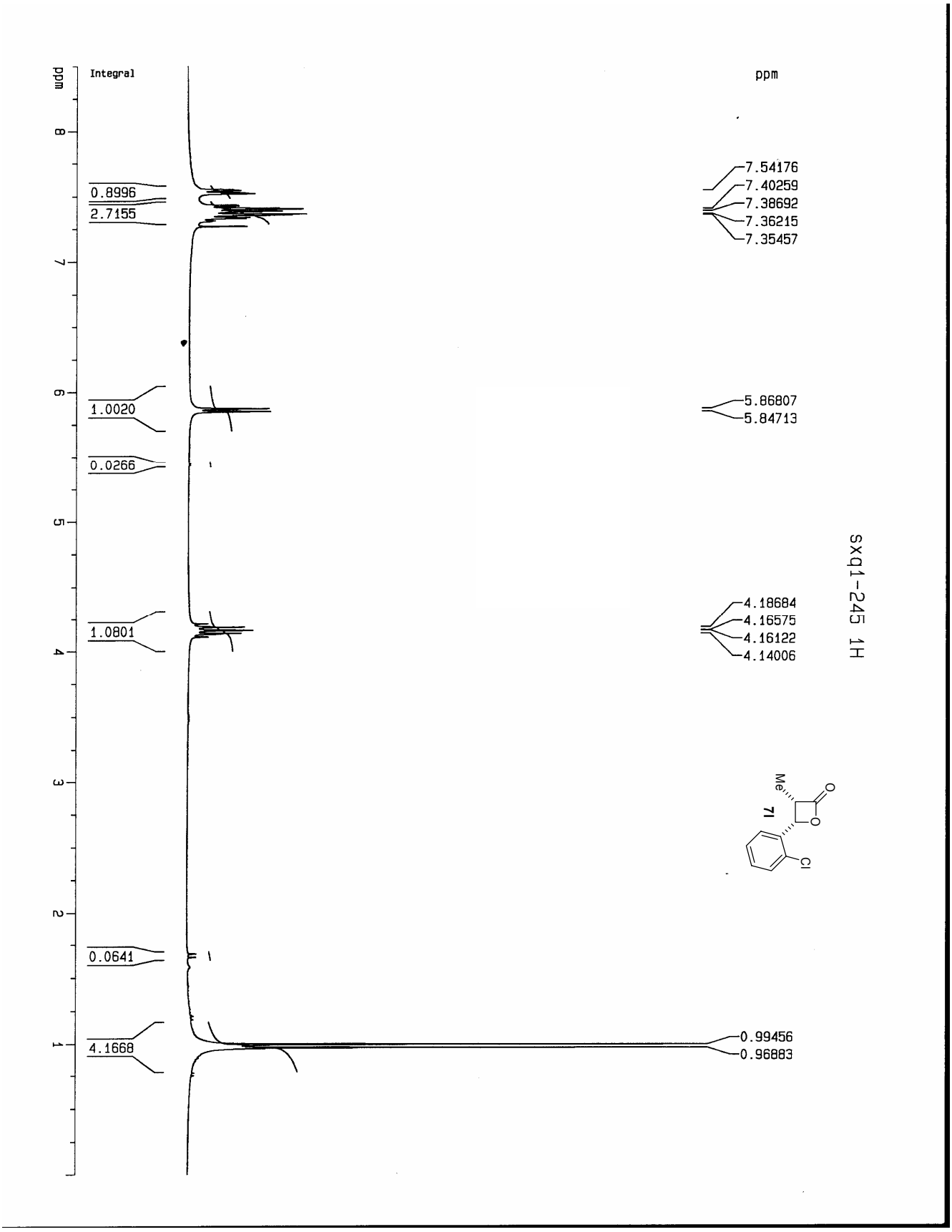
1.0000
0.9980
3.5209
ent-7k
Me
O
O
F
ppm
7.32078
7.31970
7.31373
7.30335
7.30215
7.29644
7.29123
7.28009
7.27386
7.16260
7.13403
7.12771
7.10513
7.00277
6.86384
5.89991
5.70151
5.65862
5.63716
5.51508
5.39165
5.36992
4.11242
4.10358
4.10174
4.09065
4.08644
4.06491
4.06055
4.04844
4.03905
4.03482
4.01312
4.00108
3.99557
3.99386
3.98999
2.66779
2.64272
2.61772
2.59290
1.84156
1.64724
1.63937
1.26499
1.25036
1.22767
0.97491
0.95917
0.93329
0.91138
0.90235
0.90040
0.89859
0.89685
0.89501
0.89312
0.88743
0.88528
0.88383
0.88233


ppm
180
160
140
120
100
80
60
40
20
ent-7k
Me
O
O
F
ppm
171.773
164.312
161.032
130.453
130.414
127.516
127.476
127.439
127.405
127.341
127.308
127.280
115.730
115.659
115.628
115.565
115.536
115.504
115.441
115.374
115.343
115.311
77.432
77.005
76.582
74.620
50.119
9.356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1.0801
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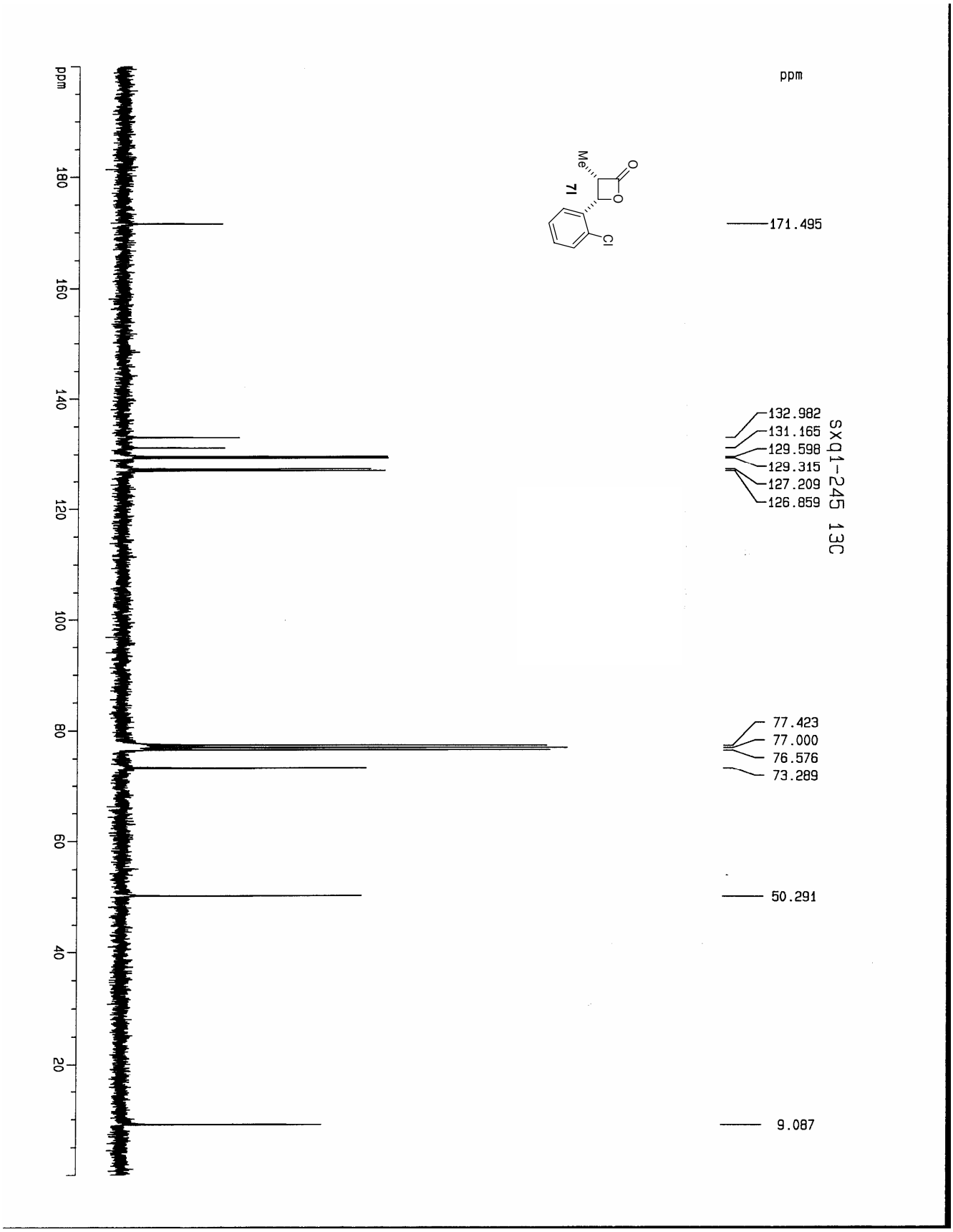
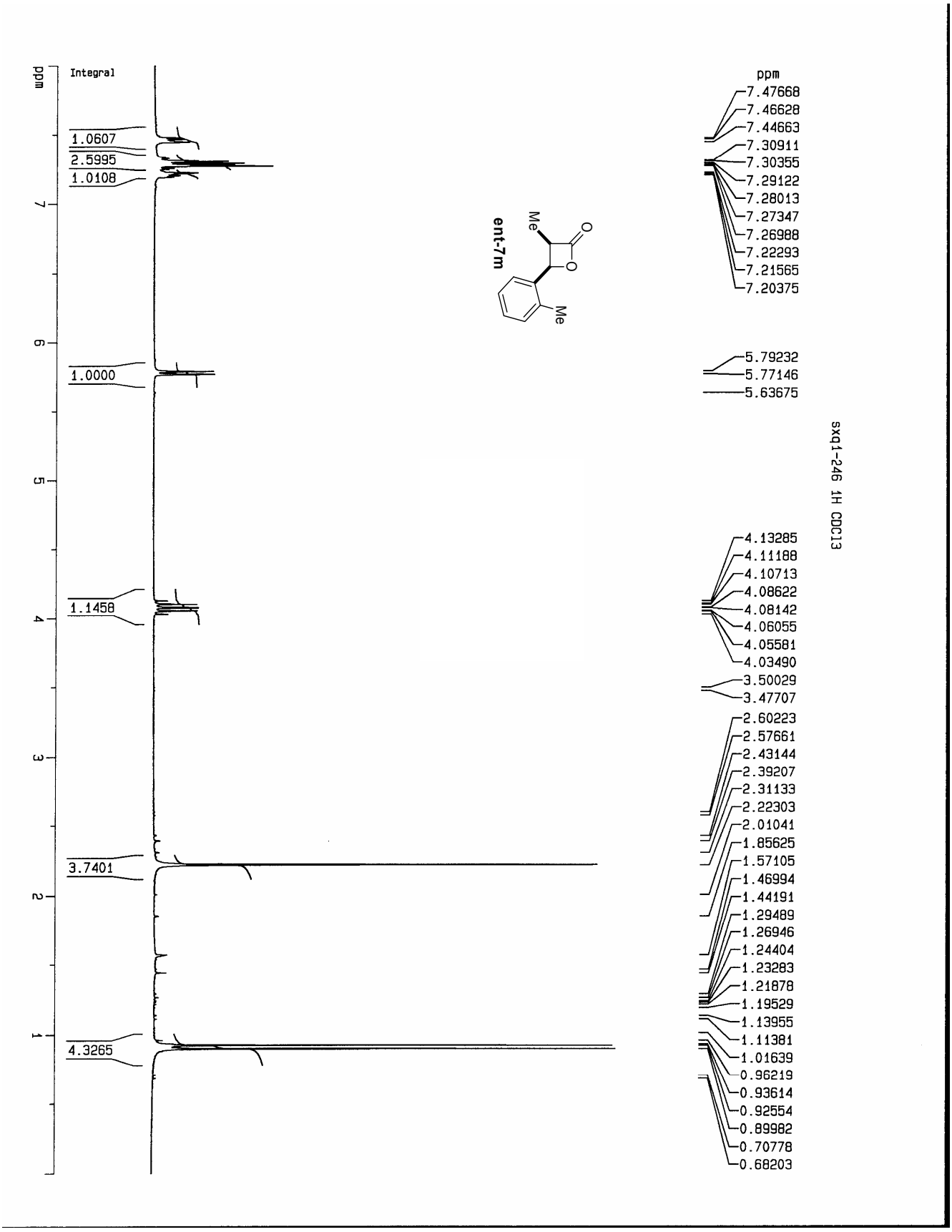
7.40259
7.38692
7.36215
7.35457
5.86807
5.84713
4.18684
4.16575
4.16122
4.14006
0.99456
0.96883
sxq1-245 1H
Me
O
O
Cl
71


ppm
180
160
140
120
100
80
60
40
20
Me
O
O
Cl
71
ppm
171.495
132.982
131.165
129.598
129.315
127.209
126.859
77.423
77.000
76.576
73.289
50.291
9.087
sxq1-245 13C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1.1458
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4.3265
ent-7m
Me
Me
O
O
ppm
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7.46628
7.44663
7.30911
7.30355
7.29122
7.28013
7.27347
7.26988
7.22293
7.21565
7.20375
5.79232
5.77146
5.63675
4.13285
4.11188
4.10713
4.08622
4.08142
4.06055
4.05581
4.03490
3.50029
3.47707
2.60223
2.57661
2.43144
2.39207
2.31133
2.22303
2.01041
1.85625
1.57105
1.46994
1.44191
1.29489
1.26946
1.24404
1.23283
1.21878
1.19529
1.13955
1.11381
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0.93614
0.92554
0.89982
0.70778
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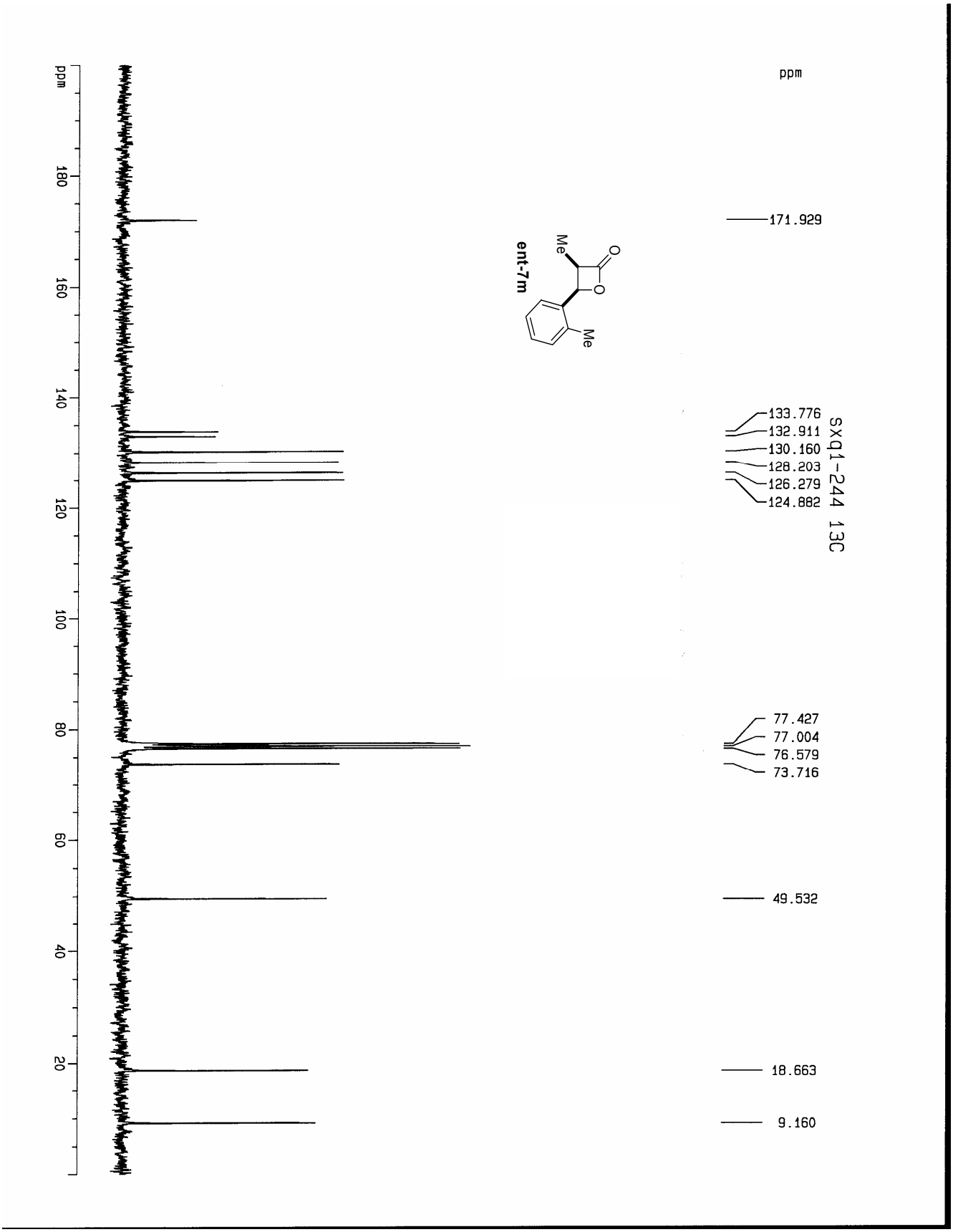
sxq1-246 1H CDCl3


ppm
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120
100
80
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40
20
ent-7m
Me
O
O
Me
ppm
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130.160
128.203
126.279
124.882
77.427
77.004
76.579
73.716
49.532
18.663
9.160
sxq1-244 13C