Concise synthesis of tricyclic isoindolinones via one-pot cascade multicomponent sequences

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Supporting Information

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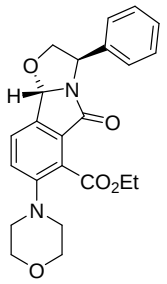
General informations: Unless otherwise noted, all starting materials were obtained from commercial suppliers and used without purification. Petroleum ether was distilled under Argon. Toluene was used without distillation. All reactions were carried out under an atmosphere of dry argon. Solutions were evaporated under reduced pressure with a rotary evaporator and the residue was purified by silica gel chromatography using an ethyl acetate-petroleum ether mixture as the eluent unless specified otherwise. NMR spectra were recorded on a 300 MHz and 200 MHz Brucker spectrometer. Chemical shifts were reported in ppm relative to the residual solvent peak (7.26 ppm for $CHCl_3$) for ^1H spectra and (77.00 ppm for $CDCl_3$) for ^{13}C spectra. Melting points were measured on a Büchi Melting-Point B-545 and were uncorrected. High Resolution Mass spectroscopy data were recorded on a Autospec Ultima (Waters/Micromass) device with a resolution of 5000 RP at 5%. Impact electronic and chemical ionisation spectroscopies were recorded on a HP5989 B device of Hewlett-Packard ; for electrospray mass spectroscopy an add-on 59 987 A was used ; Branford source-type ; 4 µL/min. Infrared spectra were recorded on a FT IR spectrometer Nicolet Impact 400 D of Nicolet Instruments in KBr for liquid and solid compounds. TLC was carried out on Silica Gel 60 F_{254} (0.5 mm thickness).

General procedure for tricyclic lactams synthesis:

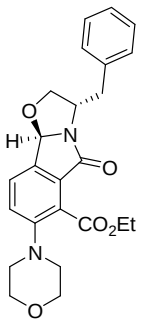
In a round bottom flask, were stirred 1 eq of 5-amino-2-furaldehyde **1**, 1 eq of amino alcohol **2** and 2 eq of diethyl maleate **3** in 8 mL of toluene in Dean–Stark conditions. After 96 hours, the reaction was allowed to cool down at RT and the solvent was evaporated under reduced pressure. The crude was purified by silica gel chromatography using Petroleum ether: EtOAc as eluent.



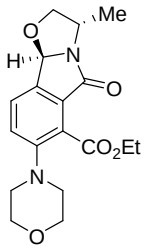
(3*R*, 9b*S*)-ethyl 2,3,5,9b-tetrahydro-7-morpholino-5-oxo-3-phenyloxazolo[2,3-*a*]isoindole-6-carboxylate 4a: Following the general procedure of the one-pot process 250 mg of **1a** (1.37 mmol), 188 mg of (*R*)-phenylglycinol **2a** (1.37 mmol) and 445 µL (2.74 mmol) of **3** led to 220 mg of colorless crystals (40%) after silica gel chromatography (Petroleum ether: EtOAc 7:3). $[\alpha]^{25}_D = -98°$ (c = 0.185, CH_2Cl_2). Mp 165.1–167.5 °C. IR (KBr, ν, cm^{-1}): 1729, 1610, 1498, 1393, 1284, 1236, 1149, 1114, 1061. ^1H NMR

($CDCl_3$, 300 MHz): δ 1.40 (t, ^3J = 7.1, 3H, $CO_2CH_2CH_3$), 3.02–3.13 (m, 4H, NCH_2), 3.78–4.11 (m, 4H, OCH_2), 4.1 (dd, J = 8.9 and 7.5, 1H, NCHPhC*H*HO), 4.40–4.52 (m, 2H, $CO_2CH_2CH_3$), 4.80 (dd, J = 8.9 and 7.5, 1H, NCHPhCH*H*O), 5.16 (t, ^3J = 7.5, 1H, NC*H*Ph), 5.99 (s, 1H, OC*H*N), 7.29–7.36 (m, 6H, Ph + C*H*=CHCCHON), 7.62 (d, ^3J = 8.1, 1H, CH=C*H*CCHON); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 14.2 ($CO_2CH_2CH_3$), 53.3 (2C, NCH_2), 58.2 (N*C*HPh), 61.9 ($CO_2CH_2CH_3$), 67.1 (2C, OCH_2), 77.9 (CHPhCH_2O), 91.2 (NCHO), 124.8 (CH), 125.7 (CH), 126.0 (3C, CH, Ph), 127.7 (CH), 128.8 (2C, CH, Ph), 131.2 (C), 136.9 (C), 139.4 (C), 152.0 (C), 166.5 (CO_2), 171.7 (CON). ESI-MS *m/z* (rel int): 431.2 (100, M + Na^+), 839.4 (40, 2M + Na^+). HR-MS m/z calcd for [($C_{23}H_{24}N_2O_5$) + H^+] 409.1763; found 409.1752.

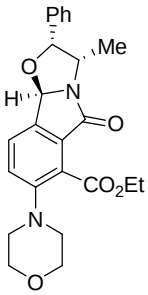
Compounds **4b** – **4g** and **5** were obtained, unless otherwise noted, following the general procedure of the one-pot process, as described for **4a** with various amino alcohols.



(3*R*, 9b*S*)-ethyl 3-benzyl-2,3,5,9b-tetrahydro-7-morpholino-5-oxooxazolo[2,3-*a*]isoindole-6-carboxylate 4b : 100 mg **1a** (0.55 mmol), 83 mg (0.55 mmol) of (*S*)-phenylalaninol **2b** and 178 µL (1.1 mmol) of **3**. Yellow solid 65% yield (Petroleum ether: EtOAc 75:25). $[\alpha]^{25}_D$ = – 12° (c = 0.139, CH_2Cl_2). Mp 150.8 °C. IR (KBr, ν, cm^{-1}): 1719, 1607, 1453, 1378, 1311, 1236, 1161, 1110, 1064, 1022. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.35 (t, ^3J = 7.1, 3H, $CO_2CH_2CH_3$), 2.85–3.13 (m, 6H, NCH_2 + $PhCH_2$), 3.66–3.80 (m, 4H, OCH_2), 3.87–3.92 (m, 1H, BnCHC*H*HOCHN), 4.18–4.24 (m, 1H, BnCHCH*H*OCHN), 4.30–4.43 (m, 3H, $CO_2CH_2CH_3$ + BnC*H*), 5.60 (s, 1H, OC*H*N), 7.19–7.27 (m, 6H, Ph + C*H*=CHCCHON), 7.47 (d, ^3J = 8.1, 1H, CH=C*H*CCHON); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 14.2 ($CO_2CH_2CH_3$), 39.6 ($PhCH_2CH$), 53.0 (2C, NCH_2), 55.5 ($PhCH_2CH$), 61.9 ($CO_2CH_2CH_3$), 67.1 (2C, OCH_2), 74.9 (CHPhCH_2O), 90.3 (OCHN), 124.8 (CH), 125.6 (CH), 126.8 (CH), 127.8 (C), 128.6 (2C, CH), 129.5 (2C, CH), 131.2 (C), 136.6 (C), 137.3 (C), 151.9 (C), 166.6 (CO_2), 171.5 (CON). ESI-MS *m/z* (rel int): 445.3 (100, M + Na^+). HR-MS m/z calcd for [($C_{24}H_{26}N_2O_5$) + H^+] 423.1920; found 423.1910.

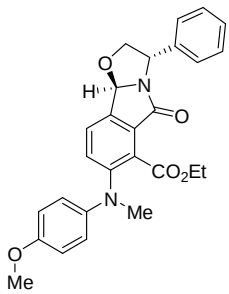


(3*S*, 9b*R*)-ethyl 2,3,5,9b-tetrahydro-3-methyl-7-morpholino-5-oxooxazolo[2,3-*a*]isoindole-6-carboxylate 4c: 100 mg of **1a** (0.55 mmol), 43 µL (0.55 mmol) of (*S*)-alaninol **2c** and 178 µL (1.1 mmol) of **3**. Yellow solid 35% yield (Petroleum ether: EtOAc 65:35). $[\alpha]^{25}_{D}$ = – 5° (c = 0.149, CH_2Cl_2). Mp 146.8 °C. IR (KBr, ν, cm^{-1}): 1729, 1699, 1614, 1405, 1285, 1232, 1113, 1022. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.37 (m, 6H, $CO_2CH_2CH_3$ + $NCHCH_3$), 3.02–3.09 (m, 4H, NCH_2), 3.77–3.81 (m, 5H, OCH_2 + $NCHCH_3CHHO$), 4.21 (q, ^3J = 6.75, 1H, $NCHCH_3$), 4.43–4.48 (m, 3H, $CO_2CH_2CH_3$ + $NCHCH_3CHHO$), 5.83 (s, 1H, OC*H*N), 7.31 (d, ^3J = 8.1, 1H, C*H*=CHCCHON), 7.57 (d, ^3J = 8.1, 1H, CH=C*H*CCHON); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 14.2 ($CO_2CH_2CH_3$), 19.5 ($CHCH_3$), 50.7 ($CHCH_3$), 53.1 (2C, NCH_2), 61.8 ($CO_2CH_2CH_3$), 67.2 (2C, OCH_2), 77.1 ($CHCH_3CH_2O$), 89.9 (OCHN), 124.7 (*C*H=CHCCHON), 125.6 (CH=*C*HCCHON), 127.9 (C), 131.5 (C), 137.1 (C), 152.0 (C), 166.6 (CO_2), 171.5 (CON). ESI-MS *m/z* (rel int): 369.2 (100, M + Na^+), 715.4 (90, 2M + Na^+). HR-MS m/z calcd for [($C_{18}H_{22}N_2O_5$) + Na^+] 369.1426; found 369.1418.

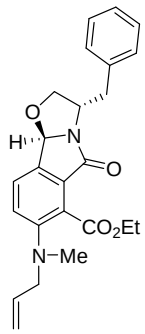


(2*R*, 3*S*, 9b*R*)-ethyl 2,3,5,9b-tetrahydro-3-methyl-7-morpholino-5-oxo-2-phenyloxazolo[2,3-*a*]isoindole-6-carboxylate 4d: 100 mg of **1a** (0.55 mmol), 83.2 mg (0.55 mmol) of (–)-norephedrine **2d** and 178 µL (1.1 mmol) of **3**. Yellow oil 45 % yield (Petroleum ether: EtOAc 8:2). $[\alpha]^{25}_{D}$ = – 16° (c = 0.37, CH_2Cl_2). IR (KBr, ν, cm^{-1}): 1720, 1675, 1608, 1496, 1453, 1390, 1281, 1230, 1136, 1017. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 0.96 (d, ^3J = 7.1, 3H, $NCHCH_3$), 1.43 (t, ^3J = 7.1, 3H, $CO_2CH_2CH_3$), 3.01–3.13 (m, 4H, NCH_2), 3.78–3.81 (m, 4H, OCH_2), 4.49 (q, ^3J = 7.1, 2H, $CO_2CH_2CH_3$), 4.64 (q, ^3J = 7.1, 1H, $NCHCH_3$), 5.05 (d, ^3J = 5.2, 1H, OC*H*Ph), 6.24 (s, 1H, OC*H*N), 7.29–7.37 (m, 6H, C*H*=CHCCHON + Ph), 7.68 (d, ^3J = 8.1, 1H, CH=C*H*CCHON); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 14.2 ($CO_2CH_2CH_3$), 14.7 ($CHCH_3CHPh$), 53.0 (2C, NCH_2), 55.5 ($CHCH_3CHPh$), 61.9 ($CO_2CH_2CH_3$), 67.1 (2C, OCH_2), 83.8 ($CHCH_3CHPhO$), 89.2 (O*C*HN), 125.1 (CH, *C*H=CHCCHON), 125.9 (2C, CH, Ph), 127.3 (C), 127.9

(CH, CH=*C*HCCHON), 128.3 (3C, CH, Ph), 130.8 (C), 136.7 (C), 139.1 (C), 151.8 (C), 166.7 (CO_2), 171.6 (CON). HR-MS m/z calcd for [($C_{24}H_{26}N_2O_5$) + H^+] 423.1920; found 423.1908.

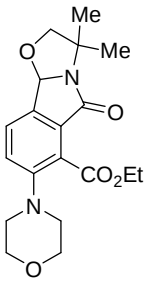


(3*S*, 9b*R*)-ethyl 7-(*N*-(4-methoxyphenyl)-*N*-methylamino)-2,3,5,9b-tetrahydro-5-oxo-3-phenyloxazolo[2,3-*a*]isoindole-6-carboxylate 4e: 150 mg of **1b** (0.64 mmol) and 88 mg of (*S*)-phenylglycinol **2e** (0.64 mmol) and 198 µL (1.28 mmol) **3**. brown crystals 34% yield (Petroleum ether: EtOAc 8:2). $[\alpha]^{25}_D$ = + 93° (c = 0.155, CH_2Cl_2). Mp 139-142° C. IR (KBr, ν, cm^{-1}): 1720, 1605, 1510, 1485, 1386, 1284, 1243, 1138, 1029. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.16 (t, ^3J = 7.1, 3H, $CO_2CH_2CH_3$), 3.25 (s, 3H, NCH_3), 3.77 (s, 3H, OCH_3), 4.05–4.18 (m, 3H, $CO_2CH_2CH_3$ + CHPhC*H*HO), 4.79–4.85 (dd, *J* = 8.9 and 7.5, 1H, CHPhCH*H*O), 5.15–5.20 (t, ^3J = 7.5, 1H, NC*H*Ph), 6.01 (s, 1H, OC*H*N), 6.78–6.86 (m, 4H, NCH_3PhOCH_3), 7.27–7.37 (m, 6H, C*H*=CHCCHON + Ph), 7.61 (d, ^3J = 9, 1H, CH=C*H*CCHON); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 13.8 ($CO_2CH_2CH_3$), 42.0 (NCH_3), 55.6 (OCH_3), 58.3 (N*C*HPh), 61.7 ($CO_2CH_2CH_3$), 77.9 (CHPhCH_2O), 91.2 (O*C*HN), 114.5 (2C, CH), 121.4 (2C, CH), 125.9 (CH), 126.1 (3C, CH, Ph), 127.7 (CH), 128.8 (2C, CH, Ph), 129.0 (C), 131.7 (C), 136.8 (C), 139.6 (C), 143.1 (C), 149.9 (C), 154.8 (C), 166.0 (CO_2), 171.7 (CON). ESI-MS *m/z* (rel int): 481.2 (100, M + Na^+), 939.7 (50, 2M + Na^+). HR-MS m/z calcd for [($C_{27}H_{26}N_2O_5$) + Na^+] 481.1739; found 481.1742.

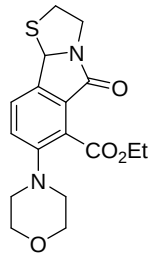


(3*S*, 9b*R*)-ethyl 7-(*N*-allyl-*N*-methylamino)-2,3,5,9b-tetrahydro-5-oxo-3-phenyloxazolo[2,3-*a*]isoindole-6-carboxylate 4f: Following the general procedure of the one-pot process 100 mg of **1c** (0.665 mmol), 100 mg of **2b** (0.665 mmol) and 197 µL (1.21 mmol) **3**. Yellow oil 33% yield (Petroleum ether: EtOAc 9:1). $[\alpha]^{25}_D$ = − 169° (c = 0.10, CH_2Cl_2). IR (KBr, ν, cm^{-1}): 1732, 1608, 1483, 1451, 1388, 1298, 1279, 1233, 1136, 1114, 1065, 1028, 1013. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.44 (t, ^3J = 7.1, 3H, $CO_2CH_2CH_3$), 2.87 (s, 3H, NCH_3), 2.99–3.05 (m, 1H, PhCH_2CH), 3.18–3.30 (m, 1H, PhCH_2CH), 3.79 (d, ^3J = 5.4, 2H, NCH_2CHCH_2), 3.98–4.01 (m, 1H, CHCH_2OCHN), 4.30–4.33 (m, 1H,

CHC*H*$_2$OCHN), 4.48–4.53 (m, 3H, CO_2C*H*$_2$$CH_3$ + $PhCH_2$C*H*), 5.20–5.27 (m, 2H, NCH_2CHC*H*$_2$), 5.28 (s, 1H, OC*H*N), 5.67–5.90 (m, 1H, NCH_2C*H*CH_2), 7.19 (d, ^3J = 8.5, 1H, C*H*=CHCCHON), 7.30–7.35 (m, 5H, Ph), 7.47 (d, ^3J = 8.3, 1H, CH=C*H*CCHON); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 14.0 (CO_2CH_2*C*H_3), 39.6 (CH*C*H_2Ph), 40.0 (NCH_3), 55.4 (*C*HCH_2Ph), 58.9 (N*C*H_2CH=CH_2), 61.9 (CO_2*C*H_2CH_3), 74.8 ($CHCH_2$Ph*C*H_2O), 90.2 (O*C*HN), 117.2 (NCH_2CH=*C*H_2), 122.5 (C), 123.1 (CH), 125.1 (CH), 126.7 (CH), 128.5 (2C, CH, Ph), 129.5 (2C, CH, Ph), 131.0 (C), 133.8 (NCH_2*C*H=CH_2), 134.0 (C), 136.7 (C), 151.2 (C), 167.4 (CO_2), 172.1 (CON). ESI-MS *m/z* (rel int): 429.3 (100, M + Na^+), 445.3 (70, M + K^+), 835.4 (15, 2M + Na^+), 851.4 (18, 2M + K^+). HR-MS m/z calcd for [($C_{24}H_{26}N_2O_4$) + H^+] 407.1971; found 407.1985.



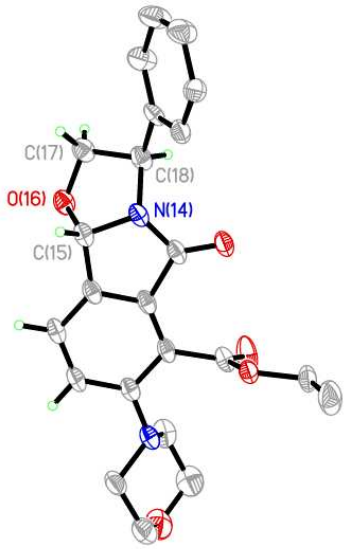
ethyl 2,3,5,9b-tetrahydro-3,3-dimethyl-7-morpholino-5-oxooxazolo[2,3-*a*]isoindole-6-carboxylate 4g: 250 mg of **1a** (1.37 mmol), 122 mg (1.37 mmol) of 2-amino-2-methylpropanol **2g** and 450 µL (2.74 mmol) of **3**. Yellow solid 35% yield (Petroleum ether: EtOAc 6:4). Mp 92.5–94 °C. IR (KBr, ν, cm^{-1}): 1730, 1710, 1602, 1450, 1386, 1280, 1227, 1119, 1062, 1027. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.38–1.62 (m, 9H, CO_2CH_2C*H*$_3$ + NCHC*H*$_3$C*H*$_3$), 3.00–3.16 (m, 4H, NC*H*$_2$), 3.78–3.81 (m, 4H, OC*H*$_2$), 4.10 (d, ^2J = 8.5, 1H, OC*H*$_2$$CCH_3CH_3$), 4.11 (d, ^2J = 8.5, 1H, OC*H*$_2$$CCH_3CH_3$), 4.40–4.51 (m, 2H, CO_2C*H*$_2$$CH_3$), 5.86 (s, 1H, OCHN), 7.35 (d, ^3J = 8.3, 1H, C*H*=CHCCHON), 7.56 (d, ^3J = 8.1, 1H, CH=C*H*CCHON); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 14.2 (CO_2CH_2*C*H_3), 23.3 (C*C*H_3CH_3), 26.6 (CCH_3*C*H_3), 53.2 (2C, NCH_2), 59.5 (C, *C*CH_3CH_3), 61.8 (CO_2*C*H_2CH_3), 67.1 (2C, OCH_2), 84.6 (CCH_3CH_3*C*H_2O), 91.3 (OCHN), 124.5 (CH, *C*H=CHCCHON), 125.2 (CH, CH=*C*HCCHON), 127.8 (C), 133.1 (C), 136.5 (C), 151.6 (C), 166.8 (CO_2), 167.5 (CON). ESI-MS *m/z* (rel int): 383.3 (90, M + Na^+), 399.2 (15, M + K^+), 743.4 (100, 2M + Na^+), 759.5 (18, 2M + K^+). HR-MS m/z calcd for [($C_{19}H_{24}N_2O_5$) + H^+] 361.1763; found 361.1751.

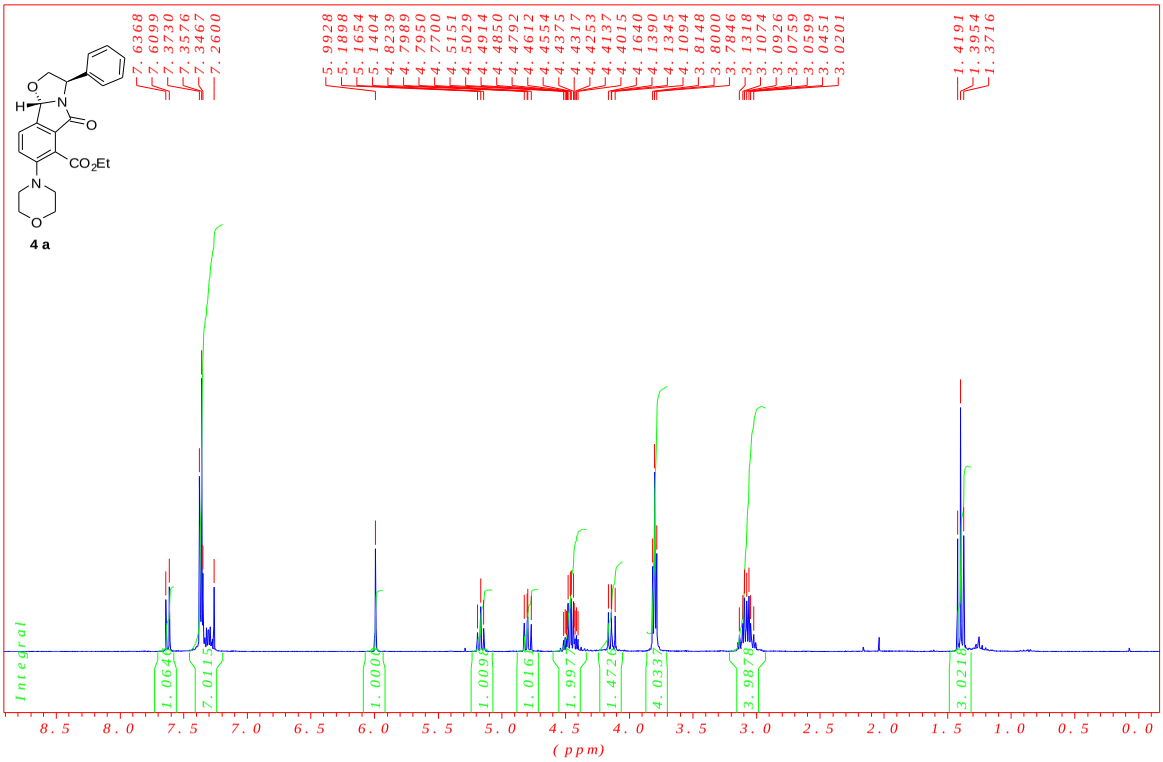


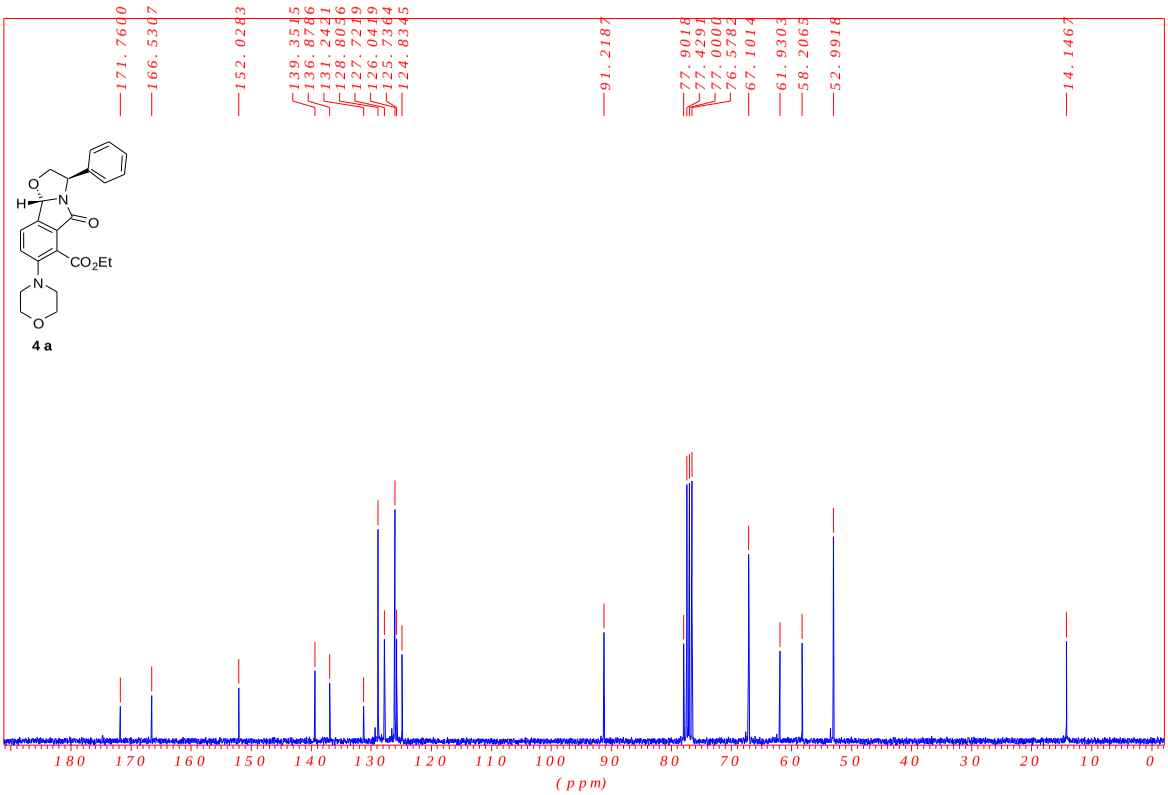
ethyl 2,3,5,9b-tetrahydro-7-morpholino-5-oxothiazolo[2,3-*a*]isoindole-6-carboxylate 5 : 100 mg **1a** (0.55 mmol), 62 mg (0.55 mmol) of 2-aminoethanethiol hydrochloride, 73 mg K_2CO_3 (0.55 mmol), and 178 µL (1.1 mmol) **3**. Yellow oil, 58% yield (Petroleum ether: EtOAc 75:25 to 7:3). IR (KBr, ν, cm^{-1}): 2955, 2924, 2853, 1730, 1709, 1610, 1483, 1450, 1372, 1291, 1266, 1234, 1135, 1114, 1021. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.41 (t, ^3J = 7.1, 3H, $CO_2CH_2CH_3$), 3.03–3.12 (m, 4H, NCH_2), 3.34–3.39 (m, 2H, NCH_2CH_2S), 3.78–3.81 (m, 4H, OCH_2), 4.39–4.51 (m, 4H, $CO_2CH_2CH_3$ + NCH_2CH_2S), 5.82 (s, 1H, SCHN), 7.40 (d, ^3J = 8.3, 1H, C*H*=CHCCHSN), 7.47 (d, ^3J = 8.3, 1H, CH=C*H*CCHSN); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 14.2 ($CO_2CH_2CH_3$), 36.5 (NCH_2CH_2S), 44.6 (NCH_2CH_2S), 53.2 (2C, NCH_2), 61.9 ($CO_2CH_2CH_3$), 65.5 (SCHN), 67.2 (2C, OCH_2), 124.9 (2C, *C*H=CHCCHSN + CH=*C*HCCHSN), 128.1 (C), 129.2 (C), 140.2 (C), 150.8 (C), 166.7 (CO_2), 168.9 (CON). ESI-MS *m/z* (rel int): 371.4 (100, M + Na^+), 719.3 (13, 2M + Na^+). HR-MS m/z calcd for [($C_{17}H_{20}N_2O_4S$) + Na^+] 371.1041; found 371.1040.

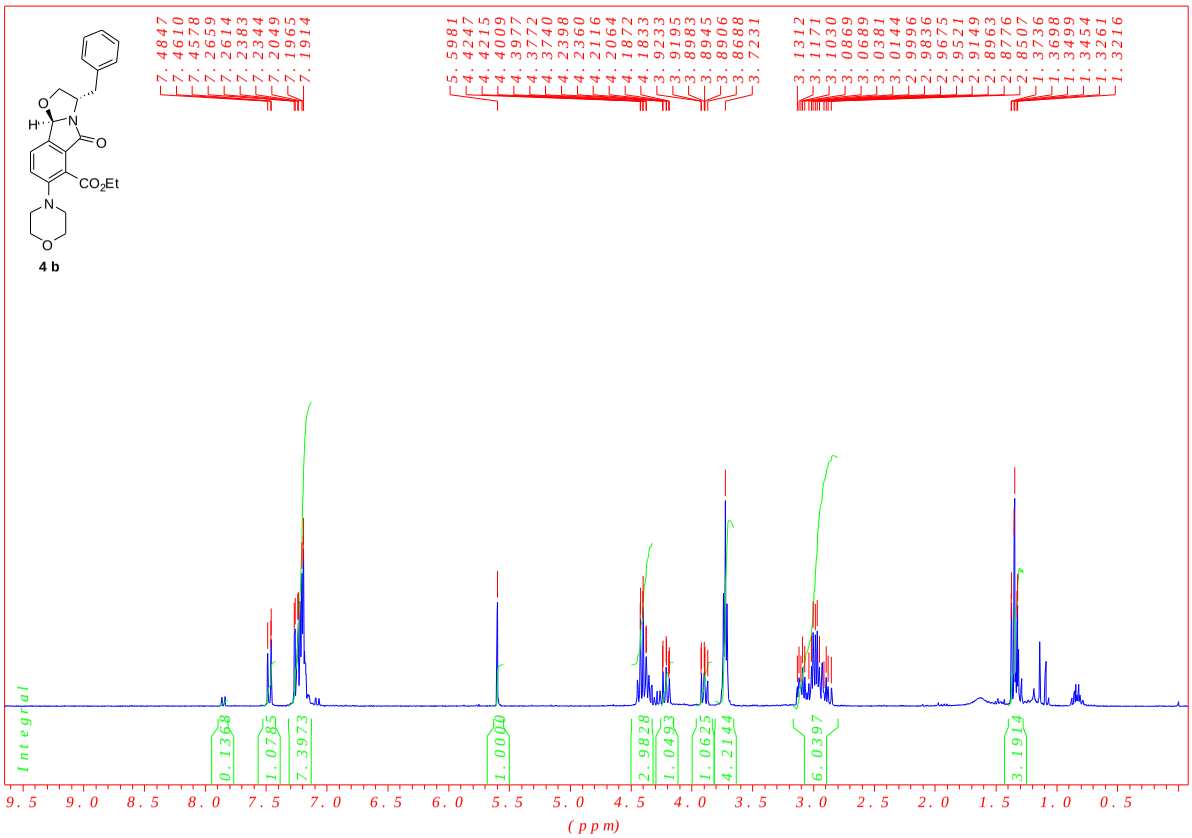
Cristallographic data and ORTEP plot for **4a**

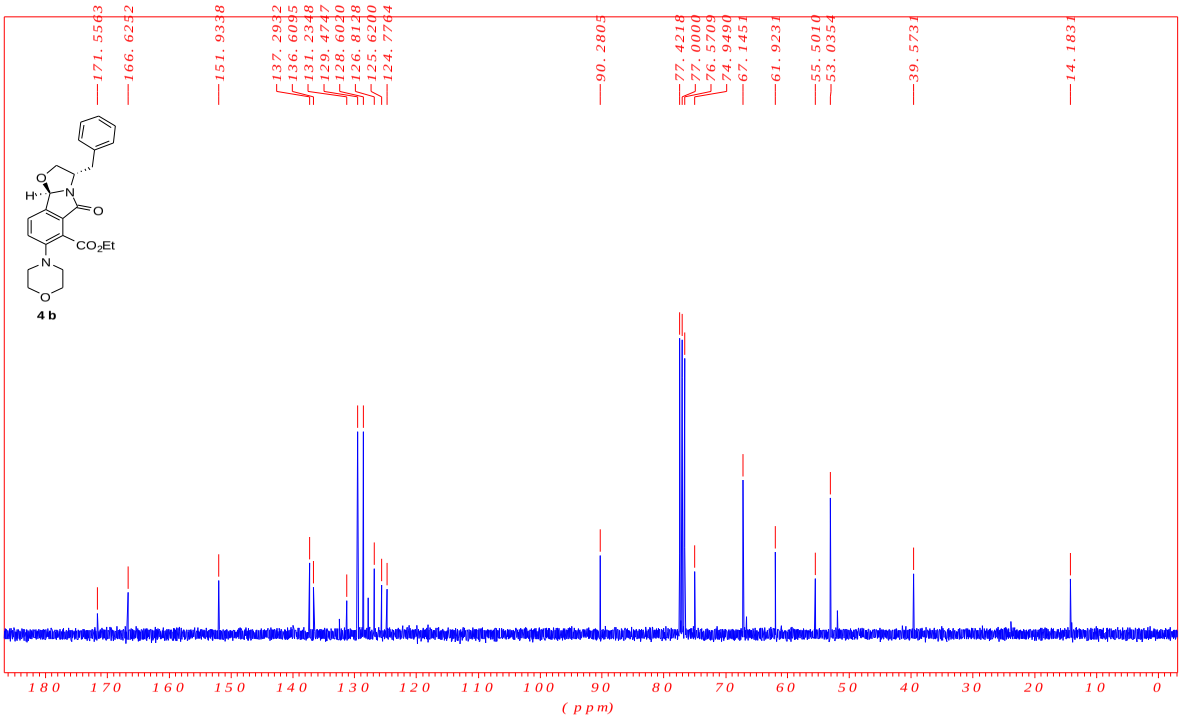
C_{23} H_{24} N_2 O_5, M_w = 408.44, orthorhombic, space group $P2_12_12_1$; dimensions: a = 8.7153(7) Å, b = 9.8625(9) Å, c = 24.055(2) Å, V = 2067.7(3) $Å^3$; Z = 4; μ = 1.312 mm^{-1}; 36210 reflections measured at 100 K; independent reflections: 3404 [2497 Fo > 4σ (Fo)]; data were collected up to a 2Θmax value of 60.10° (99.2 % coverage). Number of variables: 297; R_1 = 0.0399, wR_2 = 0.1060, S = 1.072; highest residual electron density 0.214 e.$Å^{-3}$ (all data R_1 = 0.0702, wR_2 = 0.1295). CCDC 717782.

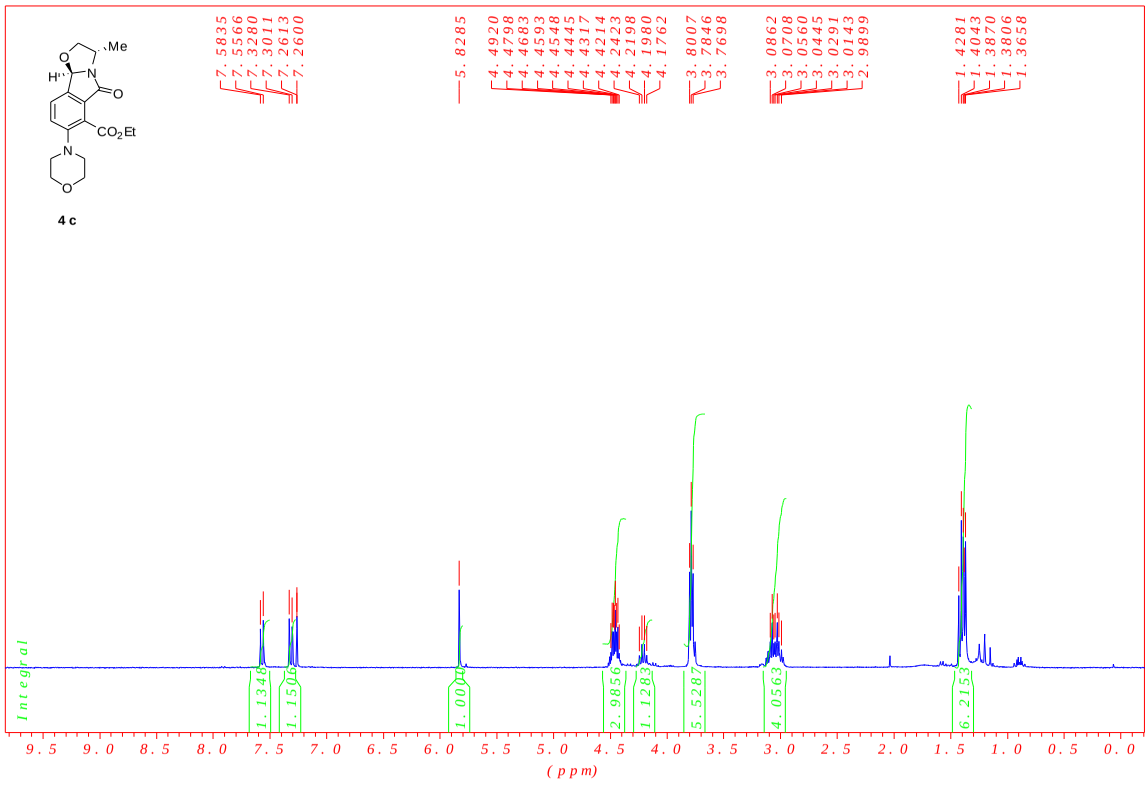


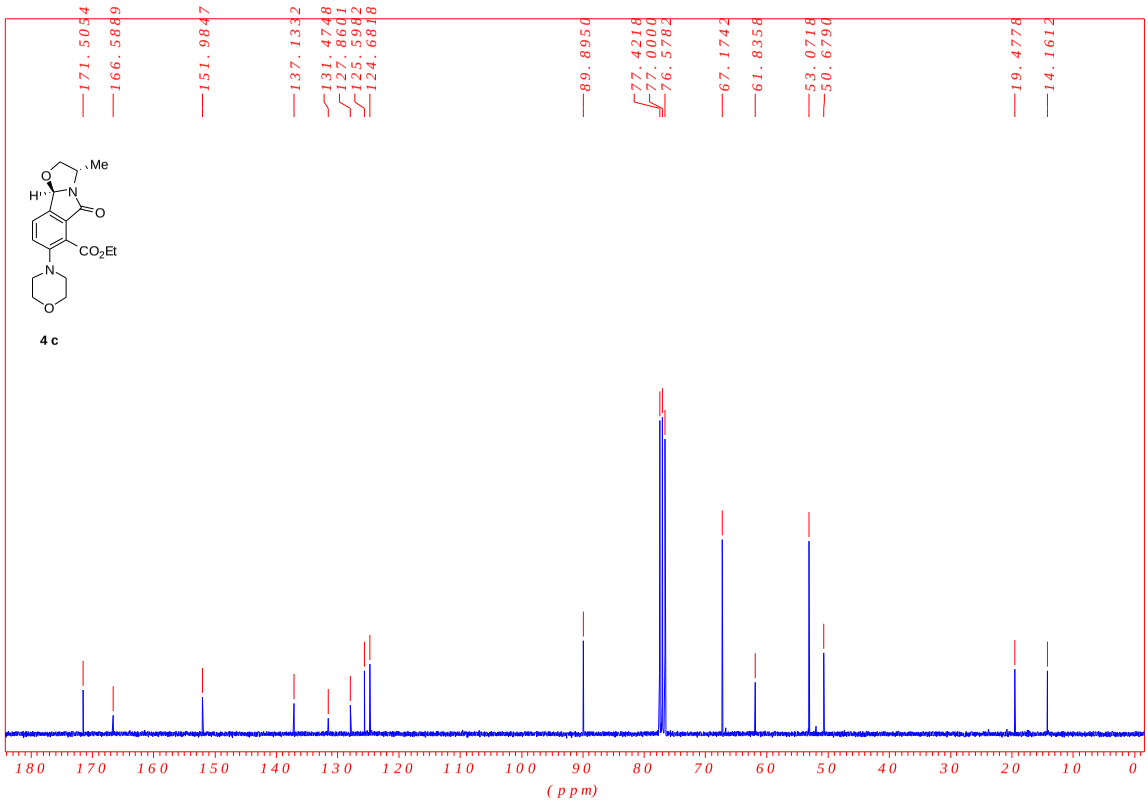

4 a
Integral
(ppm)

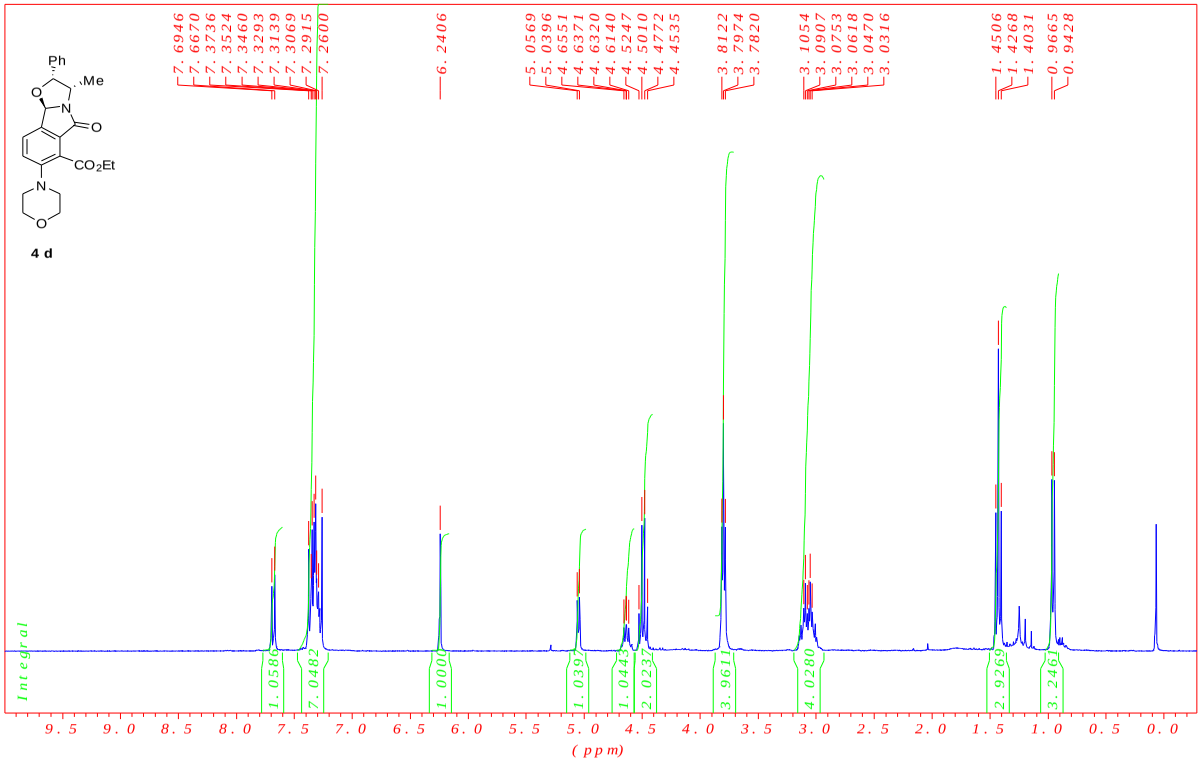

4 a
(ppm)

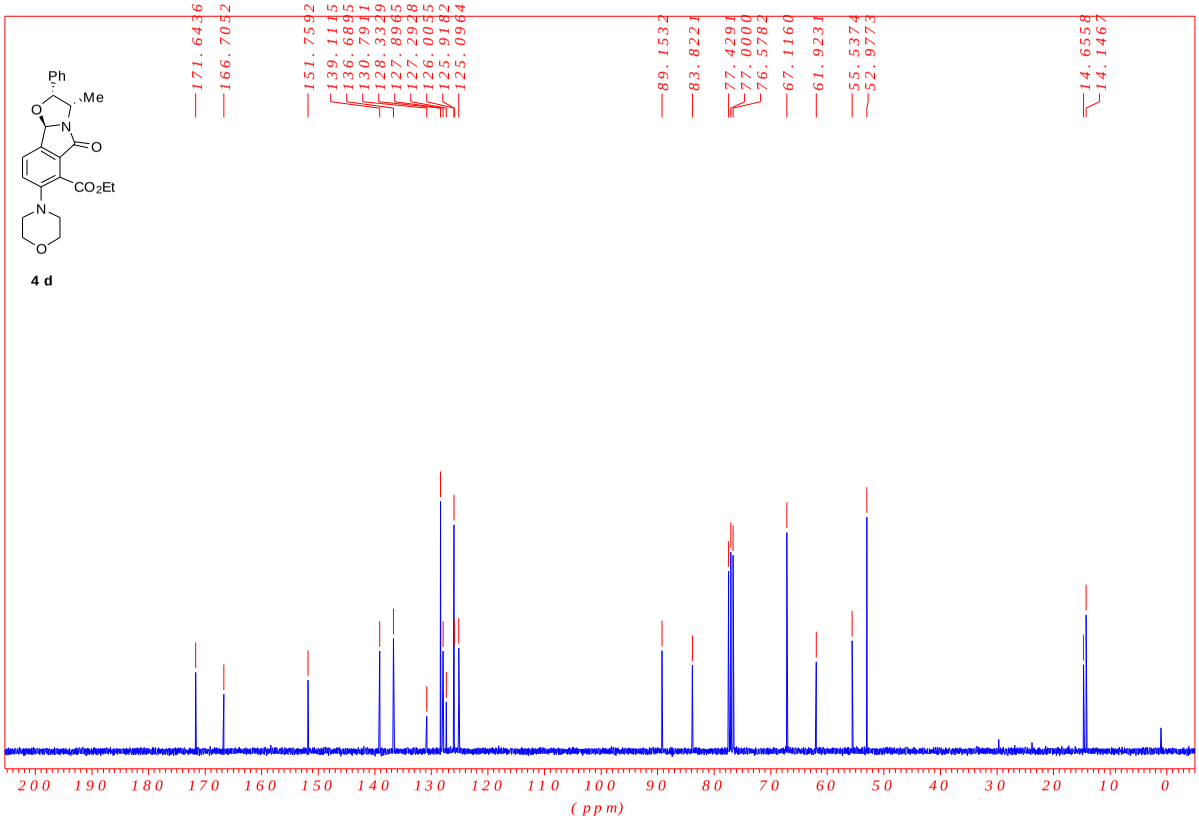

4 b
Integral
(ppm)

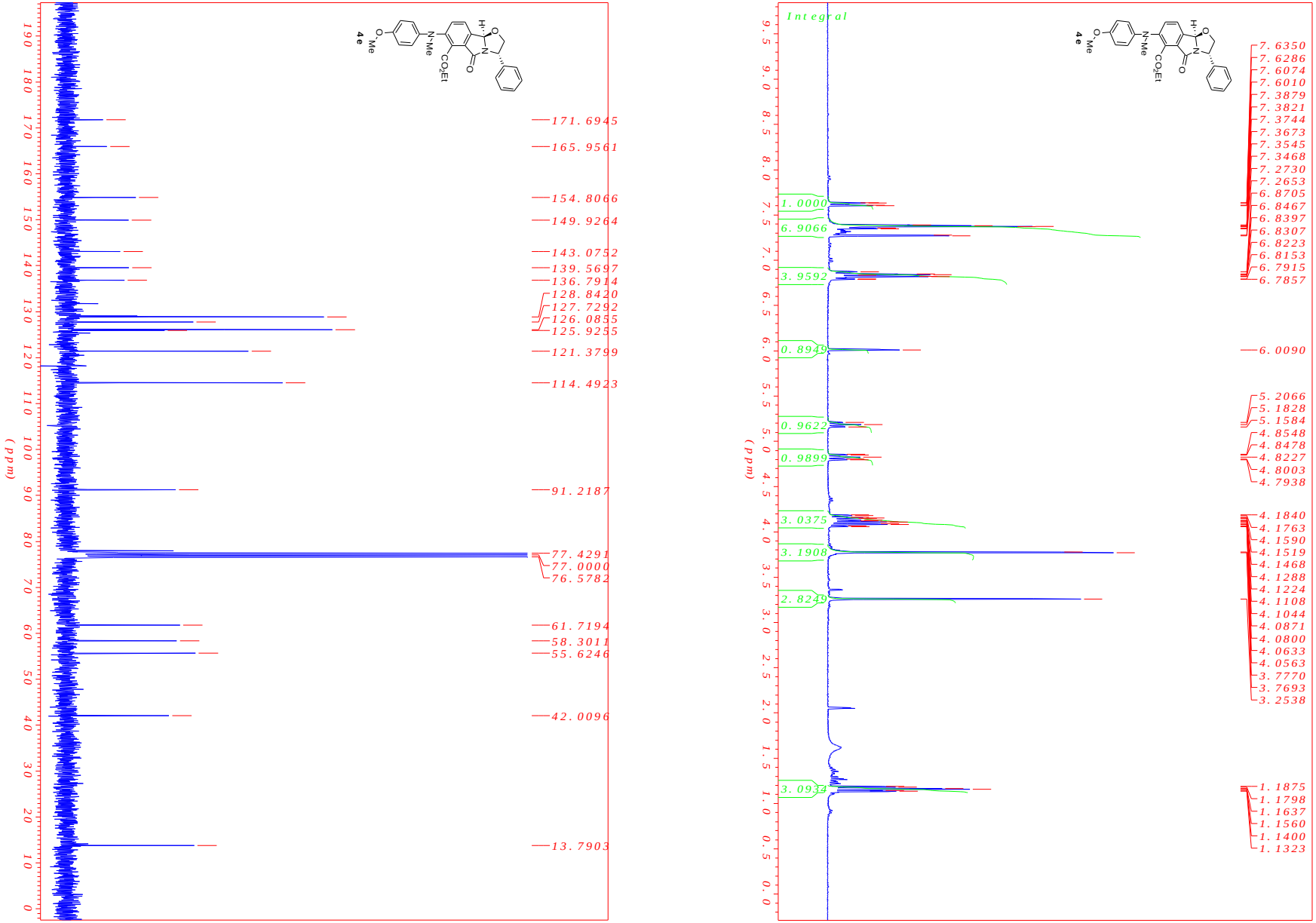

4 b
(ppm)

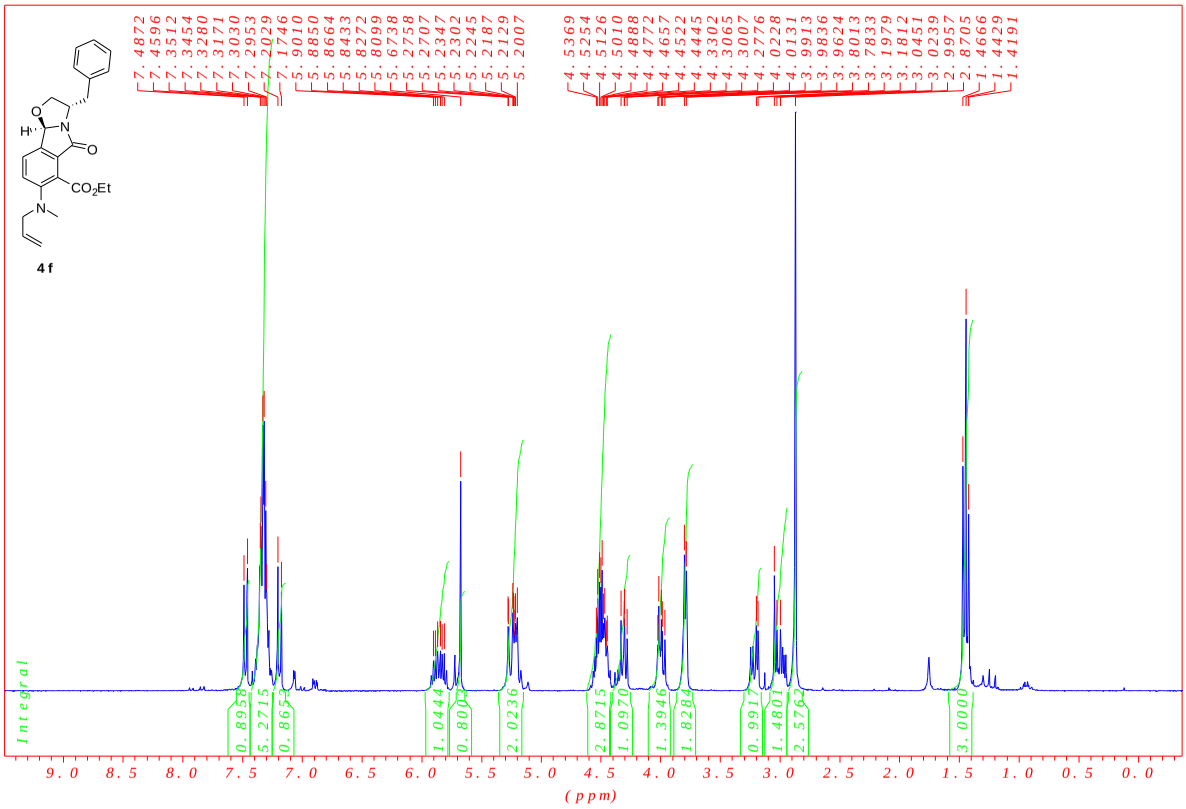

4 c
Me
H
O
N
O
CO2Et
N
O
7.5835
7.5566
7.3280
7.3011
7.2613
7.2600
5.8285
4.4920
4.4798
4.4683
4.4593
4.4548
4.4445
4.4317
4.4214
4.2423
4.2198
4.1980
4.1762
3.8007
3.7846
3.7698
3.0862
3.0708
3.0560
3.0445
3.0291
3.0143
2.9899
1.4281
1.4043
1.3870
1.3806
1.3658
Integral
1.1345
1.1500
1.0000
2.9856
1.1283
5.5287
4.0563
6.2153
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
(ppm)

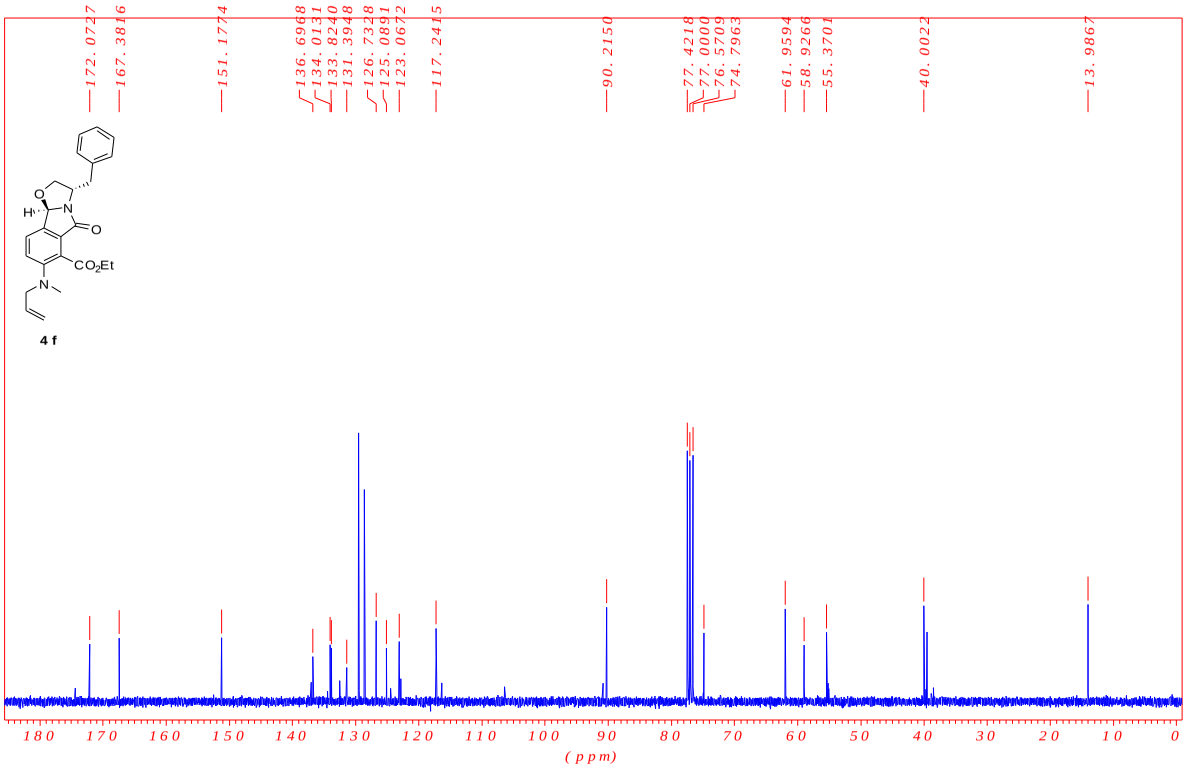

171.5054
166.5889
151.9847
137.1332
131.4748
127.8601
125.5982
124.6818
89.8950
77.4218
77.0000
76.5782
67.1742
61.8358
53.0718
50.6790
19.4778
14.1612
4 c
Me
H
O
N
O
CO2Et
N
O
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
(ppm)

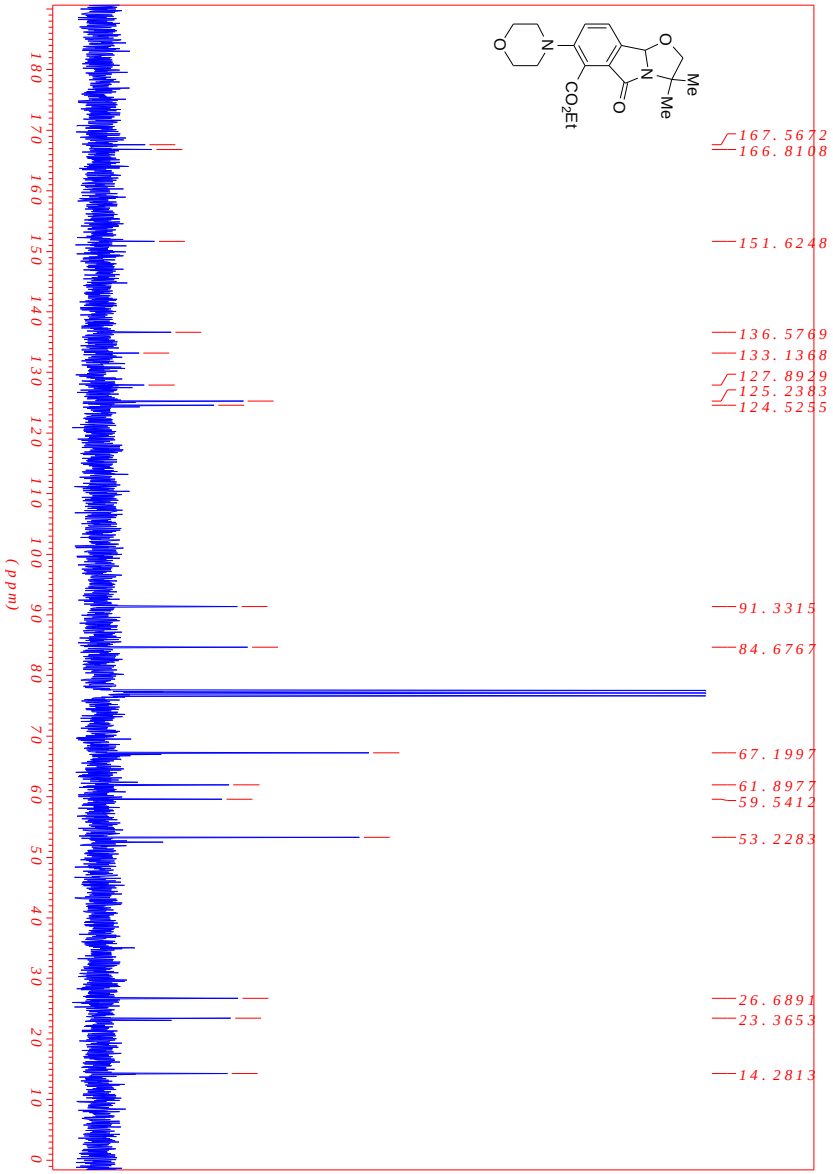

4 d
Integral
(ppm)

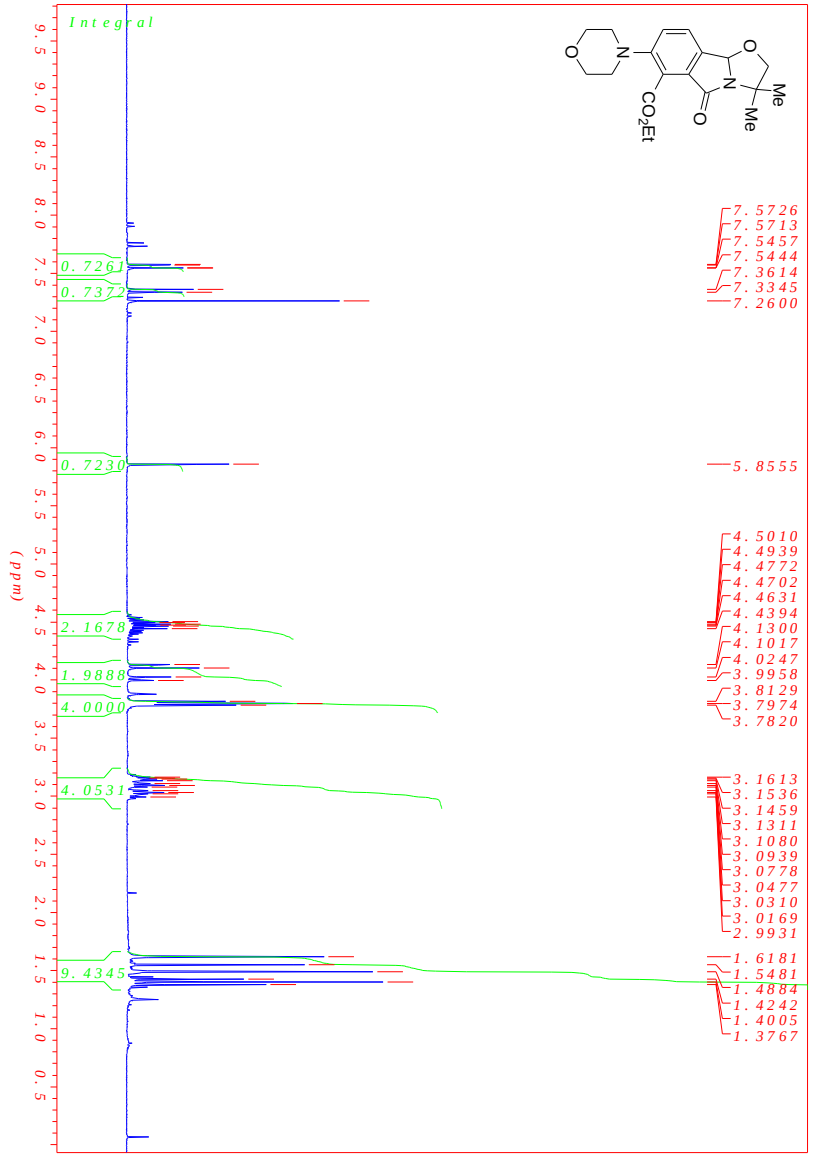

4 d
(ppm)

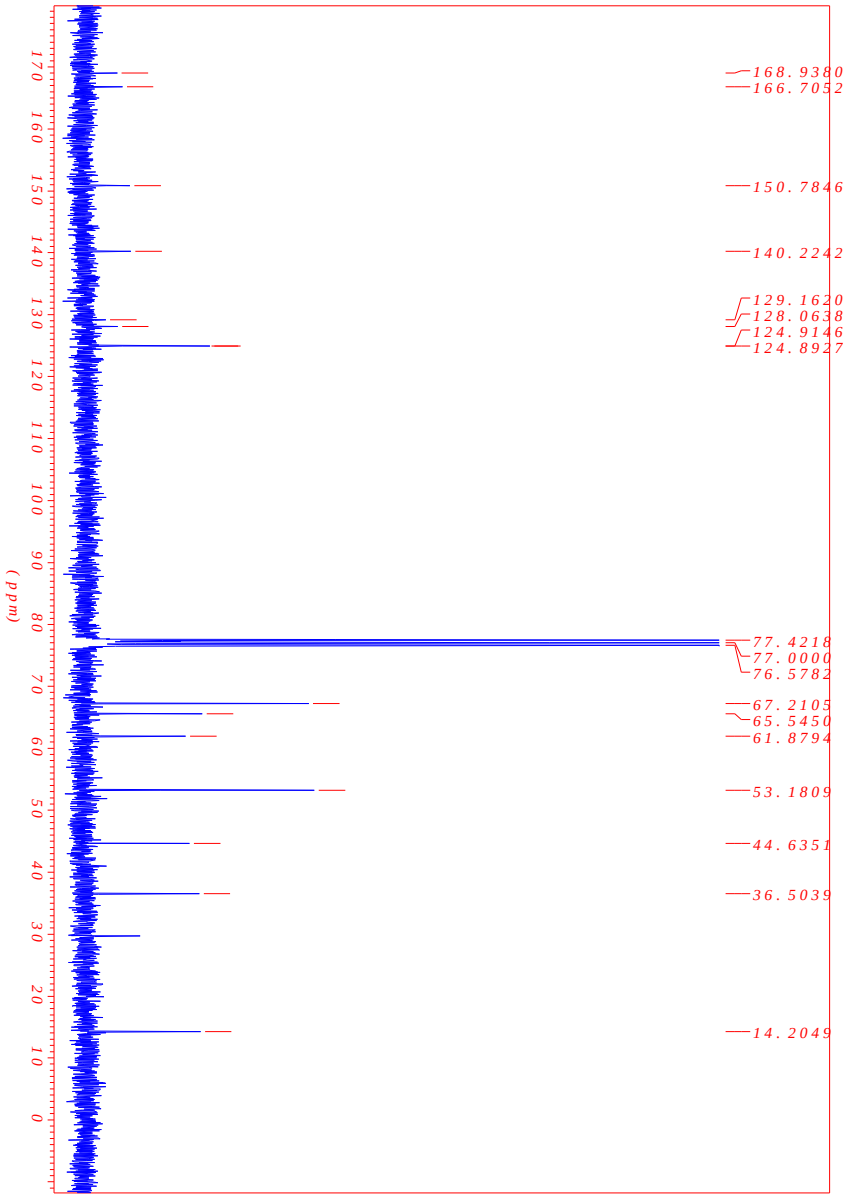

Integral
(ppm)
1.0000
6.9066
3.9592
0.8949
0.9622
0.9899
3.0375
3.1908
2.8249
3.0934
7.6350
7.6286
7.6074
7.6010
7.3879
7.3821
7.3744
7.3673
7.3545
7.3468
7.2730
7.2653
6.8705
6.8467
6.8397
6.8307
6.8223
6.8153
6.7915
6.7857
6.0090
5.2066
5.1828
5.1584
4.8548
4.8478
4.8227
4.8003
4.7938
4.1840
4.1763
4.1590
4.1519
4.1468
4.1288
4.1224
4.1108
4.1044
4.0871
4.0800
4.0633
4.0563
3.7770
3.7693
3.2538
1.1875
1.1798
1.1637
1.1560
1.1400
1.1323
4e
171.6945
165.9561
154.8066
149.9264
143.0752
139.5697
136.7914
128.8420
127.7292
126.0855
125.9255
121.3799
114.4923
91.2187
77.4291
77.0000
76.5782
61.7194
58.3011
55.6246
42.0096
13.7903

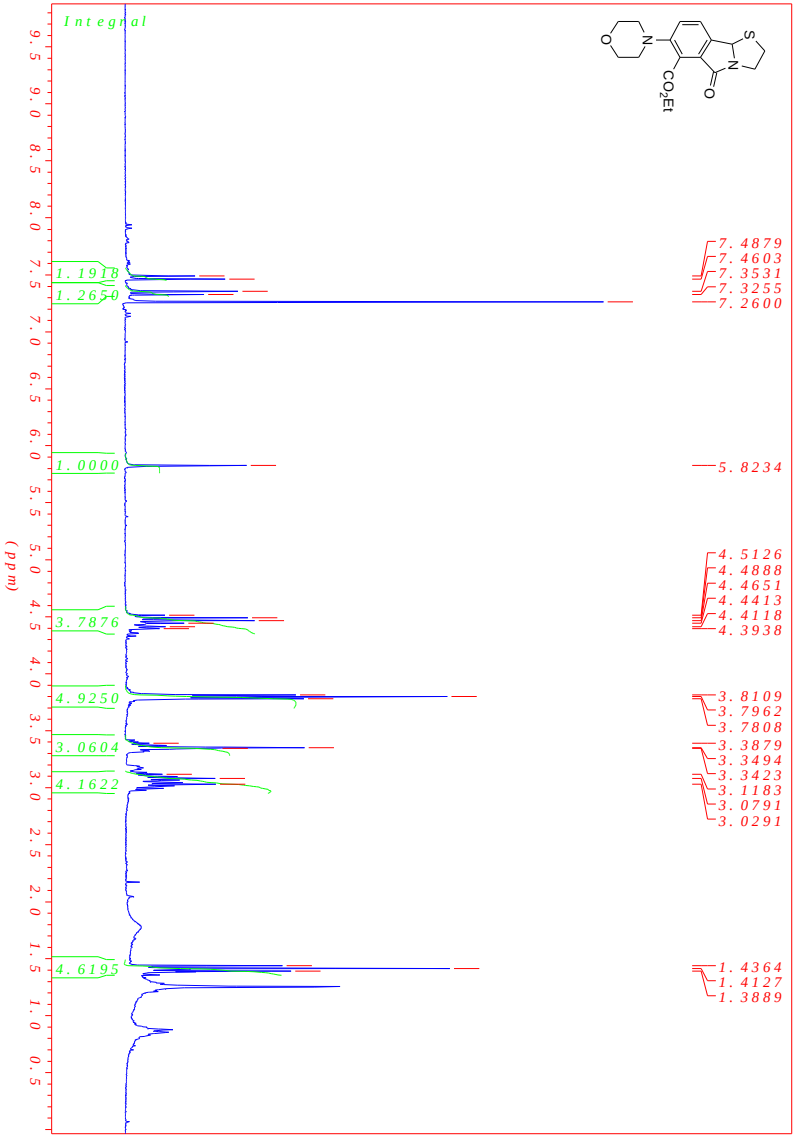

4f
Integral
(ppm)

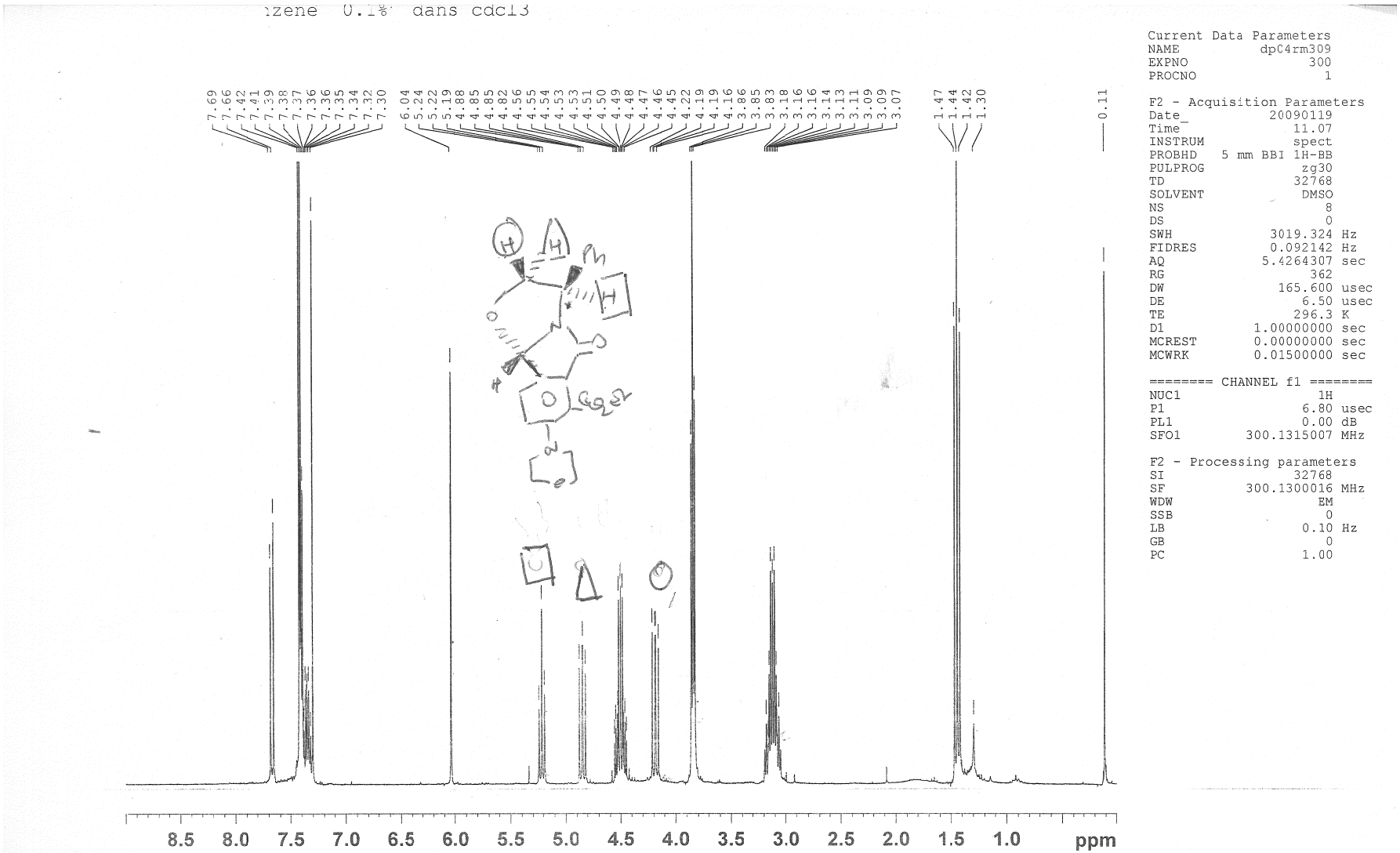

4f
(ppm)


167.5672
166.8108
151.6248
136.5769
133.1368
127.8929
125.2383
124.5255
91.3315
84.6767
67.1997
61.8977
59.5412
53.2283
26.6891
23.3653
14.2813
(ppm)


Integral
0.7261
0.7372
0.7230
2.1678
1.9888
4.0000
4.0531
9.4345
7.5726
7.5713
7.5457
7.5444
7.3614
7.3345
7.2600
5.8555
4.5010
4.4939
4.4772
4.4702
4.4631
4.4394
4.1300
4.1017
4.0247
3.9958
3.8129
3.7974
3.7820
3.1613
3.1536
3.1459
3.1311
3.1080
3.0939
3.0778
3.0477
3.0310
3.0169
2.9931
1.6181
1.5481
1.4884
1.4242
1.4005
1.3767
(ppm)


168.9380
166.7052
150.7846
140.2242
129.1620
128.0638
124.9146
124.8927
77.4218
77.0000
76.5782
67.2105
65.5450
61.8794
53.1809
44.6351
36.5039
14.2049
(ppm)


Integral
1.1918
1.2650
1.0000
3.7876
4.9250
3.0604
4.1622
4.6195
7.4879
7.4603
7.3531
7.3255
7.2600
5.8234
4.5126
4.4888
4.4651
4.4413
4.4118
4.3938
3.8109
3.7962
3.7808
3.3879
3.3494
3.3423
3.1183
3.0791
3.0291
1.4364
1.4127
1.3889
(ppm)



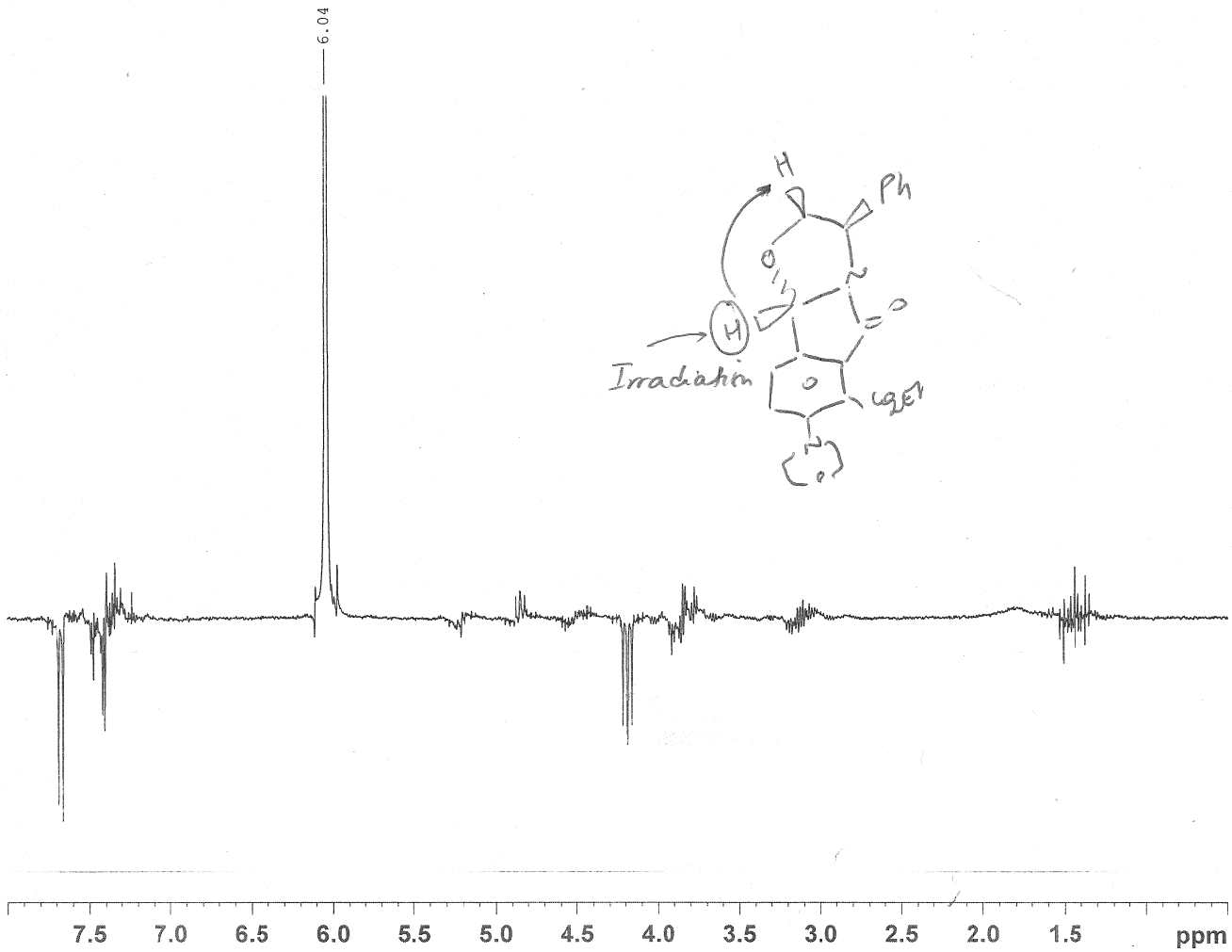

```
Current Data Parameters
NAME          dpC4rm309
EXPNO               300
PROCNO                1

F2 - Acquisition Parameters
Date_           20090119
Time              11.07
INSTRUM           spect
PROBHD   5 mm BBI 1H-BB
PULPROG            zg30
TD                32768
SOLVENT            DMSO
NS                     8
DS                     0
SWH            3019.324 Hz
FIDRES         0.092142 Hz
AQ            5.4264307 sec
RG                   362
DW               165.600 usec
DE                 6.50 usec
TE                296.3 K
D1            1.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                6.80 usec
PL1               0.00 dB
SFO1        300.1315007 MHz

F2 - Processing parameters
SI                32768
SF          300.1300016 MHz
WDW                  EM
SSB                   0
LB                 0.10 Hz
GB                    0
PC                 1.00
```

No title




```
Current Data Parameters
NAME            dp04rm309
EXPNO                 312
PROCNO                  1

F2 - Acquisition Parameters
Date_            20090119
Time               11.51
INSTRUM            spect
PROBHD   5 mm BBI 1H-BB
PULPROG           zgf2pr
TD                32768
SOLVENT            DMSO
NS                    48
DS                     4
SWH             3019.324 Hz
FIDRES          0.092142 Hz
AQ             5.4264307 sec
RG                   181
DW               165.600 usec
DE                  6.50 usec
TE                 296.4 K
D1            5.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec
NUC1                  1H
P1                  6.80 usec
PL1                 0.00 dB
SFO1         300.1315007 MHz
NUC2                  1H
PL2                120.00 dB
PL14                70.00 dB
SFO2         300.1318157 MHz

F2 - Processing parameters
SI                 32768
SF          300.1300016 MHz
WDW                   EM
SSB                    0
LB                  0.50 Hz
GB                     0
PC                  1.00
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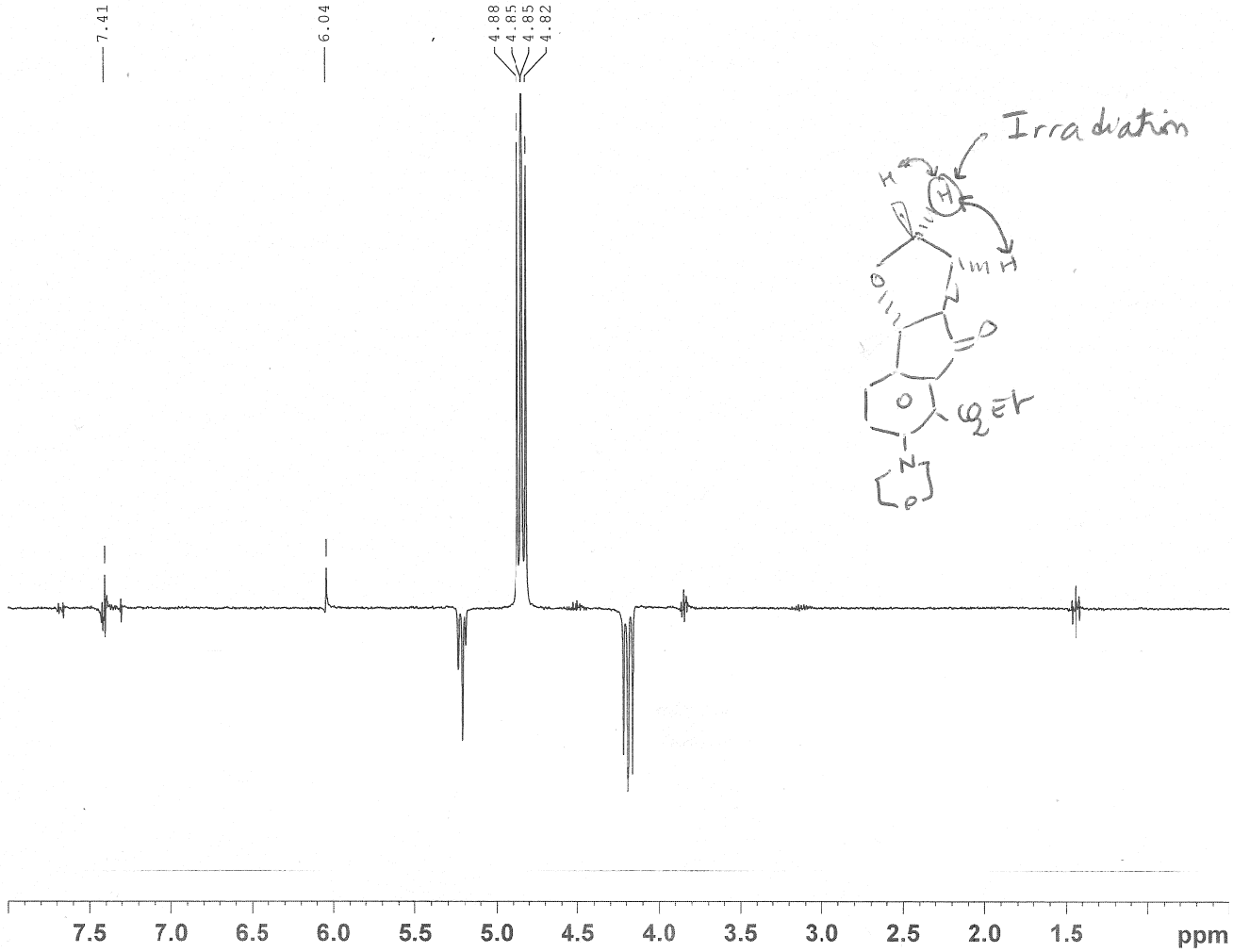
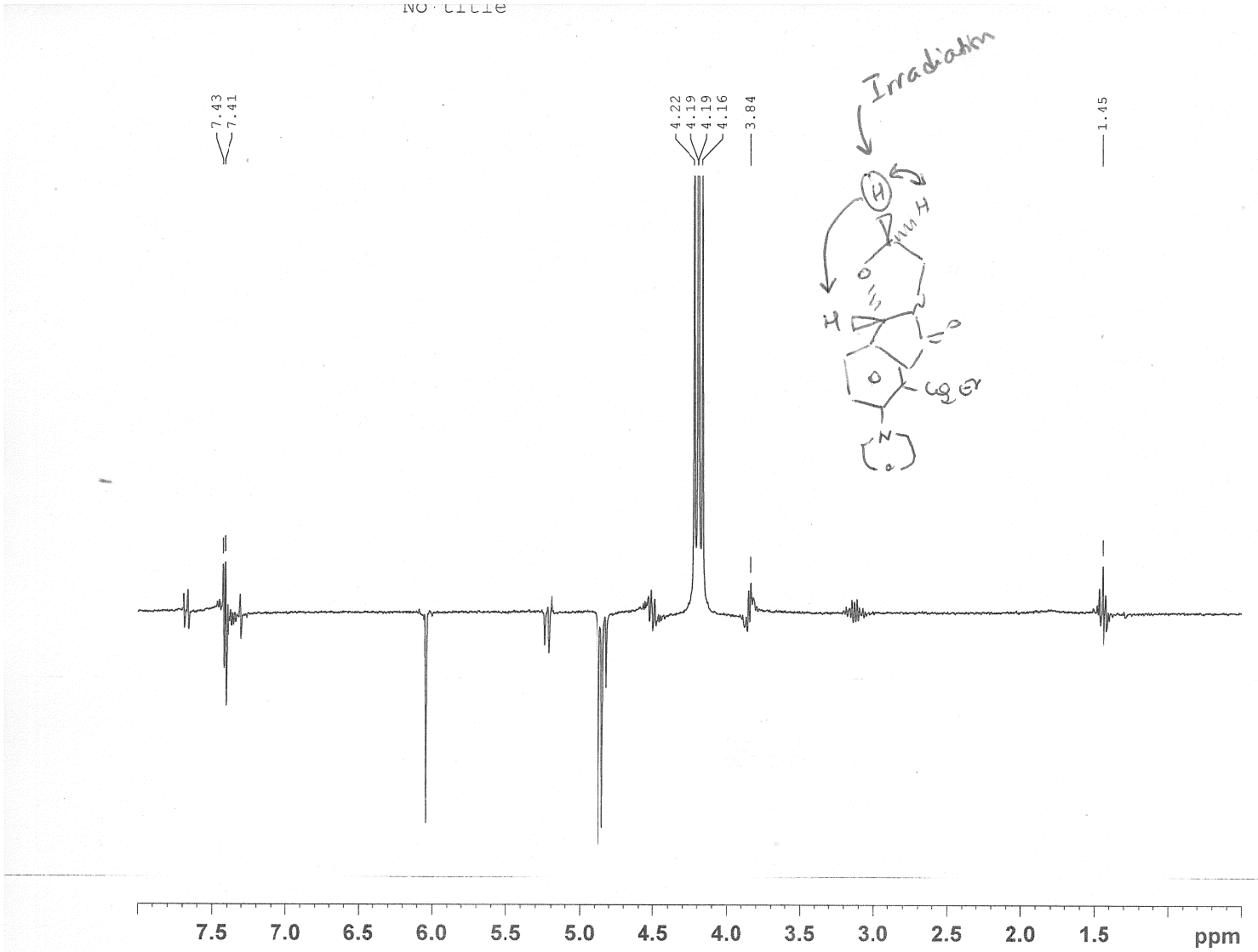
No title



Current Data Parameters
NAME dp04rm309
EXPNO 302
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090119
Time 15.40
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zgf2pr
TD 32768
SOLVENT DMSO
NS 16
DS 4
SWH 3019.324 Hz
FIDRES 0.092142 Hz
AQ 5.4264307 sec
RG 181
DW 165.600 usec
DE 6.50 usec
TE 296.5 K
D1 5.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
NUC1 1H
P1 6.80 usec
PL1 0.00 dB
SFO1 300.1315007 MHz
NUC2 1H
PL2 120.00 dB
PL14 80.00 dB
SFO2 300.1314565 MHz

F2 - Processing parameters
SI 32768
SF 300.1300016 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.00

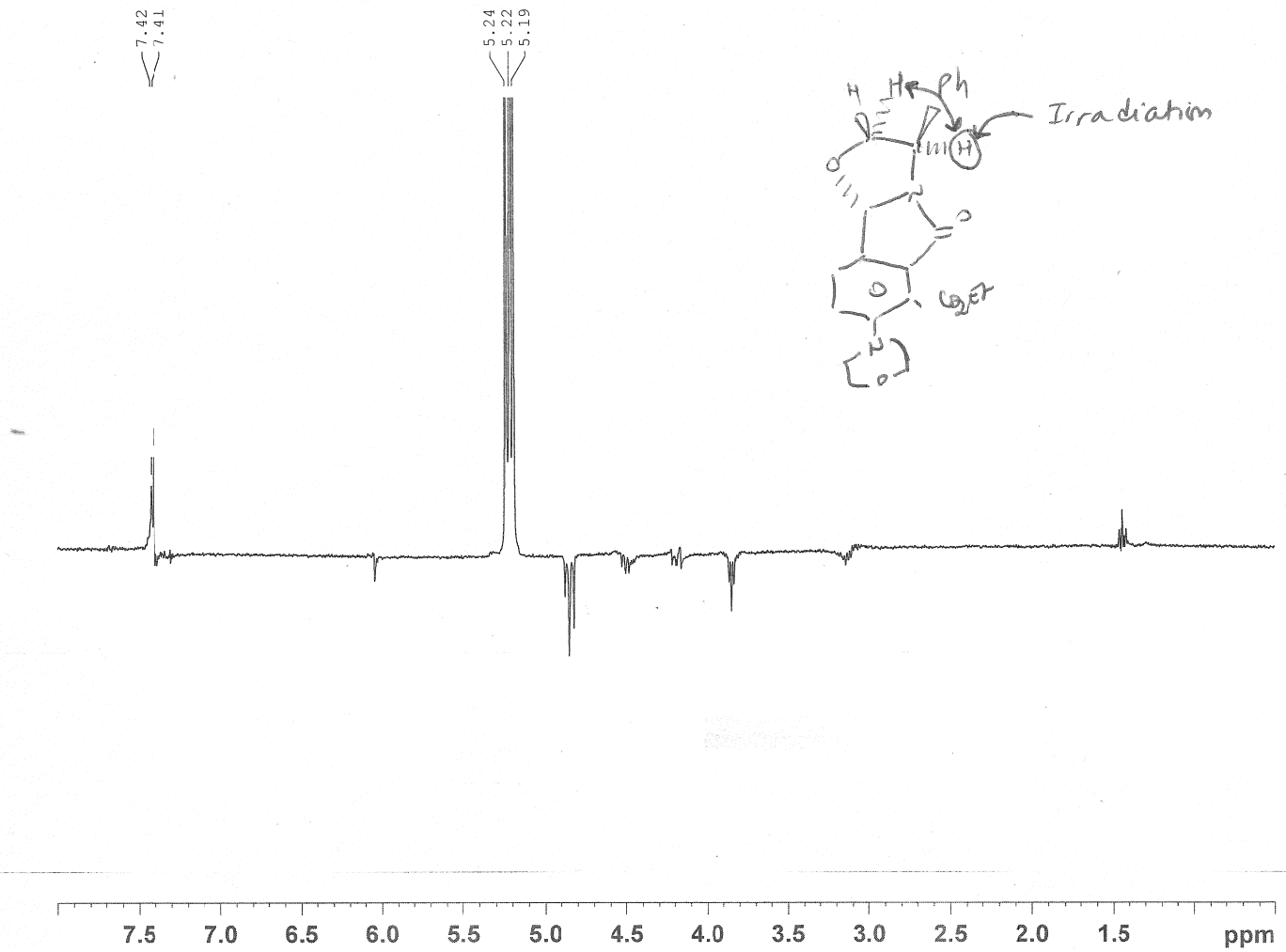



```
Current Data Parameters
NAME          dp04rm309
EXPNO               372
PROCNO                1

F2 - Acquisition Parameters
Date_          20090119
Time              16.18
INSTRUM           spect
PROBHD   5 mm BBI 1H-BB
PULPROG         zgf2pr
TD              32768
SOLVENT           DMSO
NS                   16
DS                    4
SWH            3019.324 Hz
FIDRES         0.092142 Hz
AQ            5.4264307 sec
RG                  181
DW              165.600 usec
DE                 6.50 usec
TE                296.6 K
D1           5.00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec
NUC1                 1H
P1                 6.80 usec
PL1                0.00 dB
SFO1        300.1315007 MHz
NUC2                 1H
PL2               120.00 dB
PL14               80.00 dB
SFO2        300.1312586 MHz

F2 - Processing parameters
SI                32768
SF          300.1300016 MHz
WDW                  EM
SSB                   0
LB                 0.50 Hz
GB                    0
PC                 1.00
```

No title



Current Data Parameters
NAME dp04rm309
EXPNO 362
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090119
Time 15.58
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zgf2pr
TD 32768
SOLVENT DMSO
NS 16
DS 4
SWH 3019.324 Hz
FIDRES 0.092142 Hz
AQ 5.4264307 sec
RG 181
DW 165.600 usec
DE 6.50 usec
TE 296.4 K
D1 5.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
NUC1 1H
P1 6.80 usec
PL1 0.00 dB
SFO1 300.1315007 MHz
NUC2 1H
PL2 120.00 dB
PL14 80.00 dB
SFO2 300.1315676 MHz

F2 - Processing parameters
SI 32768
SF 300.1300016 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.00