Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Electronic Supplementary Information

Synthesis of α-Sulfenyl monoketones via Metal-Free Oxidative Cross Dehydrogenative Coupling (CDC) Reaction

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Contents:

General experimental

NMR spectra were recorded at 400 MHz, using $CDCl_3$. For the spectra recorded in $CDCl_3$, TMS (tetramethylsilane) served as internal standard for ^1H NMR (0.00 ppm) and solvent signal was used as reference for ^{13}C NMR (77.00 ppm). IR spectra were measured using a FT-IR spectrometer. Mass spectra were measured using Q-Tof (ESI-HRMS) and GC-MS. Column chromatography was performed on Silica gel 100-200 mesh. Unless otherwise noted, starting materials and solvents obtained from commercial suppliers were used without further purification. The starting materials benzoxazole-2thiones were prepared as described in the literature.[1]

General and typical experimental procedures:

Method A: General experimental procedure for the synthesis of *α*-sulfenyl ketones (3a-3g, 3l, 3m-3p, 5a, 5b, 5d-5f): To a well-stirred, ice-cold heterogeneous solution of benzoxazole-2-thione derivatives (**1**, 0.25 mmol), acetophenone derivatives/aliphatic ketones (**2** or **4,** 3 equiv, 0.75 mmol), and $K_2S_2O_8$ (74.25 mg, 1.1 equiv) in DCM (1 mL) was added aq $HClO_4$ (69-72% in water, 0.11 mL, 5.0 equiv) drop wise. The reaction mixture was allowed to attain room temperature, and stirring was continued for 4 h. The reaction was then quenched by water (3-4 mL), reaction mixture was transferred into 50 mL separating funnel followed by the addition of 20 mL water. The crude compound was then extracted with DCM (10 mLx3), the combined organic fractions were dried over anhydrous Na_2SO_4 and purified on silica gel (100-200 mesh size) column by using EtOAc - petroleum ether mixture as eluent to afford the expected product and the unreacted ketone.

Method B: General experimental procedure for the synthesis of *α*-sulfenyl ketones; (3h-3k and 3q-3r): To a well-stirred, ice-cold heterogeneous solution of benzoxazole-2-thione derivatives (**1**, 0.25 mmol), Propiophenone derivatives (**2**, 1.0 mmol), and $K_2S_2O_8$ (74.25 mg, 1.1 equiv) in DCM (1 mL) was added aq. $HClO_4$ (69-72% in water, 0.11 mL, 5.0 equiv) drop wise. The reaction mixture was allowed to attain room temperature, and stirring was continued for 4 h. The reaction was then quenched by water (3-4 mL) and reaction mixture was transferred into 50 mL separating funnel followed by the addition of 20 mL water. The crude compound was then extracted with DCM (10 mLx3), the combined organic fractions were dried over anhydrous Na_2SO_4 and purified on silica gel (100-200 mesh size) column by using EtOAc-petroleum ether mixture as eluent to afford the expected product and the unreacted ketone.

Method C: General experimental experimental procedure for the synthesis of *α*-sulfenyl ketones; (7a-7h): To a well-stirred, ice-cold solution of 1,2-bis(benzo[d]thiazol-2-yl)disulfane (**6a'**, 0.25 mmol), ketones (**2 or 3**, 1.0 mmol), and $K_2S_2O_8$ (135 mg, 2.0 equiv) in CH_3CN (3 mL) was added aq $HClO_4$ (69-72% in water, 0.43 mL, 20 equiv) drop wise. The reaction mixture was allowed to attain room temperature, and stirring was continued for 4 h. The reaction was then quenched by water (3-4 mL) and reaction mixture was transferred into 50 mL separating funnel followed by the addition of 20 mL water. The crude compound was then extracted with DCM (10 mLx3), the combined organic fractions were dried over

anhydrous Na_2SO_4 and purified on silica gel (100-200 mesh size) column by using EtOAc - petroleum ether mixture as eluent to afford the expected product.

Typical experimental procedure for the synthesis of 1-((5-methylbenzo[d]oxazol-2-yl)thio)propan-2-one (5c): To a well-stirred, ice-cold heterogeneous solution of 5-methyl-benzoxazole-2-thione derivatives (**1a**, 0.25 mmol) and $K_2S_2O_8$ (74.25 mg, 1.1 equiv) in mixture of acetone (1 mL) and DCM (1 mL) solvents was added aq. $HClO_4$ (69-72% in water, 0.11 mL, 5 equiv) drop wise. The reaction mixture was allowed to attain room temperature, and stirring was continued for 4 h. The reaction was then quenched by water (3-4 mL) and reaction mixture was transferred into 50 mL separating funnel followed by the addition of 20 mL water. The crude compound was then extracted with DCM (10 mLx 3), the combined organic fractions were dried over anhydrous Na_2SO_4 and purified on silica gel (100-200 mesh size) column by using EtOAc - petroleum ether mixture as eluent to afford the expected product.

Typical experimental procedure for the synthesis of 11-(benzo[d]thiazol-2-ylthio)propan-2-one (7f): To a well-stirred, ice-cold heterogeneous solution of 5-methyl-benzoxazole-2-thione derivatives (**1a**, 0.25 mmol) and $K_2S_2O_8$ (74.25 mg, 1.1 equiv) in mixture of acetone (1 mL) and CH_3CN (2 mL) solvents was added aq. $HClO_4$ (69-72% in water, 0.43 mL, 20 equiv) drop wise. The reaction mixture was allowed to attain room temperature, and stirring was continued for 12 h. The reaction was then quenched by water (3-4 mL) and reaction mixture was transferred into 50 mL separating funnel followed by the addition of 20 mL water. The crude compound was then extracted with DCM (10 mLx 3), the combined organic fractions were dried over anhydrous Na_2SO_4 and purified on silica gel (100-200 mesh size) column by using EtOAc - petroleum ether mixture as eluent to afford the expected product.

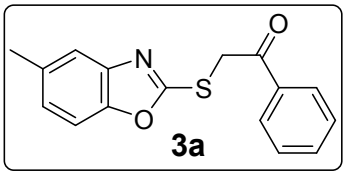
Note 1: The progress of the reactions could not be monitored by TLC. Although the starting material benzoxazole-2-thione derivatives found to disappear within an hour, the yields obtained in such cases are low. However, 3-4 h is the optimum time in case of ***method A*** and ***method B***. In case of ***method C***, the starting material disulfide disappears soon after the addition of acid and optimum reaction time found as 12 h.

Note 2: Following ***method C***, aq $HClO_4$ should be added very slowly (0.2 mL/min). Initial addition of 0.1-0.2 mL causes complete disappearance of disulfane and slowly precipitate

back to form a heterogeneous reaction mixture (off-white or light yellow in colour) with further addition of acid. Generally, low yields were observed with rapid addition of acid wherein dark yellow heterogeneous reaction mixture formed after complete addition of acid.

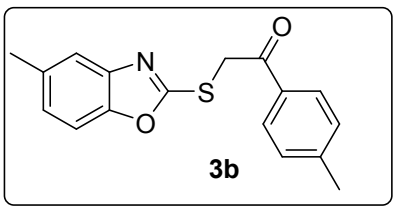
Note 3: Following ***method C,*** generally during purification stage the degradation of the product was observed in all the cases and hence the compounds **7a-7h** were isolated in low yields. However, improvement in isolated yields was observed with rapid purification and with the use of silica-gel of 100-200 mesh size.

Characterization data for products:



3a

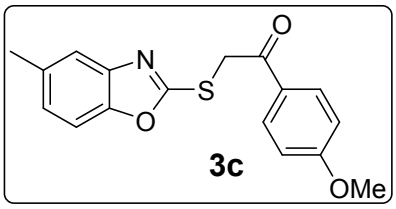
2-((5-Methylbenzo[d]oxazol-2-yl)thio)-1-phenylethanone (3a): Prepared as described in the general experimental procedure - Method A: yellow solid; **mp:** 88-90 ºC; **Yield:** 94% (67 mg); R_f (5% EtOAc/hexane) 0.4; **IR** (KBr, cm^{-1}):1680, 1426, 1378, 1253, 1147, 1103, 995; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.07 (d, *J* = 7.6 Hz, 2H), 7.63 (t, *J* = 7.4 Hz, 1H), 7.52 (t, *J* = 8.2 Hz, 2H), 7.36 (s, 1H), 7.30 (d, *J* = 8.4 Hz, 1H), 7.04 (d, *J* = 8.4 Hz, 1H), 4.95 (s, 2H), 2.43 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 192.5, 163.9, 150.3, 141.9, 135.1, 134.2, 134.0, 128.8, 128.5, 125.0, 118.4, 109.3, 41.1, 21.4; **HRESI-MS** (*m/z*): Calculated for $C_{16}H_{13}NO_2S$ (M + Na): 306.0565, found (M + Na): 306.0568.



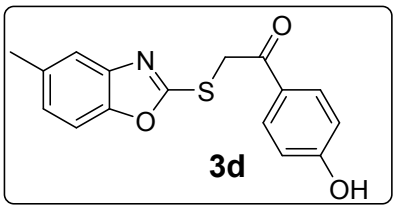
3b

2-((5-methylbenzo[d]oxazol-2-yl)thio)-1-(p-tolyl)ethan-1-one (3b): Prepared as described in the general experimental procedure - Method A: brown solid; **mp:** 131-132 ºC. **Yield:** 94% (70 mg); R_f (5% EtOAc/hexane) 0.2; **IR** (KBr, cm^{-1}): 1687, 1495, 1254, 1192, 1100, 988, 754; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.97 (d, *J*=8.4 Hz, 2 H), 7.37 (s, 1 H), 7.32-29 (m, 3 H), 7.05 (d, *J*=8.4 Hz, 1 H), 4.93 (s, 2 H), 2.44 (s, 3 H), 2.43 (s, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 192.1, 164.0, 150.3, 145.1, 141.9, 134.2, 132.7, 129.5, 128.7, 125.0, 118.6, 109.3,

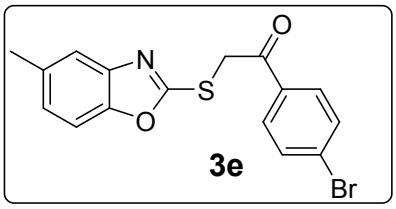
41.1, 21.6, 21.4; **HRESI-MS** (*m/z*): Calculated for $C_{17}H_{15}NO_2S$ (M + Na): 320.0721, found (M + Na): 320.0718; Recovered amount of ketone: 1.6 equiv (53 mg).



1-(4-Methoxyphenyl)-2-((5-methylbenzo[d]oxazol-2-yl)thio)ethanone (3c): Prepared as described in the general experimental procedure - Method A: White solid; **mp:** 120-121 ºC; **Yield:** 63% (49 mg); R_f (5% EtOAc/hexane) 0.1; **IR** (KBr, cm^{-1}): 1656, 1509, 1492, 1325, 1259, 1149, 1011, 991; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.04 (d, *J* = 8.0 Hz, 2H), 7.36 (s, 1H), 7.29 (d, *J* = 8.4 Hz, 1H), 7.04 (d, *J* = 8.4 Hz, 1H), 6.97 (d, *J* = 8.4 Hz, 2H), 4.90 (s, 2H), 3.88 (s, 3H), 2.43 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 190.9, 164.2, 164.1, 150.2, 141.9, 134.1, 130.9, 128.1, 124.9, 118.4, 114.0, 109.3, 55.6, 40.9, 21.4; **HRESI-MS** (*m/z*): Calculated for $C_{17}H_{15}NO_3S$ (M + Na): 336.0670, found (M + Na): 336.0670; Recovered amount of ketone: 1.9 equiv (70 mg).

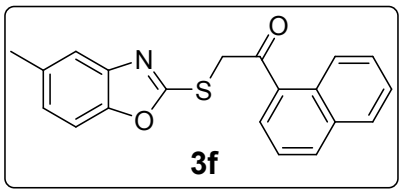


1-(4-hydroxyphenyl)-2-((5-methylbenzo[d]oxazol-2-yl)thio)ethan-1-one (3d): Prepared as described in the general experimental procedure - Method A: White solid; **mp:** 193-195 ºC; **Yield:** 45% (34 mg); R_f (30% EtOAc/hexane) 0.4; **IR** (KBr, cm^{-1}): 1665, 1469, 1308, 1237, 1161, 996; **^{1}H NMR** (400 MHz, DMSO-d_6): δ ppm 10.53 (brs. 1 H), 7.95 (d, *J*=8.4 Hz, 2 H), 7.49 (d, *J*=8.0 Hz, 1 H), 7.38 (s, 1 H), 7.11 (d, *J*=8.4 Hz, 1 H), 6.91 (d, *J*=8.4 Hz, 2 H), 5.06 (s, 2 H), 2.38 (s, 3 H); **^{13}C NMR** (100 MHz, DMSO-d_6): δ 190.4, 163.8, 162.7, 149.5, 141.4, 134.0, 131.1, 126.7, 125.0, 118.1, 115.4,109.5, 20.9; **HRESI-MS** (*m/z*): Calculated for $C_{16}H_{13}NO_3S$ (M + Na): 322.0514, found (M + Na): 322.0514. Recovered amount of ketone: 2.4 equiv (82 mg).



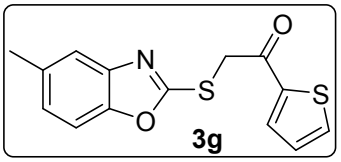
3e

1-(4-bromophenyl)-2-((5-methylbenzo[d]oxazol-2-yl)thio)ethan-1-one (3e): Prepared as described in the general experimental procedure - Method A: White solid; **mp:** 131-132 ºC; **Yield:** 93% (84 mg); R_f (5% EtOAc/hexane) 0.2; **IR** (KBr, cm^{-1}): 1677, 1580, 1497, 1390, 1254, 1146, 1068, 988, 796, 760; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.93 (d, *J*=8.4 Hz, 2 H), 7.65 (d, *J*=8.4 Hz, 2 H), 7.35 (s, 1 H), 7.30 (d, *J*=8.4 Hz, 1 H), 7.05 (d, *J*=8.4 Hz, 1 H), 4.87 (s, 2 H), 2.43 (s, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 191.61, 163.60, 150.30, 141.79, 134.23, 133.92, 132.20, 130.00, 129.35, 125.06, 118.46, 109.35, 40.63, 21.40; **HRESI-MS** (*m/z*): Calculated for $C_{16}H_{12}NO_2SBr$ (M + H): 361.9850, found (M + H): 361.9851. Recovered amount of ketone: 1.7 equiv (84 mg).

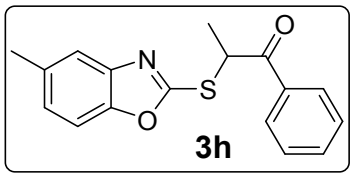


3f

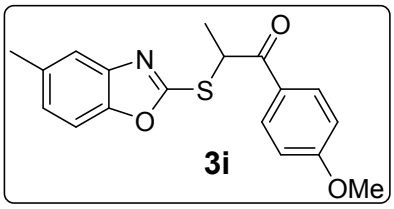
2-((5-methylbenzo[d]oxazol-2-yl)thio)-1-(naphthalen-2-yl)ethan-1-one (3f): Prepared as described in the general experimental procedure - Method A: Brown solid; **mp:** 116-119 ºC; **Yield:** 90% (75 mg); R_f (5% EtOAc/hexane) 0.25; **IR** (Neat, cm^{-1}): 1681, 1501, 1363, 1256, 1150, 1088; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.68 (d, *J*=8.4 Hz, 1 H), 8.08 (d, *J*=7.2 Hz, 1 H), 8.02 (d, *J*=7.6 Hz, 1 H),7.86 (d, *J*=7.6 Hz, 1 H), 7.48 - 7.62 (m, 3 H), 7.33 (s, 1 H), 7.26 (d, *J*=8.0 Hz, 1 H), 7.02 (d, *J*=8.0 Hz, 1 H), 4.93 (s, 2 H), 2.41 (s, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 195.6, 163.8, 150.3, 141.9, 134.1, 133.9, 133.8, 133.4, 130.2, 128.7, 128.4, 128.3, 126.7, 125.7, 124.9, 124.2, 118.4, 109.3, 43.0, 21.4; **HRESI-MS** (*m/z*): Calculated for $C_{20}H_{15}NO_2S$ (M + H): 334.0902, found (M + H): 334.0908; Recovered amount of ketone: 1.97 equiv (84 mg).



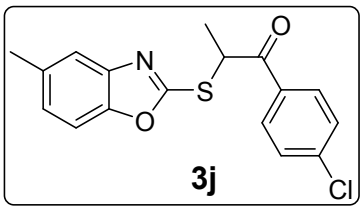
2-((5-Methylbenzo[d]oxazol-2-yl)thio)-1-(thiophen-2-yl)ethanone (3g): Prepared as described in the general experimental procedure - Method A: White solid; **Yield:** 79% (57 mg); R_f (10% EtOAc/hexane) 0.35; **IR** (Neat, cm^{-1}):1665, 1499, 1412, 1257, 1150, 1104, 1016; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.93 (dd, J_1 = 3.6 Hz, J_2 = 0.8 Hz, 1 H), 7.717 (dd, J_1 = 4.6 Hz, J_2 = 0.4 Hz, 1 H), 7.36 (s, 1 H), 7.29 (d, J = 8.0 Hz,1 H), 7.169 (dd, J_1 = 5.0 Hz, J_2 = 0.8 Hz, 1 H), 7.04 (dd, J_1 = 8.2 Hz, J_2 = 0.6 Hz, 1 H), 4.81 (s, 2 H), 2.42 (s, 3 H) ; **^{13}C NMR** (100 MHz, $CDCl_3$): δ 185.3, 163.6, 150.3, 141.9, 141.8, 135.0, 134.2, 133.3, 128.4, 125.0, 118.5, 109.4, 40.1, 21.4; **HRESI-MS** (*m/z*): Calculated for $C_{14}H_{11}NO_2S_2$ (M + Na): 312.0129, found (M + Na): 312.0131.



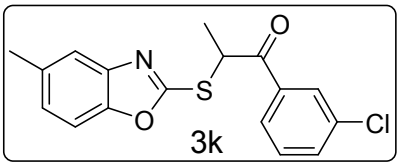
2-((5-Methylbenzo[d]oxazol-2-yl)thio)-1-phenylpropan-1-one (3h): Prepared as described in the general experimental procedure - Method B: Colourless liquid; **Yield:** 94% (70 mg); R_f (5% EtOAc/hexane) 0.3; **IR** (Neat, cm^{-1}): 1676, 1474, 1259, 1209, 1157, 1000, 805; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.08 (d, J = 8.4 Hz, 2H), 7.60 (t, J = 7.4 Hz, 1H), 7.49 (t, J = 7.6 Hz, 2H), 7.39 (s, 1H), 7.29 (d, J = 8.4 Hz, 1H), 7.05 (d, J = 8.4 Hz, 1H), 5.70 (q, J = 7.0 Hz, 1H), 2.44 (s, 3H), 1.81 (d, J = 6.8 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 197.0, 163.6, 150.2, 141.9, 134.4, 134.2, 133.8, 128.83, 128.81, 125.0, 118.5, 109.3, 47.5, 21.4, 19.1; **HRESI-MS** (*m/z*): Calculated for $C_{17}H_{15}NO_2S$ (M + Na): 320.0721, found (M + Na): 320.0722. Recovered amount of ketone: 2.0 equiv (70 mg).



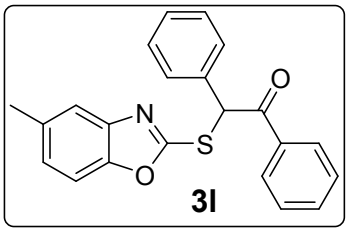
1-(4-Methoxyphenyl)-2-((5-methylbenzo[d]oxazol-2-yl)thio)propan-1-one (3i): Prepared as described in the general experimental procedure - Method B: White solid; **mp:** 120-122 ºC; **Yield:** 90% (74 mg); R_f (10% EtOAc/hexane) 0.4; **IR** (KBr, cm^{-1}): 1500, 1402, 1251, 1209, 1144, 1098, 1013, 812; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.07 (d, *J* = 8.8 Hz, 2H), 7.40 (s, 1H), 7.29 (d, *J* = 8.4 Hz, 1H), 7.05 (d, *J* = 8.0 Hz, 1H), 6.95 (d, *J* = 8.8 Hz, 2H), 5.67 (q, *J* = 8.0 Hz, 1H), 3.87 (s, 3H), 2.44 (s, 3H), 1.81 (d, *J* = 7.8 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 195.5, 164.1, 163.9, 150.2, 141.9, 134.1, 131.3, 127.2, 125.0, 118.5, 114.0, 109.3, 55.5, 47.4, 21.4, 19.4; **HRESI-MS** (*m/z*): Calculated for $C_{18}H_{17}NO_3S$ (M + Na): 350.0827, found (M + Na): 350.0832; Recovered amount of ketone: 2.1 equiv (88 mg).



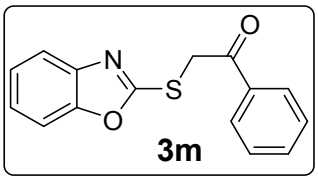
1-(4-chlorophenyl)-2-((5-methylbenzo[d]oxazol-2-yl)thio)propan-1-one (3j): Prepared as described in the general experimental procedure - Method B: Brown solid; **Yield:** 88% (73 mg); **mp:** 88-90 ºC (lit.); R_f (5% EtOAc/hexane) 0.5; **IR** (KBr, cm^{-1}): 1667, 1585,1489, 1254, 1192, 1144, 1095, 971, 835, 791; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.03 (d, *J* = 8.8 Hz, 2H), 7.45 (d, *J* = 8.8 Hz, 1H), 7.38 (s, 1H), 7.28 (d, *J* = 8.4 Hz, 1H), 7.05 (d, *J* = 8.4 Hz, 1H), 5.63 (q, *J* = 7.2 Hz, 1H), 2.44 (s, 3H), 1.78 (d, *J* = 7.2 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 195.8, 163.3, 150.3, 141.8, 140.3, 134.2, 132.8, 130.2, 129.1, 125.1, 118.5, 109.4, 47.0, 21.4, 18.8; **HRESI-MS** (*m/z*): Calculated for $C_{17}H_{14}ClNO_2S$ (M + Na): 354.0331, found (M + Na): 354.0331; Recovered amount of ketone: 2.5 equiv (105 mg).



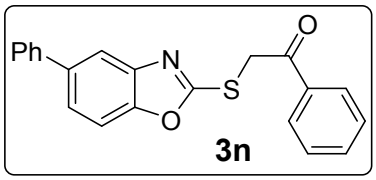
1-(3-Chlorophenyl)-2-((5-methylbenzo[d]oxazol-2-yl)thio)propan-1-one (3k): Prepared as described in the general experimental procedure - Method B: Colourless liquid; **Yield:** 95% (78 mg); R_f (10% EtOAc/hexane) 0.4; **IR** (KBr, cm^{-1}):1689, 1571, 1497,1307, 1253, 1150, 1103, 988, 799; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.09 (s, 1H), 7.97 (d, *J* = 8.0 Hz, 1H), 7.58-7.56 (m, 1H), 7.43 (t, *J* = 8.0 Hz, 1H), 7.39 (s, 1H), 7.30 (d, *J* = 8.0 Hz, 1H), 7.06 (d, *J* = 8.0 Hz, 1H), 5.61 (q, *J* = 7.2 Hz, 1H), 2.45 (s, 3H), 1.79 (d, *J* = 7.2 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 195.9, 163.2, 150.3, 141.8, 136.2, 135.2, 134.3, 133.7, 130.1, 128.9, 126.9, 125.2, 118.6, 109.4, 47.1, 21.4, 18.7; **HRESI-MS** (*m/z*): Calculated for $C_{17}H_{14}ClNO_2S$ (M + H): 332.0512, found (M + H): 332.0513; Recovered amount of ketone: 2.5 equiv (104 mg).



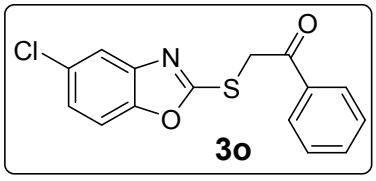
2-((5-methylbenzo[d]oxazol-2-yl)thio)-1,2-diphenylethan-1-one (3l): Prepared as described in the general experimental procedure - Method A: yellow solid; **mp:** 103-105 ºC; **Yield:** 96% (86 mg); R_f (5% EtOAc/hexane) 0.3; **IR** (Neat, cm^{-1}):1765, 1680, 1492, 1206, 1145, 1098, 996; **^{1}H NMR** (400 MHz, $CDCl_3$): δ ppm 8.04 (d, *J*=8.0 Hz, 2 H), 7.57 (d, *J*=7.2 Hz, 2 H), 7.50 (t, *J*=7.6 Hz, 1 H), 7.40 (t, *J*=7.8 Hz, 2 H), 7.32 (s, 1H), 7.29 (t, *J*=7.4 Hz, 2 H), 7.25-7.22 (m, 2 H), 6.99 (d, *J*=8.4 Hz, 1 H), 6.82 (s, 1 H), 2.38 (s, 3 H)); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 193.84, 163.45, 150.07, 141.78, 134.96, 134.93, 134.00, 133.55, 129.17, 129.13, 128.83, 128.70, 128.65, 124.90, 118.52, 109.22, 58.13, 21.31; **HRESI-MS** (*m/z*): Calculated for $C_{22}H_{17}NO_2S$ (M + Na): 382.0878, found (M + Na): 382.0879. Recovered amount of ketone: 1.8 equiv (91 mg).



2-(benzo[d]oxazol-2-ylthio)-1-phenylethan-1-one (3m)[2a]**:** Prepared as described in the general experimental procedure - Method A: brown crystalline solid; **Yield: mp:** 113-115 ºC (lit.[2b] 115-116) 71% (67 mg); R_f (5% EtOAc/hexane) 0.3; **IR** (Neat, cm^{-1}): 1675, 1493, 1447, 1189, 1130, 996, 746; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.07 (d, *J*=7.6 Hz, 2 H), 7.63 (t, *J*=7.2 Hz, 1 H), 7.57 (d, *J*=7.6 Hz, 1 H), 7.51 (t, *J*=7.6 Hz, 2 H), 7.43 (d, *J*=7.2 Hz, 1 H), 7.29 - 7.22 (m, 2 H), 4.96 (s, 2 H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 197.0, 192.35, 164.01, 151.98, 141.66, 135.12, 134.01, 128.84, 128.51, 124.33, 124.03, 118.38, 109.96, 41.08; **HRESI-MS** (*m/z*): Calculated for $C_{15}H_{11}NO_2S$ (M + Na): 292.0408, found (M + Na): 292.0408.

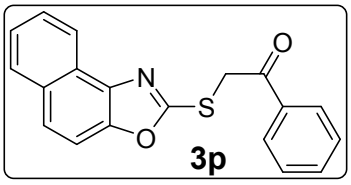


1-phenyl-2-((5-phenylbenzo[d]oxazol-2-yl)thio)ethan-1-one (3n): Prepared as described in the general experimental procedure - Method A: White solid; **mp:** 135-137 ºC; **Yield:** 90% (78 mg); R_f (5% EtOAc/hexane) 0.2; **IR** (KBr, cm^{-1}): 1680, 1494, 1454, 1255, 1190, 1099, 987, 754; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.09 (d, *J*=7.2 Hz, 2 H), 7.77 (s, 1 H), 7.64 (t, *J*=7.4 Hz, 2 H), 7.58 (d, *J*=7.6 Hz, 2 H), 7.53 (t, *J*=7.8 Hz, 2 H), 7.48 - 7.43 (m, 4 H), 7.35 (t, *J*=7.4 Hz, 2 H), 4.98 (s, 2 H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ **^{13}C NMR** (100 MHz, $CDCl_3$): δ 192.3, 164.6, 151.6, 142.3, 140.9, 138.3, 135.2, 134.0, 128.9, 128.8, 128.5, 127.4, 127.2, 123.6, 116.9, 109.9, 41.3; **HRESI-MS** (*m/z*): Calculated for $C_{21}H_{15}NO_2S$ (M + H): 346.0902, found (M + Na): 346.0901.

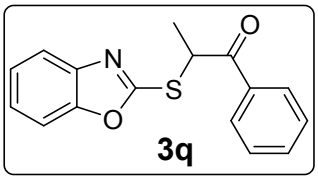


2-((5-chlorobenzo[d]oxazol-2-yl)thio)-1-phenylethan-1-one (3o): Prepared as described in the general experimental procedure - Method A: White solid; **mp:** 117-119 ºC; **Yield:** 71% (54 mg); R_f (5% EtOAc/hexane) 0.25; **IR** (KBr, cm^{-1}): 1691, 1491, 1449, 1205, 1134, 990;

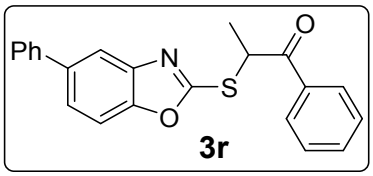
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.07 (d, *J*=8.0 Hz, 2 H), 7.64 (t, *J*=7.2 Hz, 1 H), 7.54 - 7.50 (m, 3 H), 7.34 (d, *J*= 8.4 Hz, 1 H), 7.205 (dd, J_1 = 8.6 Hz, J_2 = 4.0 Hz, 1 H), 4.94 (s, 2 H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 192.10, 165.79, 150.59, 142.82, 135.09, 134.10, 129.88, 128.90, 128.51, 124.16, 118.48, 110.58, 41.14; **HRESI-MS** (*m/z*): Calculated for $C_{15}H_{10}NO_2SCl$ (M + Na): 326.0018, found (M + Na): 326.0018.



2-(naphtho[1,2-d]oxazol-2-ylthio)-1-phenylethan-1-one (3p): Prepared as described in the general experimental procedure - Method A: Pale yellow solid; **mp:** 139-141 ºC; **Yield:** 60% (48 mg); R_f (5% EtOAc/hexane) 0.2; **IR** (KBr, cm^{-1}): 1671, 1582, 1487, 1445, 1373, 1188, 994; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.37 (d, *J*=8.4 Hz, 1 H), 8.09 (dd, *J*=8.2, 1.0 Hz, 2 H), 7.91 (d, *J*=8.0 Hz, 1 H), 7.68 (d, *J*=9.2 Hz, 1 H), 7.65 - 7.57 (m, 3 H), 7.53 - 7.47 (m, 3 H), 4.97 (s, 2 H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 192.72, 162.14, 149.32, 137.19, 135.37, 133.93, 130.97, 128.84, 128.53, 128.40, 126.78, 125.49, 125.24, 124.80, 122.00, 110.29, 40.90; **HRESI-MS** (*m/z*): Calculated for $C_{19}H_{13}NO_2S$ (M + H): 320.0745, found (M + Na): 320.0741.

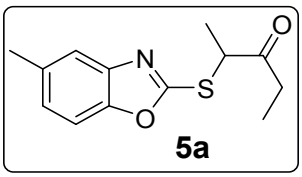


2-(Benzo[d]oxazol-2-ylthio)-1-phenylpropan-1-one (3q): Prepared as described in the general experimental procedure - Method B: Colourless liquid; **Yield**: 83% (59 mg); R_f (5% EtOAc/hexane) 0.4; **IR** (Neat, cm^{-1}): 1683, 1499, 1450, 1330, 1237, 1206, 1132; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.09 (d, *J* = 7.2 Hz, 2H), 7.61 (t, *J* = 7.0 Hz, 2H), 7.50 (t, *J* = 7.6 Hz, 2H), 7.43 (d, *J* = 7.6 Hz, 1H), 7.31-7.263 (m, 2H), 5.72 (q, *J* = 7.1 Hz, 1H), 1.83 (d, *J* = 7.2 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 197.0, 163.8, 152.0, 141.7, 134.4, 133.9, 128.9, 128.9, 124.4, 124.1, 118.5, 110.0, 47.6, 19.2; **HRESI-MS** (*m/z*): Calculated for $C_{16}H_{13}NO_2S$ (M + Na): 306.0565, found (M + Na): 306.0567.



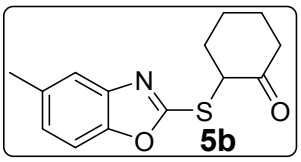
3r

1-Phenyl-2-((5-phenylbenzo[d]oxazol-2-yl)thio)propan-1-one (3r): Prepared as described in the general experimental procedure - Method B: Pale yellow liquid; **Yield:** 90% (81 mg); R_f (5% EtOAc/hexane) 0.2; **IR** (Neat, cm^{-1}): 1682, 1497, 1459, 1255, 1191, 1104, 1016; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.10 (d, *J* = 7.6 Hz, 2H), 7.80 (s, 1H), 7.63-7.58 (m, 3H), 7.52-7.43 (m, 6H), 7.36 (t, *J* = 7.4 Hz, 1H), 5.74 (q, *J* = 7.2 Hz, 1H), 1.84 (d, *J* = 6.8 Hz, 3H) ; **^{13}C NMR** (100 MHz, $CDCl_3$): δ 197.0, 164.4, 151.5, 142.3, 140.9, 138.3, 134.4, 133.9, 128.9, 128.8, 127.4, 127.3, 123.6, 117.0, 110.0, 47.7, 19.2; **HRESI-MS** (*m/z*): Calculated for $C_{22}H_{17}NO_2S$ (M + Na): 382.0878, found (M + Na): 382.0879.



5a

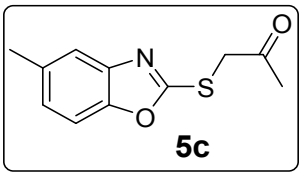
2-((5-methylbenzo[d]oxazol-2-yl)thio)pentan-3-one (5a): Prepared as described in the general experimental procedure - Method A: Colourless liquid; **Yield:** 92% (57 mg); R_f (5% EtOAc/hexane) 0.5; **IR** (Neat, cm^{-1}): 1717, 1500, 1256, 1150; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.38 (s, 1 H), 7.30 (d, *J*=8.4 Hz, 1 H), 7.05 (d, *J*=8.0 Hz, 1 H), 4.65 (q, *J*=7.2 Hz, 1 H), 2.82 (dq, J_1=18.2, J_2 = 7.4 Hz, 1 H), 2.685 (dq, J_1=18.0, J_2 = 7.2 Hz, 1 H), 2.44 (s, 3 H), 1.64 (d, *J*=7.2 Hz, 3 H), 1.12 (t, *J*=7.6 Hz, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 207.65, 163.23, 150.17, 141.83, 134.20, 125.09, 118.57, 109.31, 50.02, 33.32, 21.37, 17.20, 7.90; **HRESI-MS** (*m/z*): Calculated for $C_{13}H_{15}NO_2S$ (M + Na): 272.0721, found (M + Na): 272.0719.



5b

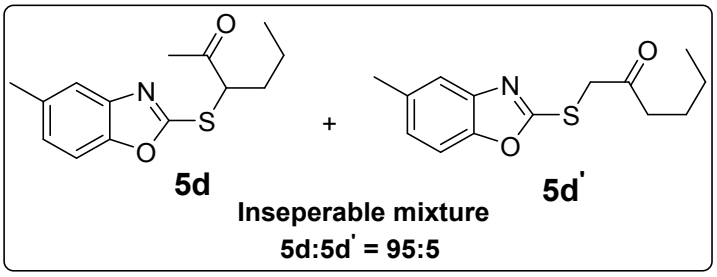
2-((5-methylbenzo[d]oxazol-2-yl)thio)cyclohexan-1-one (5b): Prepared as described in the general experimental procedure - Method A: yellow liquid; **Yield:** 64% (42 mg); R_f (5% EtOAc/hexane) 0.2; **IR** (Neat, cm^{-1}): 1780, 1714, 1499, 1256, 1150; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.36 (s, 1 H) , 7.29 (d, *J*=8.4 Hz, 1 H), 7.03 (d, *J*=8.4 Hz, 1 H), 4.755 (dd, J_1 = 10.8

Hz, J_2 = 5.6 Hz, 1 H), 2.80 - 2.75 (m, 1 H), 2.70 - 2.66 (m, 1 H), 2.52 (td, J_1 = 13.0 Hz, J_2 = 6.0 Hz, 1 H), 2.43 (s, 3 H), 2.19 - 2.14 (m, 1 H), 2.00 - 1.96 (m, 1 H), 1.93-1.88 (m, 2 H), 1.85-1.77 (m, 1 H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 205.09, 163.49, 149.97, 141.92, 134.03, 124.87, 118.43, 109.23, 56.93, 41.62, 35.69, 27.55, 25.18, 21.37; **HRESI-MS** (*m/z*): Calculated for $C_{14}H_{15}NO_2S$ (M + H): 284.0721, found (M + H): 284.0719.



5c

1-((5-methylbenzo[d]oxazol-2-yl)thio)propan-2-one (5c): Prepared as described in the typical experimental procedure for synthesis of **5c** - brown solid; 73-75 ºC; **mp:** 73-75 ºC; **Yield:** 60% (33 mg); R_f (5% EtOAc/hexane) 0.1; **IR** (Neat, cm/$^{-1}$): 1773, 1715, 1500, 1355, 1251, 1145, 1100, 803; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.36 (s, 1 H), 7.30 (d, *J*=8.4 Hz, 1 H), 7.05 (d, *J*=8.4 Hz, 1 H), 4.21 (s, 2 H), 2.43 (s, 3 H), 2.39 (s, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 201.04, 163.46, 150.33, 141.78, 134.23, 125.05, 118.51, 109.33, 42.67, 28.79, 21.38; **HRESI-MS** (*m/z*): Calculated for $C_{11}H_{11}NO_2S$ (M + Na): 244.0408, found (M + Na): 244.0411.

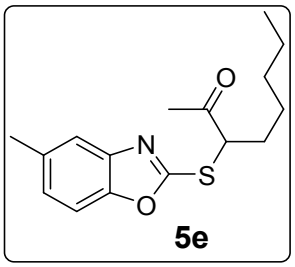


5d **5d'**

Inseperable mixture

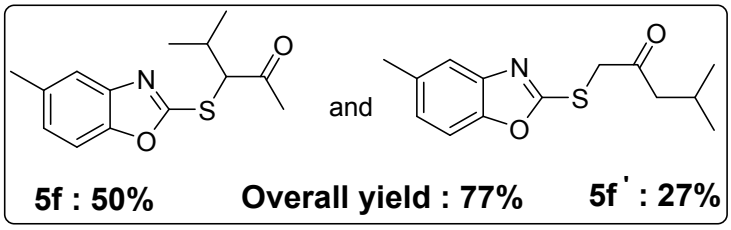
5d:5d' = 95:5

Mixture of 3-((5-methylbenzo[d]oxazol-2-yl)thio)hexan-2-one (5d) and 1-((5-methylbenzo[d]oxazol-2-yl)thio)hexan-2-one (5d'): Prepared as described in the general experimental procedure - Method A: Colourless liquid; **Over all yield:** 94% (60 mg, 5d:5d′ = 5:95); R_f (5% EtOAc/hexane) 0.3; **IR** (Neat, cm^{-1}): 1717, 1500, 1256, 1150, 1101, 11047.37 (s, 1 H), 7.30 (d, *J*=8.4 Hz, 1 H), 7.05 (d, *J*=8.0 Hz, 1 H), 4.572 (t, *J*=7.0 Hz, 1 H), 4.20 (s, 0.11 H), 2.43 (s, 3 H), 2.38 (s, 3 H), 2.05-1.96 (m, 1 H), 1.89 - 1.80 (m, 1 H), 0.97 (t, *J*=7.2 Hz, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 204.76, 163.09, 150.18, 141.79, 134.19, 125.10, 118.61, 109.28, 55.47, 32.57, 27.84, 21.34, 20.13, 13.66; **HRESI-MS** (*m/z*): Calculated for $C_{14}H_{17}NO_2S$ (M + Na): 286.0878, found (M + Na): 286.0879.



5e

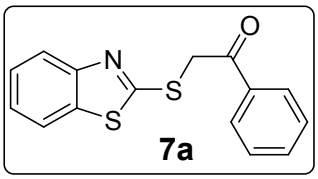
3-((5-methylbenzo[d]oxazol-2-yl)thio)octan-2-one (5e): Prepared as described in the general experimental procedure - Method A: Colourless liquid; **Yield:** 81% (59 mg); R_f (5% EtOAc/hexane) 0.4; **IR** (Neat, cm^{-1}): 1718, 1500, 1355, 1256, 1150, 1103; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.38 (s, 1 H), 7.30 (d, *J*=8.4 Hz, 1 H), 7.05 (d, *J*=8.4 Hz, 1 H), 4.56 (t, *J*=7.2 Hz, 1 H), 2.43 (s, 3 H), 2.38 (s, 3 H), 2.07-1.98 (m, 1 H), 1.90-1.80 (m, 1 H), 1.52-1.41 (m, 2 H), 1.34 - 1.26 (m, 4 H), 0.88 (t, *J*=6.8 Hz, 4 H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 204.77, 163.12, 150.19, 141.80, 134.18, 125.09, 118.62, 109.28, 55.71, 31.31, 30.52, 27.85, 26.47, 22.27, 21.35, 13.85; **HRESI-MS** (*m/z*): Calculated for $C_{16}H_{21}NO_2S$ (M + Na): 314.1191, found (M + Na): 314.1191.



4-Methyl-3-((5-methylbenzo[d]oxazol-2-yl)thio)pentan-2-one (5f) and 4-methyl-1-((5-methylbenzo[d]oxazol-2-yl)thio)pentan-2-one (5f'): Prepared as described in the general experimental procedure - Method A: The reaction performed in 0.5 mmol scale: Overall yield: 77% (101 mg)

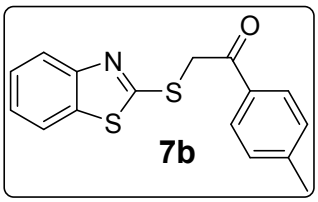
Compound 5f : Colourless liquid; **Yield:** 50% (66 mg); R_f (5% EtOAc/hexane) 0.4; **IR** (Neat, cm^{-1}): 1716, 1500, 1425, 1355, 1256, 1217, 1150, 1104, 1000, 936; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.37 (s, 1 H), 7.30 (d, *J*=8.4 Hz, 1 H), 7.05 (d, *J* = 8.0 Hz, 1 H), 4.51 (d, *J*=6.8 Hz, 1 H), 2.43 (s, 3 H), 2.40 (s, 3 H), 2.43-2.33 (m, 1H), 1.12 (d, *J*=6.8 Hz, 3 H), 1.10 (d, *J*=6.8 Hz, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 205.0, 163.6, 150.2, 141.8, 134.2, 125.1, 118.5, 109.3, 62.6, 29.6, 29.0, 21.4, 20.8, 19.5; **HRESI-MS** (*m/z*): Calculated for $C_{14}H_{17}NO_2S$ (M + Na): 286.0878, found (M + Na): 286.0874.

Compound 5f′: White solid; **mp:** 71-73 ºC (lit.) **Yield:** 27% (35 mg); R_f (5% EtOAc/hexane) 0.25; **IR** (Neat, cm^{-1}): 1719, 1502, 1467, 1360, 1253, 1147, 1101, 1034; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.35 (s, 1 H), 7.29 (d, *J*=8.4 Hz, 1 H), 7.04 (dd, J_1 = 8.4 Hz, J_2 = 0.8 Hz, 1 H), 4.19 (s, 2 H), 2.54 (d, *J*=6.8 Hz, 2 H), 2.43 (s, 3 H), 2.29-2.18 (m, 1 H), 0.96 (d, *J*=6.8 Hz, 6 H)xv; **^{13}C NMR** (100 MHz, $CDCl_3$): δ 202.87, 163.64, 150.32, 141.87, 134.19, 124.98, 118.48, 109.31, 50.51, 42.67, 24.75, 22.49, 21.40; **HRESI-MS** (*m/z*): Calculated for $C_{14}H_{17}NO_2S$ (M + Na): 286.0878, found (M + Na): 286.0876.



7a

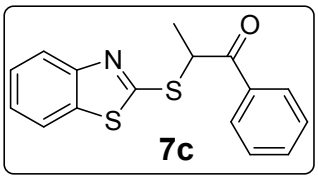
2-(benzo[d]thiazol-2-ylthio)-1-phenylethan-1-one (7a)[3]: Prepared as described in the general experimental procedure - Method C. Pale yellow solid; **Yield:** 85% (121 mg); **mp**: 107-108 ºC (lit.[3] 109-111 ºC); R_f (5% EtOAc/hexane) 0.2; **IR** (Neat, cm^{-1}): 1674, 1449, 1419, 1376, 1316, 1285, 1186, 994, 750; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.08 (d, *J*=7.6 Hz, 2 H), 7.81 (d, *J*=8.0 Hz, 1 H), 7.74 (d, *J*=8.0 Hz, 1 H), 7.62 (t, *J*=7.2 Hz, 1 H), 7.39 (t, *J*=7.6 Hz, 1 H), 7.29 (t, *J*=7.4 Hz, 1 H), 4.97 (s, 2 H **^{13}C NMR** (100 MHz, $CDCl_3$): δ 193.0, 165.2, 152.8, 135.5 (2C: ipso carbon of phenyl ring and ipso carbon attached to sulfur atom), 133.8, 128.8, 128.6, 126.0, 124.4, 121.5, 121.0, 41.0; **HRESI-MS** (*m/z*): Calculated for $C_{15}H_{11}NS_2O$ (M + Na): 308.0180, found (M + Na): 308.0175.



7b

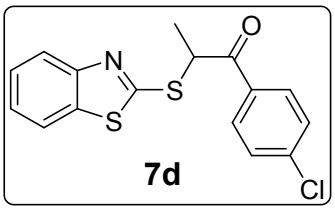
2-(benzo[d]thiazol-2-ylthio)-1-(p-tolyl)ethan-1-one (7b)[2a]: Prepared as described in the general experimental procedure - Method C: White solid; **mp**: 88-90 ºC (lit.[4] 94-95 ºC); **Yield:** 65% (97 mg); R_f (5% EtOAc/hexane) 0.2; **IR** (KBr, cm^{-1}): 1678, 1601, 1510, 1459, 1424, 993; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.97 (d, *J*=8.0 Hz, 2 H), 7.82 (d, *J*=8.0 Hz, 1 H), 7.74 (d, *J*=8.0 Hz, 1 H), 7.40 (t, *J*=7.6 Hz, 1 H), 7.30 (d, *J*=8.0 Hz, 2 H), 7.29 (t, *J*=8.4 Hz, 1 H), 4.97; **^{13}C NMR** (100 MHz, $CDCl_3$): δ 192.4, 165.3, 152.9, 144.8, 135.5, 132.9, 129.4,

128.6, 126.0, 124.3, 121.4, 121.0, 40.9, 21.7; **HRESI-MS** (*m/z*): Calculated for $C_{16}H_{13}NOS_2$ (M + Na): 322.0336, found (M + Na): 322.0338.



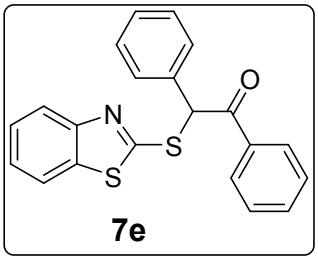
7c

2-(Benzo[d]thiazol-2-ylthio)-1-phenylpropan-1-one (7c): Prepared as described in the general experimental procedure - Method C: Colourless liquid; **Yield**: 67% (100 mg); R_f (5% EtOAc/hexane) 0.6; **IR** (Neat, cm^{-1}): 1682, 1594, 1455, 1426, 1308, 1238, 1201, 1077, 995; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.09 (d, *J*=7.6 Hz, 2 H), 7.82 (d, *J* = 7.6 Hz, 1 H), 7.73 (dd, , J_1 = 8.0 Hz, J_2 = 0.4 Hz, 1 H), 7.57 (t, *J* = 7.4 Hz, 1 H), 7.46 (t, *J* = 7.6 Hz, 2 H), 7.40 (t, *J* = 7.4 Hz, 2 H), 7.28 (t, *J* = 7.6 Hz, 1 H), 5.87 (q, *J* = 7.12 Hz, 1 H), 1.77 (d, *J* = 7.02 Hz, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 197.1, 164.6, 152.8, 135.6, 134.9, 133.5, 128.8, 128.7, 126.0, 124.4, 121.5, 121.0, 47.0, 18.3; **HRESI-MS** (*m/z*): Calculated for $C_{16}H_{13}NOS_2$ (M + Na): 322.0336, found (M + Na): 322.0338.



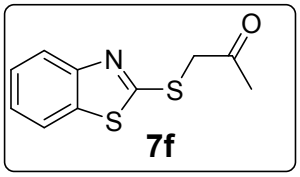
7d

2-(benzo[d]thiazol-2-ylthio)-1-(4-chlorophenyl)propan-1-one (7d): Prepared as described in the general experimental procedure - Method C: Colourless liquid; **Yield:** 73% (122 mg); R_f (5% EtOAc/hexane) 0.5; **IR** (Neat, cm^{-1}): 1683, 1588, 1457, 1426, 1238, 1201, 993; **^{1}H NMR** (400 MHz, $CDCl_3$): 8.05 (d, *J* = 8.4 Hz, 2 H), 7.82 (d, *J* = 8.0 Hz, 2 H), 7.75 (d, *J* = 7.6 Hz, 1 H), 7.44 - 7.40 (m, 3 H), 7.30 (t, *J* = 7.6 Hz, 1 H), 5.82 (q, *J* = 7.2 Hz, 1 H), 1.75 (d, *J* = 6.8 Hz, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 196.0, 164.4, 152.7, 140.1, 135.7, 133.4, 130.2, 129.0, 126.1, 124.6, 121.6, 121.1, 46.6, 18.0; **HRESI-MS** (*m/z*): Calculated for $C_{16}H_{12}ClNOS_2$ (M + Na): 355.9947, found (M + Na): 355.9945.



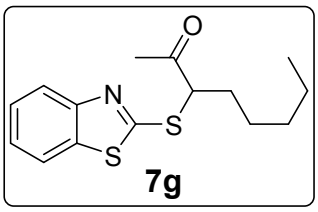
7e

2-(benzo[d]thiazol-2-ylthio)-1,2-diphenylethan-1-one (7e): Prepared as described in the general experimental procedure - Method C: White Solid; **mp:** 106-108 ºC; **Yield:** 72% (130 mg); R_f (5% EtOAc/hexane) 0.6; **IR** (Neat, cm^{-1}): 1684, 1586, 1452, 1420, 1268, 1199, 1074, 994; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.06 (d, *J* = 8.0 Hz, 2 H), 7.71 (d, *J* = 8.0 Hz, 1 H), 7.67 (d, *J* = 8.0 Hz, 1 H), 7.55 (d, *J* = 7.6 Hz, 2 H), 7.51 (t, *J* = 7.4 Hz, 1 H), 7.41 (t, *J* = 7.0Hz, 2 H), 7.39-7.28 (m, 3 H), 7.26 - 7.21 (m, 2 H), 6.95 (s, 1 H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 194.2, 164.7, 152.7, 135.5, 135.5, 134.6, 133.3, 129.1, 129.02, 129.0, 128.6, 128.6, 125.8, 124.3, 121.4, 121.00, 58.1; **HRESI-MS** (*m/z*): Calculated for $C_{21}H_{15}NOS_2$ (M + Na): 384.0493, found (M + Na): 384.0493.



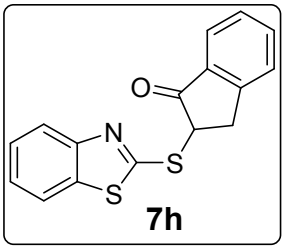
7f

1-(benzo[d]thiazol-2-ylthio)propan-2-one (7f)[5]: Prepared as described in the typical experimental procedure for synthesis of **7f** - Method E: brown Solid; 68-70 ºC (lit.[5] 67-68 ºC) **Yield:** 68% (76 mg); R_f (5% EtOAc/hexane) 0.1; **IR** (Neat, cm^{-1}): 1718, 1459, 1420, 1351, 1151, 992; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.83 (d, *J* = 8.4 Hz, 1 H), 7.74 (d, *J* = 8.0 Hz, 1 H), 7.41 (t, *J*=7.8 Hz, 1 H), 7.29 (t, *J*=7.6 Hz, 1 H), 4.23 (s, 2 H), 2.39 (s, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 201.7, 164.9, 152.7, 135.5, 126.1, 124.4, 121.5, 121.1, 43.0, 28.8; **HRESI-MS** (*m/z*): Calculated for $C_{10}H_9NOS_2$ (M + Na): 246.0023, found (M + Na): 246.0027.



7g

3-(benzo[d]thiazol-2-ylthio)octan-2-one (7g): Prepared as described in the general experimental procedure - Method C: Pale yellow liquid; **Yield:** 82% (120 mg); R_f (5% EtOAc/hexane) 0.5; **IR** (Neat, cm^{-1}): 1714, 1623, 1460, 1426, 1354, 1196, 1180, 995; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.85 (d, *J*=8.0 Hz, 1 H), 7.75 (d, *J*=8.0 Hz, 1 H), 7 .41 (t, *J*=8.0 Hz, 1 H), 7.30 (t, *J*=7.6 Hz, 1 H), 4.69 (t, *J*=7.2 Hz, 1 H), 2.39 (s, 3 H), 2.06-1.96 (m, 1 H), 1.88 - 1.79 (m, 1 H), 1.56-1.31 (m, 2 H), 1.34 - 1.31 (m, 4 H), 0.90-0.87 (m, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 205.2, 164.7, 152.9, 135.6, 126.1, 124.5, 121.6, 121.0, 56.3, 31.4, 30.4, 27.9, 26.70, 22.3, 13.90; **HRESI-MS** (*m/z*): Calculated for $C_{15}H_{19}NOS_2$ (M + Na): 316.806, found (M + Na): 316.814.



7h

2-(benzo[d]thiazol-2-ylthio)-2,3-dihydro-1H-inden-1-one (7h): Prepared as described in the general experimental procedure - Method C: Pale yellow liquid; **Yield:** 69% (102 mg); R_f (10% EtOAc/hexane) 0.3; **IR** (Neat, cm^{-1}): 1718, 1607, 1461, 1426, 1237, 1238, 1207, 995; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.85 (d, *J* = 8.0 Hz, 1 H), 7.73 (dd, *J* = 8.0, 0.4 Hz, 1 H), 7.68 - 7.63 (m, 2 H), 7.48 - 7.42 (m, 2 H), 7.35 (t, *J* = 8.0 Hz, 1 H), 7.29 - 7.25 (m, 1 H), 4.70 (dd, J_1 = 8.0, J_2 = 4.8 Hz, 1 H), 3.94 (dd, J_1 = 17.60, J_2 = 8.0 Hz, 1 H), 3.42 (dd, J_1 = 17.70, J_2 = 4.8 Hz, 1 H) ; **^{13}C NMR** (100 MHz, $CDCl_3$): δ 200.6, 164.0, 152.8, 152.1, 135.8, 135.6, 135.4, 127.8, 126.3, 125.9, 124.4, 124.4, 121.6, 121.0, 50.2, 35.6; **HRESI-MS** (*m/z*): Calculated for $C_{16}H_{11}NOS_2$ (M + Na): 320.0180, found (M + Na): 320.0180.

References

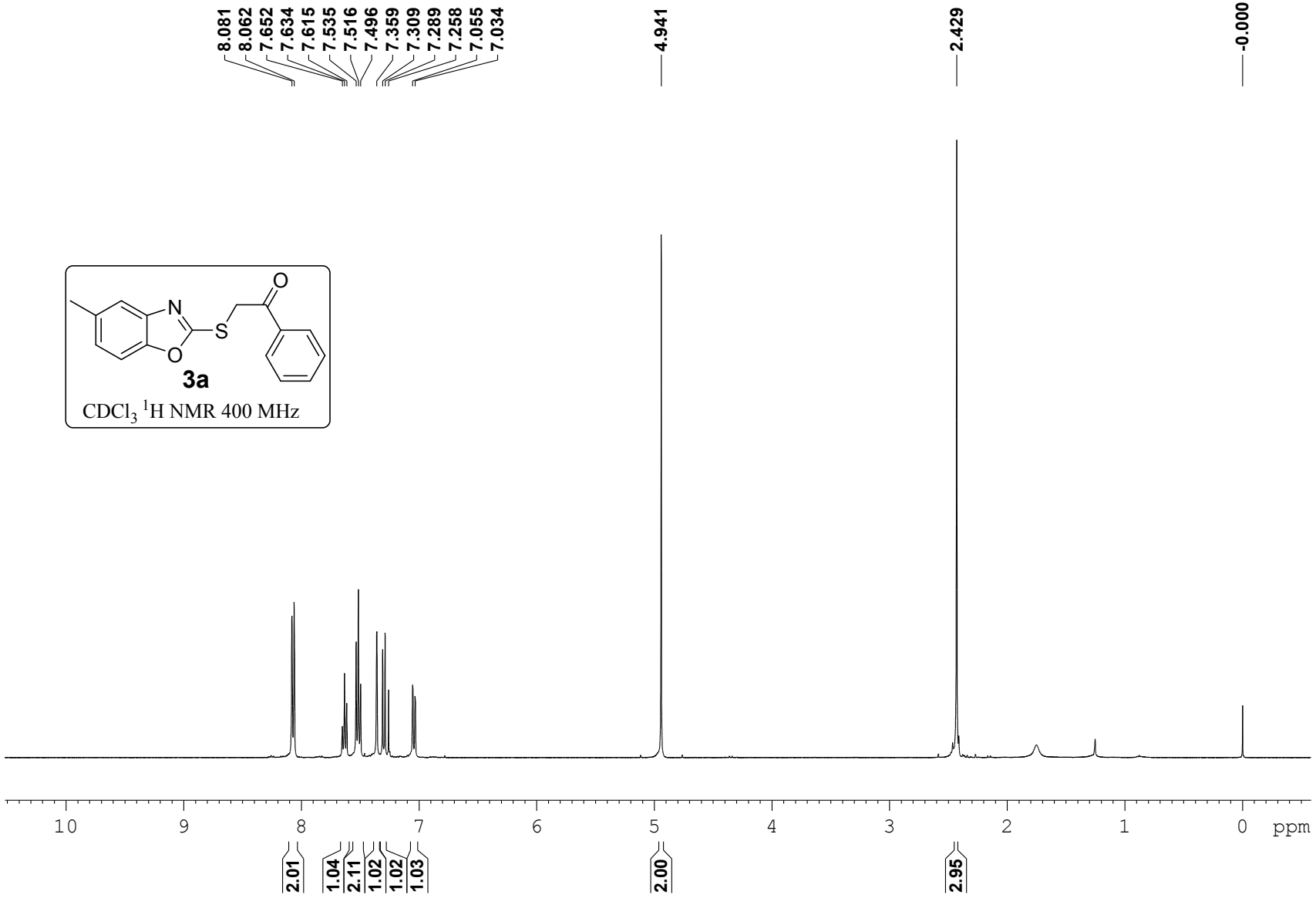
(1) (a) B. V. Varun and K. R. Prabhu, *J. Org. Chem.* 2014, **79,** 9655; (b) B. V. Varun, K. Gadde and K. R. Prabhu, *Org. Lett.* 2015, **17,** 2944.

(2) (a) H. Loghmani-Khouzani and D. Hajiheidari *J. Fluorine Chem.,* 2010, **131,** 561; (b) S. N. Dehuri and A. Nayak, *J. Indian Chem. Soc.,* 1982, **59,** 1170.

(3) N. Yamada, M. Mizuochi and H. Morita, *Tetrahedron*, 2007, **63,** 3408.

(4) A. R. Katritzky, J. M. Aurrecoechea and L. M. V. Vazquez de Miguel, *J. Chem. Soc. Perkin Trans. I* 1987, 769.

(5) F. Al-Omran and A. A. El-Khair, *J Chem Res* 2009, **7**, 433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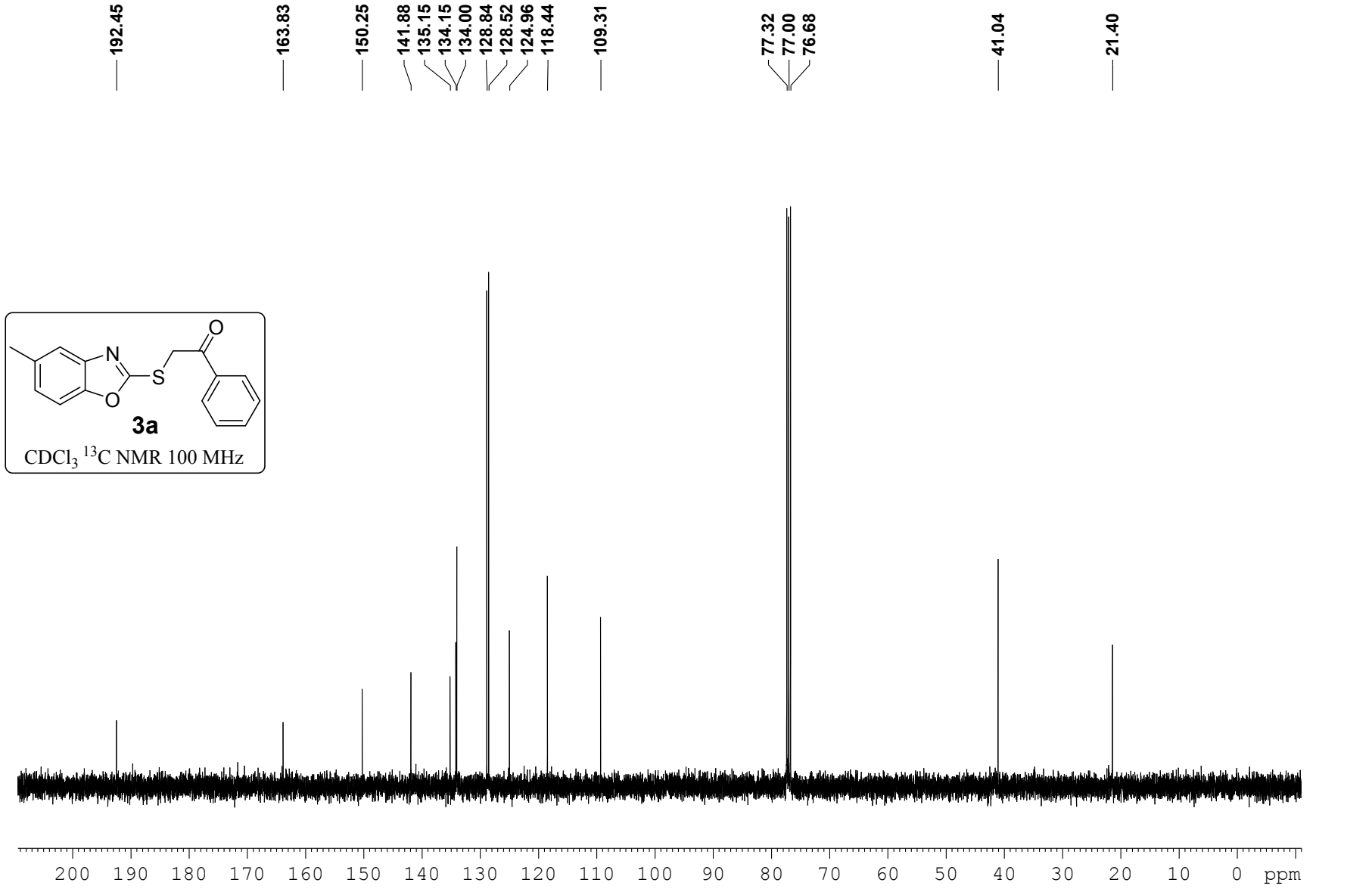
7.652
7.634
7.615
7.535
7.516
7.496
7.359
7.309
7.289
7.258
7.055
7.034
4.941
2.429
-0.000
O
N
S
O
3a
CDCl3 1H NMR 400 MHz
10
9
8
7
6
5
4
3
2
1
0
ppm
2.01
1.04
2.11
1.02
1.02
1.03
2.00
2.95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134.15
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77.32
77.00
76.68
41.04
21.40
O
N
S
O
3a
CDCl3 13C NMR 100 MHz
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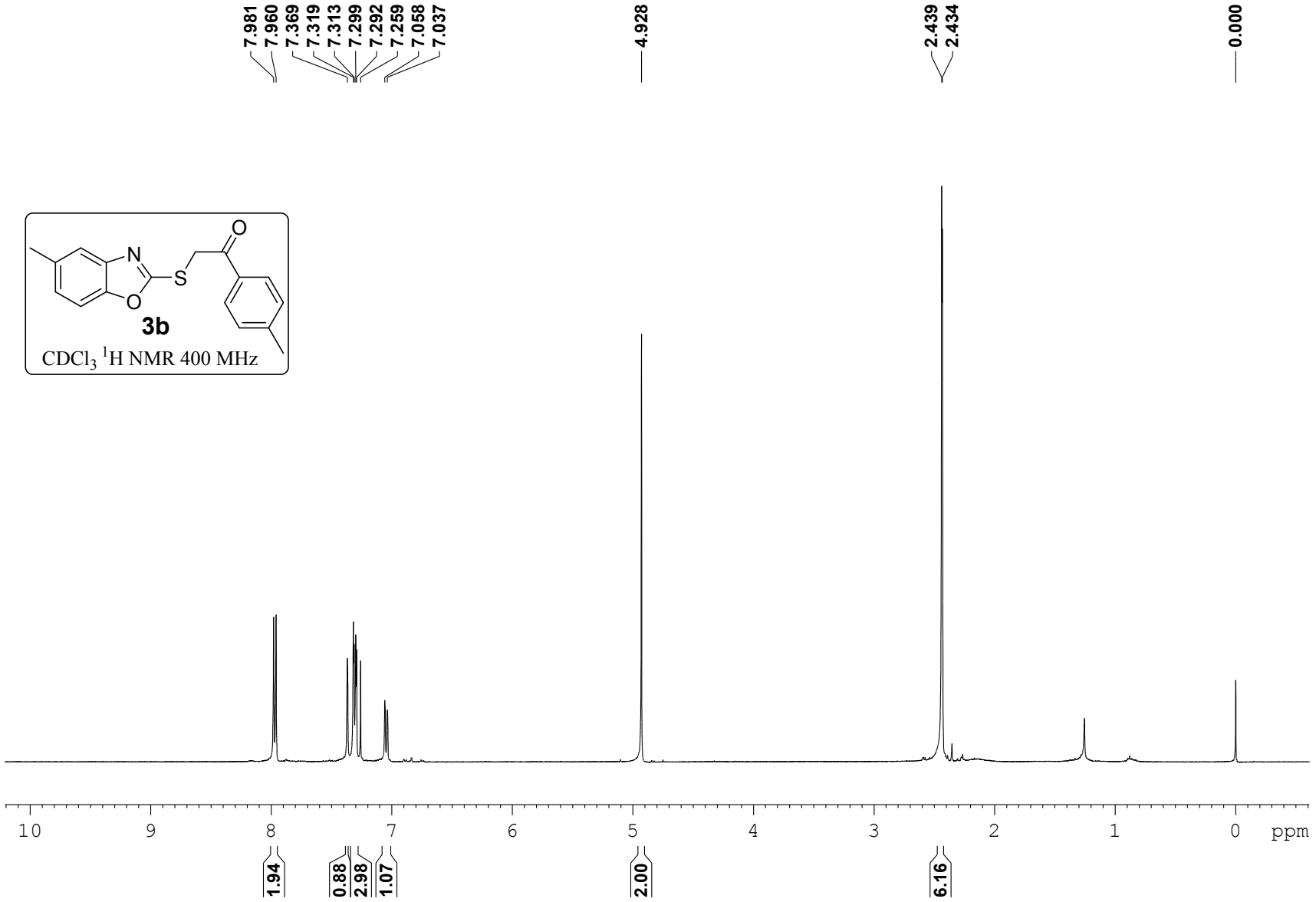
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7.292
7.259
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7.037
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2.434
0.000
O
N
S
O
3b
CDCl3 1H NMR 400 MHz
10
9
8
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4
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0
ppm
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1.07
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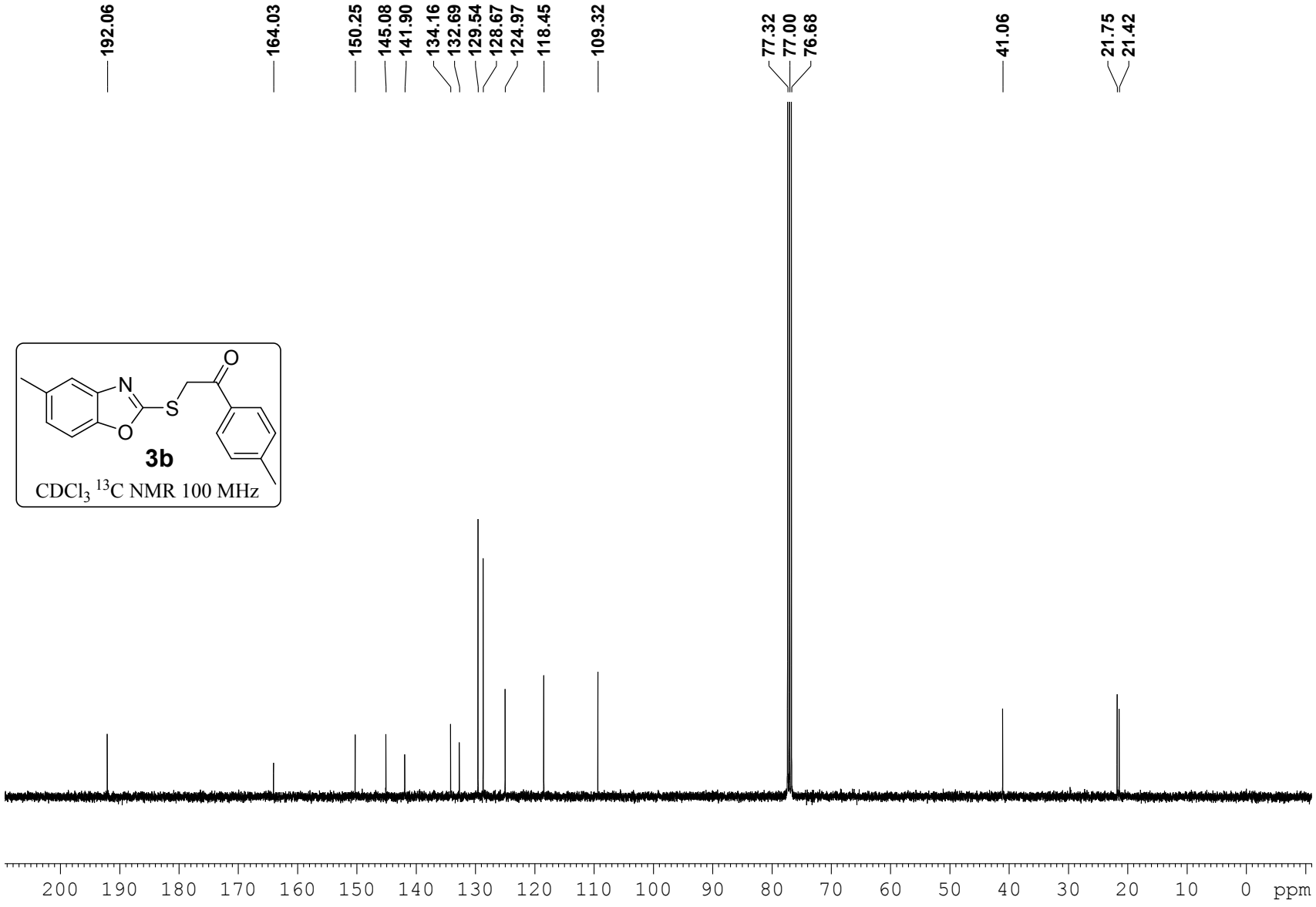
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77.00
76.68
41.06
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N
S
O
3b
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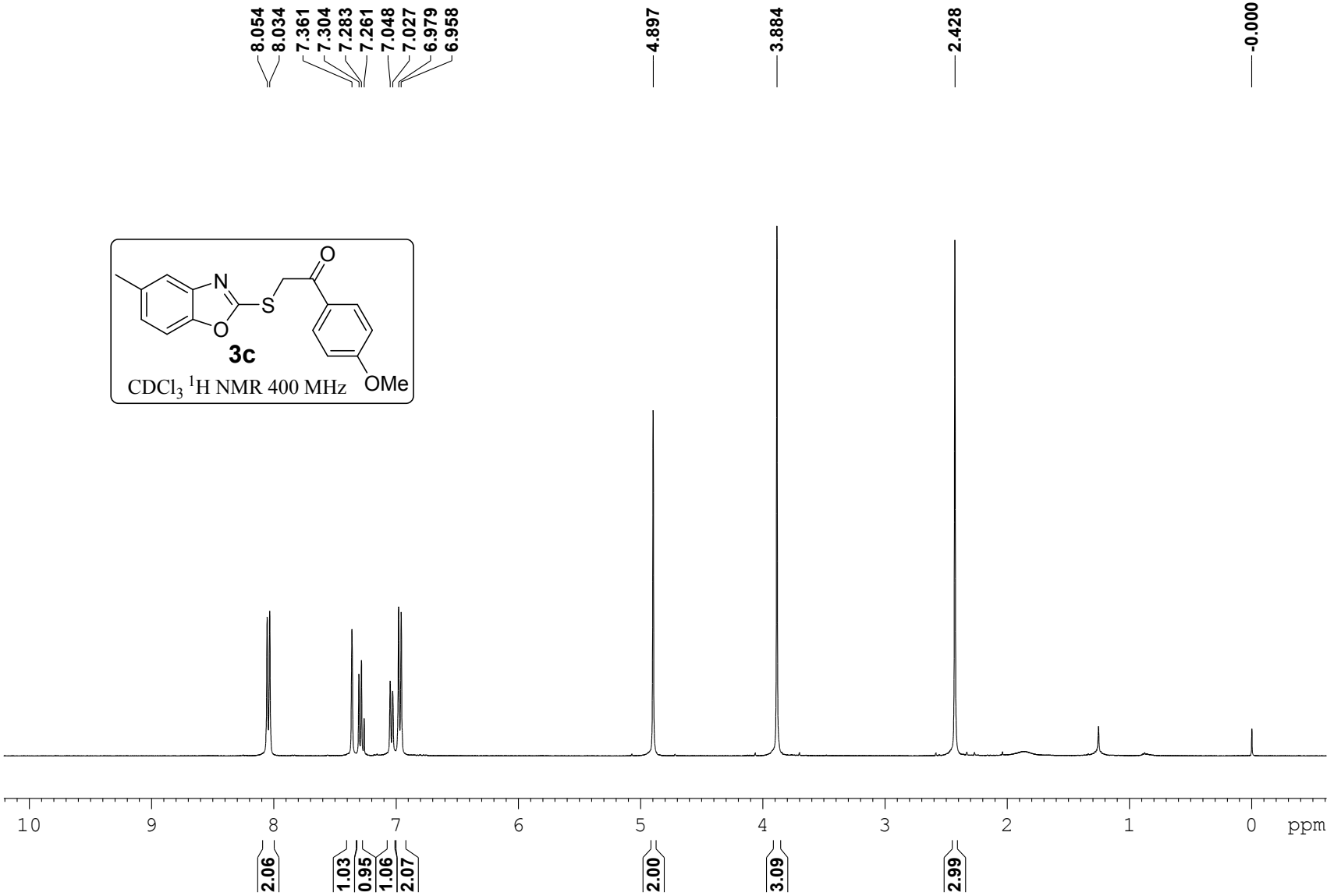
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O
N
S
O
3c
CDCl3 1H NMR 400 MHz
OMe
10
9
8
7
6
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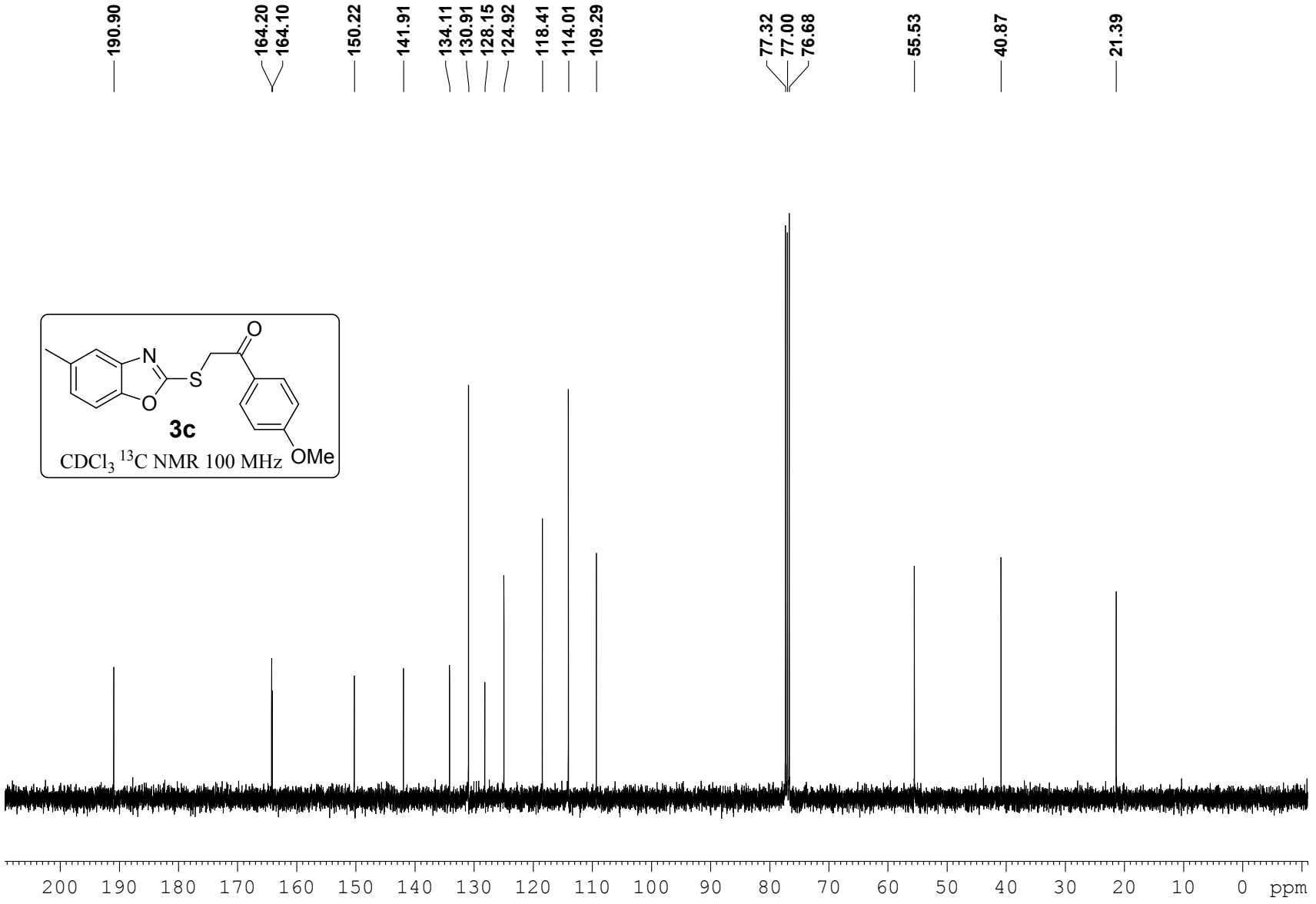
2.99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77.00
76.68
55.53
40.87
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O
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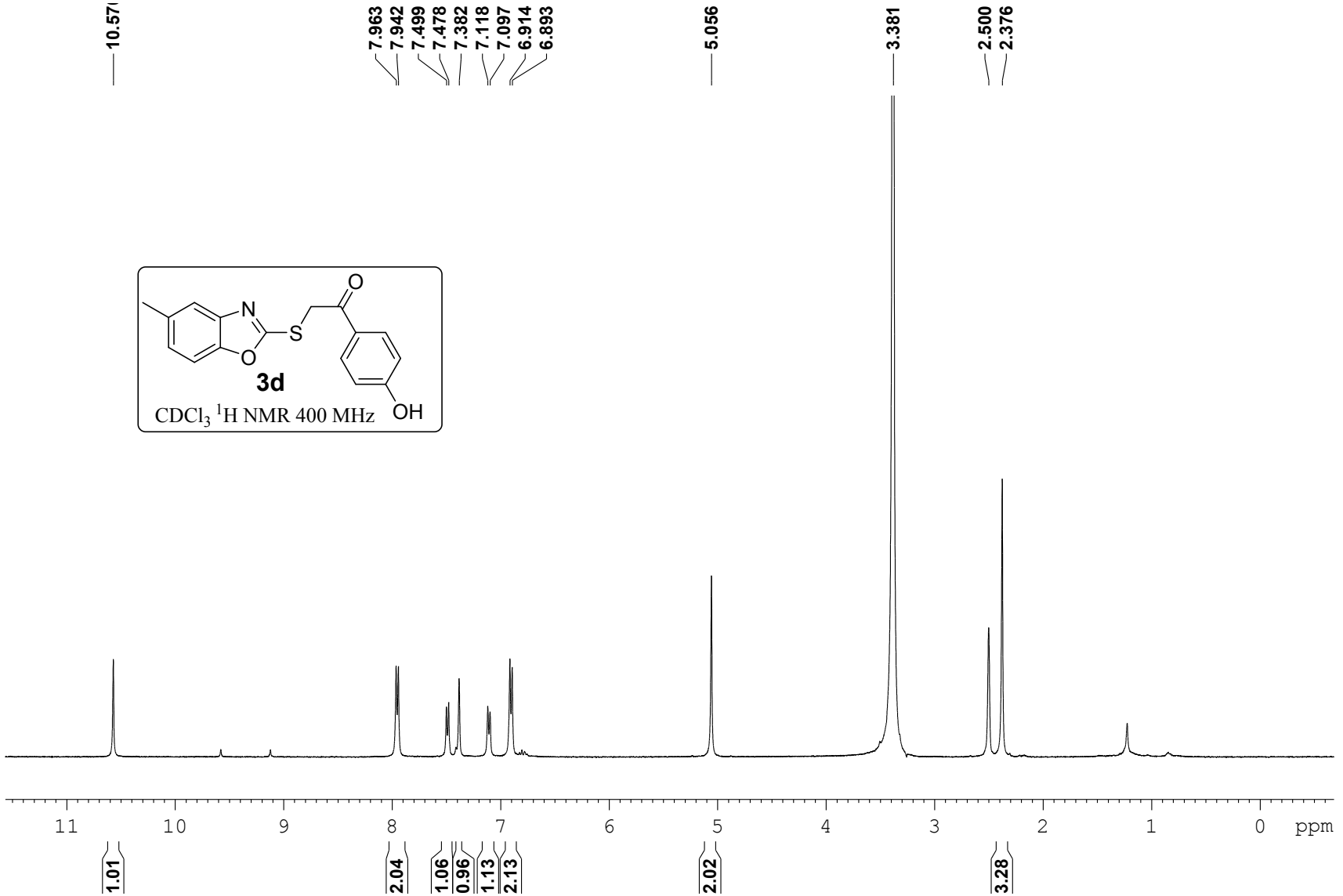
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2.376
O
N
S
O
3d
CDCl3 1H NMR 400 MHz
OH
11
10
9
8
7
6
5
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1
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ppm
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1.06
0.96
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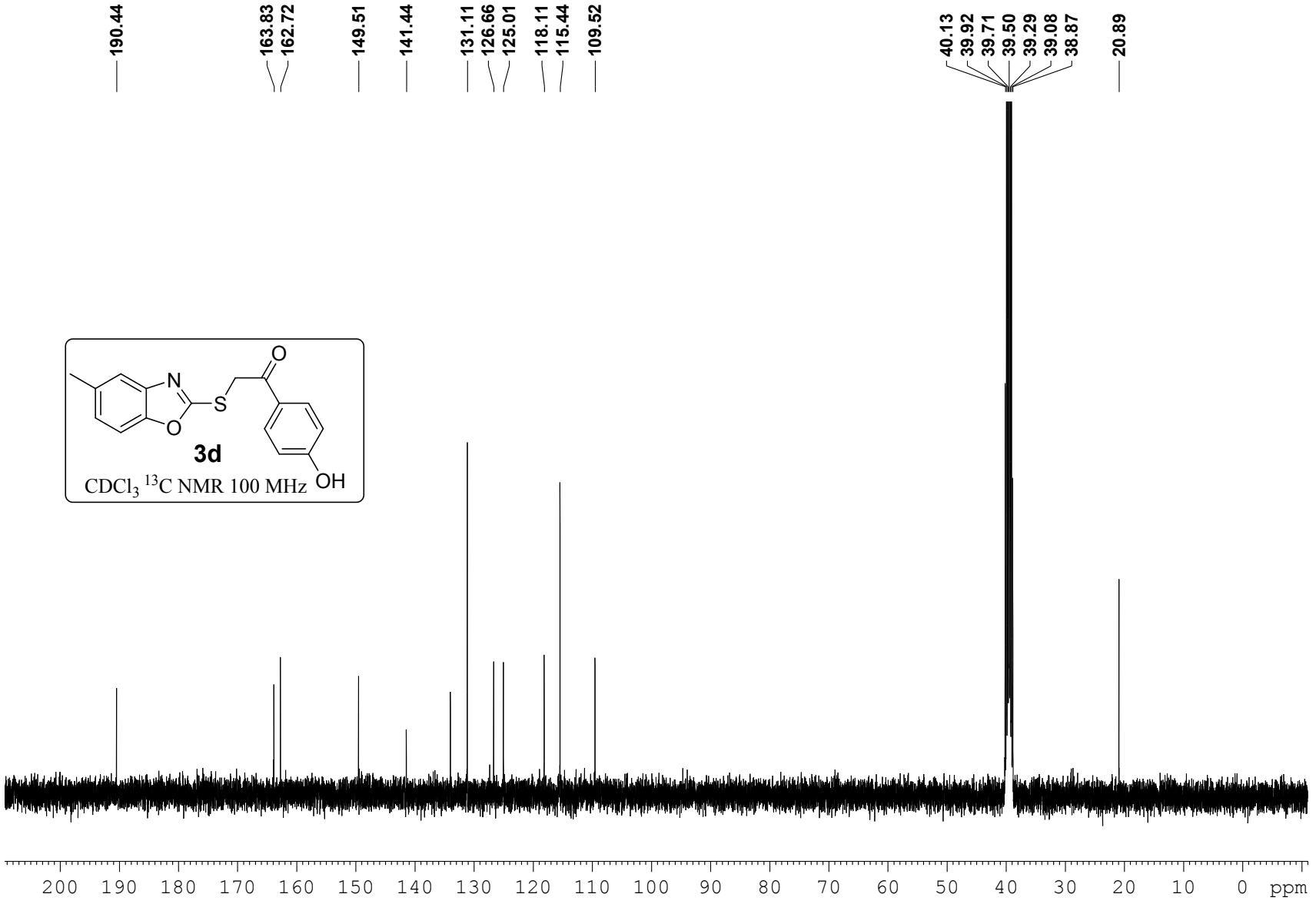
2.13
2.02
3.28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109.52
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39.71
39.50
39.29
39.08
38.87
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N
S
O
3d
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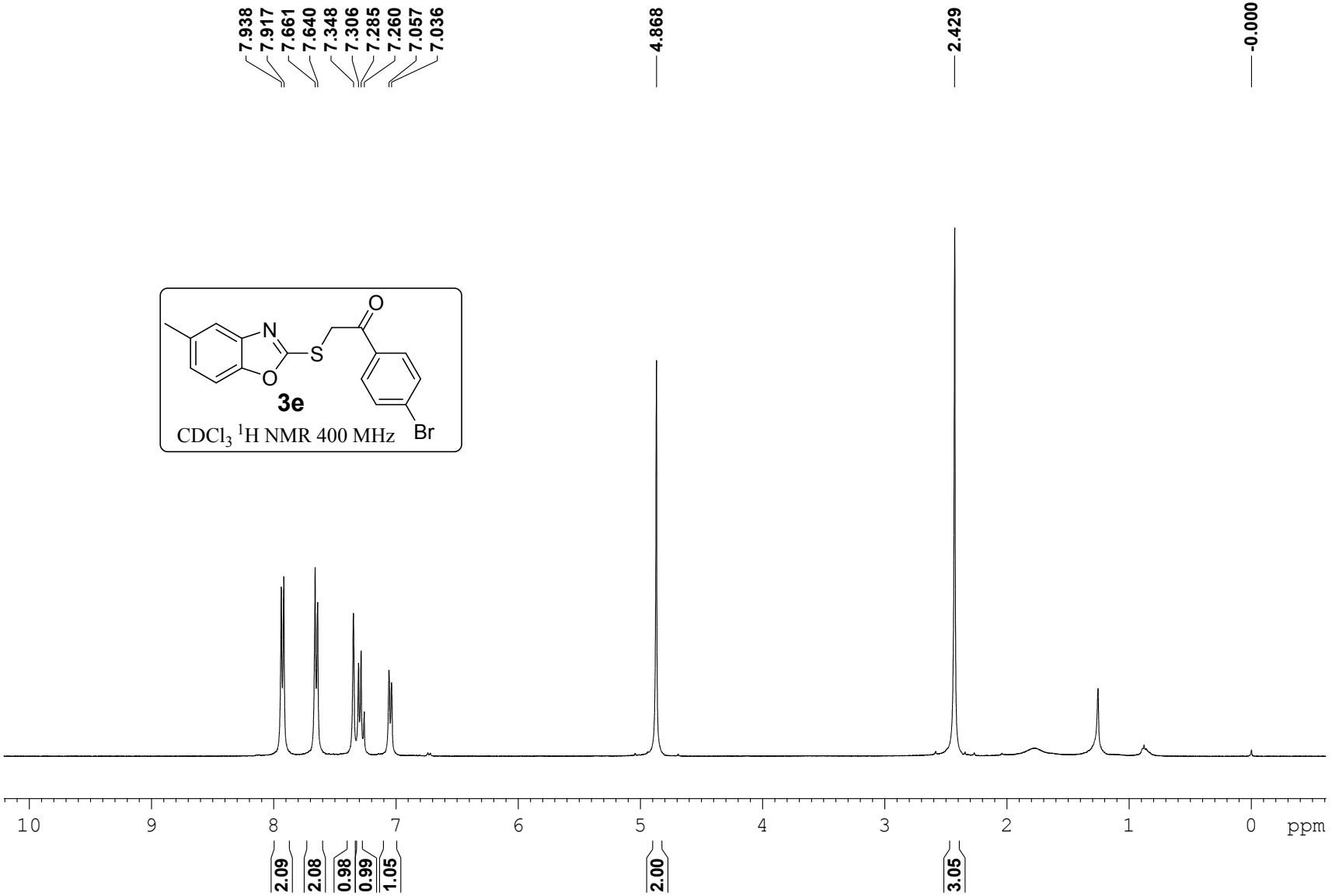
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-0.000
O
N
S
O
3e
CDCl3 1H NMR 400 MHz
Br
10
9
8
7
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2
1
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ppm
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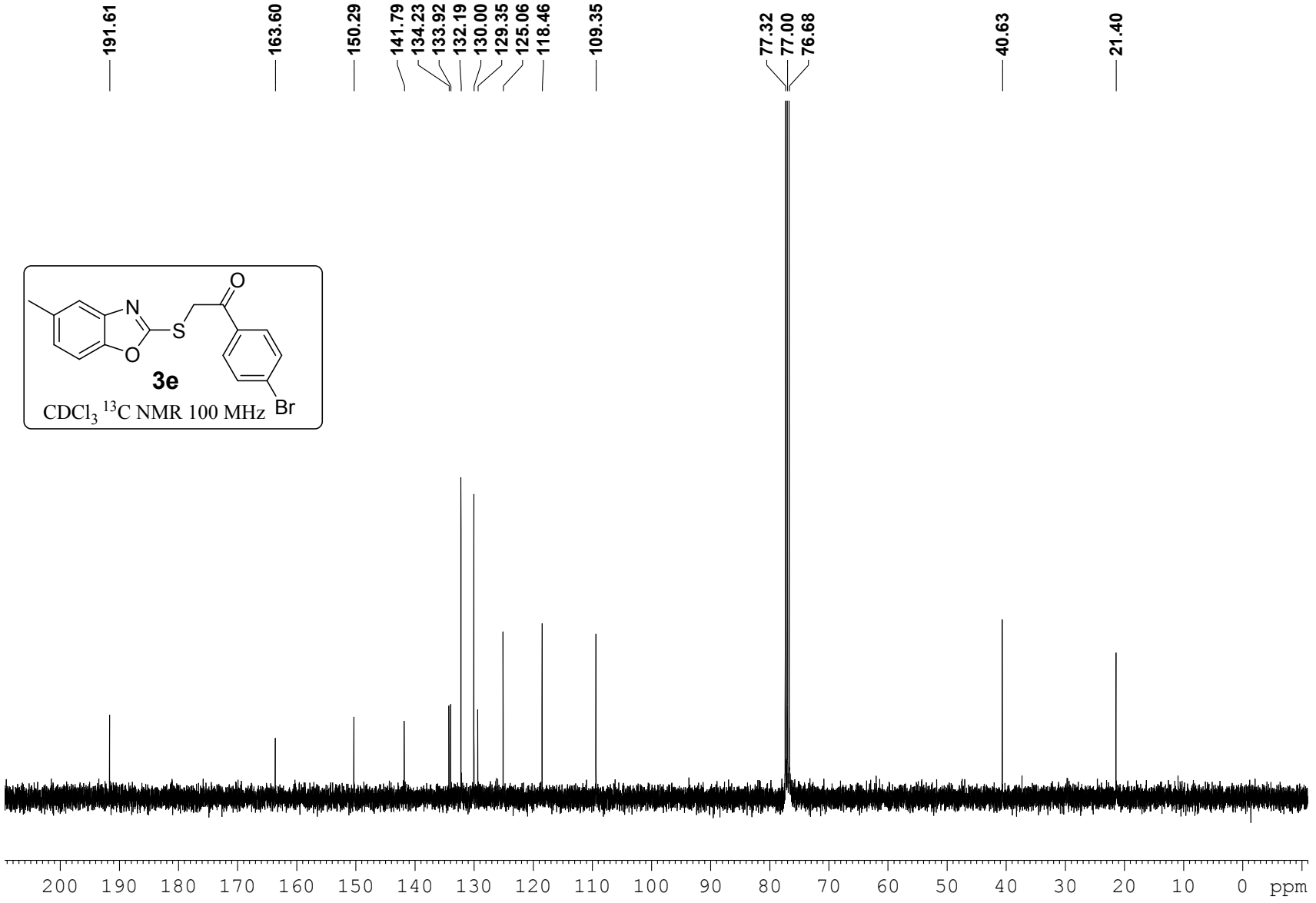
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77.00
76.68
40.63
21.40
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N
S
O
Br
3e
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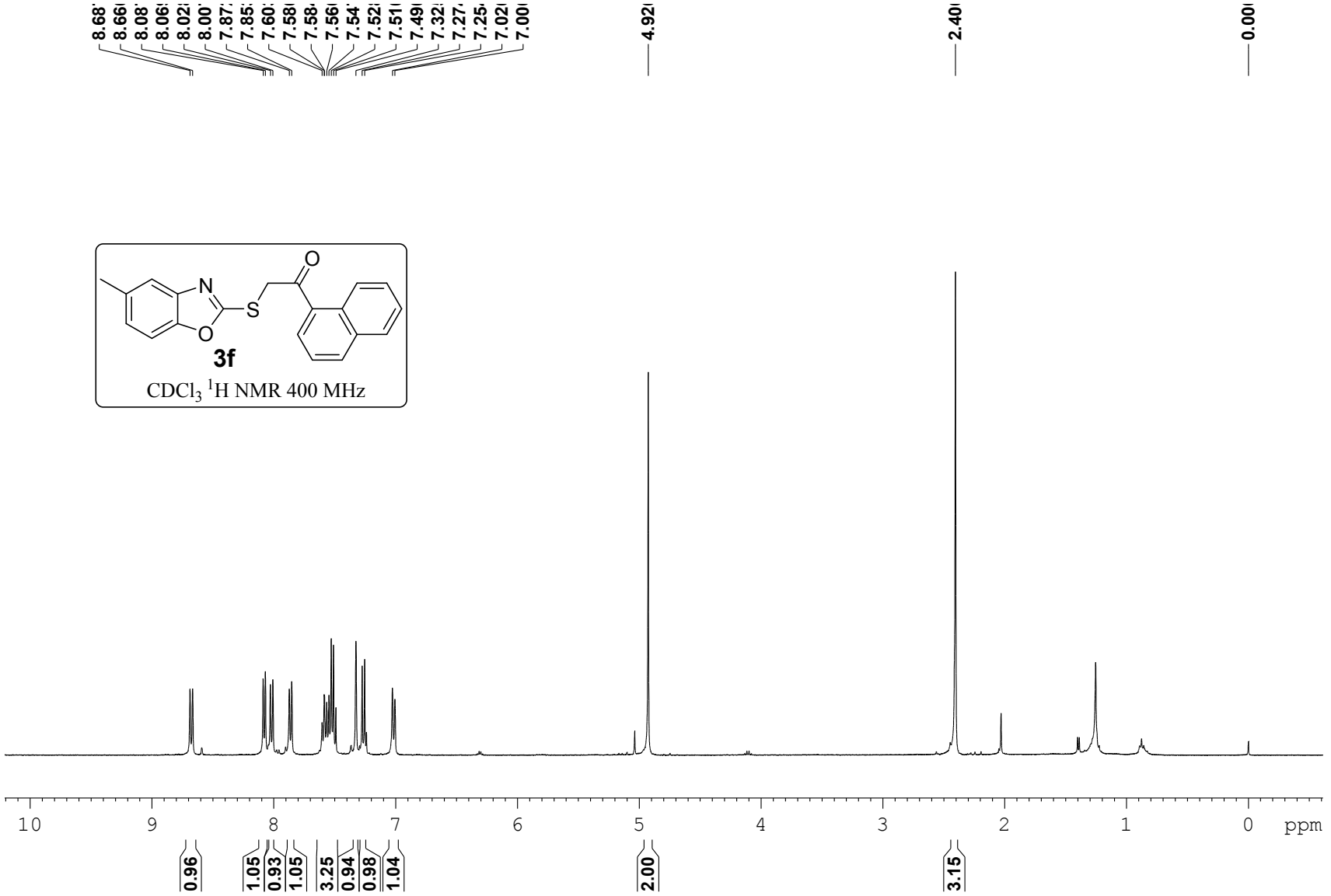
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7.52
7.51
7.49
7.32
7.27
7.25
7.02
7.00
4.92
2.40
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O
N
S
O
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CDCl3 1H NMR 400 MHz
10
9
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7
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5
4
3
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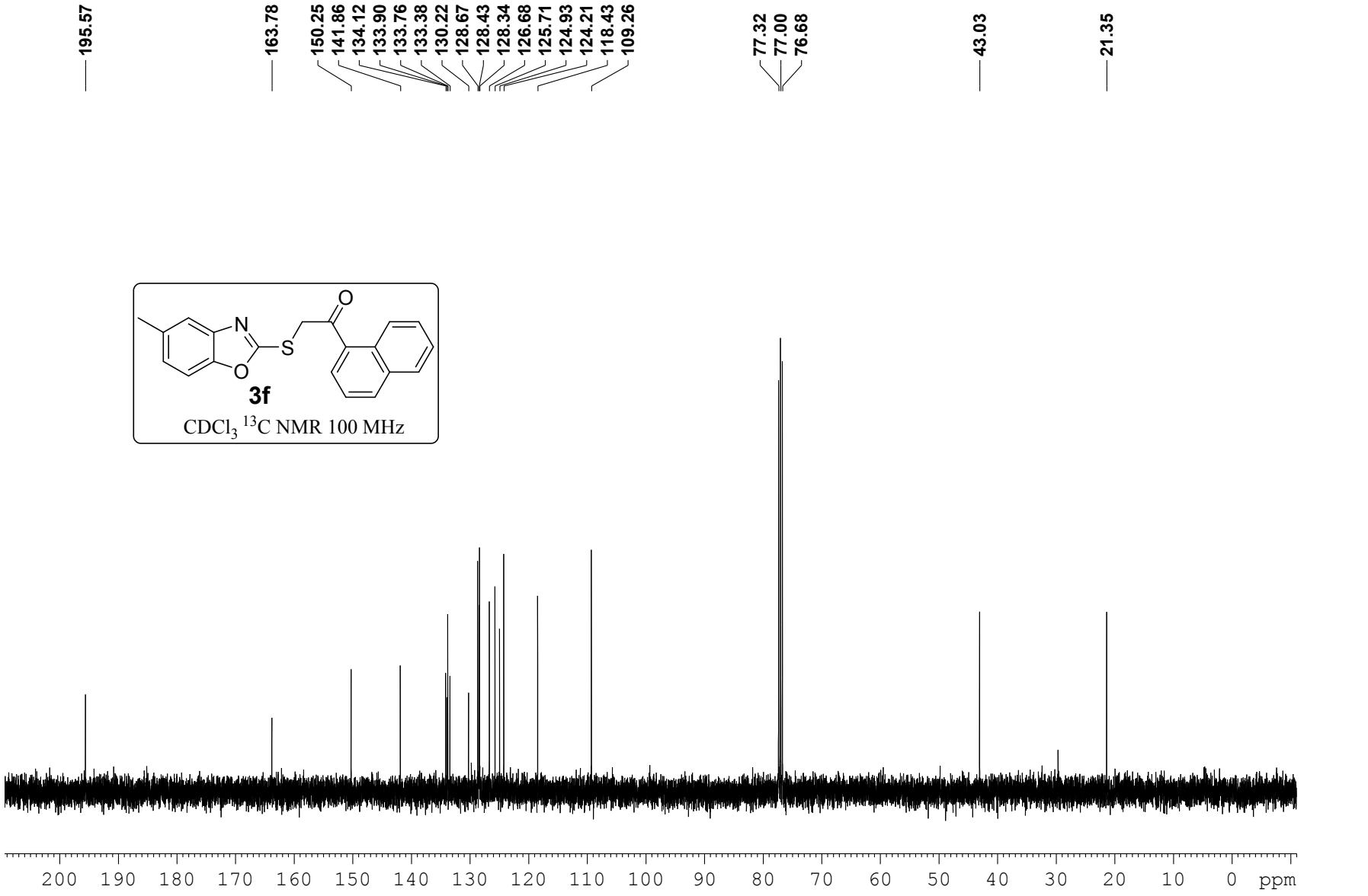
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124.93
124.21
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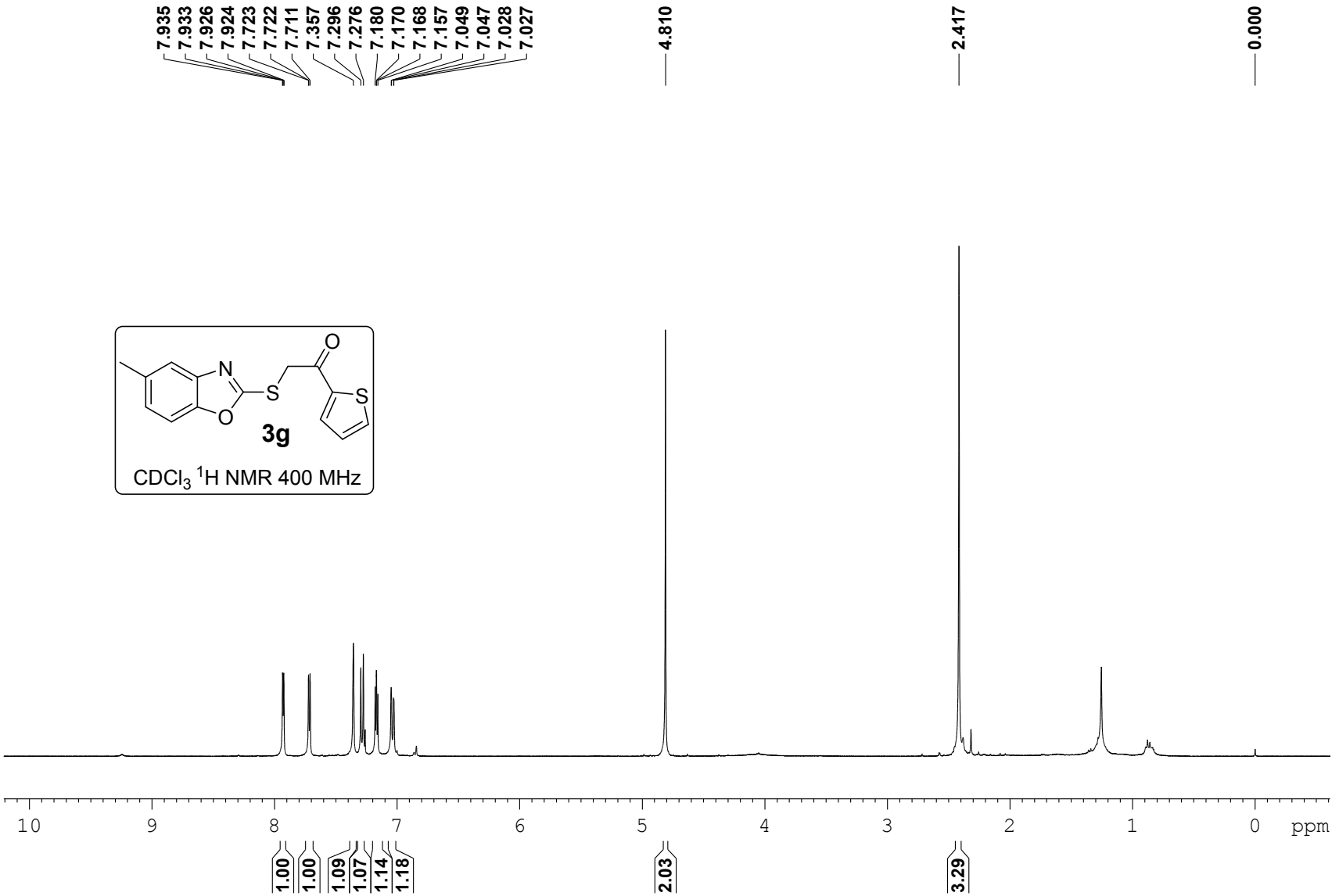
77.32
77.00
76.68
43.03
21.35
O
N
S
O
3f
CDCl3 13C NMR 100 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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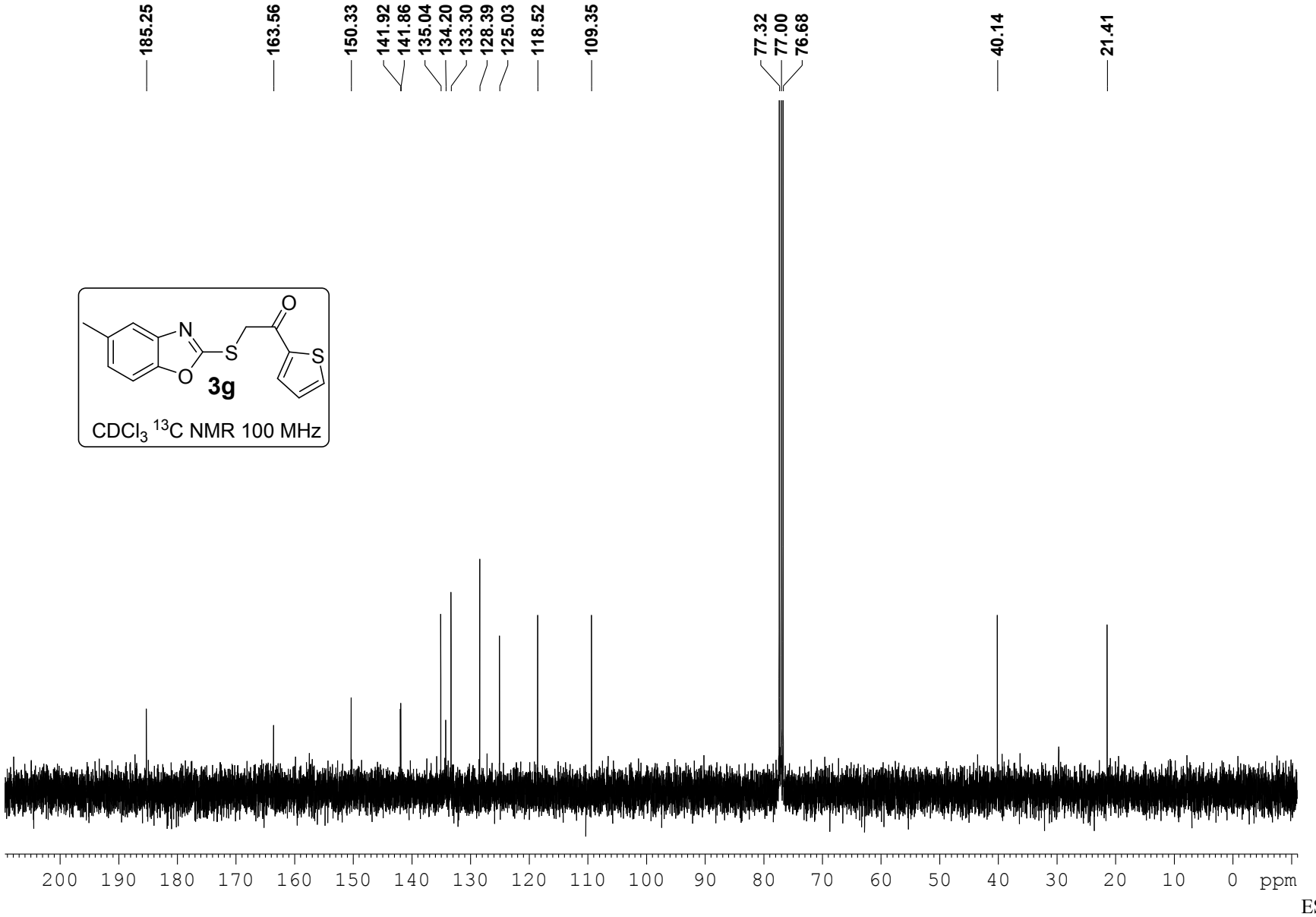
7.933
7.926
7.924
7.723
7.722
7.711
7.357
7.296
7.276
7.180
7.170
7.168
7.157
7.049
7.047
7.028
7.027
4.810
2.417
0.000
O
N
S
S
O
3g
CDCl3 1H NMR 400 MHz
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.00
1.09
1.07
1.14
1.18
2.03
3.29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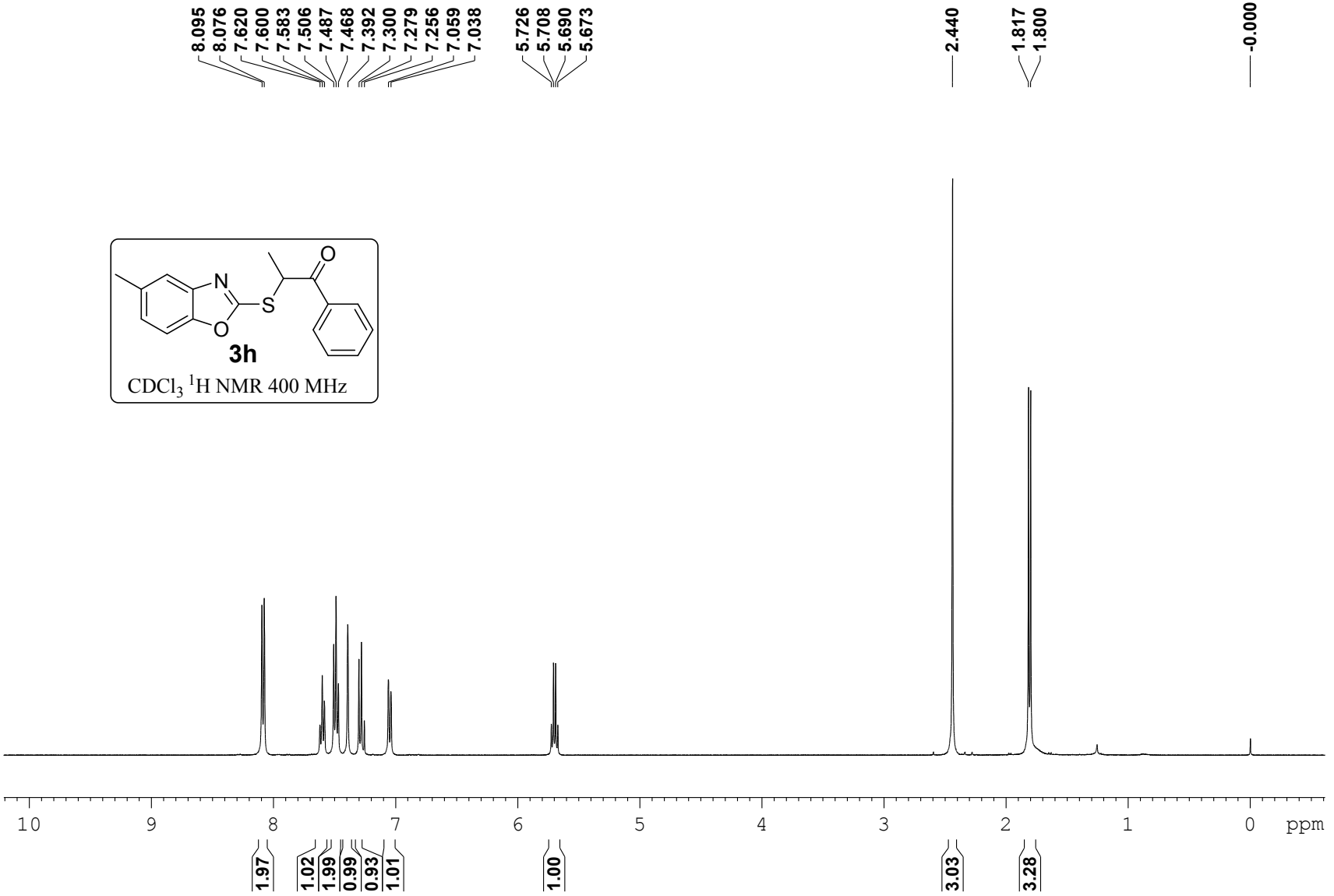
163.56
150.33
141.92
141.86
135.04
134.20
133.30
128.39
125.03
118.52
109.35
77.32
77.00
76.68
40.14
21.41
O
N
S
S
O
3g
CDCl3 13C NMR 100 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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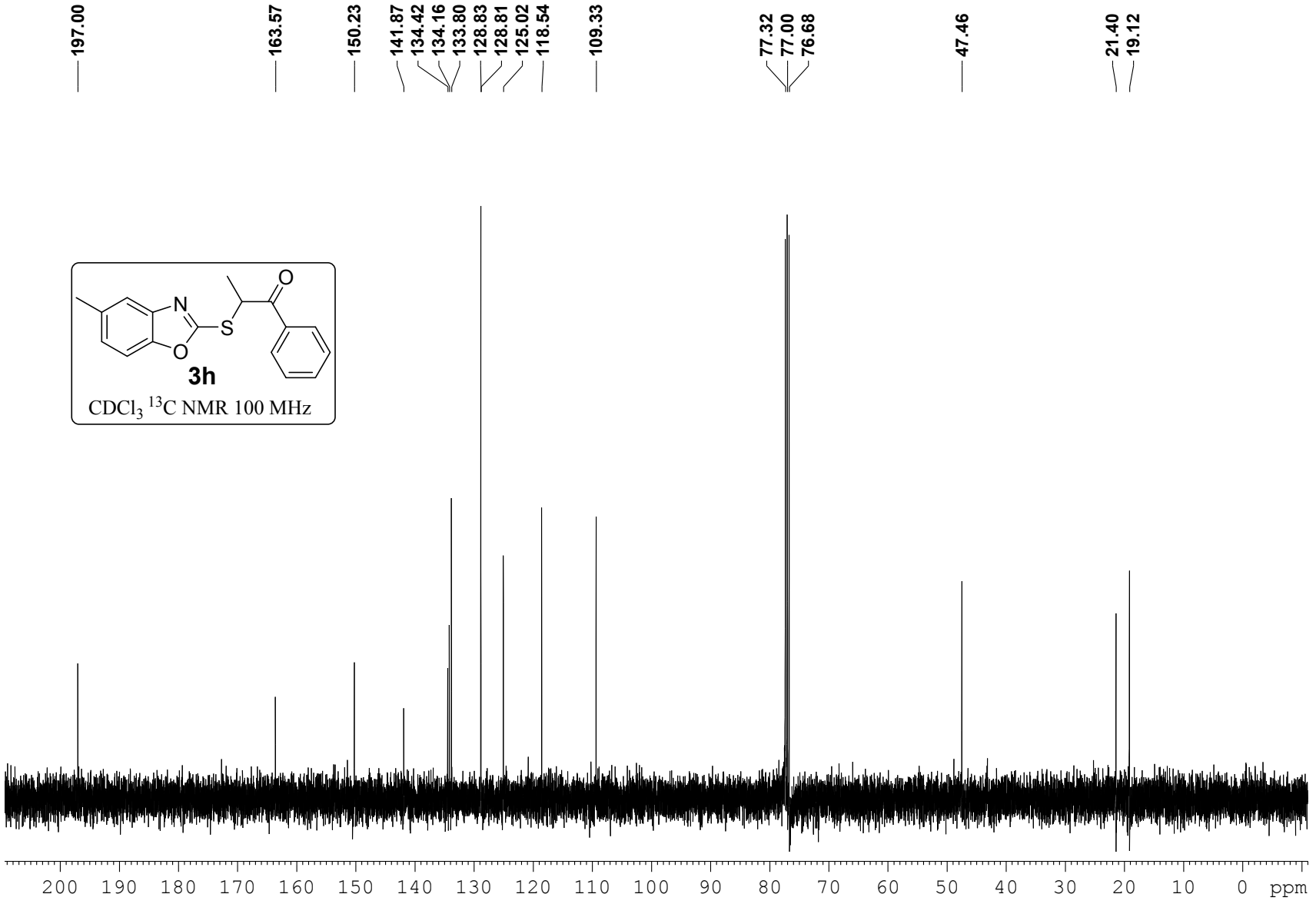
8.076
7.620
7.600
7.583
7.506
7.487
7.468
7.392
7.300
7.279
7.256
7.059
7.038
5.726
5.708
5.690
5.673
2.440
1.817
1.800
-0.000
N
S
O
O
3h
CDCl3 1H NMR 400 MHz
10
9
8
7
6
5
4
3
2
1
0
ppm
1.97
1.02
1.99
0.99
0.93
1.01
1.00
3.03
3.28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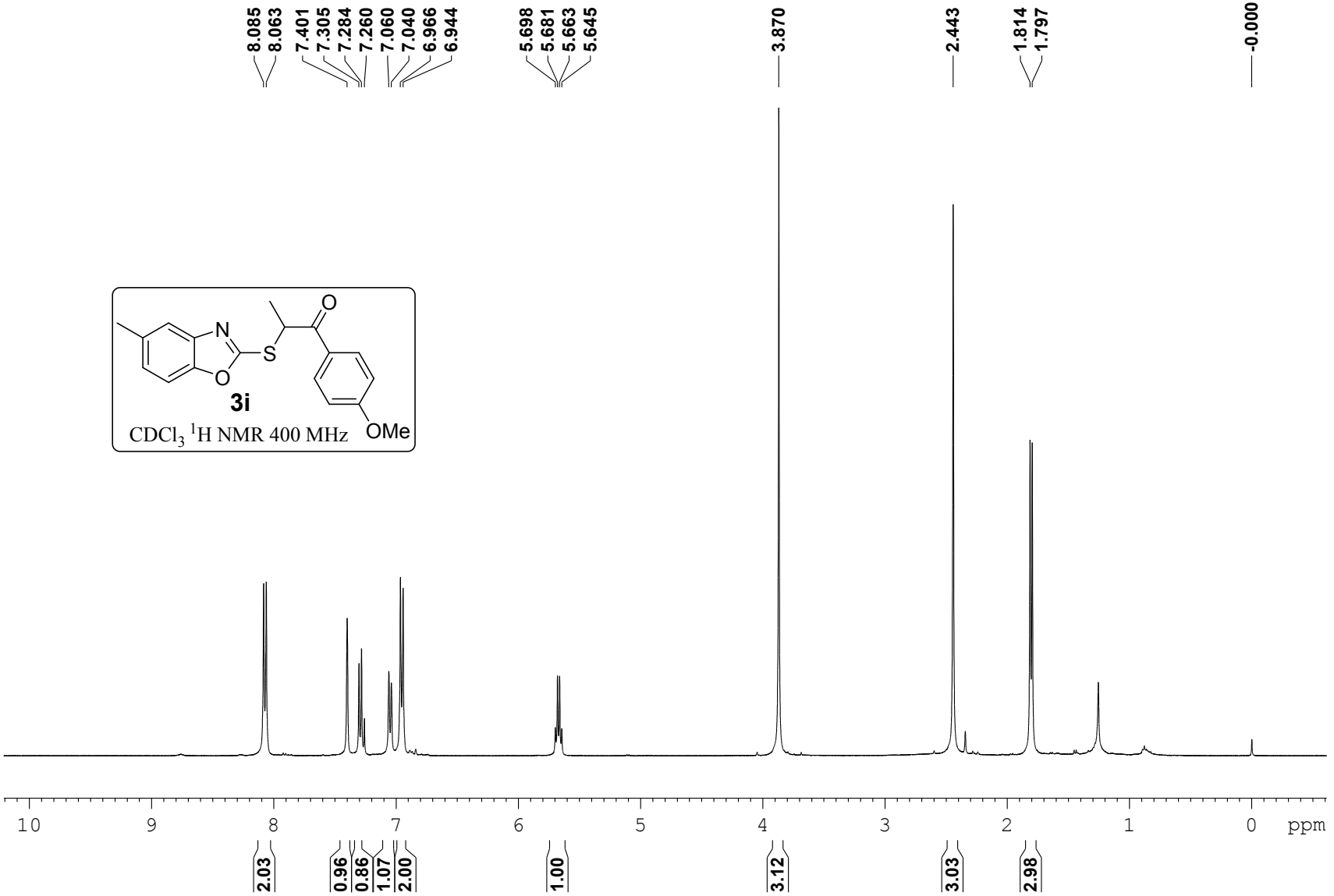
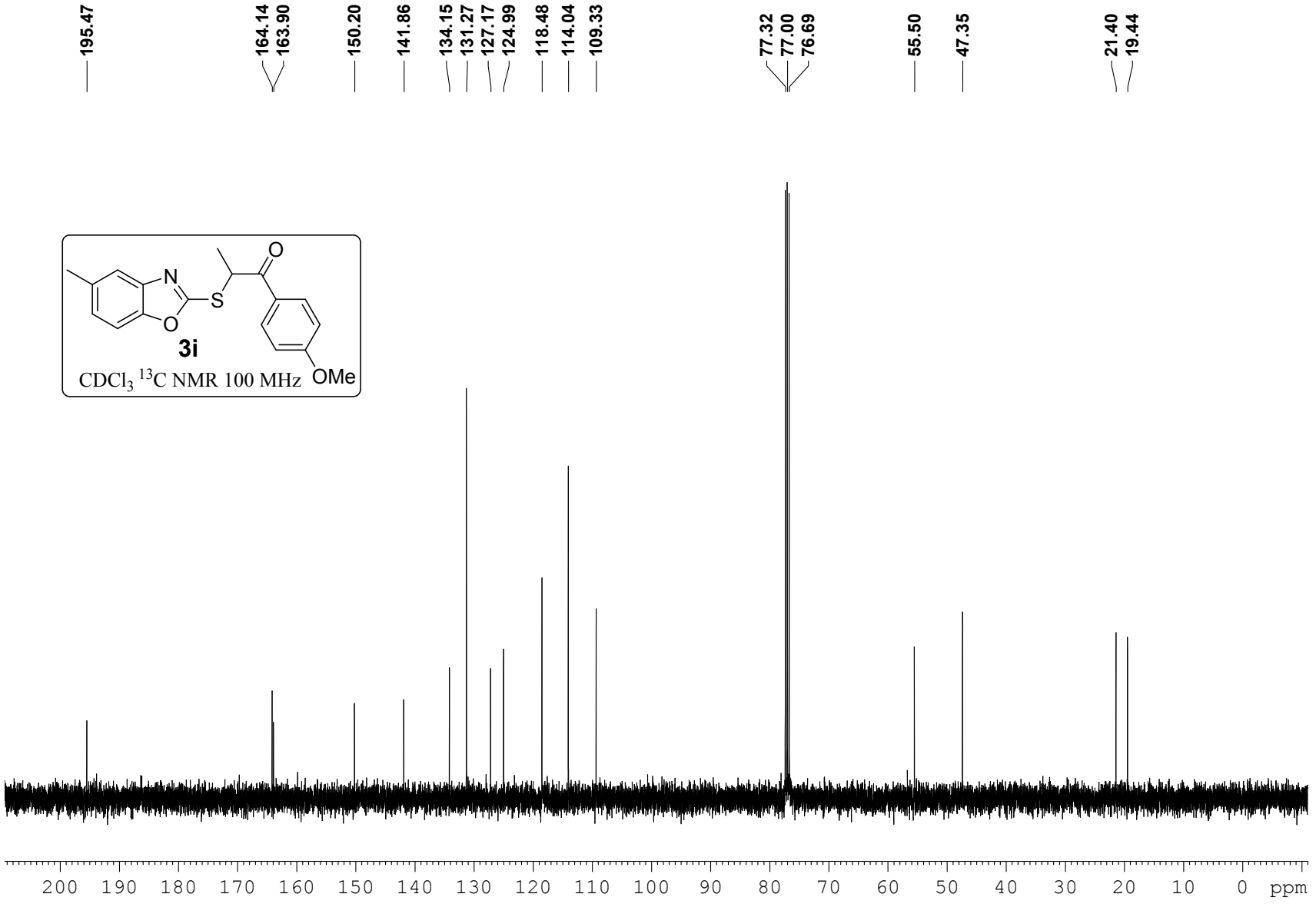
141.87
134.42
134.16
133.80
128.83
128.81
125.02
118.54
109.33
77.32
77.00
76.68
47.46
21.40
19.12
O
N
S
O
3h
CDCl3 13C NMR 100 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


8.085
8.063
7.401
7.305
7.284
7.260
7.060
7.040
6.966
6.944
5.698
5.681
5.663
5.645
3.870
2.443
1.814
1.797
-0.000
O
N
S
O
3i
CDCl3 1H NMR 400 MHz
OMe
10
9
8
7
6
5
4
3
2
1
0
ppm
2.03
0.96
0.86
1.07
2.00
1.00
3.12
3.03
2.98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164.14
163.90
150.20
141.86
134.15
131.27
127.17
124.99
118.48
114.04
109.33
77.32
77.00
76.69
55.50
47.35
21.40
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O
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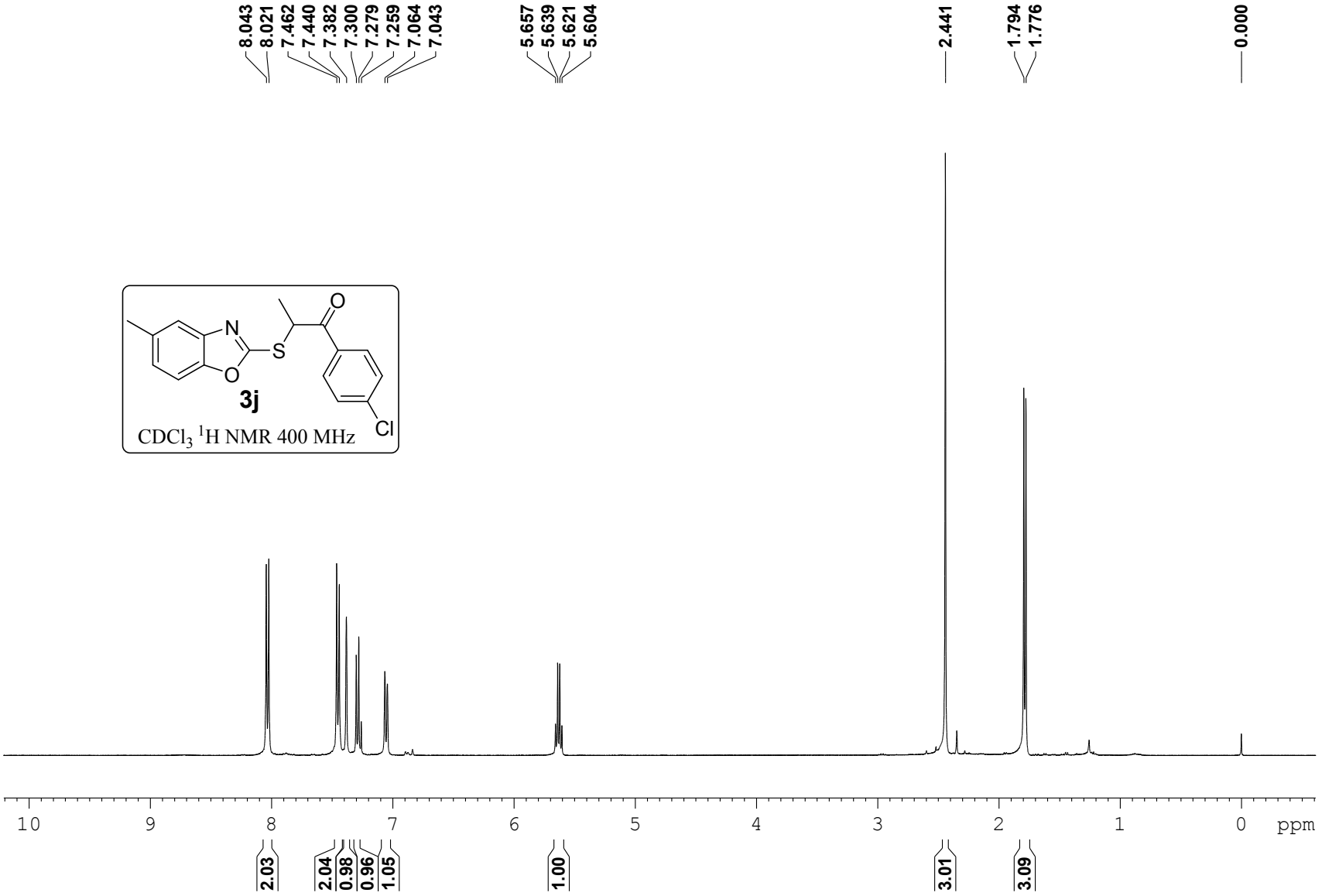
OMe
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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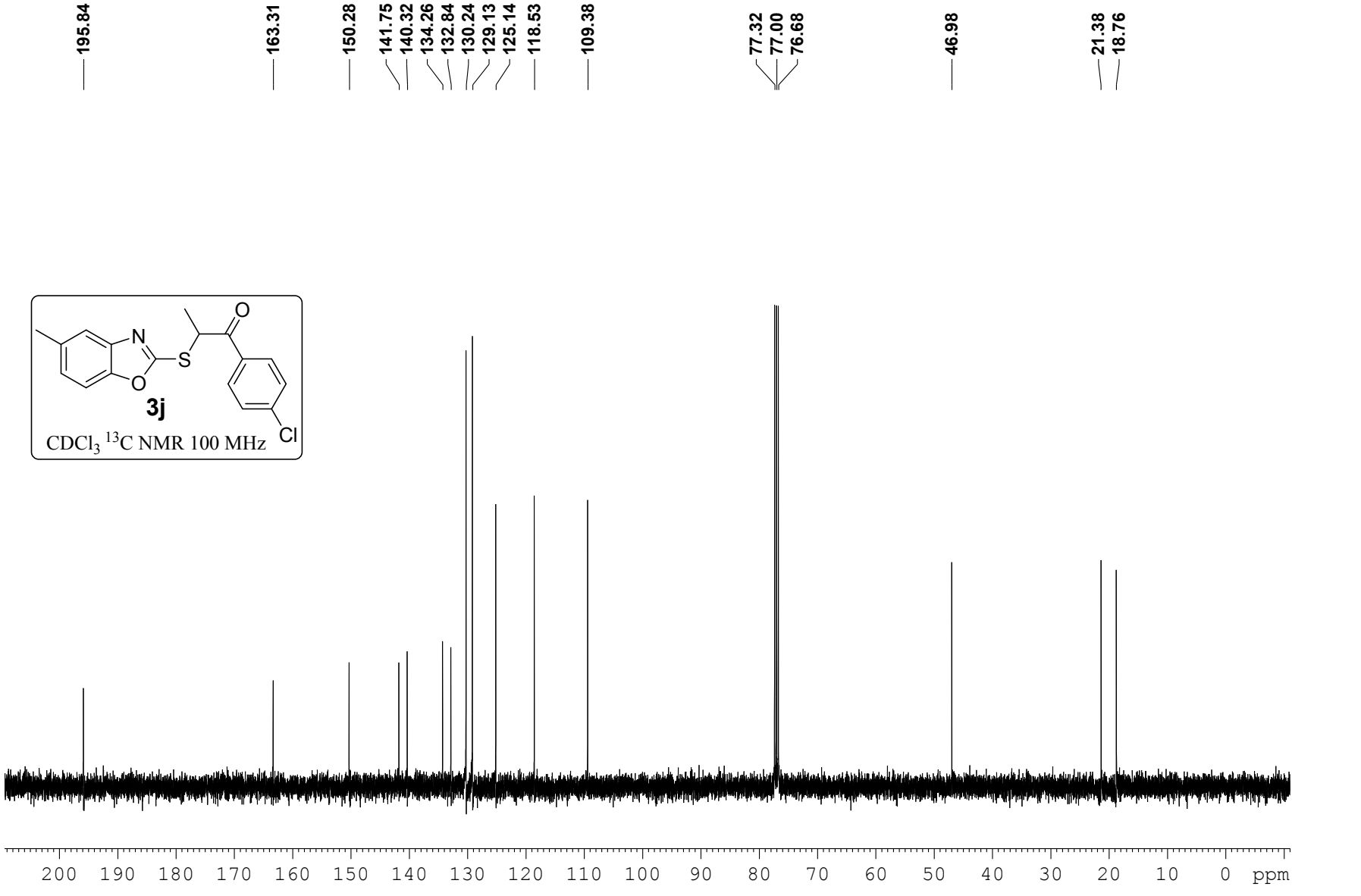
8.021
7.462
7.440
7.382
7.300
7.279
7.259
7.064
7.043
5.657
5.639
5.621
5.604
2.441
1.794
1.776
0.000
O
N
S
O
Cl
3j
CDCl3 1H NMR 400 MHz
10
9
8
7
6
5
4
3
2
1
0
ppm
2.03
2.04
0.98
0.96
1.05
1.00
3.01
3.09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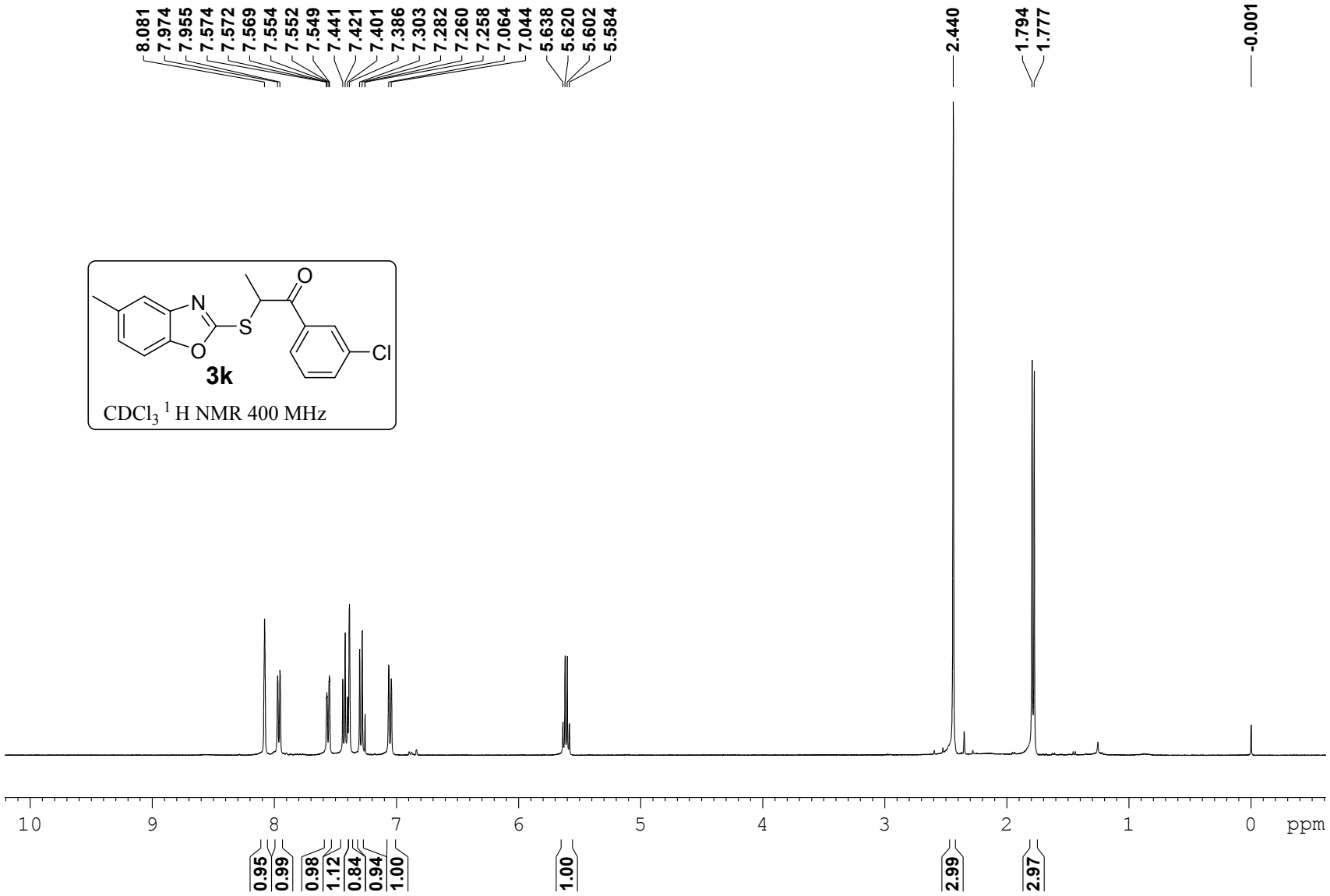
150.28
141.75
140.32
134.26
132.84
130.24
129.13
125.14
118.53
109.38
77.32
77.00
76.68
46.98
21.38
18.76
N
S
O
O
Cl
3j
CDCl3 13C NMR 100 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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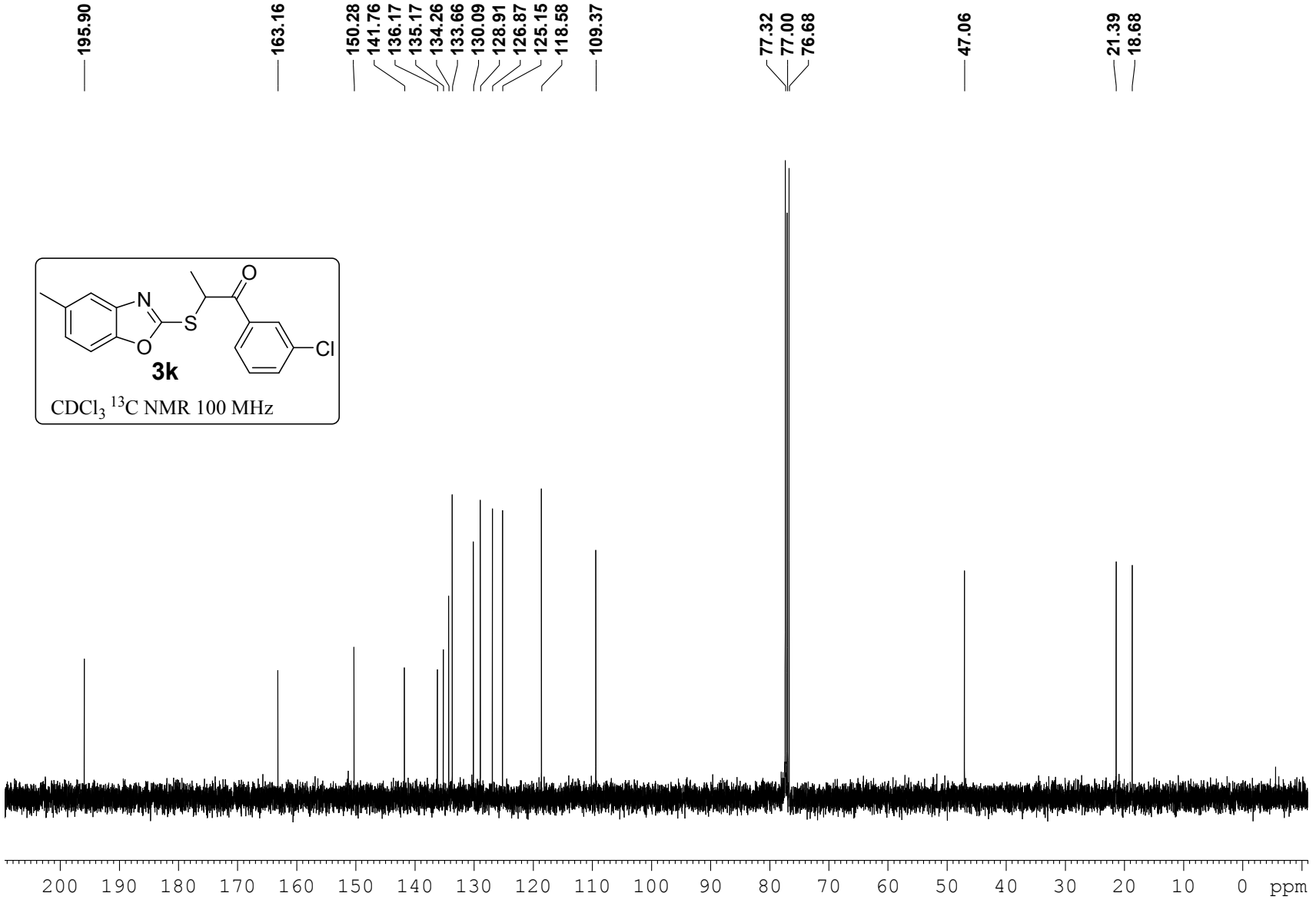
7.574
7.572
7.569
7.554
7.552
7.549
7.441
7.421
7.401
7.386
7.303
7.282
7.260
7.258
7.064
7.044
5.638
5.620
5.602
5.584
2.440
1.794
1.777
-0.001
O
N
S
O
Cl
3k
CDCl3 1 H NMR 400 MHz
10
9
8
7
6
5
4
3
2
1
0
ppm
0.95
0.99
0.98
1.12
0.84
0.94
1.00
1.00
2.99
2.97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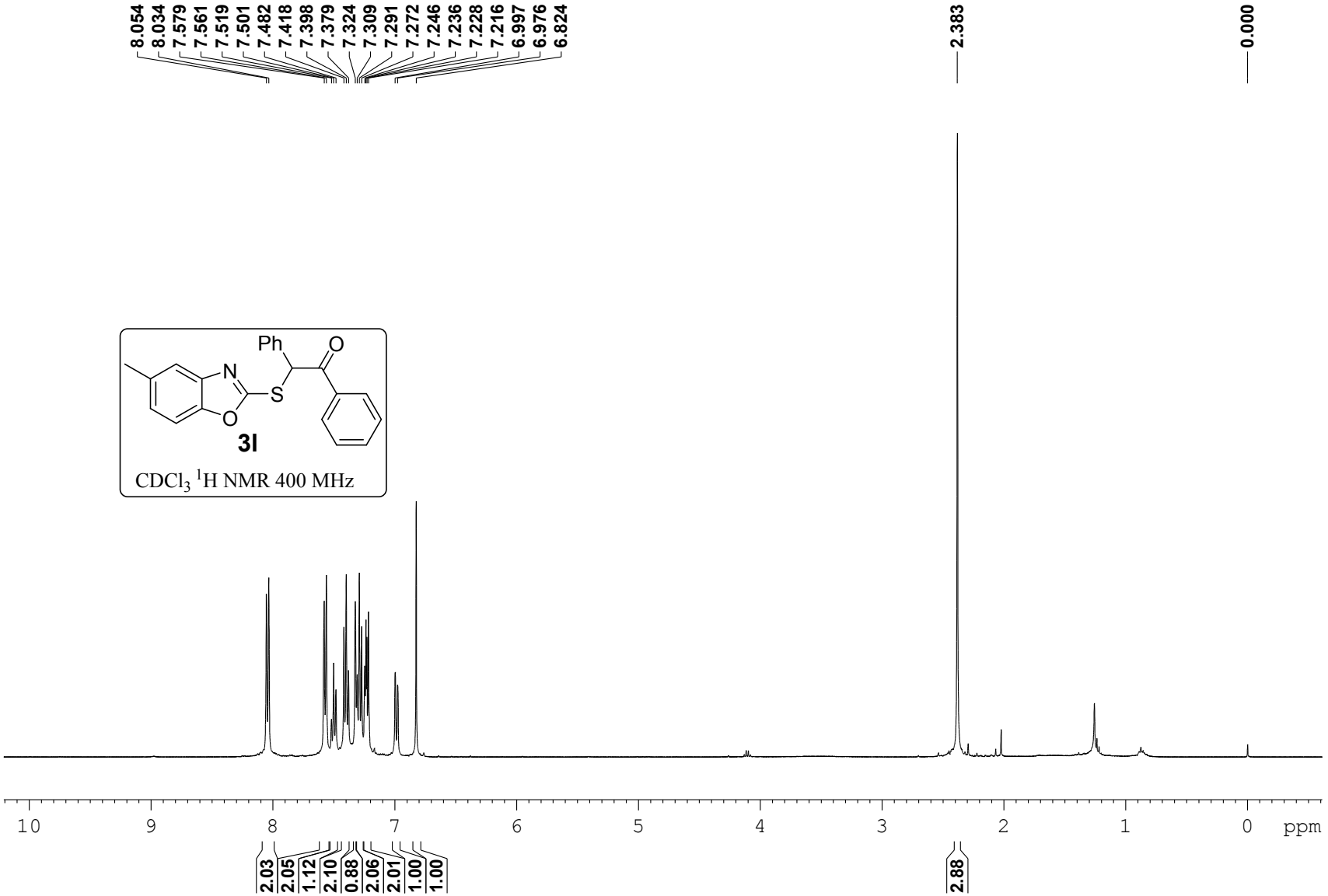
136.17
135.17
134.26
133.66
130.09
128.91
126.87
125.15
118.58
109.37
77.32
77.00
76.68
47.06
21.39
18.68
O
N
S
O
Cl
3k
CDCl3 13C NMR 100 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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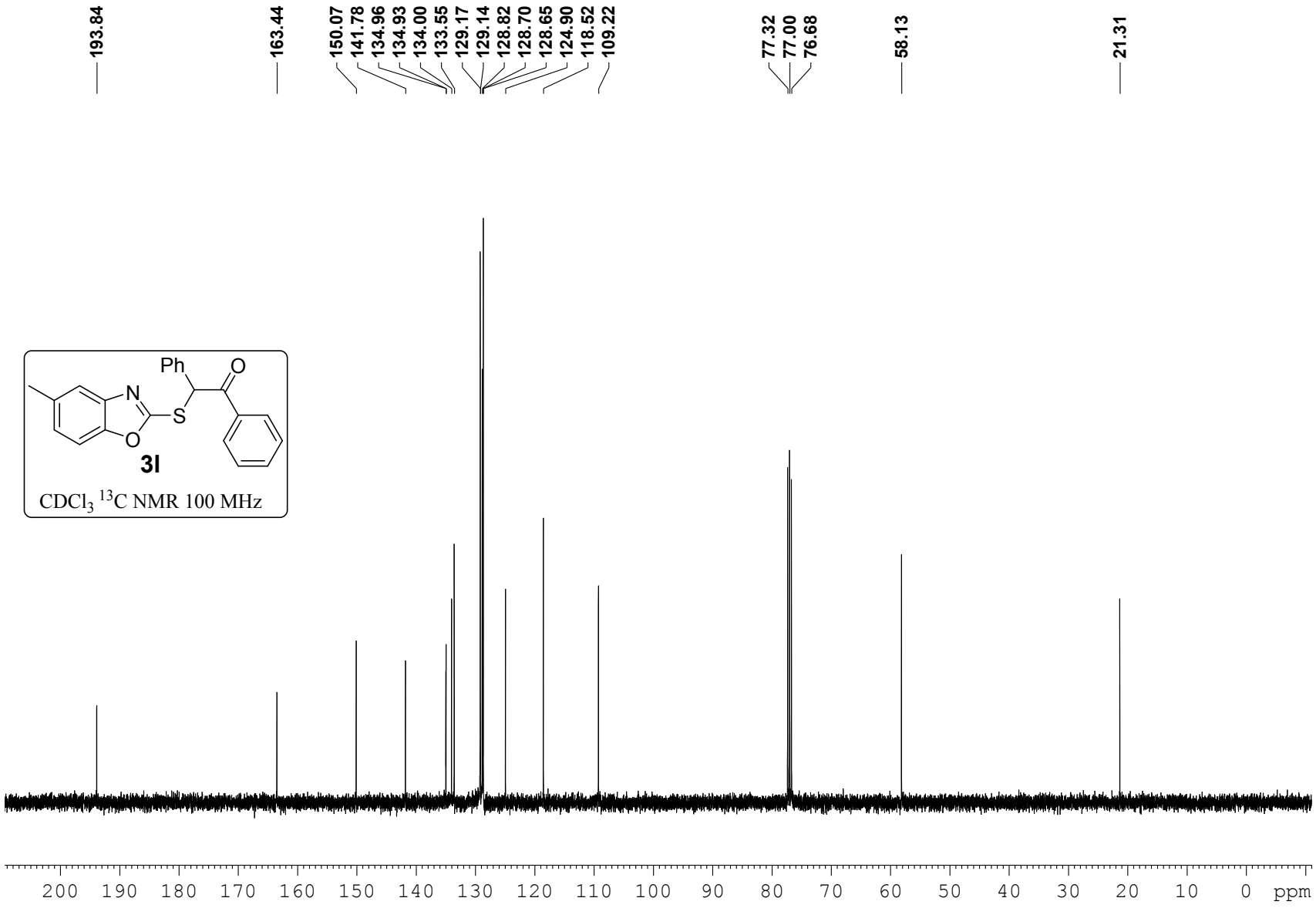
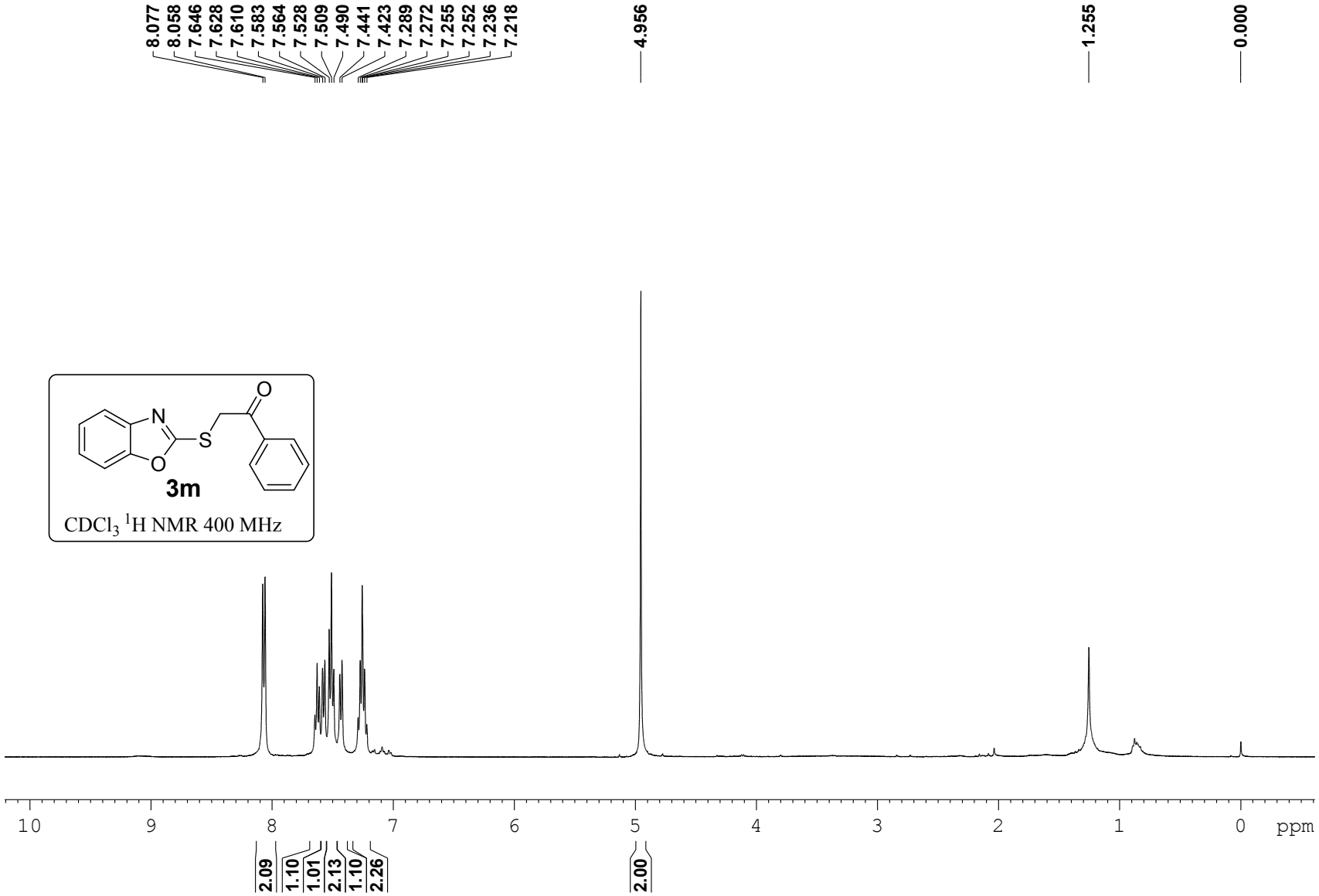
7.561
7.519
7.501
7.482
7.418
7.398
7.379
7.324
7.309
7.291
7.272
7.246
7.236
7.228
7.216
6.997
6.976
6.824
2.383
0.000
Ph
O
N
S
O
3l
CDCl3 1H NMR 400 MHz
10
9
8
7
6
5
4
3
2
1
0
ppm
2.03
2.05
1.12
2.10
0.88
2.06
2.01
1.00
1.00
2.88


193.84
163.44
150.07
141.78
134.96
134.93
134.00
133.55
129.17
129.14
128.82
128.70
128.65
124.90
118.52
109.22
77.32
77.00
76.68
58.13
21.31
Ph
O
N
S
O
3l
CDCl3 13C NMR 100 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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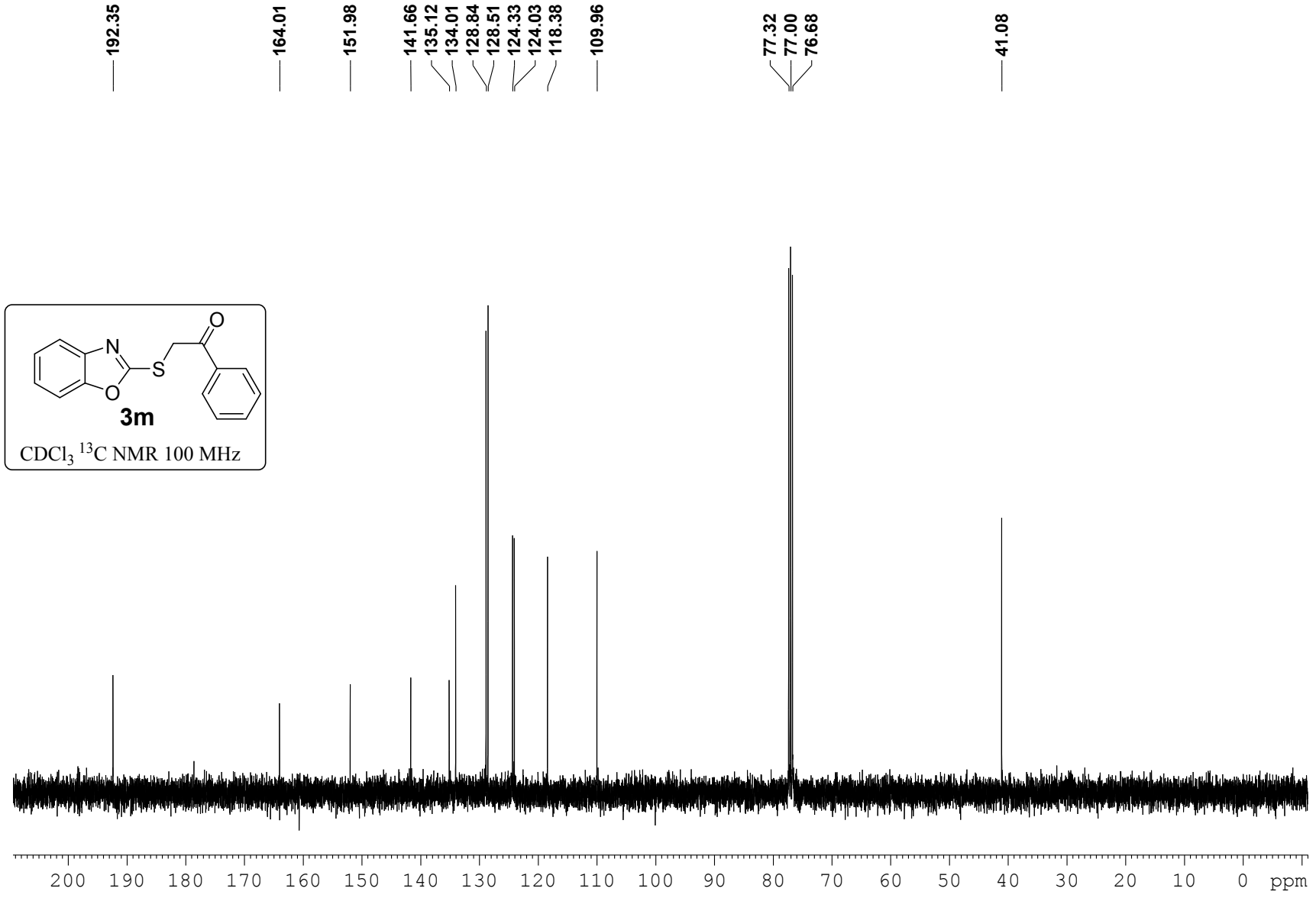
8.058
7.646
7.628
7.610
7.583
7.564
7.528
7.509
7.490
7.441
7.423
7.289
7.272
7.255
7.252
7.236
7.218
4.956
1.255
0.000
O
N
S
O
3m
CDCl3 1H NMR 400 MHz
10
9
8
7
6
5
4
3
2
1
0
ppm
2.09
1.10
1.01
2.13
1.10
2.26
2.00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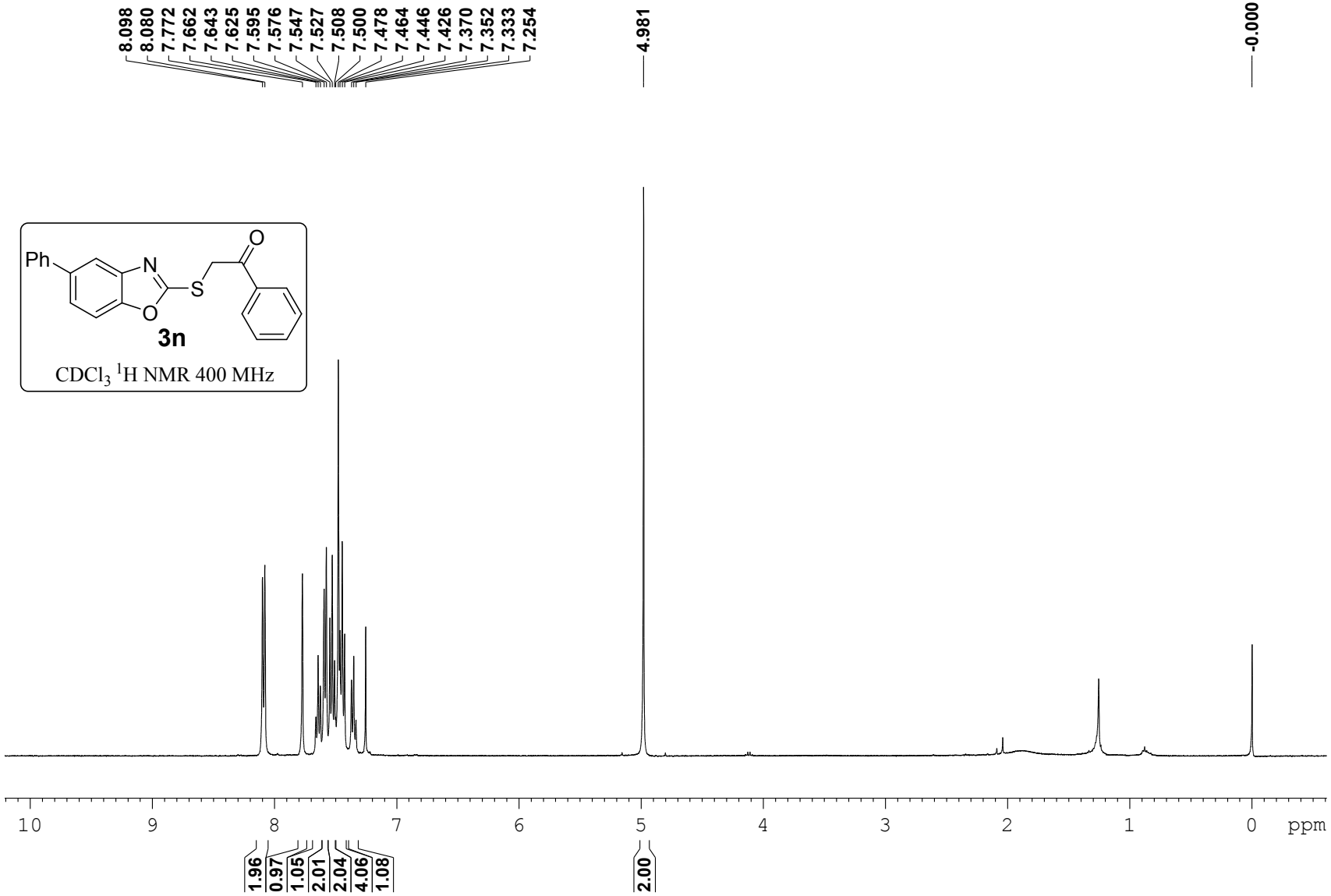
141.66
135.12
134.01
128.84
128.51
124.33
124.03
118.38
109.96
77.32
77.00
76.68
41.08
O
N
S
O
3m
CDCl3 13C NMR 100 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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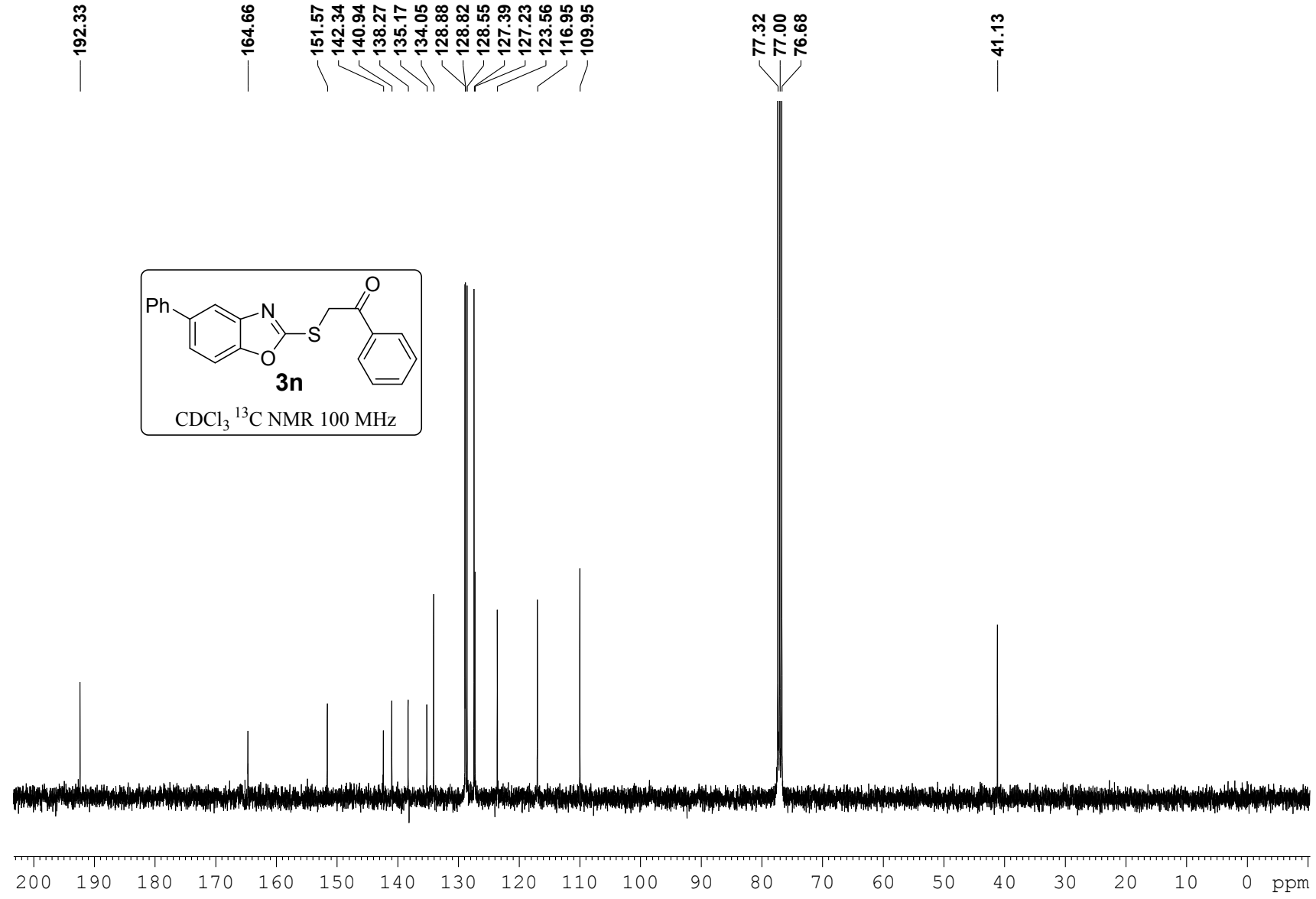
8.080
7.772
7.662
7.643
7.625
7.595
7.576
7.547
7.527
7.508
7.500
7.478
7.464
7.446
7.426
7.370
7.352
7.333
7.254
4.981
-0.000
Ph
N
S
O
O
3n
CDCl3 1H NMR 400 MHz
10
9
8
7
6
5
4
3
2
1
0
ppm
1.96
0.97
1.05
2.01
2.04
4.06
1.08
2.00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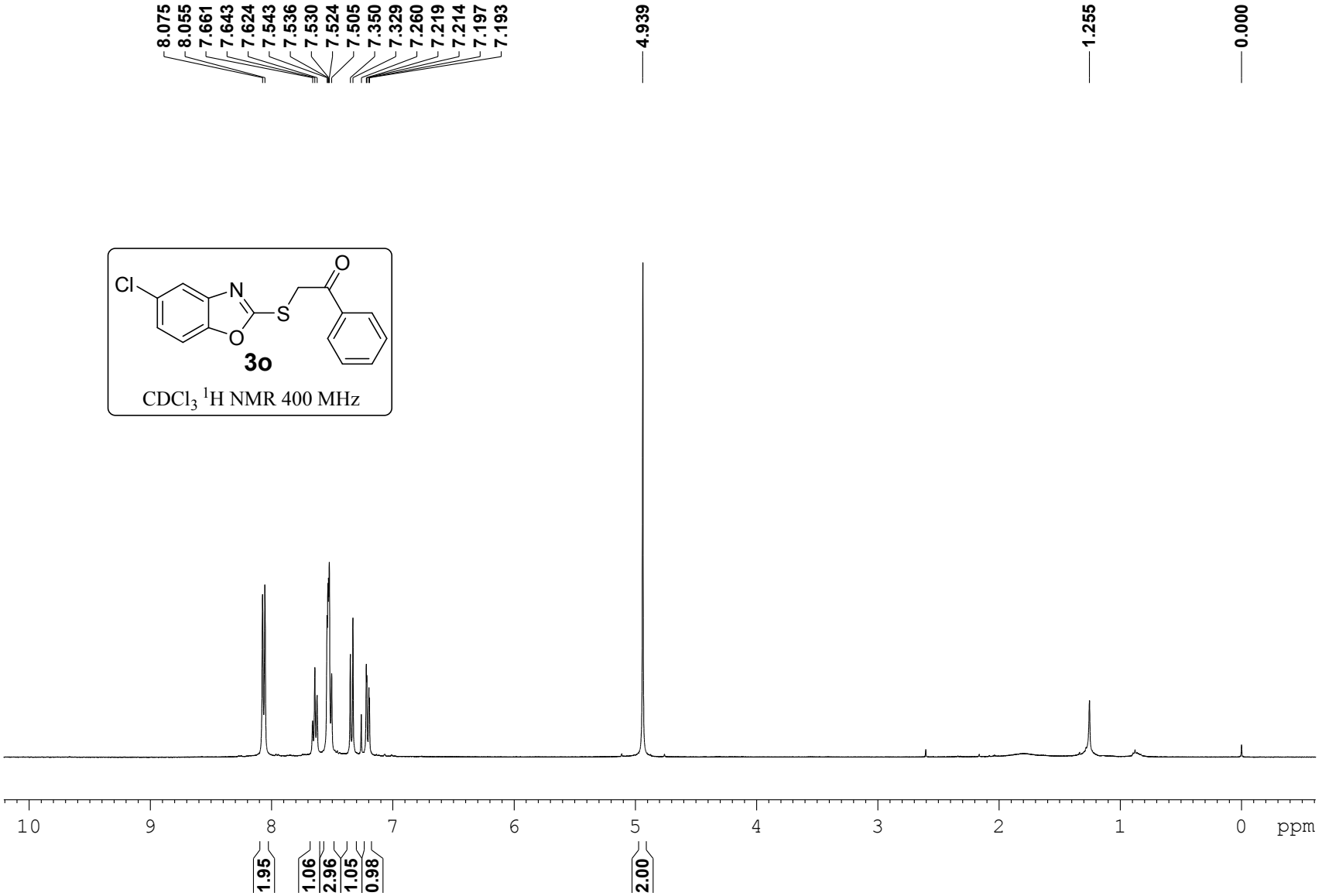
151.57
142.34
140.94
138.27
135.17
134.05
128.88
128.82
128.55
127.39
127.23
123.56
116.95
109.95
77.32
77.00
76.68
41.13
Ph
N
S
O
O
3n
CDCl3 13C NMR 100 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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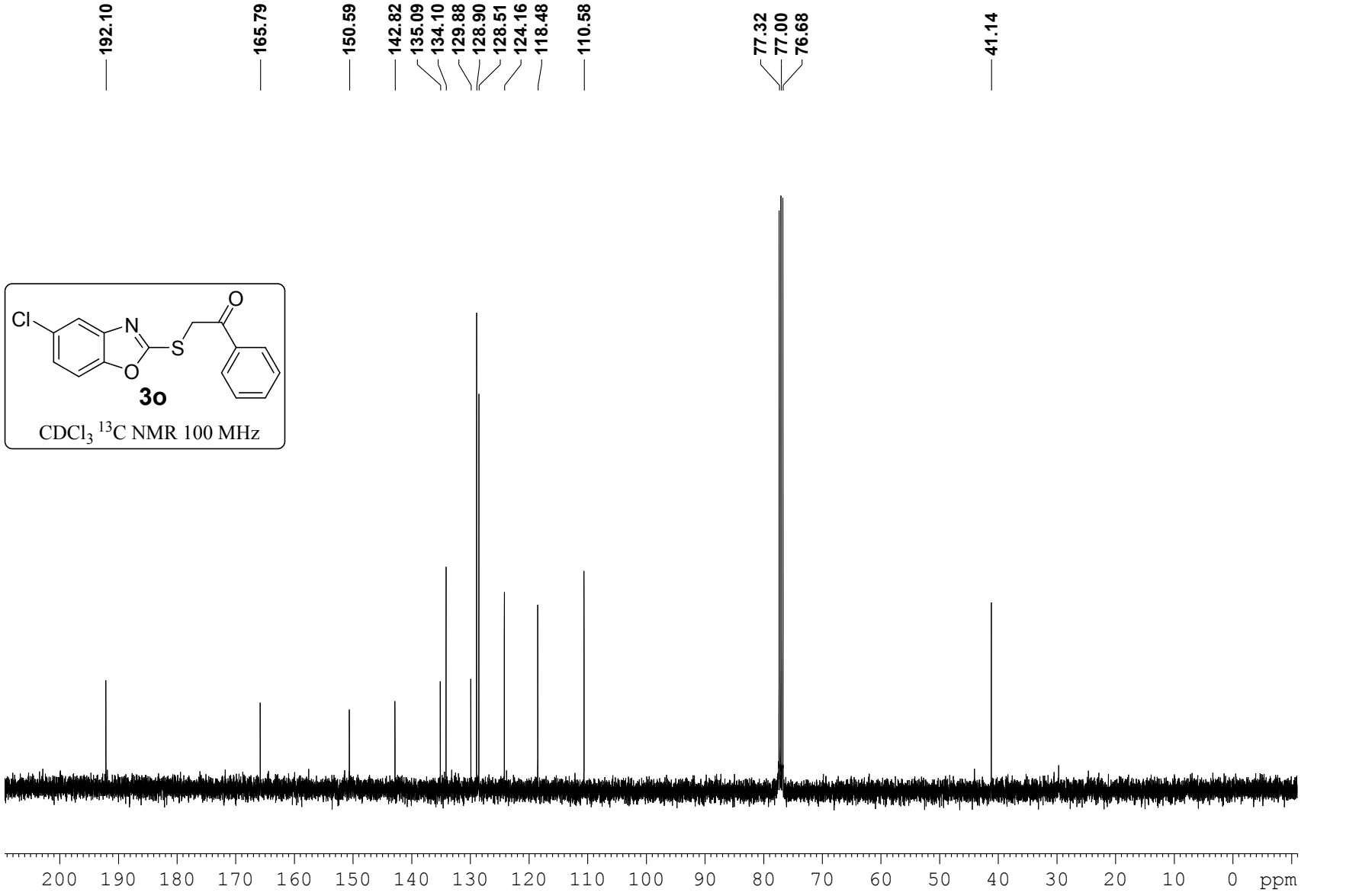
8.055
7.661
7.643
7.624
7.543
7.536
7.530
7.524
7.505
7.350
7.329
7.260
7.219
7.214
7.197
7.193
4.939
1.255
0.000
Cl
N
S
O
O
3o
CDCl3 1H NMR 400 MHz
10
9
8
7
6
5
4
3
2
1
0
ppm
1.95
1.06
2.96
1.05
0.98
2.00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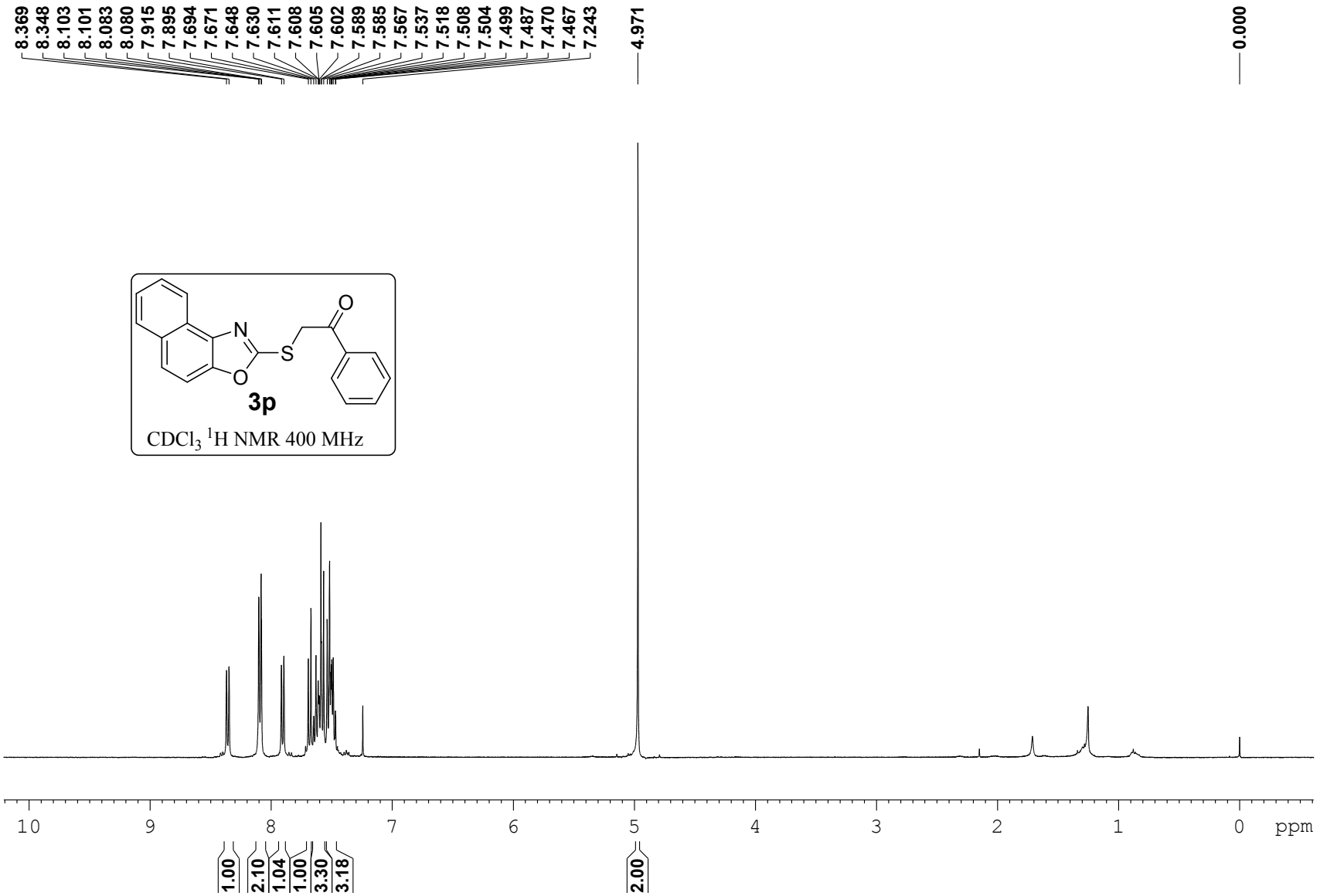
134.10
129.88
128.90
128.51
124.16
118.48
110.58
77.32
77.00
76.68
41.14
Cl
N
S
O
O
3o
CDCl3 13C NMR 100 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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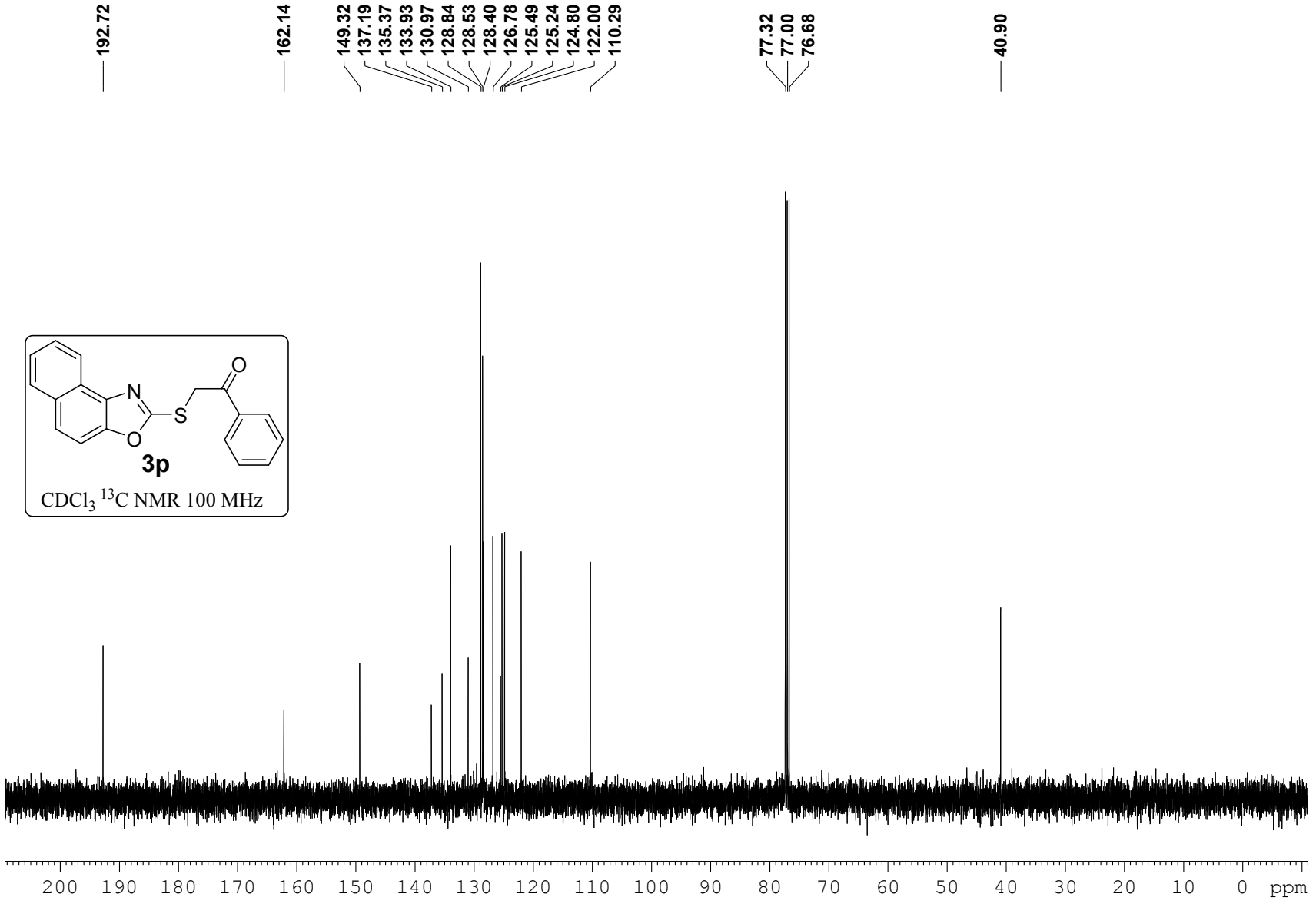
8.101
8.083
8.080
7.915
7.895
7.694
7.671
7.648
7.630
7.611
7.608
7.605
7.602
7.589
7.585
7.567
7.537
7.518
7.508
7.504
7.499
7.487
7.470
7.467
7.243
4.971
0.000
O
N
S
O
3p
CDCl3 1H NMR 400 MHz
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.10
1.04
1.00
3.30
3.18
2.00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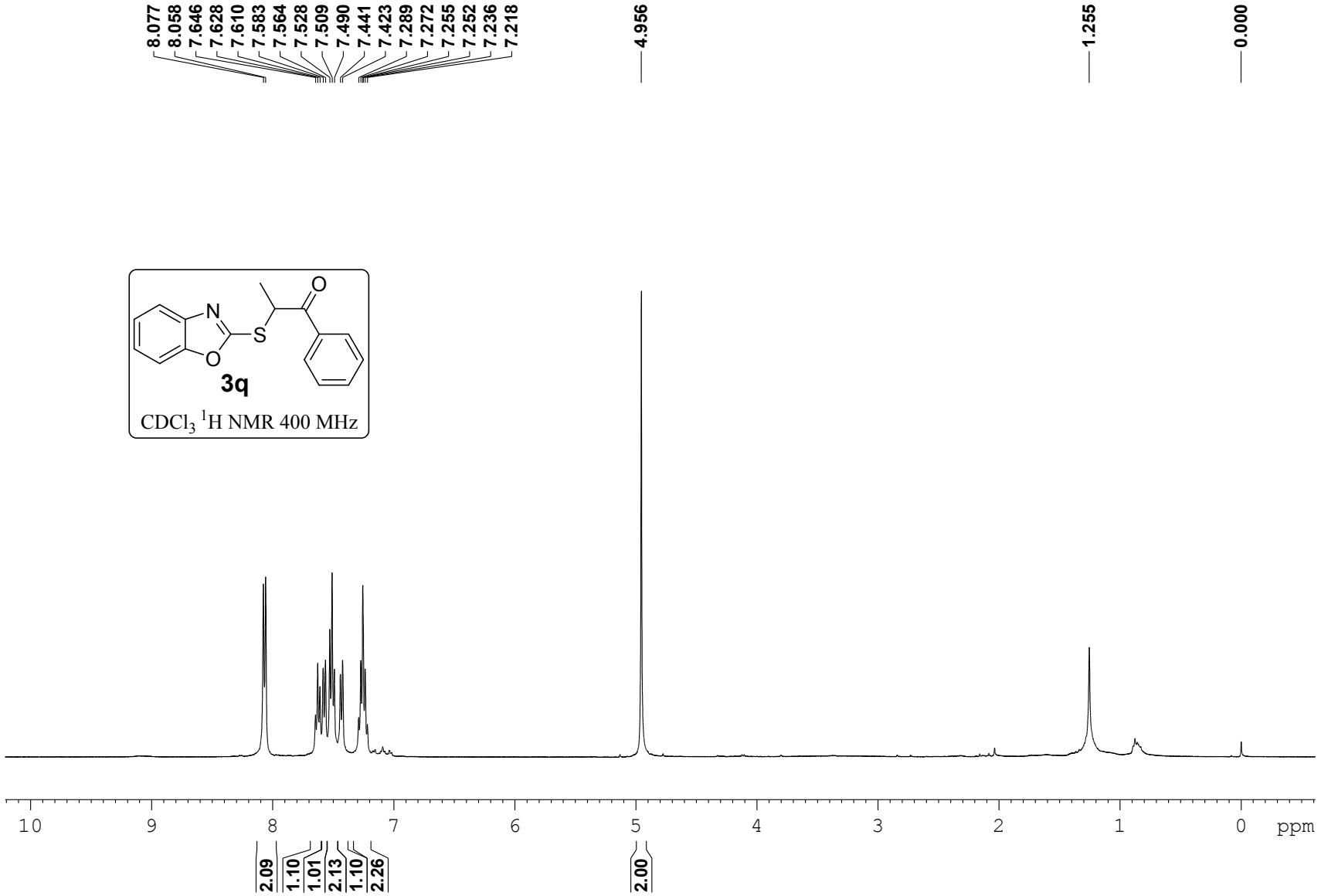
137.19
135.37
133.93
130.97
128.84
128.53
128.40
126.78
125.49
125.24
124.80
122.00
110.29
77.32
77.00
76.68
40.90
O
N
S
O
3p
CDCl3 13C NMR 100 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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8.058
7.646
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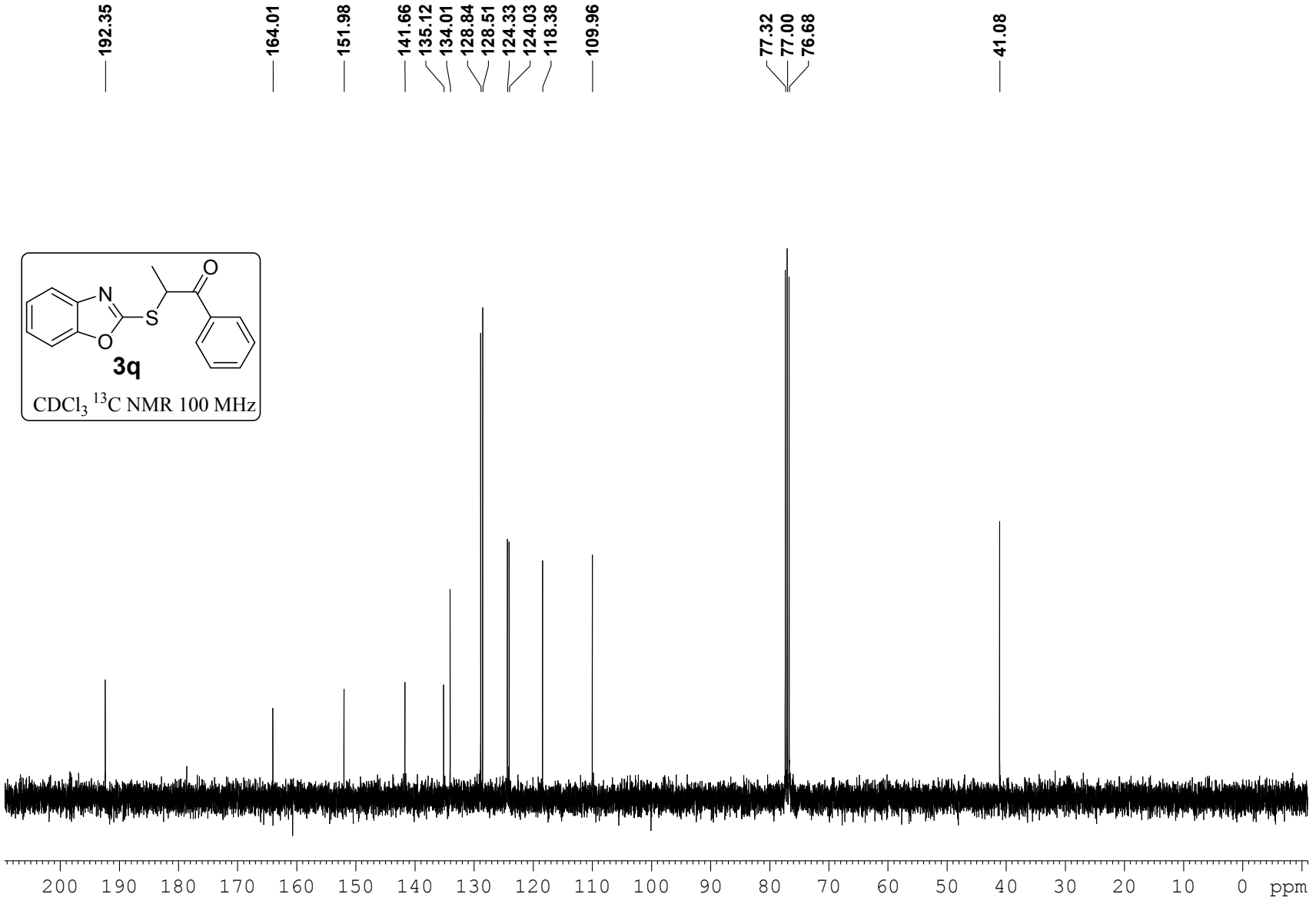
7.583
7.564
7.528
7.509
7.490
7.441
7.423
7.289
7.272
7.255
7.252
7.236
7.218
4.956
1.255
0.000
N
S
O
O
3q
CDCl3 1H NMR 400 MHz
10
9
8
7
6
5
4
3
2
1
0
ppm
2.09
1.10
1.01
2.13
1.10
2.26
2.00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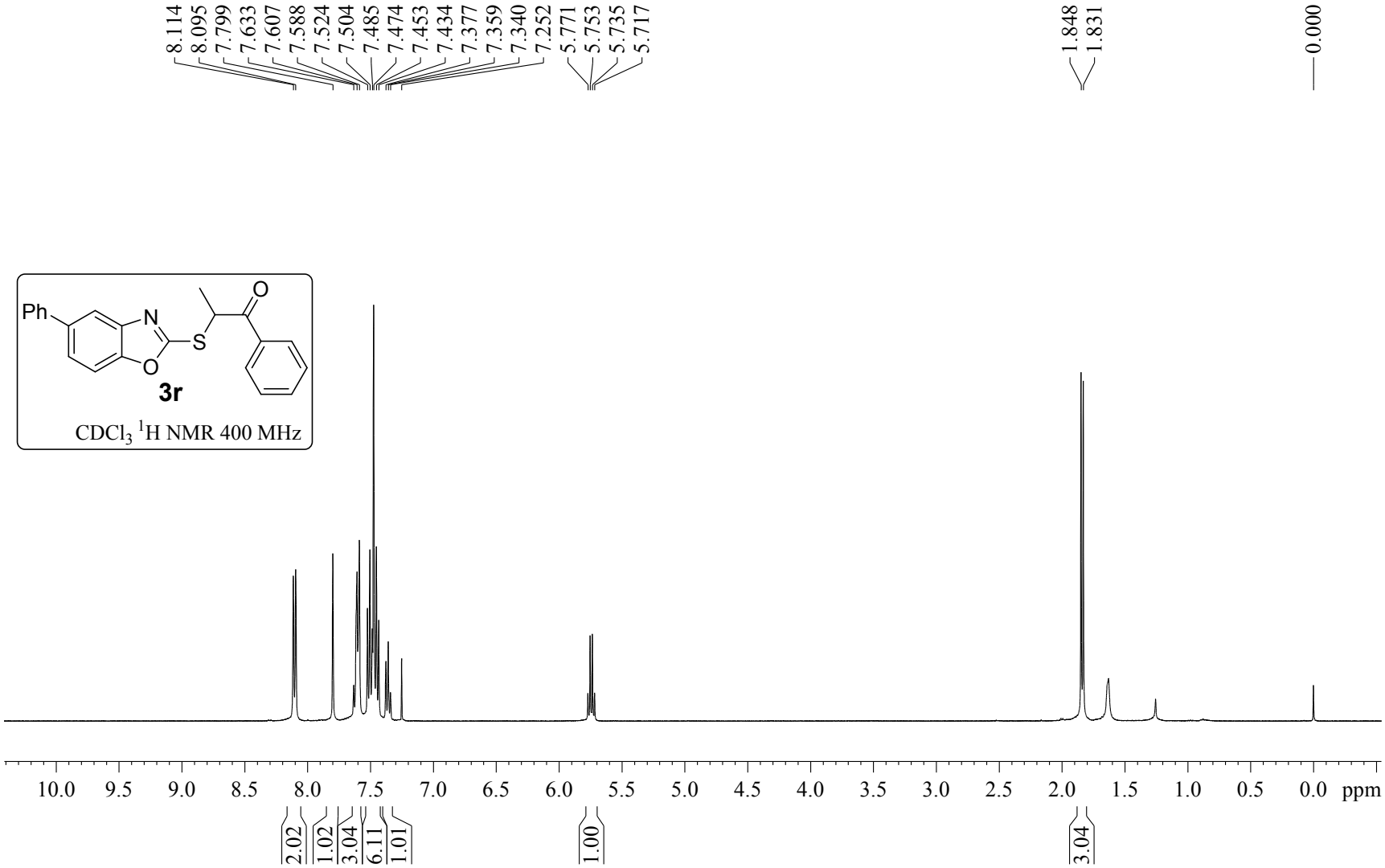
141.66
135.12
134.01
128.84
128.51
124.33
124.03
118.38
109.96
77.32
77.00
76.68
41.08
N
S
O
O
3q
CDCl3 13C NMR 100 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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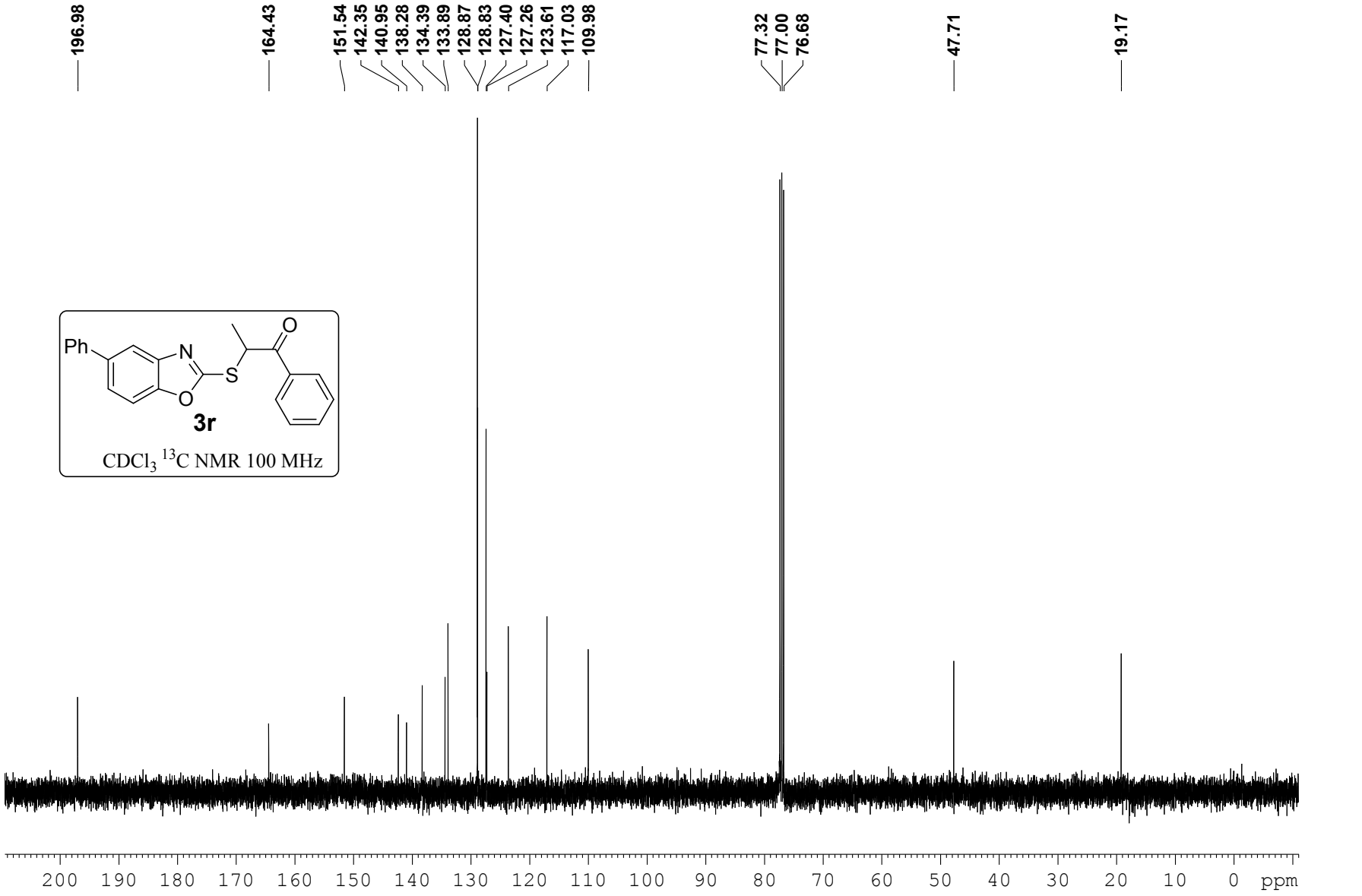
7.633
7.607
7.588
7.524
7.504
7.485
7.474
7.453
7.434
7.377
7.359
7.340
7.252
5.771
5.753
5.735
5.717
1.848
1.831
0.000
Ph
N
S
O
O
3r
CDCl3 1H NMR 400 MHz
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.02
1.02
3.04
6.11
1.01
1.00
3.04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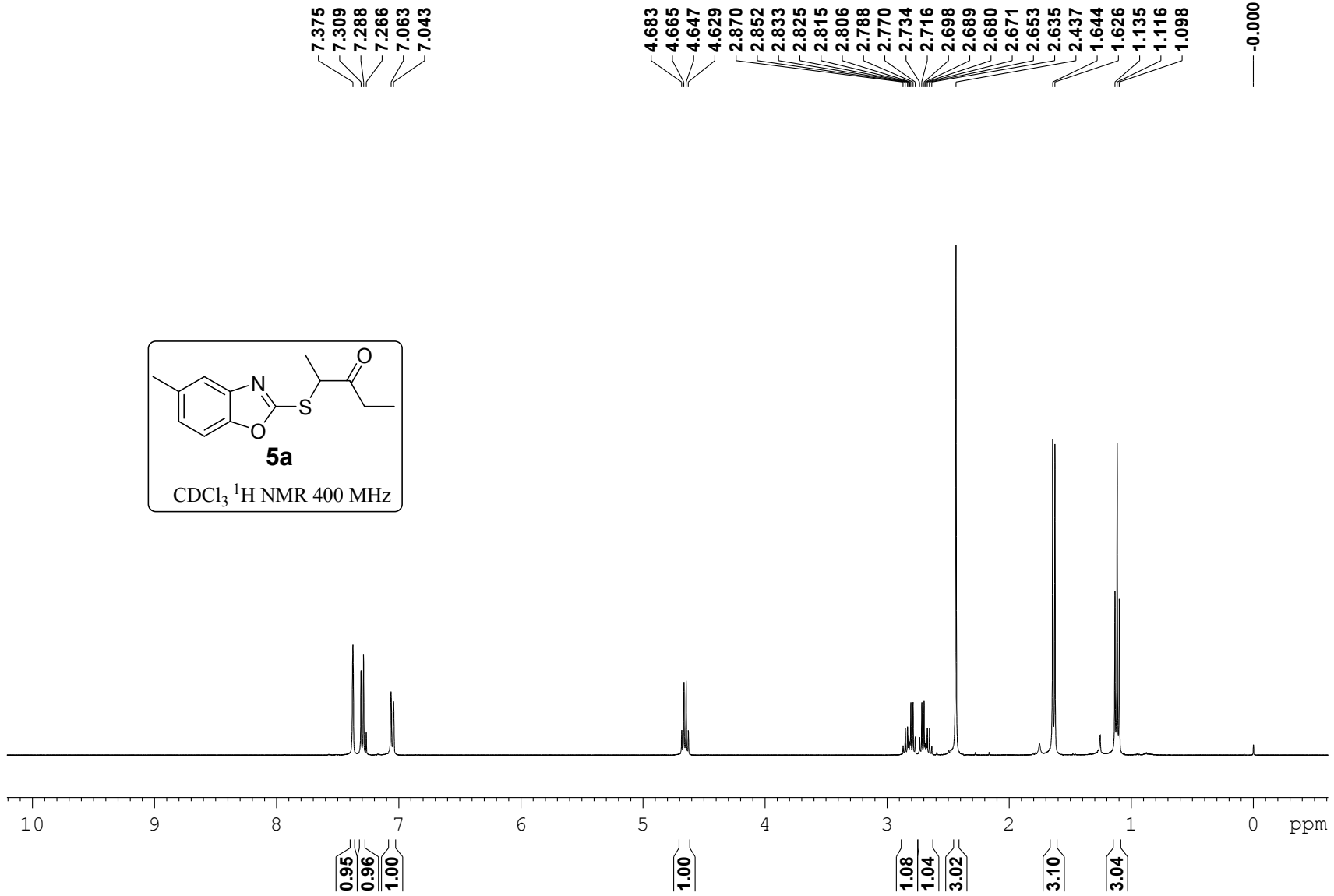
133.89
128.87
128.83
127.40
127.26
123.61
117.03
109.98
77.32
77.00
76.68
47.71
19.17
Ph
N
S
O
O
3r
CDCl3 13C NMR 100 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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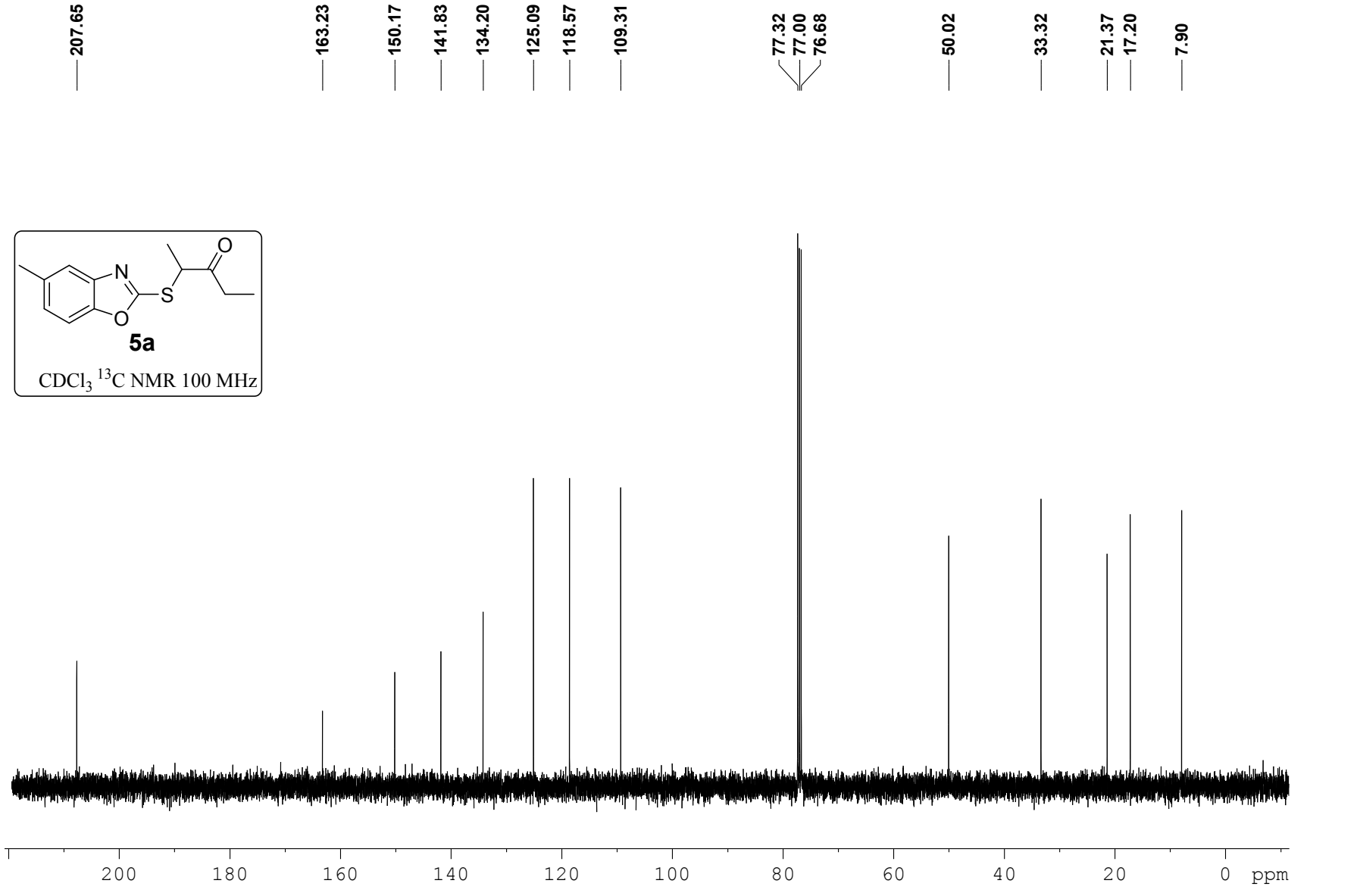
7.309
7.288
7.266
7.063
7.043
4.683
4.665
4.647
4.629
2.870
2.852
2.833
2.825
2.815
2.806
2.788
2.770
2.734
2.716
2.698
2.689
2.680
2.671
2.653
2.635
2.437
1.644
1.626
1.135
1.116
1.098
-0.000
O
N
S
O
5a
CDCl3 1H NMR 400 MHz
10
9
8
7
6
5
4
3
2
1
0
ppm
0.95
0.96
1.00
1.00
1.08
1.04
3.02
3.10
3.04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163.23
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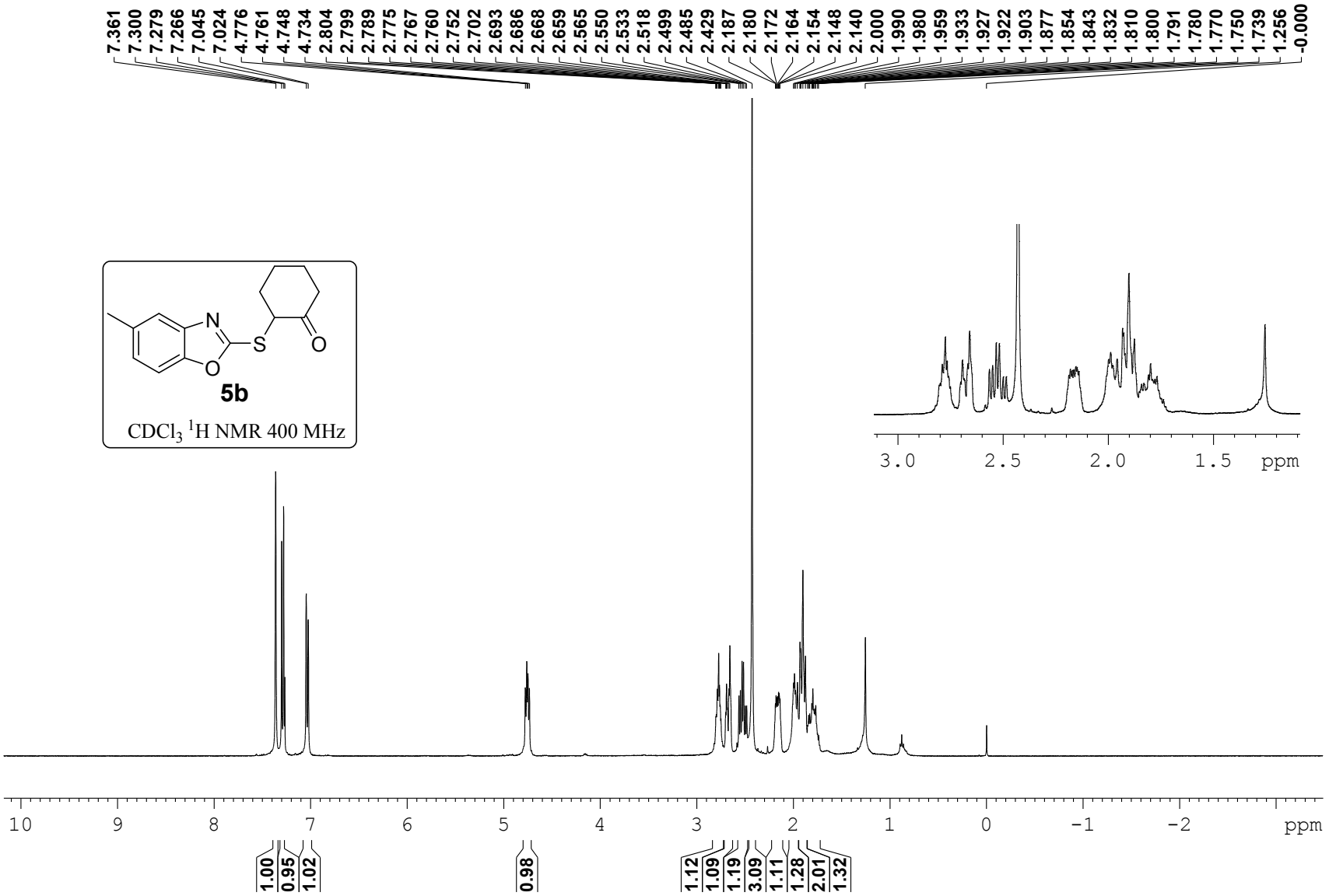
141.83
134.20
125.09
118.57
109.31
77.32
77.00
76.68
50.02
33.32
21.37
17.20
7.90
N
S
O
O
5a
CDCl3 13C NMR 100 MHz
200
180
160
140
120
100
80
60
40
20
0
ppm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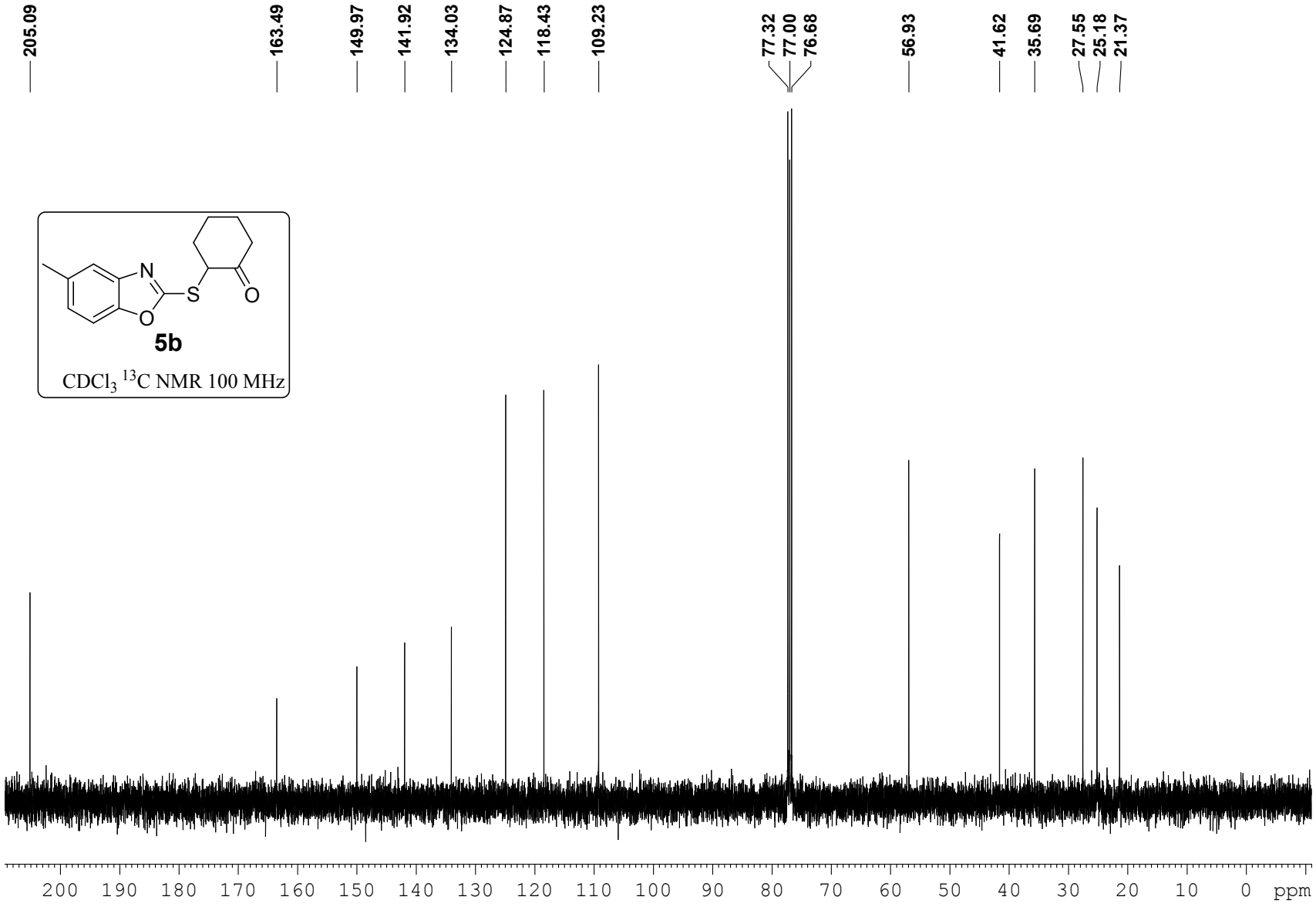
7.300
7.279
7.266
7.045
7.024
4.776
4.761
4.748
4.734
2.804
2.799
2.789
2.775
2.767
2.760
2.752
2.702
2.693
2.686
2.668
2.659
2.565
2.550
2.533
2.518
2.499
2.485
2.429
2.187
2.180
2.172
2.164
2.154
2.148
2.140
2.000
1.990
1.980
1.959
1.933
1.927
1.922
1.903
1.877
1.854
1.843
1.832
1.810
1.800
1.791
1.780
1.770
1.750
1.739
1.256
-0.000
N
S
O
O
5b
CDCl3 1H NMR 400 MHz
3.0
2.5
2.0
1.5
ppm
10
9
8
7
6
5
4
3
2
1
0
-1
-2
ppm
1.00
0.95
1.02
0.98
1.12
1.09
1.19
3.09
1.11
1.28
2.01
1.32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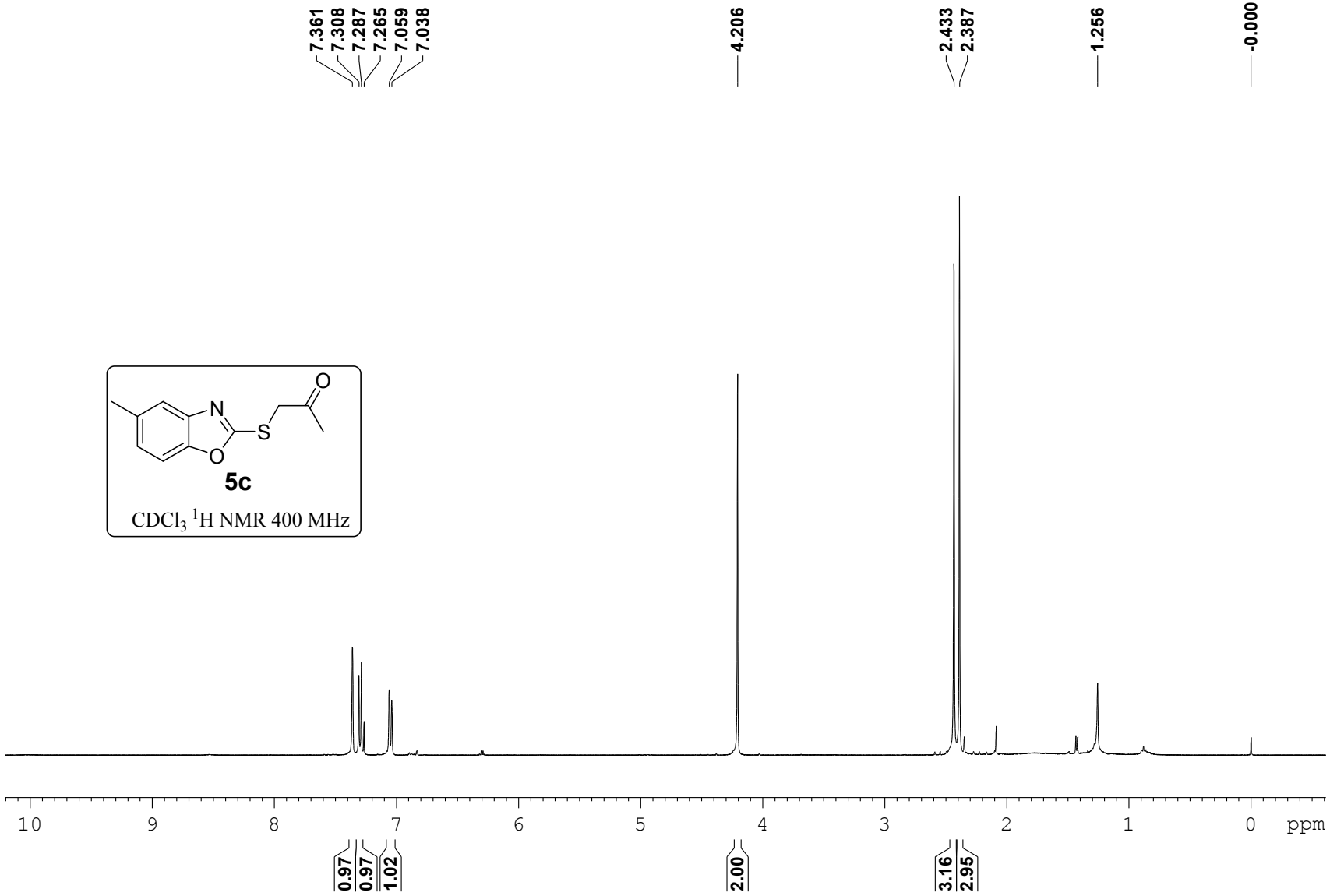
163.49
149.97
141.92
134.03
124.87
118.43
109.23
77.32
77.00
76.68
56.93
41.62
35.69
27.55
25.18
21.37
N
S
O
O
5b
CDCl3 13C NMR 100 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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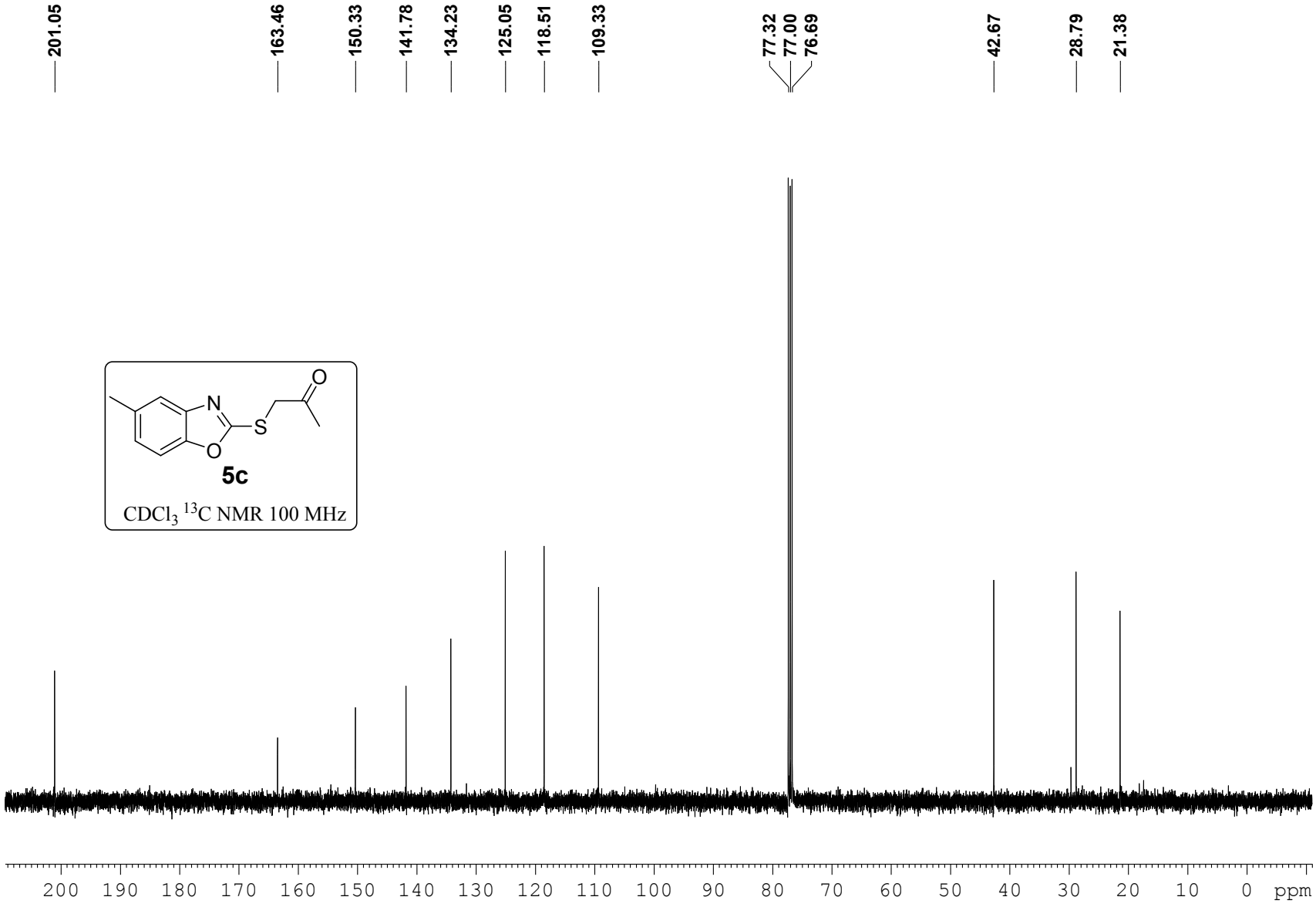
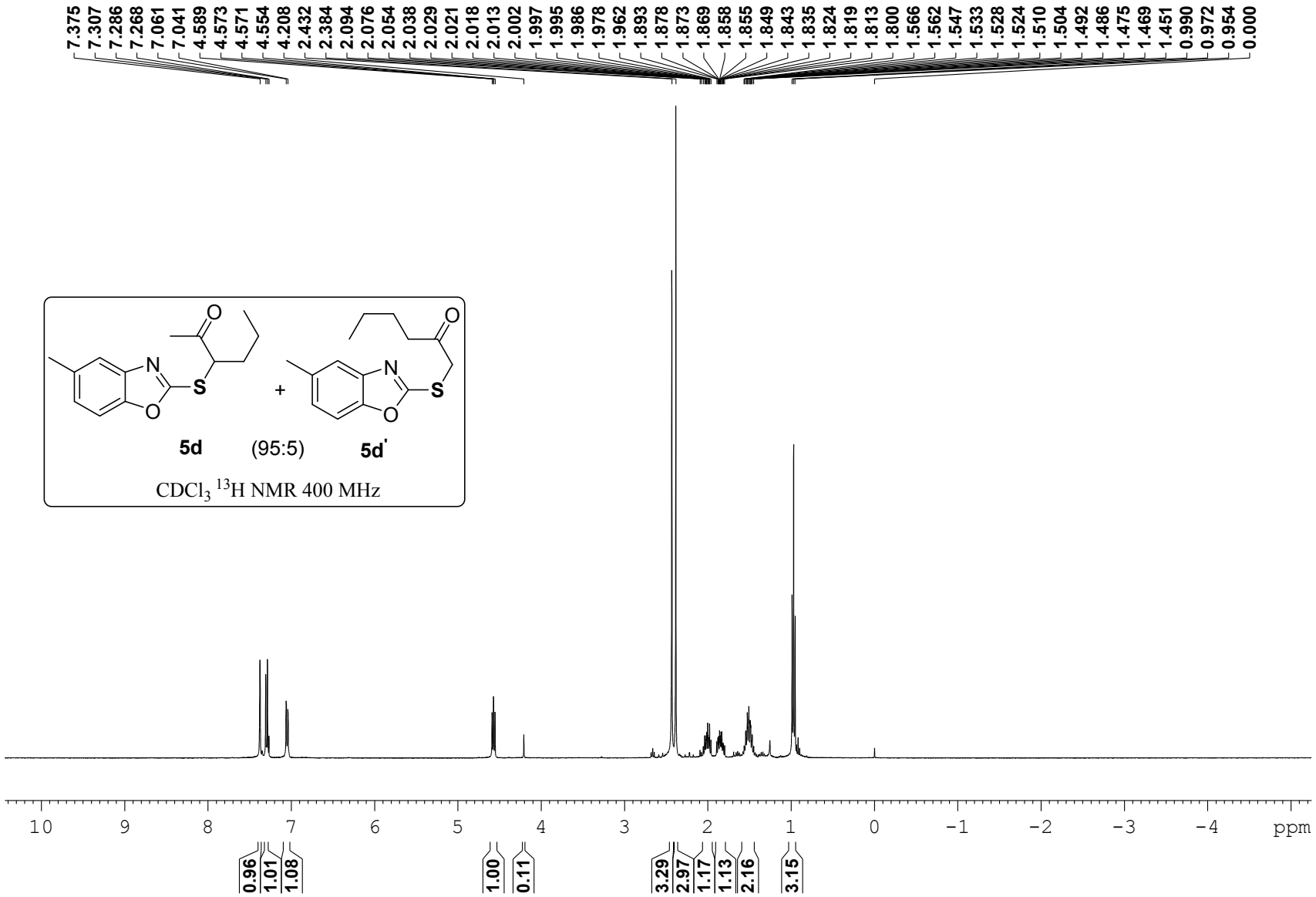
7.308
7.287
7.265
7.059
7.038
4.206
2.433
2.387
1.256
-0.000
O
N
S
O
5c
CDCl3 1H NMR 400 MHz
10
9
8
7
6
5
4
3
2
1
0
ppm
0.97
0.97
1.02
2.00
3.16
2.95


201.05
163.46
150.33
141.78
134.23
125.05
118.51
109.33
77.32
77.00
76.69
42.67
28.79
21.38
O
N
S
O
5c
CDCl3 13C NMR 100 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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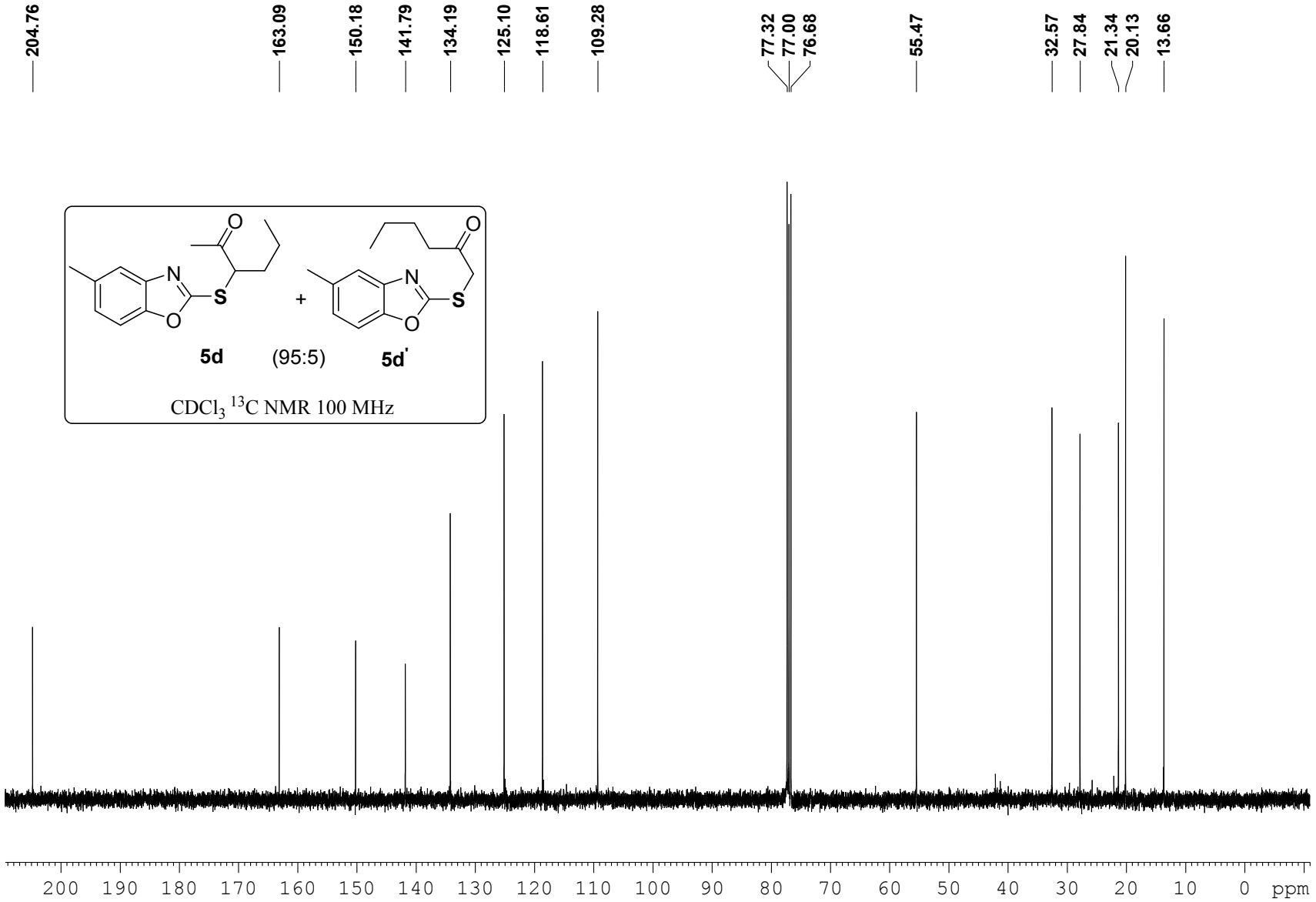
7.307
7.286
7.268
7.061
7.041
4.589
4.573
4.571
4.554
4.208
2.432
2.384
2.094
2.076
2.054
2.038
2.029
2.021
2.018
2.013
2.002
1.997
1.995
1.986
1.978
1.962
1.893
1.878
1.873
1.869
1.858
1.855
1.849
1.843
1.835
1.824
1.819
1.813
1.800
1.566
1.562
1.547
1.533
1.528
1.524
1.510
1.504
1.492
1.486
1.475
1.469
1.451
0.990
0.972
0.954
0.000
O
N
S
O
+
O
N
S
O
5d
(95:5)
5d'
CDCl3 13H NMR 400 MHz
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4
ppm
0.96
1.01
1.08
1.00
0.11
3.29
2.97
1.17
1.13
2.16
3.15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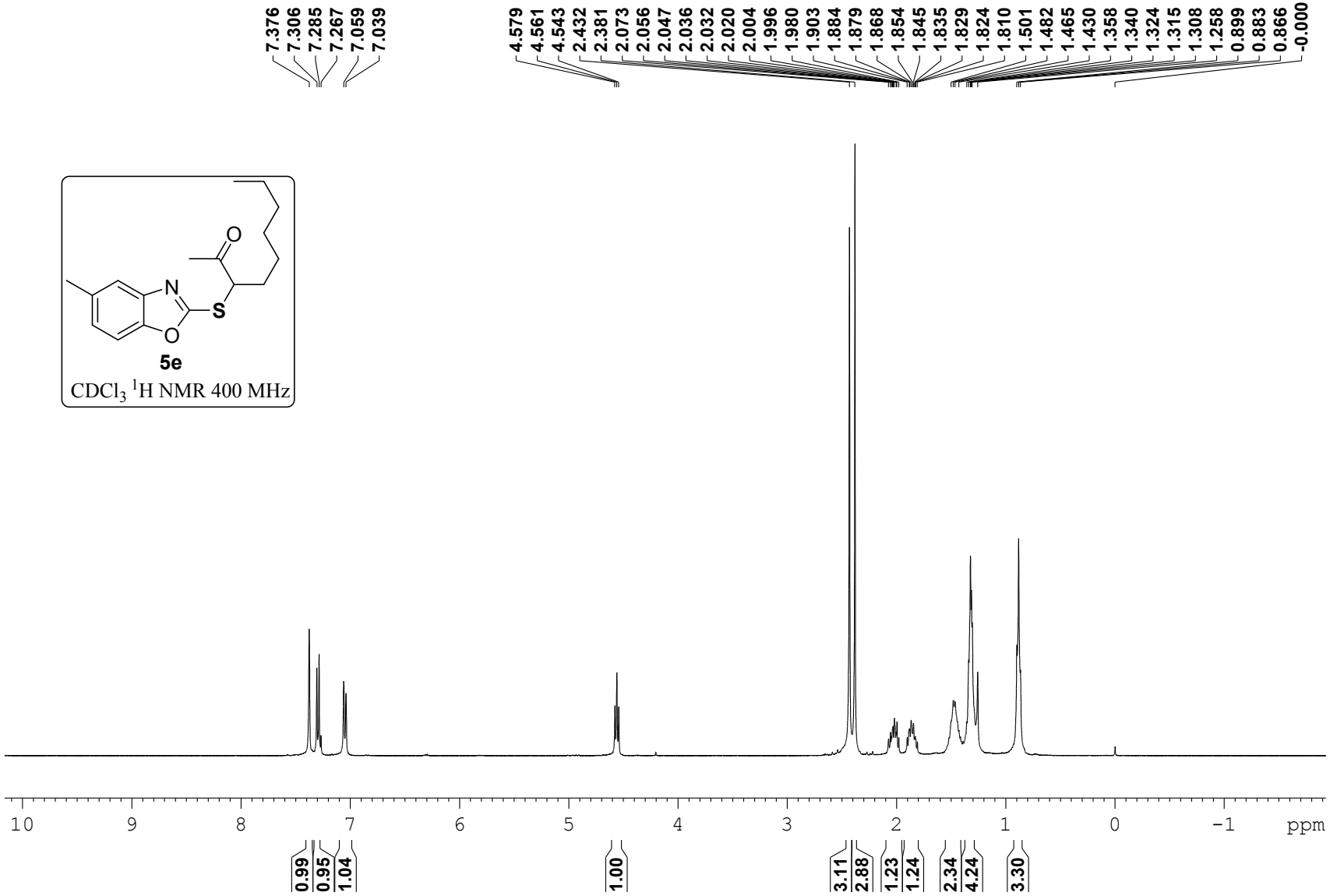
163.09
150.18
141.79
134.19
125.10
118.61
109.28
77.32
77.00
76.68
55.47
32.57
27.84
21.34
20.13
13.66
5d (95:5) 5d'
CDCl3 13C NMR 100 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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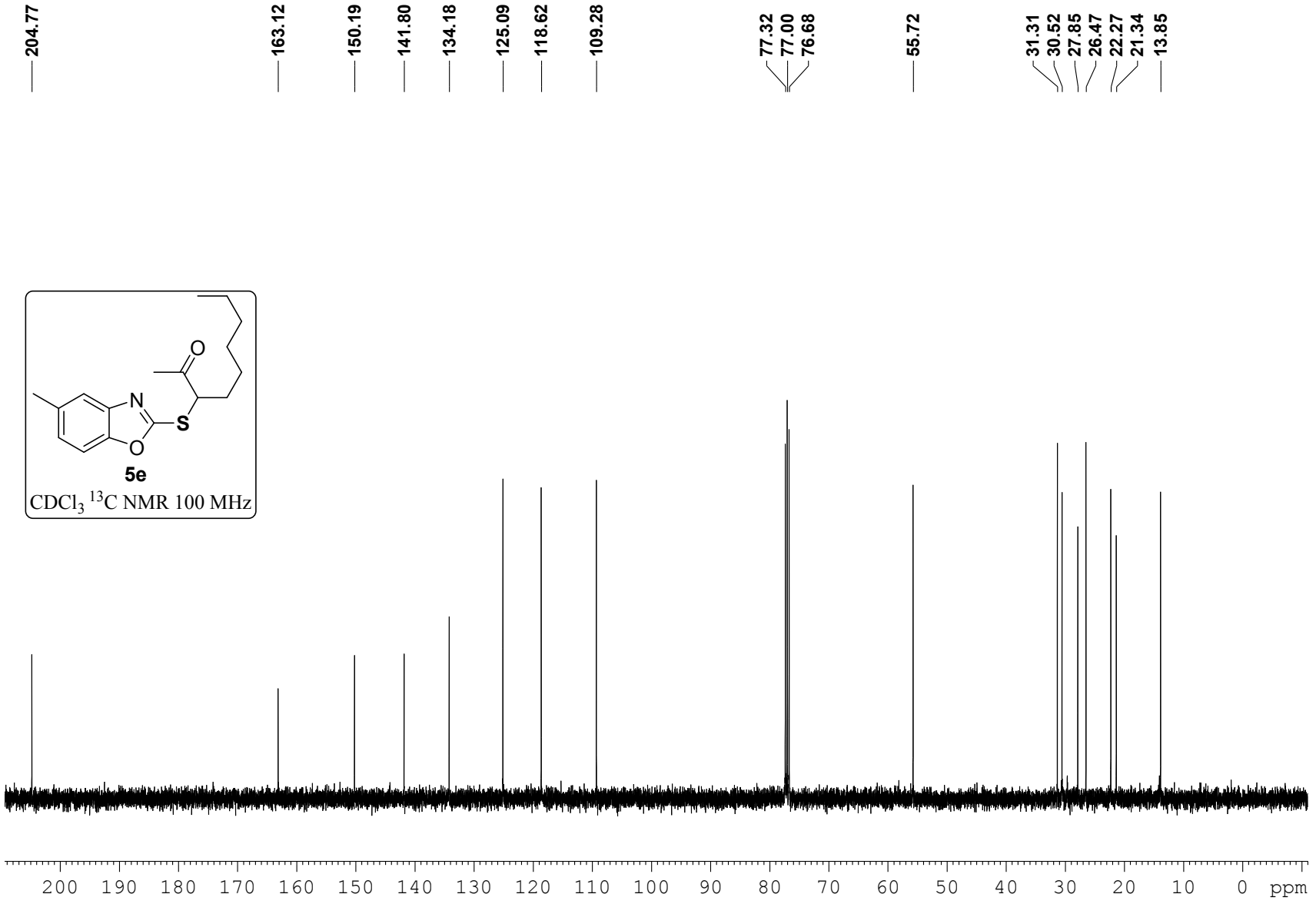
7.306
7.285
7.267
7.059
7.039
4.579
4.561
4.543
2.432
2.381
2.073
2.056
2.047
2.036
2.032
2.020
2.004
1.996
1.980
1.903
1.884
1.879
1.868
1.854
1.845
1.835
1.829
1.824
1.810
1.501
1.482
1.465
1.430
1.358
1.340
1.324
1.315
1.308
1.258
0.899
0.883
0.866
-0.000
O
N
S
O
5e
CDCl3 1H NMR 400 MHz
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
0.99
0.95
1.04
1.00
3.11
2.88
1.23
1.24
2.34
4.24
3.30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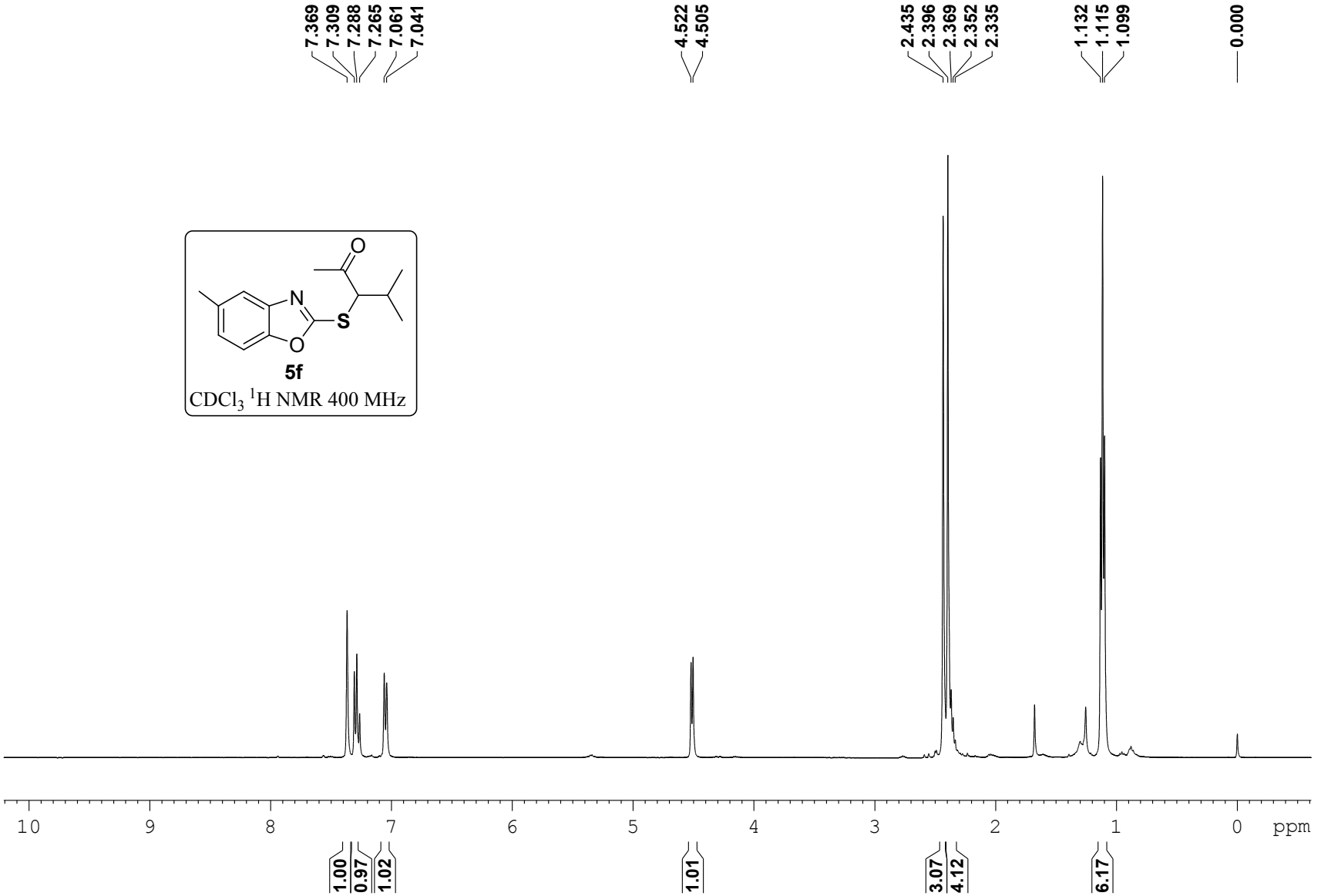
163.12
150.19
141.80
134.18
125.09
118.62
109.28
77.32
77.00
76.68
55.72
31.31
30.52
27.85
26.47
22.27
21.34
13.85
O
N
S
O
5e
CDCl3 13C NMR 100 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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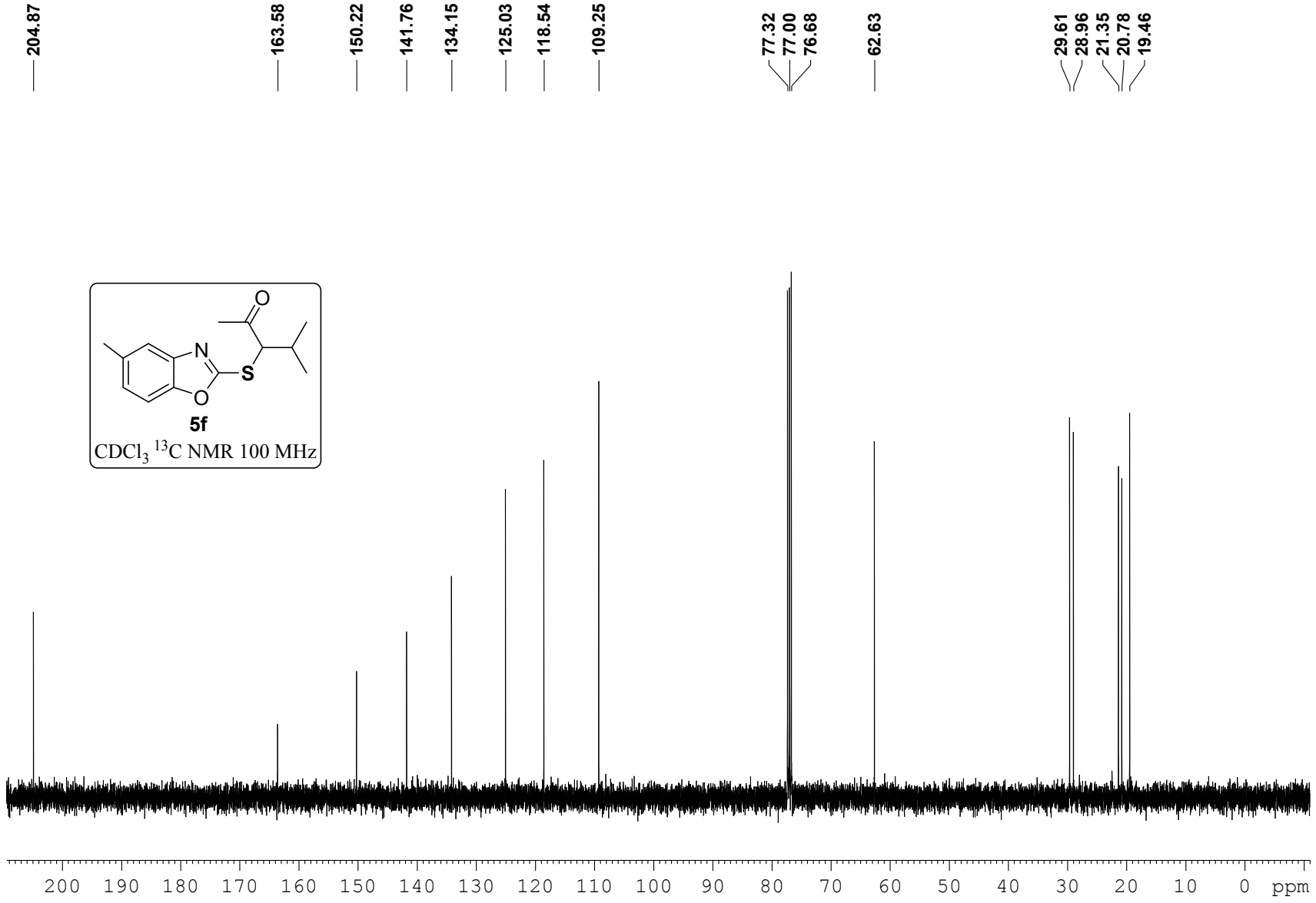
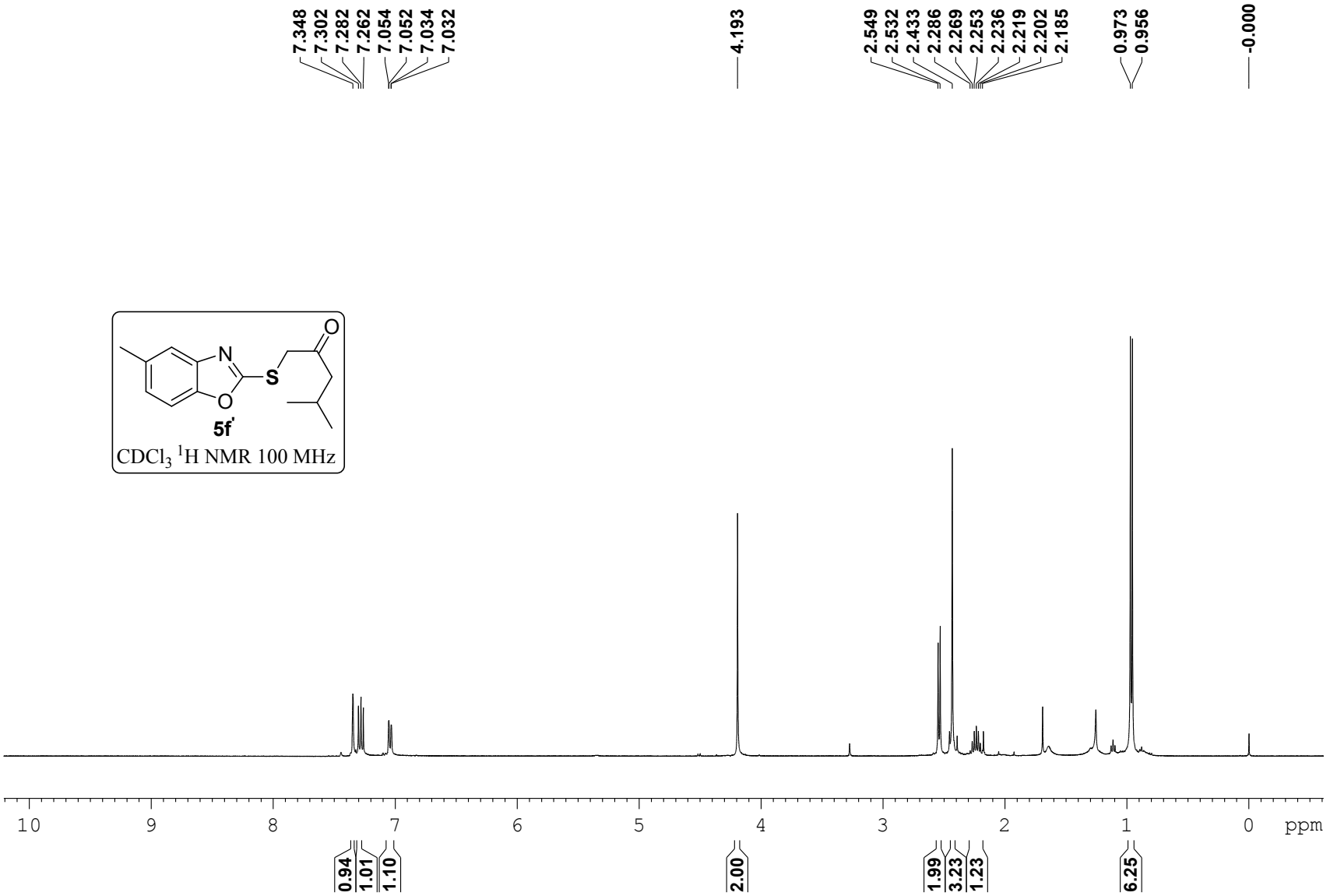
7.309
7.288
7.265
7.061
7.041
4.522
4.505
2.435
2.396
2.369
2.352
2.335
1.132
1.115
1.099
0.000
O
N
S
O
5f
CDCl3 1H NMR 400 MHz
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
0.97
1.02
1.01
3.07
4.12
6.17


204.87
163.58
150.22
141.76
134.15
125.03
118.54
109.25
77.32
77.00
76.68
62.63
29.61
28.96
21.35
20.78
19.46
O
N
S
O
5f
CDCl3 13C NMR 100 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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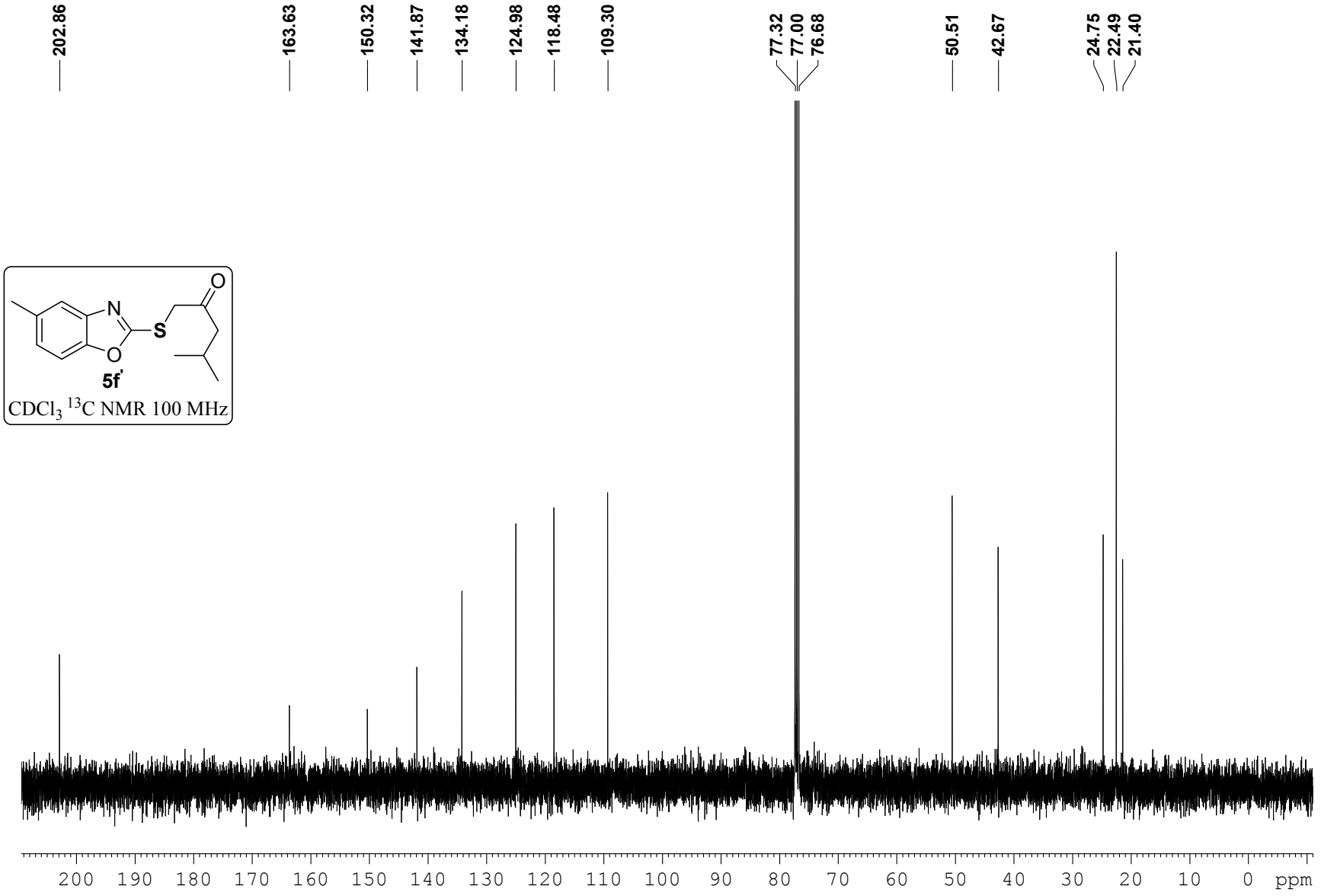
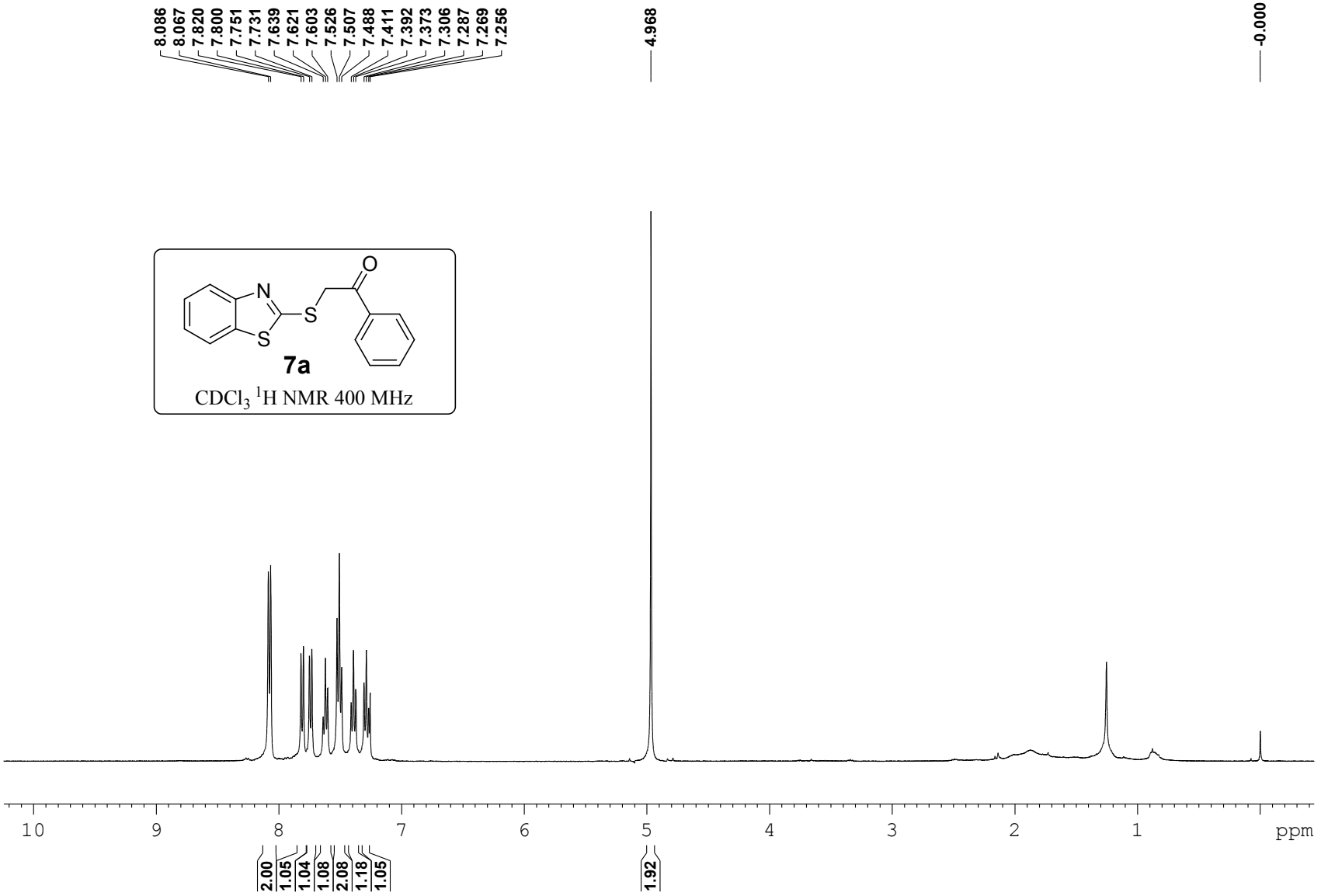
7.302
7.282
7.262
7.054
7.052
7.034
7.032
4.193
2.549
2.532
2.433
2.286
2.269
2.253
2.236
2.219
2.202
2.185
0.973
0.956
-0.000
O
N
S
O
5f'
CDCl3 1H NMR 100 MHz
10
9
8
7
6
5
4
3
2
1
0
ppm
0.94
1.01
1.10
2.00
1.99
3.23
1.23
6.25


202.86
163.63
150.32
141.87
134.18
124.98
118.48
109.30
77.32
77.00
76.68
50.51
42.67
24.75
22.49
21.40
O
N
S
O
5f'
CDCl3 13C NMR 100 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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8.067
7.820
7.800
7.751
7.731
7.639
7.621
7.603
7.526
7.507
7.488
7.411
7.392
7.373
7.306
7.287
7.269
7.256
4.968
-0.000
N
S
S
O
7a
CDCl3 1H NMR 400 MHz
10
9
8
7
6
5
4
3
2
1
ppm
2.00
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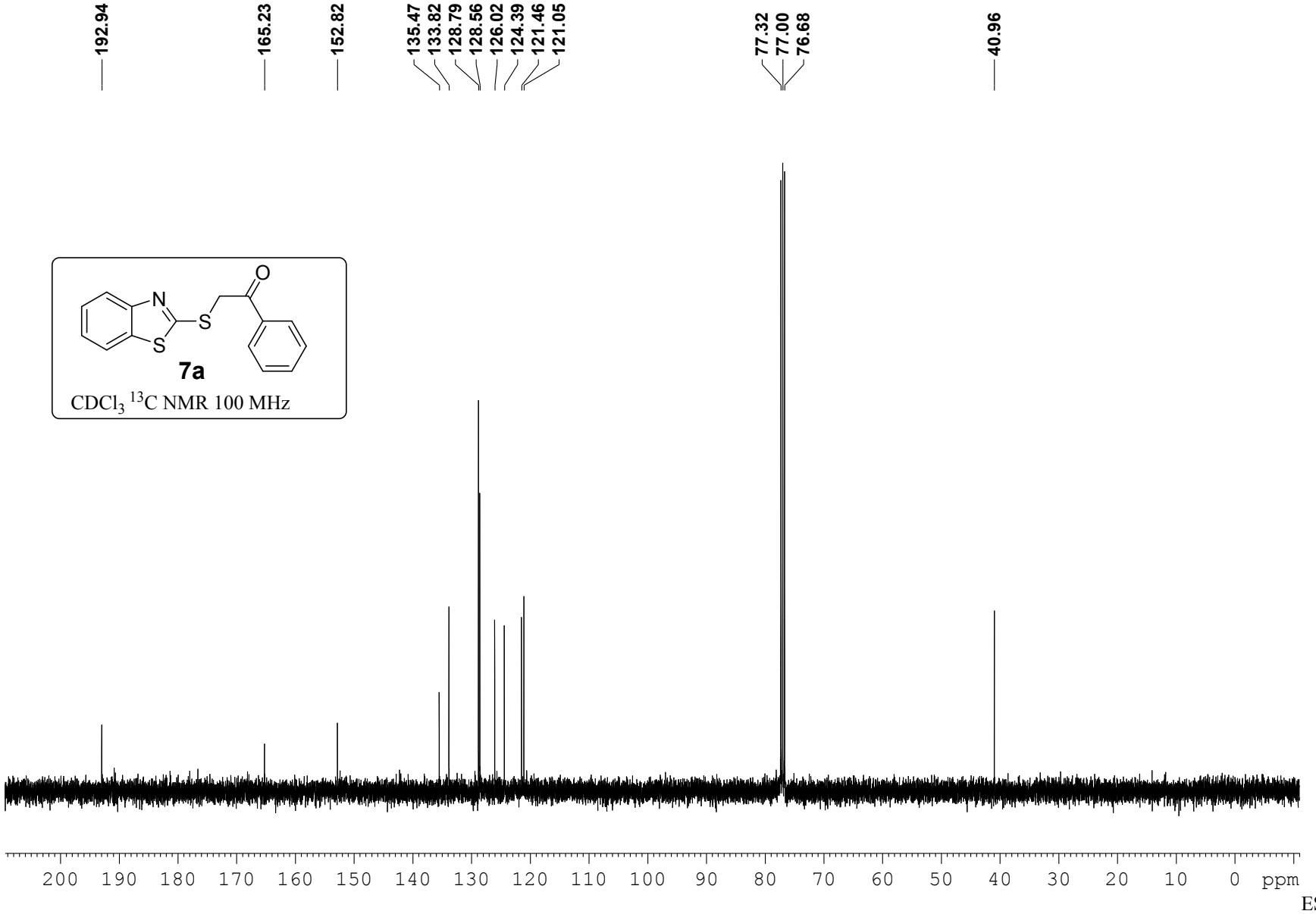
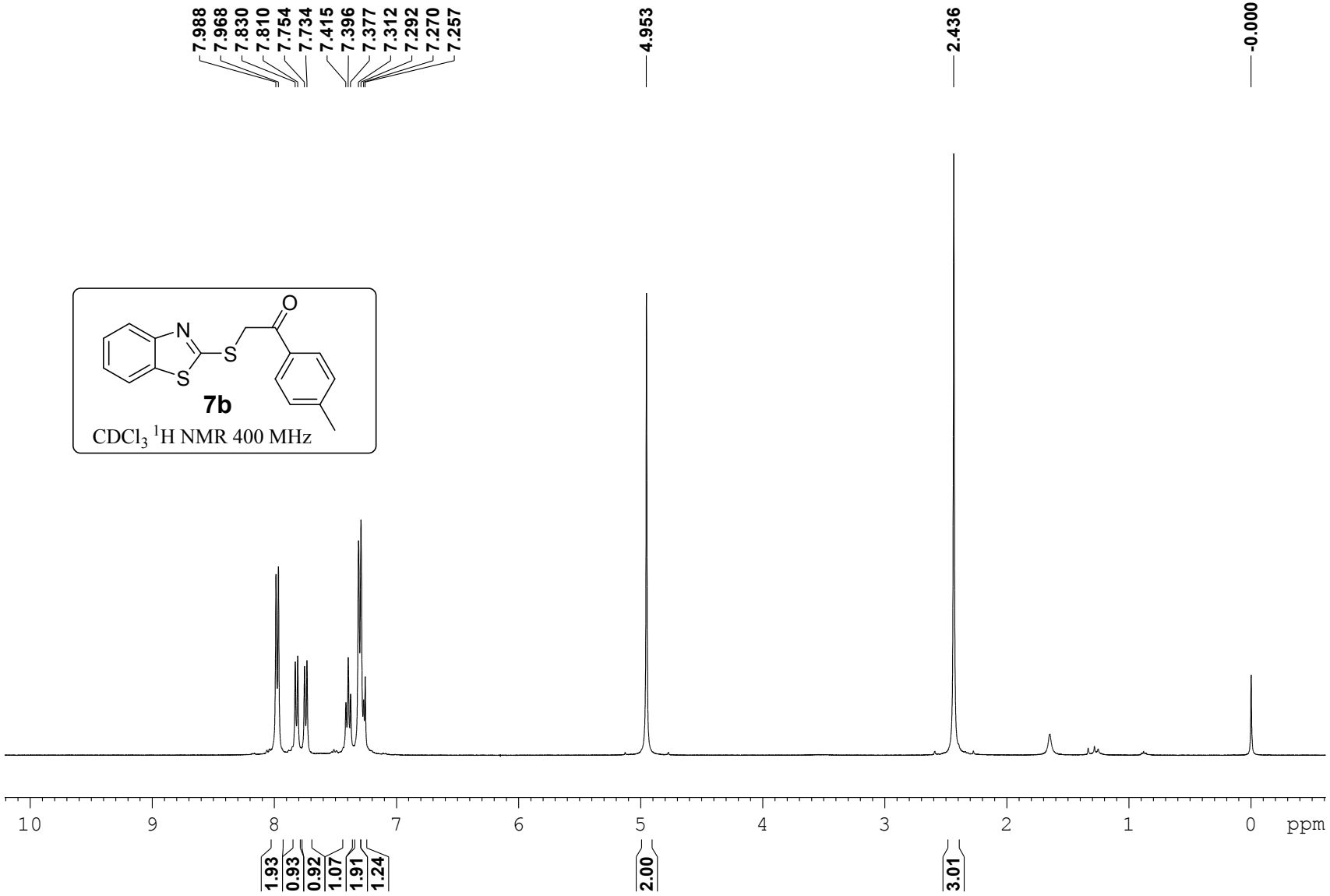
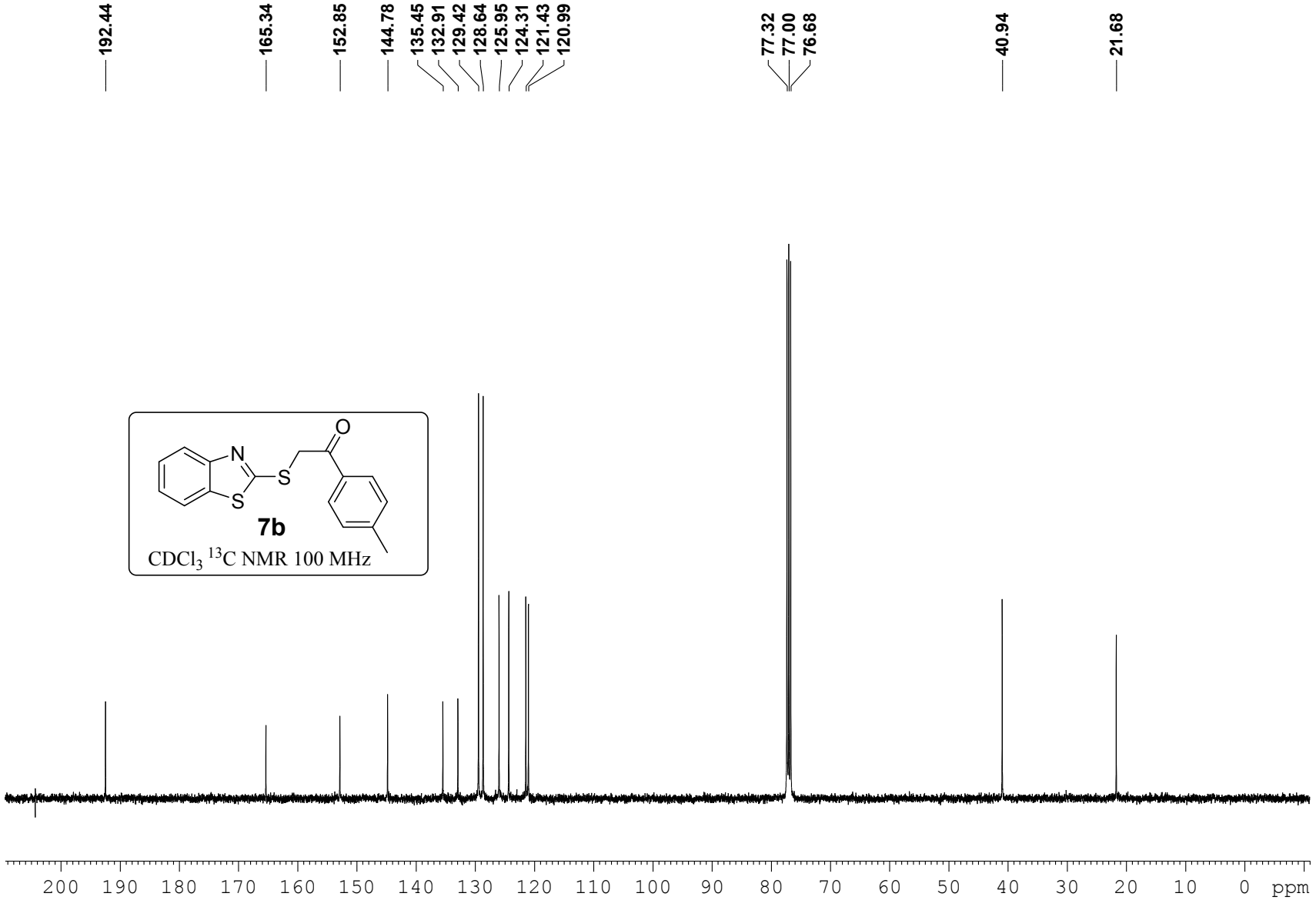
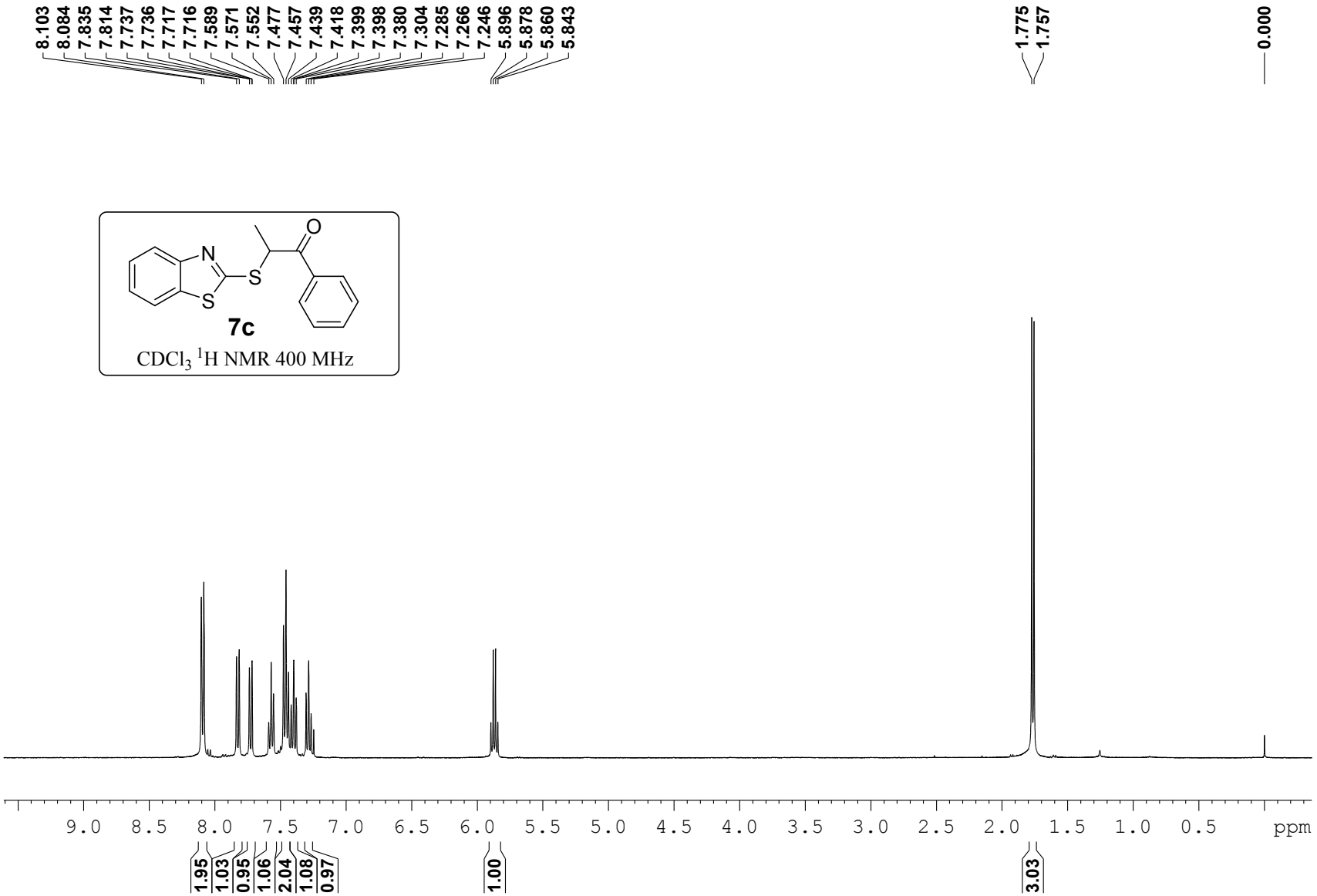
1.04
1.08
2.08
1.18
1.05
1.92


192.94
165.23
152.82
135.47
133.82
128.79
128.56
126.02
124.39
121.46
121.05
77.32
77.00
76.68
40.96
7a
CDCl3 13C NMR 100 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.988
7.968
7.830
7.810
7.754
7.734
7.415
7.396
7.377
7.312
7.292
7.270
7.257
4.953
2.436
-0.000
O
N
S
S
7b
CDCl3 1H NMR 400 MHz
10
9
8
7
6
5
4
3
2
1
0
ppm
1.93
0.93
0.92
1.07
1.91
1.24
2.00
3.01


192.44
165.34
152.85
144.78
135.45
132.91
129.42
128.64
125.95
124.31
121.43
120.99
77.32
77.00
76.68
40.94
21.68
O
N
S
S
7b
CDCl3 13C NMR 100 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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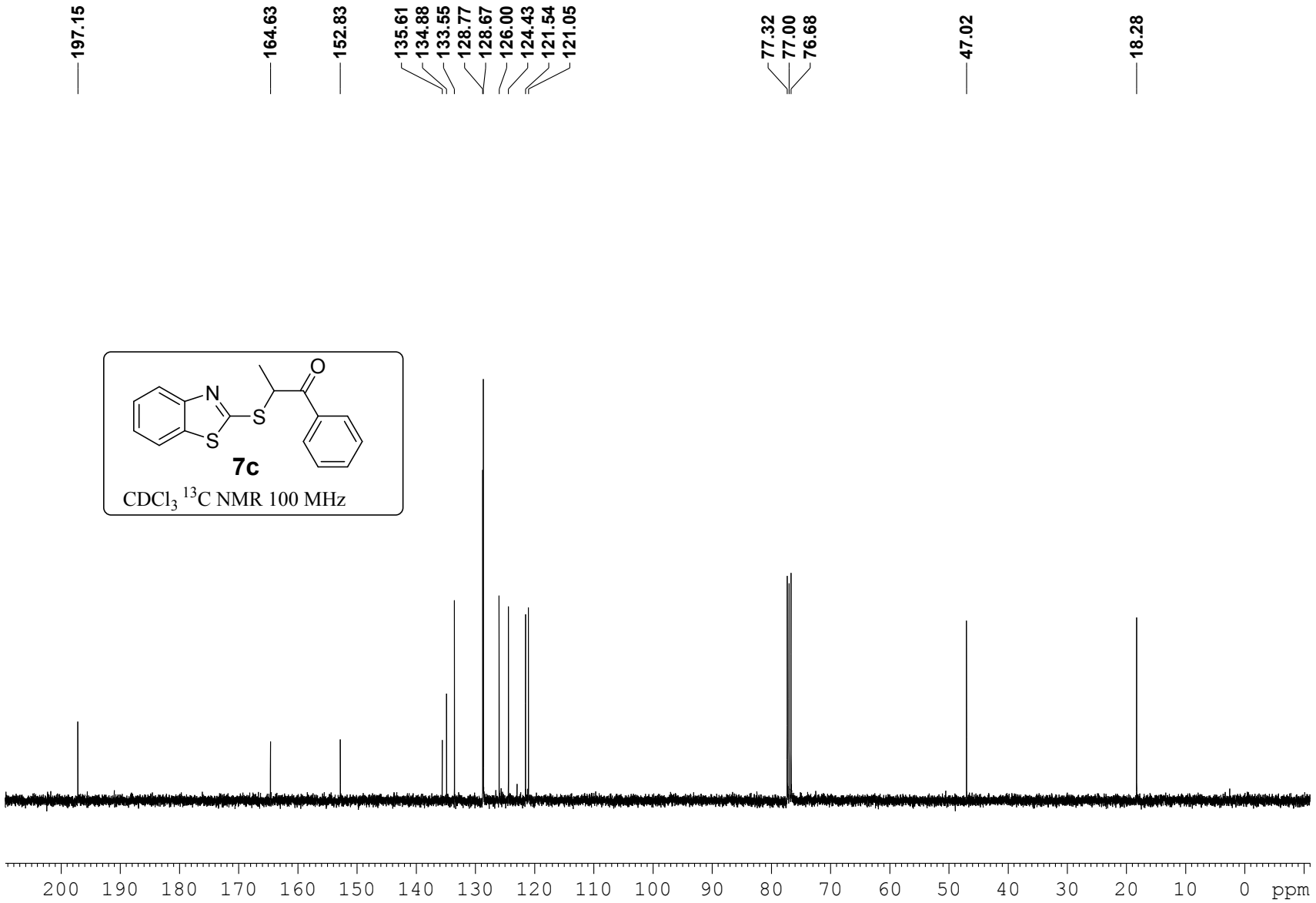
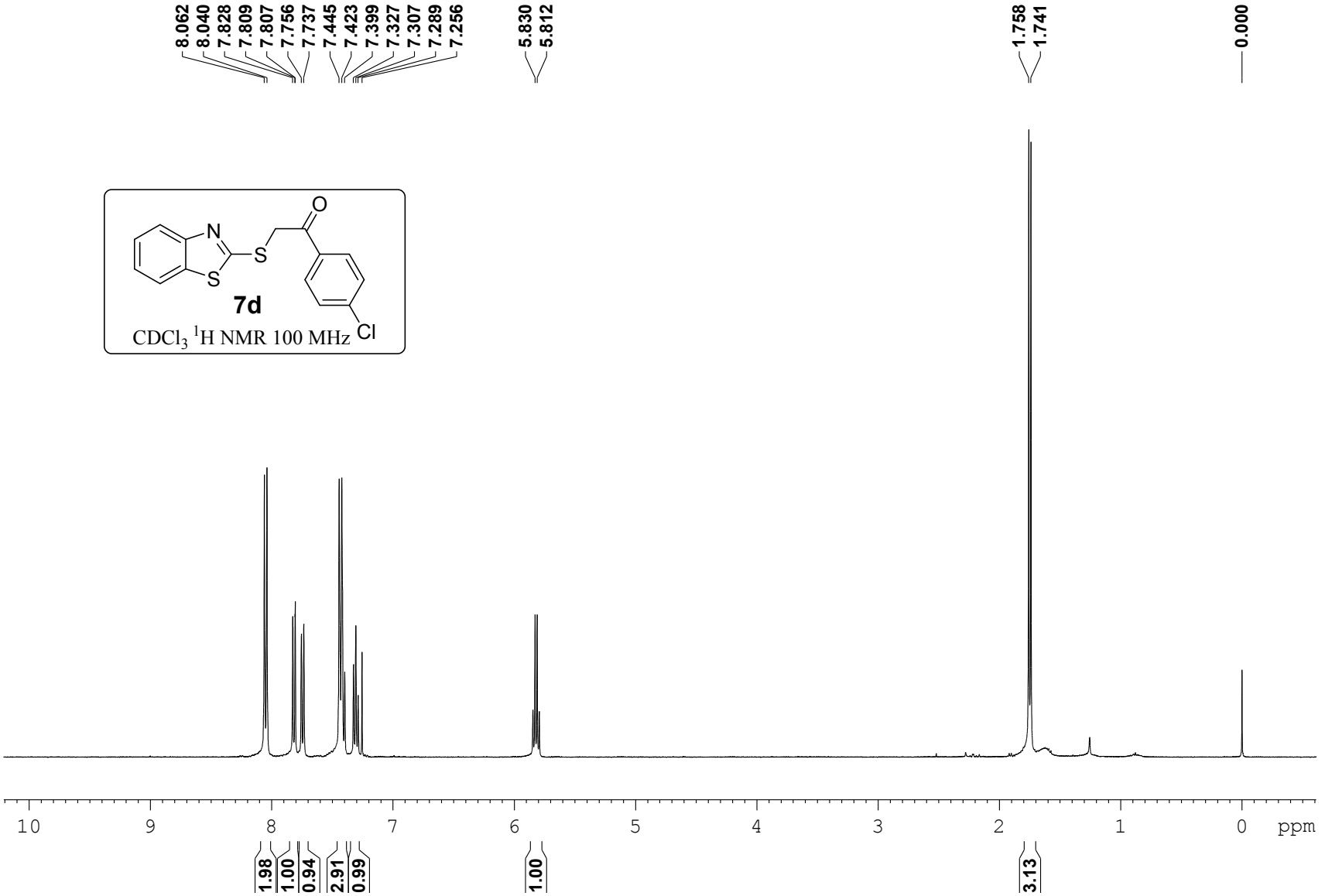
8.084
7.835
7.814
7.737
7.736
7.717
7.716
7.589
7.571
7.552
7.477
7.457
7.439
7.418
7.399
7.398
7.380
7.304
7.285
7.266
7.246
5.896
5.878
5.860
5.843
1.775
1.757
0.000
N
S
S
O
7c
CDCl3 1H NMR 400 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.95
1.03
0.95
1.06
2.04
1.08
0.97
1.00
3.03


197.15
164.63
152.83
135.61
134.88
133.55
128.77
128.67
126.00
124.43
121.54
121.05
77.32
77.00
76.68
47.02
18.28
7c
CDCl3 13C NMR 100 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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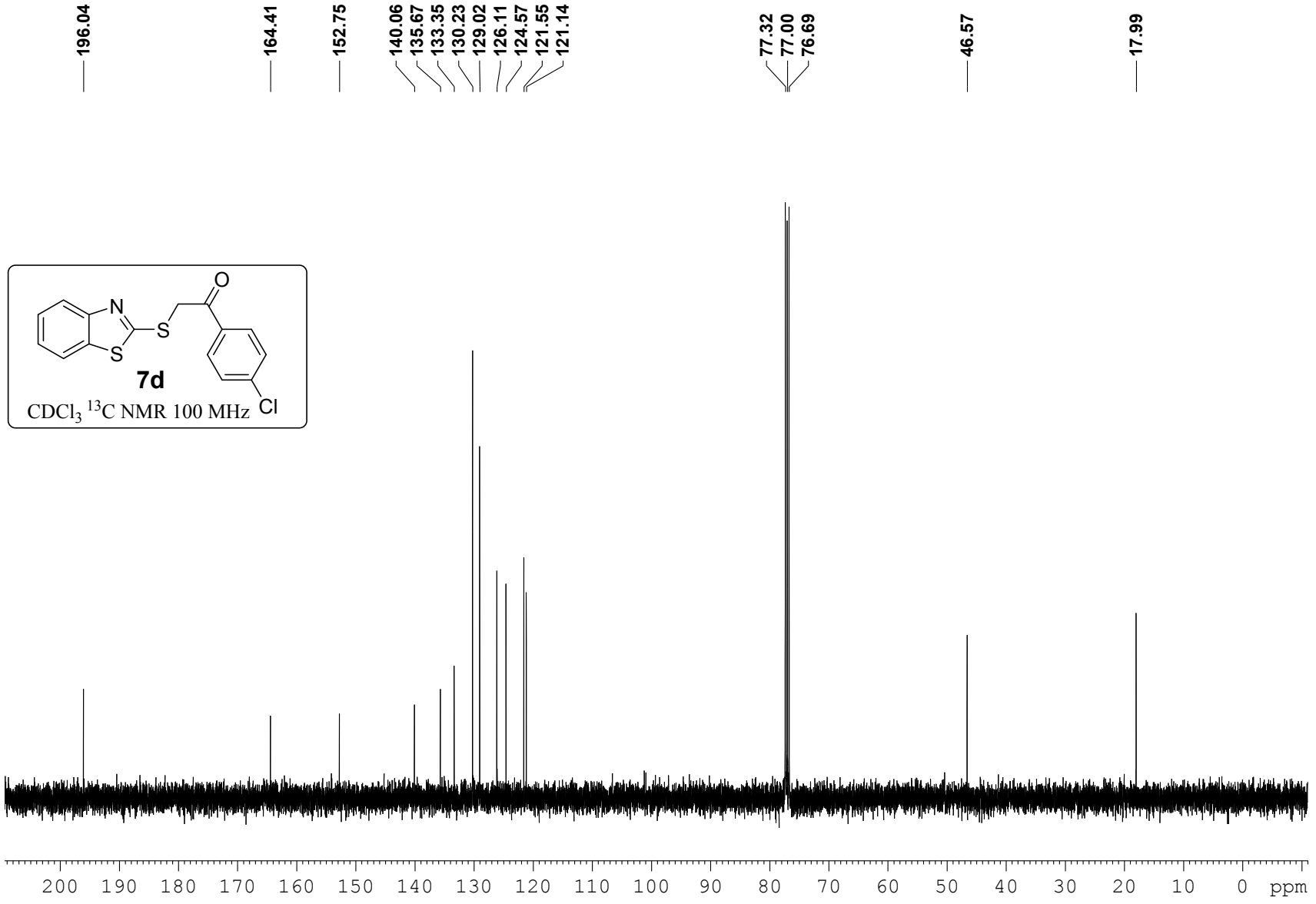
8.040
7.828
7.809
7.807
7.756
7.737
7.445
7.423
7.399
7.327
7.307
7.289
7.256
5.830
5.812
1.758
1.741
0.000
N
S
S
O
Cl
7d
CDCl3 1H NMR 100 MHz
10
9
8
7
6
5
4
3
2
1
0
ppm
1.98
1.00
0.94
2.91
0.99
1.00
3.13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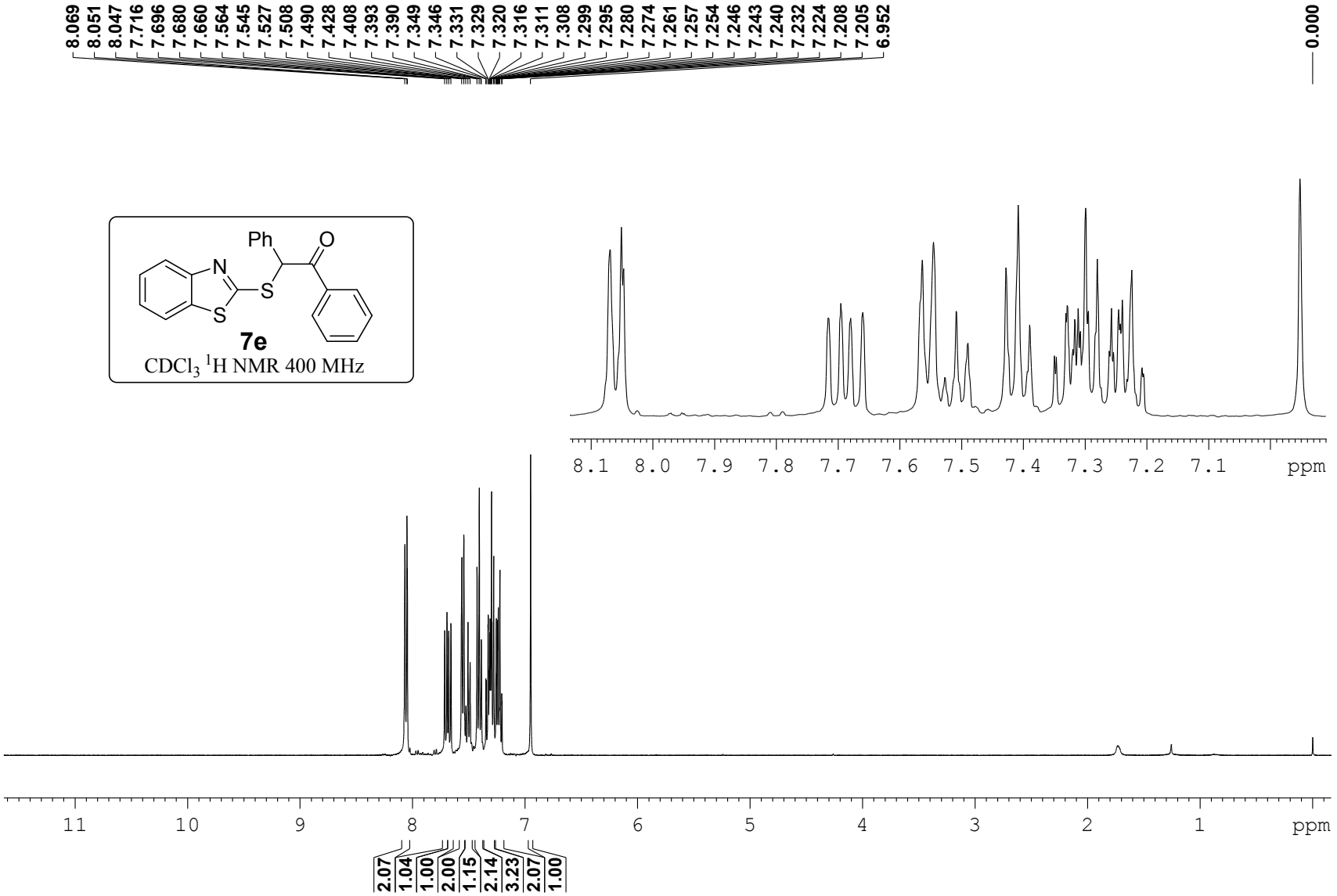
164.41
152.75
140.06
135.67
133.35
130.23
129.02
126.11
124.57
121.55
121.14
77.32
77.00
76.69
46.57
17.99
O
N
S
S
Cl
7d
CDCl3 13C NMR 100 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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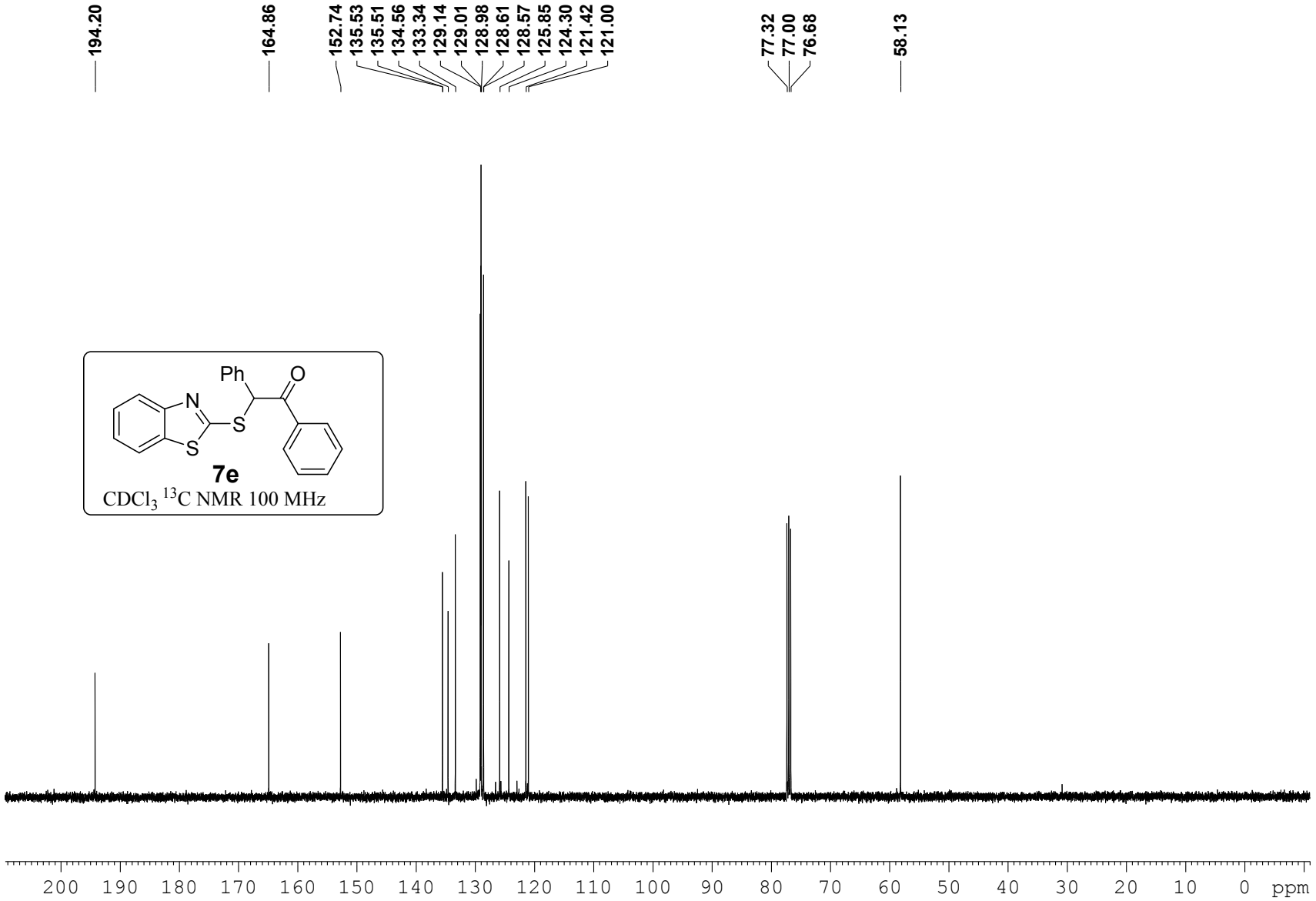
8.051
8.047
7.716
7.696
7.680
7.660
7.564
7.545
7.527
7.508
7.490
7.428
7.408
7.393
7.390
7.349
7.346
7.331
7.329
7.320
7.316
7.311
7.308
7.299
7.295
7.280
7.274
7.261
7.257
7.254
7.246
7.243
7.240
7.232
7.224
7.208
7.205
6.952
0.000
Ph
O
N
S
S
7e
CDCl3 1H NMR 400 MHz
8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 ppm
11 10 9 8 7 6 5 4 3 2 1 ppm
2.07
1.04
1.00
2.00
1.15
2.14
3.23
2.07
1.00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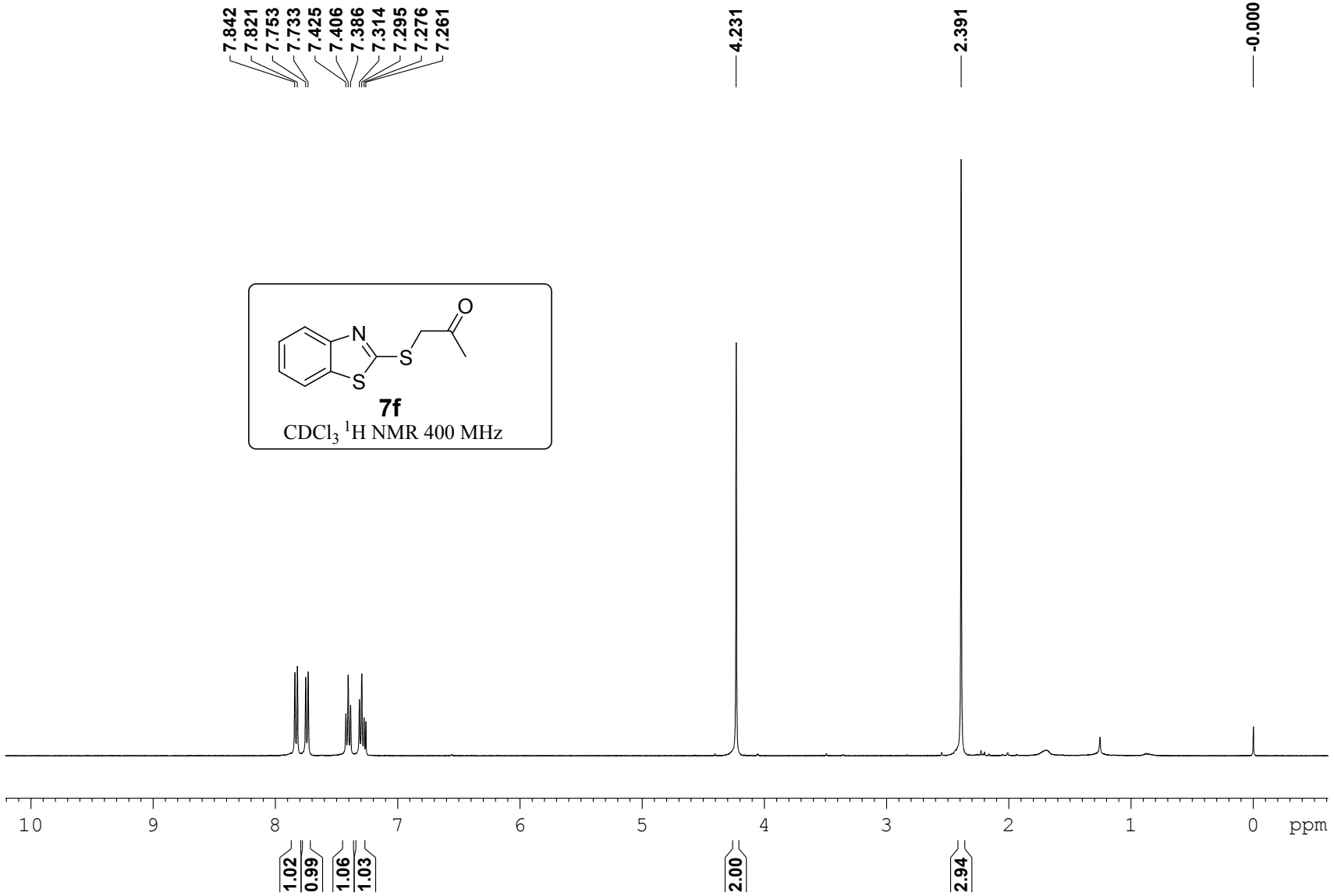
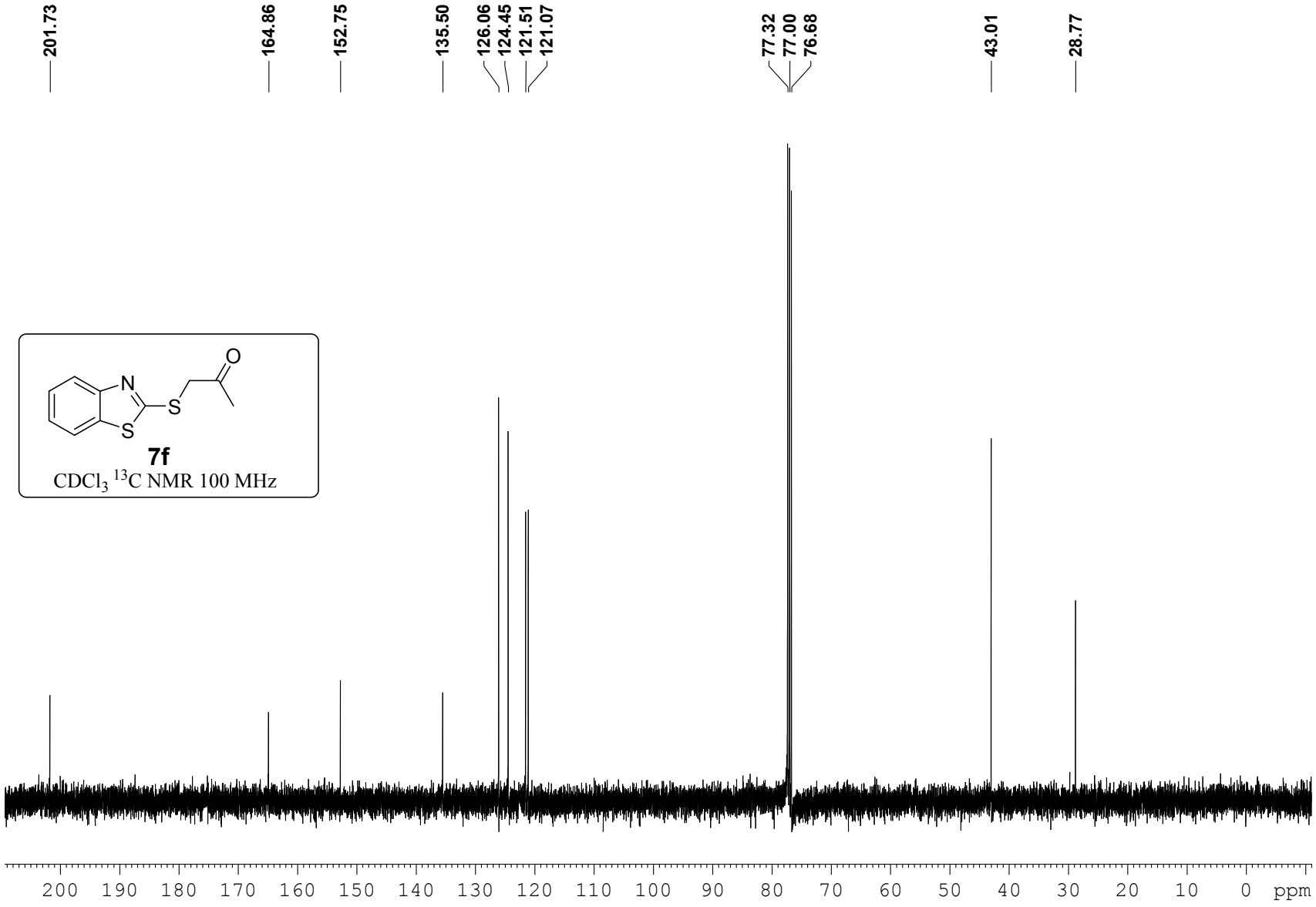
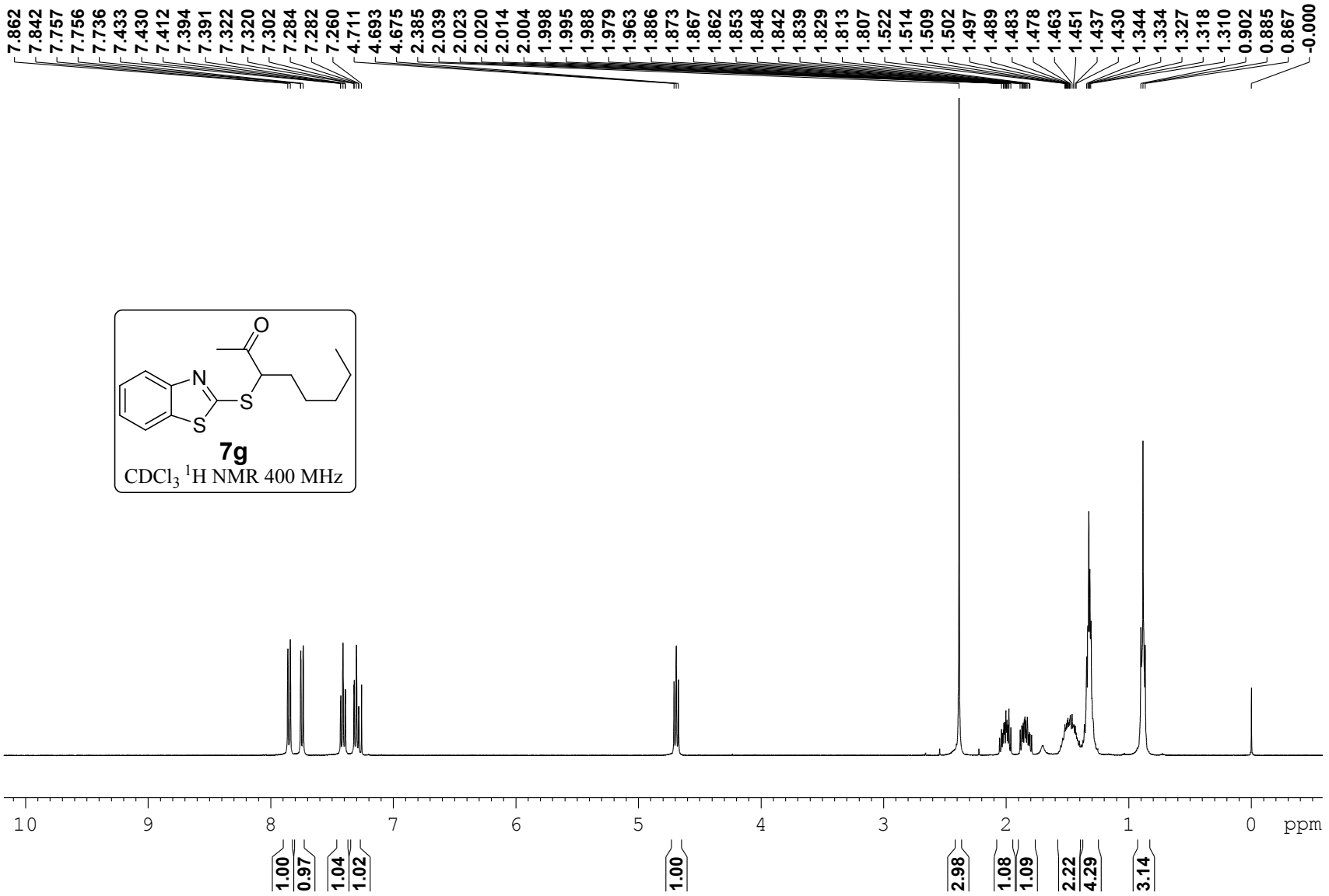
152.74
135.53
135.51
134.56
133.34
129.14
129.01
128.98
128.61
128.57
125.85
124.30
121.42
121.00
77.32
77.00
76.68
58.13
Ph
O
N
S
S
7e
CDCl3 13C NMR 100 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.842
7.821
7.753
7.733
7.425
7.406
7.386
7.314
7.295
7.276
7.261
4.231
2.391
-0.000
N
S
S
O
7f
CDCl3 1H NMR 400 MHz
10
9
8
7
6
5
4
3
2
1
0
ppm
1.02
0.99
1.06
1.03
2.00
2.94


201.73
164.86
152.75
135.50
126.06
124.45
121.51
121.07
77.32
77.00
76.68
43.01
28.77
N
S
S
O
7f
CDCl3 13C NMR 100 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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7.430
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7.302
7.284
7.282
7.260
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2.039
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1.853
1.848
1.842
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1.514
1.509
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1.497
1.489
1.483
1.478
1.463
1.451
1.437
1.430
1.344
1.334
1.327
1.318
1.310
0.902
0.885
0.867
-0.000
O
N
S
S
7g
CDCl3 1H NMR 400 MHz
10
9
8
7
6
5
4
3
2
1
0
ppm
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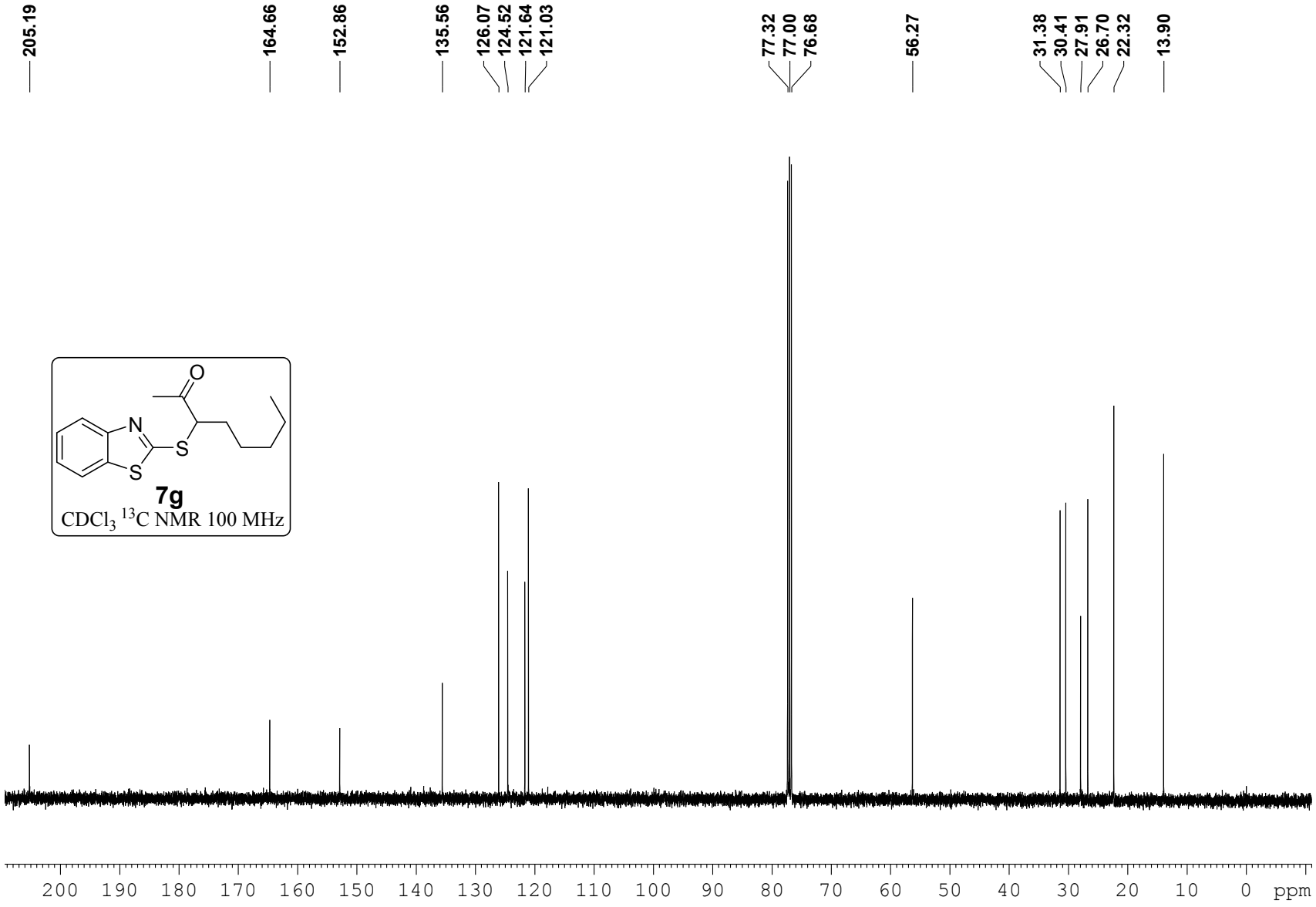
1.04
1.02
1.00
2.98
1.08
1.09
2.22
4.29
3.14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135.56
126.07
124.52
121.64
121.03
77.32
77.00
76.68
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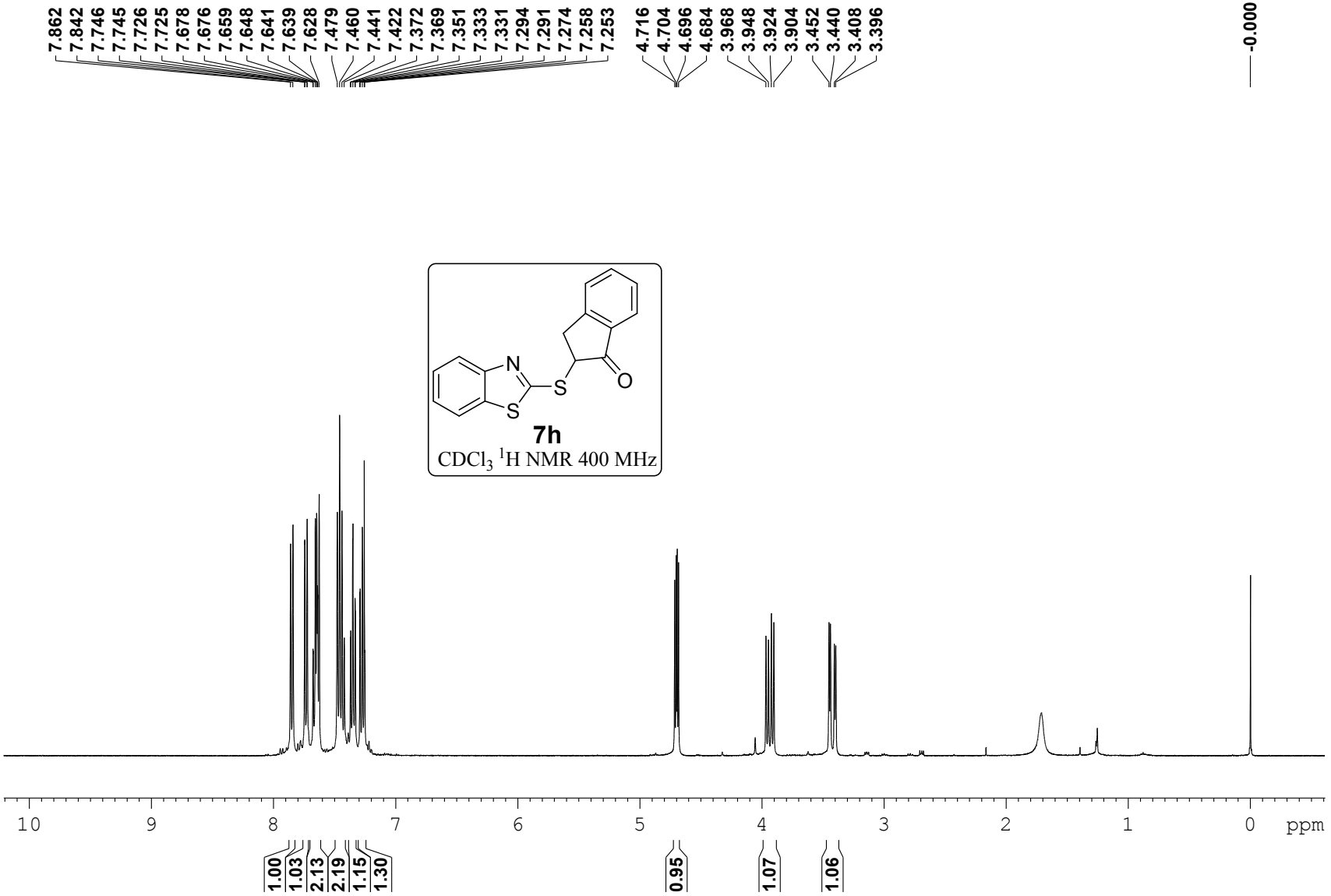
31.38
30.41
27.91
26.70
22.32
13.90
O
N
S
S
7g
CDCl3 13C NMR 100 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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7.628
7.479
7.460
7.441
7.422
7.372
7.369
7.351
7.333
7.331
7.294
7.291
7.274
7.258
7.253
4.716
4.704
4.696
4.684
3.968
3.948
3.924
3.904
3.452
3.440
3.408
3.396
-0.000
N
S
S
O
7h
CDCl3 1H NMR 400 MHz
10
9
8
7
6
5
4
3
2
1
0
ppm
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2.13
2.19
1.15
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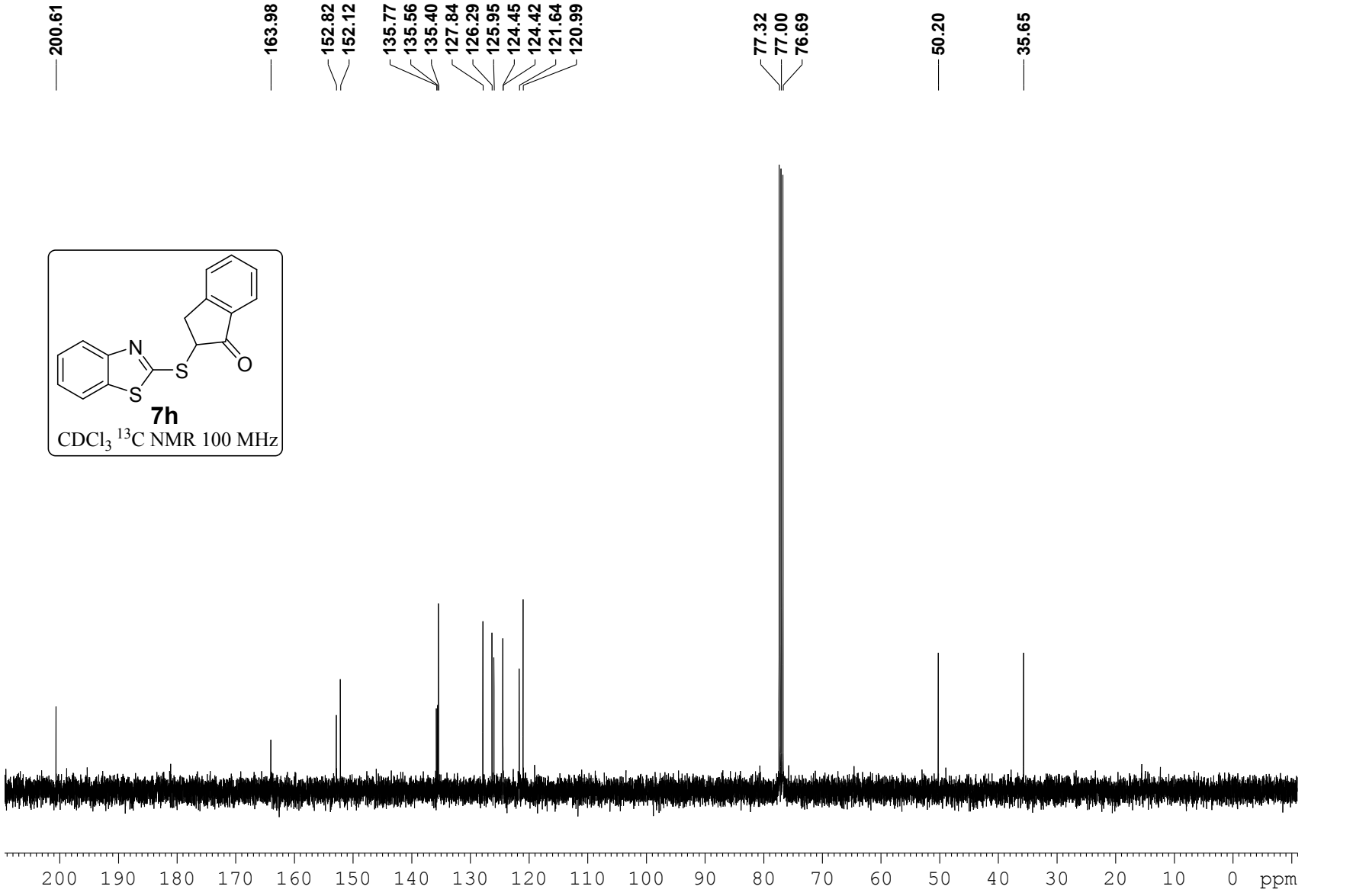
0.95
1.07
1.06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152.82
152.12
135.77
135.56
135.40
127.84
126.29
125.95
124.45
124.42
121.64
120.99
77.32
77.00
76.69
50.20
35.65
N
S
S
O
7h
CDCl3 13C NMR 100 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm